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ARTICLES

Luminescence and defect formation by visible and near-infrared irradiation of vitreous silica

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We report the observation of photoluminescence (PL) originating from defects created by the visible and near-infrared irradiation of vitreous silica. Three PL bands at 283, 468, and 558 nm exhibited excitation spectra peaking at 250 nm, which corresponds to the oxygen vacancy defect V_O absorption. The observed PL bands can be attributed to the following transitions: the singlet-singlet transition of oxygen vacancy for 283 nm, and the radiative recombination of separated carriers on the vacancy-interstitial pairs (V_O;O_i) and (V_O;$(O_2)_i$) for 468 and 558 nm, respectively. [S0163-1829(99)07337-3]

I. INTRODUCTION

Since the first observation[1] of intrinsic defect photoluminescence (PL) in annealed and neutron-irradiated amorphous (a-)SiO_2, much effort has been made to determine the origins and structure of defects in different kinds of silica: crystalline (c-)SiO_2;[2] wet and dry (a-)SiO_2;[3–5] electron-,[6] γ-ray–,[7–9] and neutron-irradiated (Ref. 1); ultraviolet irradiation[10] and hydrostatic pressure[11] densified; and Ge-doped[12–14] and Si-rich.[9,15] The formation of defects is of particular importance in applications where SiO_2 is grown over Si because the dioxide layers are strained under such conditions[16] and are less resistant to defect formation under irradiation.[7,17] The production of luminescent porous Si and silica containing Si nanocrystals[18] cannot be achieved without understanding the role of defect formation within the Si-SiO_2 interface.[19,20] Issues related to the degradation of ultraviolet (UV)-grade optics[10] and optical fibers[14,21] under conditions of elevated irradiation powers are also of interest in this regard. Despite the significant progress that has been made in these areas, a full understanding of defect generation and structure in different glasses has not yet been developed and these issues remain a matter of debate.

Recently, the fabrication of optical waveguides by induced damage in different glasses[22,23] using the focused irradiation of visible light has been reported; in addition, glass has been proposed as a media for three-dimensional optical memory with multilayered bit recording.[24–26] These last applications, in particular, require a deeper understanding of the behavior of induced defects than has been developed thus far.

Our aim in this study was to explore PL originating from optically damaged areas created by focused visible and near-infrared light inside vitreous silica (v-)SiO_2. Well-defined spectral positions and the form factor of PL bands allow us to discuss the structure of corresponding defects on the basis of photoluminescence excitation (PLE) spectra, the defects' generation power law, and the time decay of the PL bands.

A comment should be made regarding the terminology used for the defects because even different defects can be named in the same way; primarily, the naming of the defect takes into account the E' center and the oxygen vacancy.[6,7] Hereafter, we will refer to a $\equiv$Si—Si$\equiv$ defect as an oxygen vacancy (V_O) that is an electrically neutral diamagnetic defect[3] (hereafter - denotes a bond, $\cdot$ and $\circ$ denote an unpaired electron and a trapped hole, respectively). Other, historical names for an oxygen deficiency defect can also be found.[27,28] The defect can be in either an unrelaxed $\equiv$Si$\cdot\cdot$Si$\equiv$ or a relaxed $\equiv$Si—Si$\equiv$ state[27–29] according to the different Si-to-Si distance. Tight-binding calculations show that two types of oxygen vacancies with different Si-Si distances give optical transitions at 5.85 eV (212 nm) for an unrelaxed defect (Si-Si distance of 3 Å) and 7.6 eV (163 nm) for a relaxed defect.[30] Experimentally, the absorption band for the relaxed oxygen vacancy was found to be at 7.6 eV,[9,28,29] while the absorption for the unrelaxed vacancy appears at 5.1 eV (243 nm),[28] which is referred to as the $B_2\alpha$ (Ref. 5) or B_2 (Refs. 3 and 4) absorption band. The E' (or E_1') center is another well-known defect with an absorption band around 210 nm (Ref. 31) that, as is now well accepted, conforms to a trapped hole on an oxygen vacancy[32] and usually acts as a nonradiative recombination center.[15] Different signatures of this defect can be found: $\equiv$Si$\cdot\circ$Si$\equiv$,[7] which shows the defect structure, or $\equiv$Si$\cdot$,[33] which indicates the paramagnetic nature of the defect, which has been well explored by electron-spin-resonant measurements.[9,33] The counterpart of this defect is interstitial oxygen in the form of O_i^-.[10]

II. EXPERIMENTAL PROCEDURES

Two kinds of dry v-SiO$_2$, *ED-C* and *ED-B* (Nippon Silica Glass Co.), which were classified according to the concentration of the hydroxyl group OH, being less than 1.0 ppm or less than 10 ppm, respectively, were irradiated in an optical microscope by focused pulsed laser shots through an oil-immersed objective lens of 100× magnification and numerical aperture, $A_N = 1.30$. In the case of picosecond (ps) fabrication, we employed an Optiphot 2, Nikon microscope, while for the femtosecond (fs) fabrication, an Olympus IX 70 inverted-type microscope was used. A laser system that generates and amplifies 800-nm wavelength pulses at a repetition rate of 5 kHz with a pulse with (FWHM value) of approximately 130 fs was used as a damage-bit-inducing light source. This light source was composed of a mode-locked Ti:sapphire laser Tsunami pumped by argon-ion laser Beamlok 2080, and a kilohertz-pulsed Ti:sapphire regenerative amplifier Spitfire pumped by a multikilohertz intracavity doubled Nd:YLF laser Merlin (all from Spectra Physics). This system can deliver radiation up to 1.2-W power at a fundamental wavelength and up to 100-mW in the second harmonic at 400 nm (HGS-T, Spectra Physics) with an energy stability of 3% (root-mean-square estimate). Another laser source used for the ps fabrication was the Nd:YAG laser Ekspla PL2143A with an operation wavelength of 532 nm, a repetition rate of 10 Hz, and a pulse width FWHM of 30 ps.

By controlling the scanning stage velocity, damage bits were uniformly distributed in plane with a spacing of several micrometers inside a v-SiO$_2$ plate. Hereafter the term ''damage bit'' is used to refer to any observable silica transparency change recognizable under a conventional optical microscope. PL and PLE spectra were measured by means of a fluorescence spectrometer (FP-4500, Hitachi) furnished with a photomultiplier used as a detector and a 150-W Xe lamp used as an excitation source.

PL time decay was measured by the time-correlated single-photon-counting method. The PL excitation light source was the Ti:sapphire fs-laser Tsunami pumped by the solid-state laser Millennia (Spectra Physics) with subsequent frequency doubler and tripler stages. As a monochromator we utilized the ARC300i, Acton spectrometer and as a detector the multichannel-plate photomultiplier R3809-50U Hamamatsu Photonics. The recorded PL transients were deconvoluted as multiexponential decays.

A charge-coupled device camera was installed on the microscope to monitor the fabrication. Following the procedure described elsewhere,[34] the actual laser pulse energy experienced by the sample E_T was calculated by measurement of the microscope transmission T and the incident laser pulse energy E_I, according to $E_T = TE_I$.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Photoluminescence spectra

Typical PL spectra of damaged v-SiO$_2$ are presented in Fig. 1 for different damage-inducing irradiation wavelengths, pulse duration, and power. Three distinct bands at 283, 468, and 558 nm appear, in contrast to the featureless PL spectrum from undamaged silica. The distinct feature of all the PL data is that exactly the same bands were found in spectra from damaged v-SiO$_2$ when damage was induced by different visible and near-infrared focused irradiation of 130 fs (at 400 and 800 nm) and 30 ps (at 532 nm), as presented in Fig. 1(b). The common conditions of the damage-bit creation were: (i) sub-band-gap irradiation quanta; (ii) energy density exceeding the optical damage threshold of a few GW/cm^2 (Ref. 21) at the focal point where the damage was caused; and (iii) single-shot irradiation for every damage bit, which was realized by proper adjustment of the velocity of the optical stage of the microscope with the repetition rate of the laser pulses. Identical PL band spectral positions were found from the damage bit areas in both kinds of dry v-SiO$_2$ used (Fig. 1).

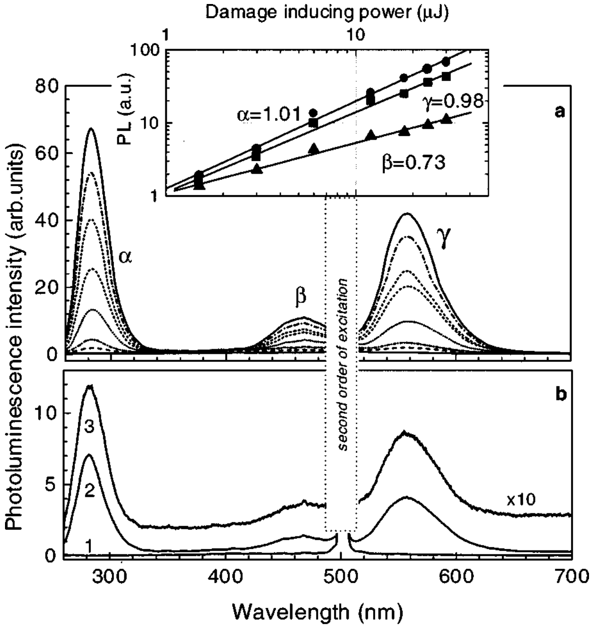


FIG. 1. (a) Photoluminescence spectra of dry v-SiO$_2$ (*ED-B*) optically damaged by 800 nm, 130-fs pulses of 0.75–30 μJ energy as measured at the entrance of the microscope. The corresponding eight damage-inducing irradiation energies per pulse as experienced by the sample were 9 nJ (no damaged induced), 30, 60, 90, 240, 360, 480, and 600 nJ, in accordance with a light attenuation of 0.02 introduced by the microscope. (b) Spectra of dry *ED-B* v-SiO$_2$ before (1) and after modification by 400-nm irradiation (2). The spectrum of *ED-C* v-SiO$_2$ damaged by 532 nm, 30-ps pulses (3) is presented 10 times multiplied. The power laws of α, β, and γ PL band intensities are presented in the inset: intensity of photoluminescene bands vs damage-induced irradiation energy at 800 nm. The PL was excited by 250-nm irradiation. The second order of excitation at 500-nm is covered by a boxed area. Up to 11% of PL in γ band can be caused by a second diffraction order of 280-nm band.

The power law of defects generation vs the damage-inducing pulse energy can be extracted from the PL band intensity [Fig. 1(a)]. As can be seen in the results plotted in the inset of Fig. 1(a) linear dependencies were found valid for the 283- and 558-nm PL bands, while a slope of 0.73 was found for the 468-nm band (detailed consideration in Sec. III C). Since the irradiation of the silica was carried out with

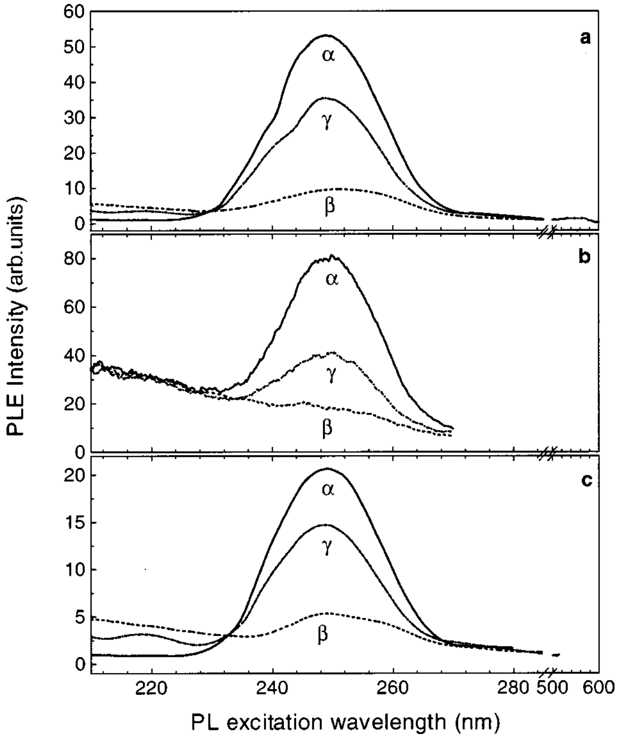


FIG. 2. Photoluminescence excitation spectra of v-SiO$_2$ where the bit damage areas were created by 800 nm, 24 μJ energy per pulse irradiation in *ED-B* v-SiO$_2$ (a), by 532 nm, 0.2 μJ energy per pulse irradiation in *ED-C* v-SiO$_2$ (b), and by 400 nm, 0.5 μJ energy per pulse irradiation in *ED-B* (c). The PL was excited by 250-nm irradiation.

sub-band-gap energy photons i.e., the highest energy per quantum was 3.1 eV (400-nm irradiation), the defect generation was expected to occur due to multiphoton (MP) absorption. The deposited laser shot power creates damage at the peak of a focused Gaussian beam profile where the intensity is sufficient to produce MP excitation of free carriers in v-SiO$_2$. These free carriers are heated further by a one-photon absorption of the same laser pulse.[35] The excess energy of free carriers is coupled with the lattice on the ps scale, which is what is responsible for the difference between fs and ps fabrication of damage bits. In the case of fs pulses, the lattice gains the energy after the end of the pulse, while for ps irradiation the lattice is heated up during the pulse itself. The eventual damage area has been found to be different in size for ps and fs fabrication.[24] In both cases, the area is composed of two regions: a central part constrained within a focal point with an approximate diameter of $\lambda/2A_N$ in accordance with the diffraction-limited spot size, where λ is the irradiation wavelength, and an outer shell of densified v-SiO$_2$ approximately 1 μm in diameter. The described process of defects creation is consistent with our observed first (α and γ PL bands) or lower (β band) order power law (inset of Fig. 1), since the process corresponds to the one-photon absorption by light-generated carriers (the nature of the defects is discussed in Sec. III C).

The new experimental findings suggest that PLE spectra originating from damaged areas created by fs and ps sub-band-gap irradiation exhibit peaks at 250 nm with a bandwidth of 18–20 nm, as depicted in Fig. 2. The PLE maximum of all three PL bands at 250 nm is due to a primary absorption at the oxygen vacancy V_O center.[15,29] The same spectral positions of the PL bands and their excitation spectra imply that the same silica structural modifications were introduced by 130-fs or 30-ps pulse irradiation at different wavelengths; nevertheless, it has been reported[35] that the morphology of the bit area created inside the glass is different from that on the surface, where fracture and ablation are observed in the case of pulses shorter than 10 ps, with melting occurring for longer pulses. Again, this phenomenon can be explained by defect generation originating from a one-photon absorption process.

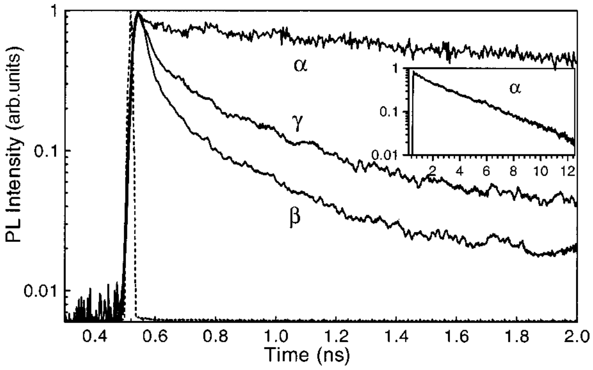


FIG. 3. PL time decays for α, β, and γ PL bands. The single exponential decay (3.7 ns) of PL at 283 nm on an extended time scale of 11 ns is presented in the inset. The instrumental response is depicted by a dashed line.

B. Photoluminescence time decay

The PL time decay is presented in Fig. 3 for all three PL bands. There was no difference between the time decays of PL from damaged areas introduced by different wavelengths or pulse duration, or made in different kinds of dry v-SiO$_2$. We can see that after a 250-nm excitation, only the 283-nm band decays exponentially with a time constant of 3.7 ns, which is consistent with the reported value of 4.2 ns for the $S_1 \rightarrow S_0$ transition of oxygen vacancy.[15,29] The other two bands show more complex stretched exponential decay resembling the PL decay of the α-PL band over a longer period (tens of ns). Since PL was excited by an initial absorption at the V_O defect, it is reasonable to expect that different PL bands occur due to the recombination of electrons and holes distributed over neighboring defect sites. However, initial trapping was not resolved within our temporal resolution of 50 ps. Later these separated carriers recombine yielding in PL, which decays by stretched exponential law. Of the defects involved, most are probably oxygen interstitials in the form of O$_i$ and (O$_2$)$_i$, which are the counterparts and nearest neighbors of V_O and which act as electron traps due to the electronegativity of oxygen.

C. Defect generation

One of the most probable mechanisms for V_O generation could be: Si-O-Si$+N\times h\nu \rightarrow (V_O;O_i)$, where the oxygen vacancy–interstitial pair is the result of silica optical damage induced by N photons of energy $h\nu$ (O$_i$ is expected to form a weak donor-acceptor linkage with the nearest normally coordinated O atom). Here, we assume that the consumed photons heat the damage area and favor stable (separated enough) vacancy-interstitial generation. Therefore, the number N is not directly related to the N-power law of defect generation, but rather to the efficiency of the one-photon absorption (first power-law) process. A similar reaction for the creation of another pair of defects (E';O_i^-) has been reported.[10] In addition, it has been shown that annealing at 600 °C annihilates the vacancy-interstitial pair.[15] At our employed silica irradiation conditions, the temperature was as high as the melting point of silica at about 1710 °C (Ref. 36) in the focal point. After such an annealing, the presence of the vacancy interstitial, (V_O;O_i), is more common in the area surrounding the damage bit where the temperature was somewhat lower. This can explain, in part, the lower power law (slope 0.73) for the PL β band, when some of vacancy-interstitial pairs relax into the initial form of Si-O-Si due to such an annealing.

Another more chemically stable pair (V_O;$(O_2)_i$) can be created in the central part of the damage, since longer vacancy-interstitial distances can be induced. For this to occur, it is necessary for two oxygen interstitials O_i to be able to react with each other to form $(O_2)_i$. This defect pair can be created by the following reaction: $2(\text{Si-O-Si})+N\times h\nu \rightarrow V_O+(V_O;(O_2)_i)$, which implies the generation of a V_O defect and a pair (V_O;$(O_2)_i$) in equal quantities. We found that the same power law of defect generation for α and γ bands (inset of Fig. 1), which implies that there is a proportional density for these defects. On the basis of the presented experimental data, we propose that the presence of PL bands is due to a recombination of electrons and holes separated on the defect pairs (V_O;$(O_2)_i$) and (V_O;O_i) for PL bands γ and β, respectively.

The intradefect origin of the PL band at 468 nm is supported by observation of nonexponential PL decay[10] probed at this wavelength, which is due to the disorder-influenced distance distribution between the reacting defect pairs. The almost twice-broader PL bandwidth observed at 558 and 468 nm as compared with the 283-nm band (Fig. 1) reflects the distance distribution between the defects involved.

Under the irradiation intensity considered, ~100 GW/cm^2, the generation of permanent defects such as E', V_O, $(O_2)_i$, or O_i^- is possible. The creation of metastable Frenkel defect pairs in c-SiO_2 and fused silica by electron irradiation has been reported.[6] The possible mechanism of the defect generation has been proposed in terms of the nonluminescent recombination of self-trapped excitons.[6,32] This concept was originally introduced to explain defect generation in alkali halides and has been shown to be suitable for glasses.[37] The generation of electrons and holes by MP absorption causes a relaxation of material in response to the creation of a pair of positively and negatively charged defects, which eventually traps the generated carriers.

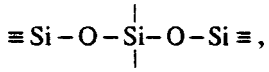
The densification of the glass in the vicinity of the focal point is a natural consequence of the microexplosion[25] that takes place inside glass during irradiation; i.e., melted glass is accelerated sideways from the focal point and is then thermally quenched, freezing in the defects. The resulting defects induced in the densified glass are stable on a yearly scale, according to our experience; these results suggest that it is possible to create stable Frenkel pairs[6] since elevated temperatures during the sample preparation are favorable for large interstitial-vacancy distances.

D. Photoluminescence bands

Discrepant data on the defects in silica and related PL bands are available, e.g., the 290-nm band has been attributed to the spin selection rule forbidden triplet-to-singlet $T_1 \rightarrow S_0$ transition,[3,38] but more recent experimental data suggest the presence of a singlet-singlet $S_1 \rightarrow S_0$ transition[15,29] for the oxygen vacancy. Physically, this means a transition between the localized states that result from the bonding and antibonding combination of the *sp* orbitals of two Si atoms[15] yielding a 283-nm luminescence.

Similar to our observed PL band, a band 50 nm in width at 468 nm has been reported when silica is excited by UV irradiation of 7.9 eV (Ref. 29) or by electrons[6] or γ rays.[7–9] Such luminescence has been attributed to the radiative decay of self-trapped excitons after throughout–band-gap excitation by electrons[10] or UV irradiation.[5] Self-trapped excitons are considered to be an interstitial-vacancy pair, for which the interstitial takes the form of an O_2 molecule according to the optically detected–magnetic-resonance data.[39] In addition, PL in the spectral region of 460–520 nm have been attributed to the triplet state decay of V_O,[15] although the triplet-state lifetime should exceed that of the singlet state. We, however, have observed a faster decay of the β band than that of the α band (Fig. 3). The PL excitation pulses were tested at different repetition rates to avoid overexcitation, but PL transients longer than those shown in Fig. 3 were not obtained.

Similar 2.2 eV (564 nm) and 0.44 eV bandwidth PL bands have been reported in γ-rays–irradiated glass[9] when the PL is excited by 3.8-eV (326-nm) He-Cd laser irradiation. This PL band has been attributed to the radiation of a paramagnetic defect, where one electron is delocalized over a cluster of five Si atoms. The generation process of V_O and $(O_2)_i$ defects when the oxygens are misplaced into an interstitial location after a breakage of the four nearest Si-O bonds of the same Si atom, which is in the middle of the fragment



$$\equiv \text{Si}-\text{O}-\overset{|}{\underset{|}{\text{Si}}}-\text{O}-\text{Si}\equiv,$$

results in a structure resembling the paramagnetic defect of the five Si atoms considered above. Moreover, the Si-O-Si or Si-V_O-Si intertetrahedral bond angle is known to be 144 in silica,[7,38,40] although it has been found to be reduced in densified glasses.[7] Then, under the conditions of optical silica damage, some of the Si from neighboring Si-O-Si chains can come near enough for the pairs to resemble Si-Si unrelaxed bonds. Such modification is consistent with the well-accepted view[41] that glass densification is caused by a reorganization of the ringlike structure of -SiO_4- ion tetrahedrons into a similar structure consisting of similar rings.[7] The V_O defect acts as a ring breaking-closing point. It has been reported that even a new phase of silica can be formed at the high-pressure (>21 GPa) and elevated-temperature (~600 K) conditions[42] that are expected to be met during the optical damage of silica.[25] Under the high-intensity irradiation we employed to damage the v-SiO_2, the generation of (V_O,$(O_2)_i$) could produce a cluster of five Si atoms. Nevertheless, whether this cluster belongs to the luminescent species itself is not obvious. According to the model proposed here, PL arises from the recombination of carriers trapped on the pair of oxygen interstitial vacancy, but it is not due to the bimolecular recombination of carriers inside the Si cluster.

Another proposed explanation for the 2.2-eV PL band that appears after a band-to-band excitation is that the band is due to a self-trapped exciton radiative decay, by which interstitial oxygen is returned into a stoichiometric Si-O-Si position,[10] annihilating the defect itself. A similar origin has been attributed to the 2.8-eV PL band of c-SiO_2.[2] It has been reported that these defects are unstable when induced at cryogenic temperatures.[10] Based on our PL data, it follows that the β and γ bands (Fig. 1) are evidently related to the initial absorption at the V_O center due to their similar PLE spectra (Fig. 2). The observed PL band at 558 nm was excited by subband-gap excitation, which excludes the self-trapped exciton from consideration. Also, our proposed model in which the oxygen interstitial-vacancy pairs are responsible for the PL bands is consistent with the established fact of a low rate of O_2 diffusion in densified glasses with an activation energy of 1.3 eV.[7,17]

It is noteworthy to mention, that a PL band at 650 nm, which is usually attributable to the PL of the nonbridging oxygen hole center $\equiv$Si—O·,[9] was observed only in the silica irradiated by the intensities 2–3 times higher than that utilized in the experiments described.

IV. CONCLUSIONS

We report, to our knowledge, the first observation of PL and PLE spectra originating from optically damaged dry v-SiO_2. PL bands at 283, 468, and 558 nm were observed. These bands can be induced by visible (400 nm, 532 nm) and near-infrared (800 nm) irradiation of silica with fs (400 nm, 800 nm), or ps (532 nm) focused pulses and are attributed to the following transitions: $S_1 \rightarrow S_0$ of the oxygen vacancy defect for 283 nm, and the radiative recombination of separated carriers on the pair of defects (V_O;O_i) and (V_O;$(O_2)_i$) for 468 and 558 nm. The proposed model of defect structures can explain observed PLE spectra and PL time-decay data, and it is also consistent with the data reported thus far. However, the distinct location of the defects in the silica's band gap needs to be established.

The unique conditions of optical silica modification employed in our experiments, namely: high-temperature annealing of nonradiative centers in the region of optical damage; no contact of the damaged area with the outside atmosphere; no average stoichiometry changes induced during irradiation; and the high purity of the silica are favorable in using such optically damaged silica as a reference material for investigating defects in modified glasses.

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