Research Article

Influence of Radiation on the Luminescence of Silicon Nanocrystals Embedded into SiO$_2$ Film

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Influence of γ-irradiation on light emission properties of silicon nanocrystals imbedded into SiO$_2$ film is investigated. It was shown that small doses of γ-irradiation (10^3–10^5 rad) lead to enhancement of photoluminescence intensity in the nc-Si/SiO$_2$ samples. This effect was explained by radiation induced passivation of recombination active centers on the nanocrystals surface. High doses of irradiation ($\sim 10^7$ rad) lead to the photoluminescence intensity decrease up to 2 times. Radiation treatment of silicon oxide films with embedded amorphous silicon inclusions resulted only in the decrease of the photoluminescence intensity within the whole range of doses (10^3–5×10^7 rad). Radiation defects resulting in partial quenching of photoluminescence are characterized with the distributed activation energy of annealing with the peak position at ~ 0.96 eV and the frequency factor 10^7 s^{-1}. The nature of such defects and the mechanisms of their creation are discussed.

1. Introduction

It is well known that intensive irradiation of silicon dioxide films in vacuum is capable to form silicon nanocrystals in the result of radiation induced reduction of SiO$_2$ [1, 2]. This phenomenon and the corresponding mechanisms have been studied in detail. At the same time, study of influence of ionizing radiation on the properties of SiO$_2$ films with imbedded Si nanocrystals (nc-Si/SiO$_2$ film structures) was attended much less. On the one hand, Si nanocrystals are predicted to have high radiation hardness [3]. In fact, significant degradation of photoluminescence (PL) of the porous silicon, connected with radiation defects creation, was observed after irradiation by rather high doses (2×10^9–10^{10} rad) [4, 5]. Effects of PL intensity enhancement after exposure by lower doses (4.3×10^6–3×10^8 rad) [6] were observed only in the case of irradiation in air atmosphere [5] and were attributed to additional oxidation of porous Si surface under corrosive action of ozone, atomic oxygen, and hydrogen that were formed by illumination. This led to reduction of the

concentration of nonradiative recombination centers on the surface of silicon nanocrystals and sometimes to decrease of their size.

On the other hand, one may expect that ionizing irradiation with quanta or particles (X- and γ-rays, electrons) is capable of substantially changing the emitting characteristics of the nc-Si/SiO$_2$ film structures, taking into account a high radiation sensitivity of the Si–SiO$_2$ interface. The latter is known results in growth of the fast surface states density, these states being related with silicon to broken bonds [7–9]. In fact, certain enhancement (~ 1.5 times) of luminescent intensity was observed by us earlier [10] after low dose ($\sim 10^4$ rad) ionizing irradiation (γ-ray photons) of SiO$_2$ films with embedded Si nanocrystals. Contrary to the case of porous silicon, this effect in nc-Si/SiO$_2$ structures was observed after radiation treatment even in the inert gas or in vacuum; hence, it had another nature. Recently, the authors of [11] reported that proton irradiation (the energy 3.5 MeV and the total fluence 10^{12}–10^{15} cm^{-2}) of nc-Si in SiO$_2$ multilayers leads to significant decrease of the intensity of luminescence

centered near 730 nm, and in appearance of the luminescence band near ~500 nm, assigned to radiative recombination at the radiation induced centers within a-SiO$_2$ matrix. Larger exposure doses (~10^6–10^7 rad), similarly to the case of the porous silicon, led to a partial quenching of PL of the nc-Si/SiO$_2$ films [10]. Investigation of just this effect to elucidate the nature of the radiation defects created under ionizing exposure of nc-Si/SiO$_2$ films has been done in the present paper, study of the process of radiation damage annealing being mainly attended.

2. Experimental

SiO$_x$ films ($x \approx 1.12$) have been produced by thermal evaporation of SiO powder (Cerac Inc., purity of 99.9%) in vacuum (2×10^{-3} Πa). SiO$_x$ film thickness was measured after the deposition by the microinterferometer MII-4 and amounted to 450 nm. Several SiO$_x$ films were implanted with nitrogen ions N$^+$ with the energies of 70 and 140 кeV and with the total dose of 8.4×10^{15} cm^{-2} (further nc-Si/SiO$_2$:N structures). SIMS measurements have confirmed a uniform distribution of the nitrogen in SiO$_x$ film with depth.

SiO$_x$ films were thermally treated in the argon ambient at 700°C or at 1100°C for 30 min. As it was demonstrated by us earlier [12] using electron diffraction and the high resolution transmission electron microscopy, these thermal treatments resulted in creation of amorphous Si nanoinclusions (na-Si) imbedded into oxide matrix SiO$_y$ ($y \approx 1.24$) or of crystalline Si nanoinclusions (nc-Si) about 3 nm in size homogeneously distributed within the SiO$_2$ matrix, correspondingly.

Infrared (IR) transmission spectra were measured using FTIR spectrometer Spectrum BXII PerkinElmer. A *silicon* substrate (without oxide film) served as the reference sample. The absorption band related to Si–O bonds (maximum position within the range of 1000–1100 cm^{-1} depending on the oxygen content in the oxide film) was under investigation.

Photoluminescence (PL) spectra were measured at room temperature within the spectral range of 550–1000 nm under the excitation at the 473 nm from the semiconductor laser. The excitation power was about 50 mW. Measured PL spectra were corrected with respect to the spectral sensitivity of measurement setup.

Creation of Si nanocrystals in the nc-Si/SiO$_2$ and nc-Si/SiO$_2$:N structures due to high temperature annealing resulted in appearance of rather strong red PL band (peak position in the vicinity of 800 nm, see curve 1 in Figure 1(a)), corresponding value of the energy of light emission (~1.6 eV) being in agreement with that calculated or found experimentally for nc-Si with the size of ~3 nm [13]. PL intensity for the samples doped with nitrogen was by ~3 times larger (see curve 1 in Figure 1(b)), which is also agreed with the data [14]. In the case of na-Si/SiO$_y$ samples, PL band was blue shifted (peak position at ~680 nm, see curve 1 in Figure 1(c)) that is in agreement with the known results [15]. The value of corresponding energy (E) enabled us to estimate the size (d) of amorphous Si inclusions, using phenomenological dependence $E = 1.56 + 2.40d^{-2}$ [16]: it was ~3.2 nm.

na-Si/SiO$_y$, nc-Si/SiO$_2$, and nc-Si/SiO$_2$:N samples were irradiated with γ-quanta (^{60}Co) with the intensity 36.77 rad·s^{-1} and the energies 1.17 and 1.33 MeV. The dose of exposure varied in the range of $10^3 \div 5 \times 10^7$ rad. The temperature of the samples during γ-irradiation did not exceed 30°C. Irradiation was carried out both in air atmosphere and in vacuum (10^{-3} Torr).

Set of irradiated samples was subjected to isochronous ($50 \leq T \leq 300°C$, $\Delta T = 10°C$) and isothermal ($\Delta t = 2$ min) anneals in argon atmosphere.

3. Results and Discussion

Figure 1 demonstrates influence of radiation on the PL red band for the nc-Si/SiO$_2$ (a), nc-Si/SiO$_2$:N (b), and na-Si/SiO$_y$ (c) samples. Dose dependences of normalized PL intensity (I_{PL}^R/I_{PL}^i, where I_{PL}^R and I_{PL}^i are the values of PL intensity for irradiated and initial samples, resp.) for all investigated structures are presented in Figure 2. It is seen that behavior of the samples under irradiation is different depending on the type of structure: the na-Si/SiO$_y$ samples are the less sensitive to influence of exposure. Moreover, for Si/SiO$_2$:N and na-Si/SiO$_y$ structures PL intensity monotonically decays with the time of irradiation within the whole range of exposure doses. In contrast to this the dependence of I_{PL} on the exposure dose for the nc-Si/SiO$_2$ structures is nonmonotonous. In the range of irradiation doses less than ~10^5 rad the "low dose effect" was observed. This effect consisted in the remarkable (~1.7 times) increase of PL intensity with respect to that for the initial sample. Further irradiation resulted in decrease in the PL intensity, which becomes smaller than the initial one at the irradiation doses above 5×10^5 rad. "Low dose effect" was observed in the nc-Si/SiO$_2$ samples irradiated both in air atmosphere and in vacuum. This indicates (contrary to the case of porous silicon) the mechanism related to the interaction of γ-quanta with the sample, but not atmosphere influence.

The "low dose effect" was explained earlier [10] by passivation of silicon broken bonds on the surface of nc-Si inclusions with hydrogen atoms and hydroxyls, which are released in oxide matrix due to decomposition of SiH and SiOH complexes under irradiation. Data obtained here for the case of irradiation of nc-Si/SiO$_2$:N structures seem to confirm such a hypothesis. In these structures silicon broken bonds at nc-Si–SiO$_2$ interface have been partially or completely passivated by nitrogen atoms during the process of sample formation [14, 17, 18], that is why initial PL intensity for nc-Si/SiO$_2$:N samples was three times higher comparing to ordinary nc-Si/SiO$_2$ ones. Additional passivation under irradiation of a rather small amount of remaining defects is negligible and does not influence I_{PL}; thus the irradiation of nc-Si/SiO$_2$:N systems led to monotonous I_{PL} decrease only (see Figures 1(b) and 2).

High doses of irradiation resulted in essential decay of the PL intensity (see Figures 1 and 2). It was expected and may be connected with creation of radiation defects at nc-Si/SiO$_2$ interface, which are the centers of nonradiative recombination. Such processes were widely studied earlier in planar
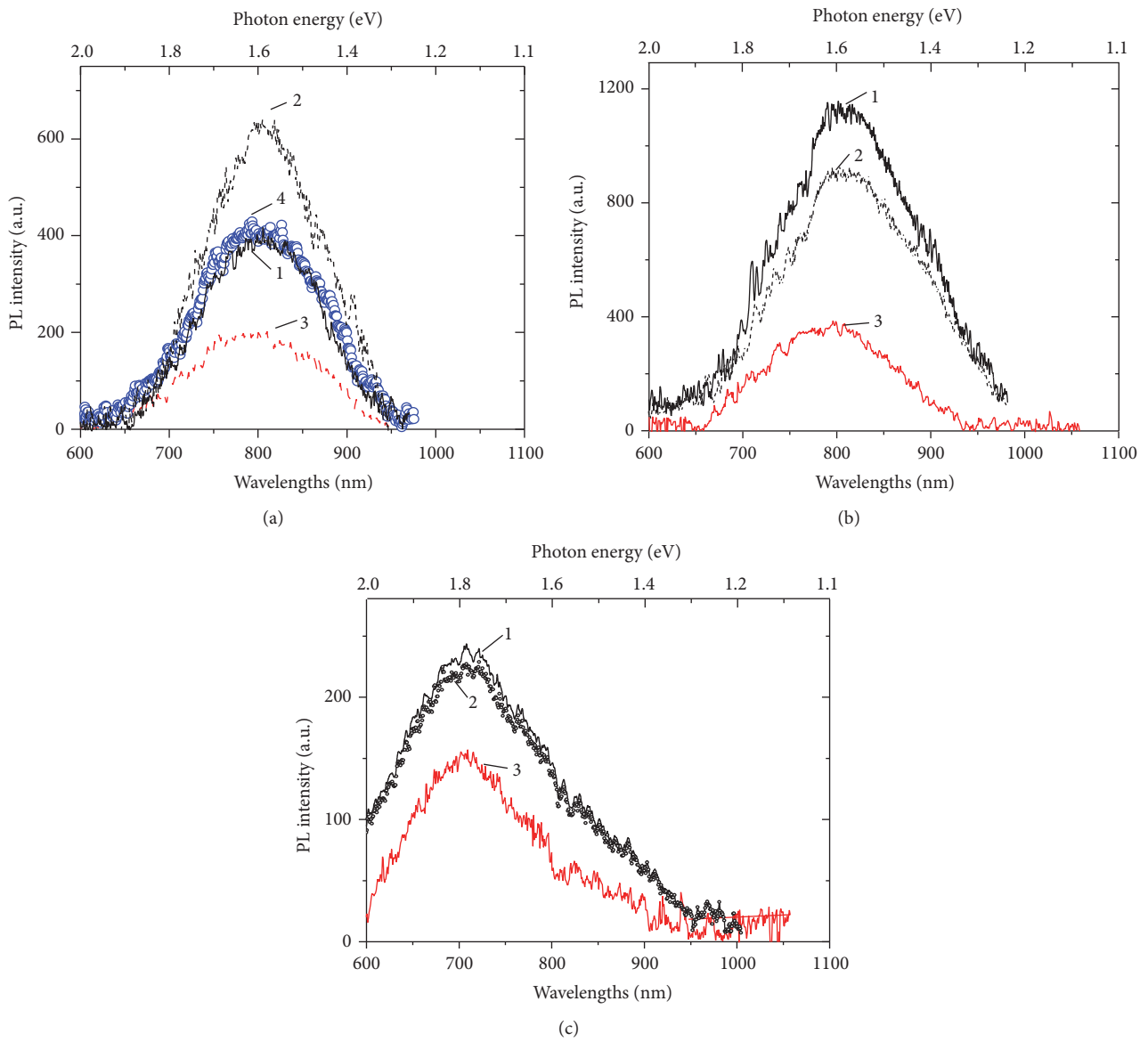
Figure 1: PL spectra for nc-Si/SiO$_2$ (a), nc-Si/SiO$_2$:N (b), and na-Si/SiO$_y$ (c) samples. Curves notation: 1, initial samples; 2 and 3, samples irradiated with the doses of 2×10^4 rad and 2×10^7 rad, respectively; 4, sample irradiated (2×10^7 rad) and annealed at $T = 300°$C.
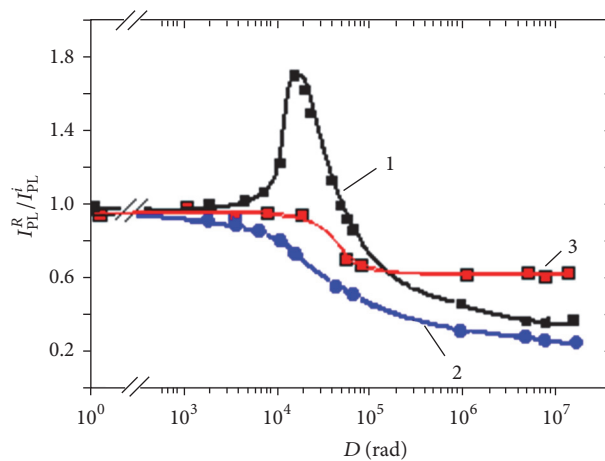

Figure 2: Dose dependence of PL band intensity for nc-Si/SiO$_2$ (1), nc-Si/SiO$_2$:N (2), and na-Si/SiO$_y$ (3) structures.

Si/SiO$_2$ systems and, naturally, the general corresponding approaches may be spread for the case of oxide films with embedded silicon nanoinclusions. However, observation of low dose effect in nc-Si/SiO$_2$ structures enabled us to suppose that there are two competing processes, namely, those leading to enhancement of I_{PL} and those quenching it (formation of nonradiative recombination centers). Low doses of ionizing radiation lead to radiation stimulated passivation of Si dangling bonds at the nc-Si–SO$_2$ interface, which is more efficient comparing to formation of nonradiative recombination centers. Increase of I_{PL} is observed in result. Passivation can be provided by, for example, the hydrogen atoms and hydroxyls, which are formed in the oxide matrix due to decomposition of SiH and SiOH complexes under the irradiation. Si nanocrystals in nc-Si/SiO$_2$ systems play a role of the gettering centers, leading to rise in the efficiency of collection on their surfaces of mentioned passivating agent already at the room temperature.

Defect formation leading to decrease of I_{PL} becomes dominating at higher irradiation doses ($D > 2 \times 10^4$ rad). The equilibrium in both defect formation processes is obtained at the irradiation dose of 2×10^4 rad. At this dose, the concentration of passivated Si broken bonds becomes approximately equal to the concentration of formed nonradiative recombination centers.

Defects formed by irradiation may be eliminated due to low-temperature treatment in vacuum or in inert ambient, resulting in restoration of the PL intensity (see curve 4 in Figure 1(a)). At the same time, IR absorption band connected with stretching vibrations of oxygen atoms in Si-O-Si units (maximum position near ~1090 cm^{-1}) with the shape inherent for SiO$_2$ phase [19] practically did not vary after irradiation within the whole dose range and low-temperature heat treatments in inert ambient both of the initial and irradiated nc-Si/SiO$_2$. This fact should mean that irradiation and thermal treatments used in this study did not affect the concentration and structural arrangement of the bridging oxygen atoms in the oxide matrix.

For clarification of the nature of radiation defects, which lead to PL quenching, as well as the mechanism of their formation, detailed study of the thermal annealing processes of radiation induced defects in nc-Si/SiO$_2$ structures was carried out.

In Figure 3 the results of isochronal annealing (temperature step $\Delta T = 10°$C, duration of anneal at every temperature $\Delta t = 10$ minutes) are presented. It is seen that heat treatment of initial sample did not affect PL intensity within the whole range of temperatures (50–300°C). In the case of irradiated sample, restoration of I_{PL} up to the initial state takes place in the temperature range of $200 \div 300°$C (Figure 3).

Isothermal annealing ($200 < T < 300°$C) demonstrated that defect elimination at every temperature is only partial. The temperature determines not only the annealing rate but also the amount of nonannealed portion N of the radiation damage. This result is important and means [20] that the radiation defect annealing in nc-Si/SiO$_2$ structures is characterized by not single activation energy but that distributed in some energy interval.
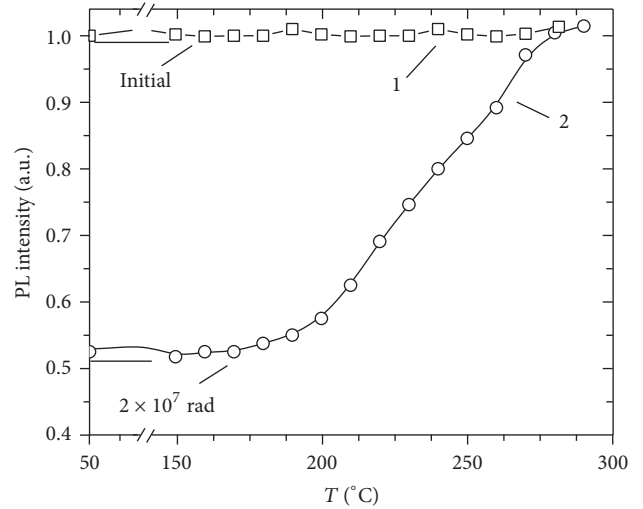

Figure 3: Dependence of the PL intensity on the annealing temperature for initial nc-Si/SiO$_2$ sample (1) and nc-Si/SiO$_2$ sample irradiated with the dose of 2×10^7 rad (2).
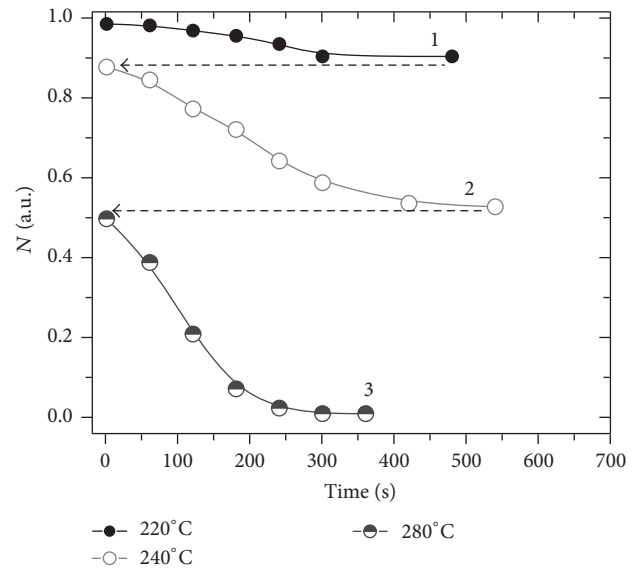

Figure 4: Curves of isothermal annealing for irradiated nc-Si/SiO$_2$ sample.

To determine this activation energy distribution according to [20] a combined heat treatment of one sample was performed, consisting of three successive isothermal annealing cycles at 220, 240, and 280°C. The results are presented in Figure 4. Nonannealed portion of radiation damage was determined as

$$N = \frac{I_{PL}^a - I_{PL}^i}{I_{PL}^R - I_{PL}^i} \tag{1}$$

with I_{PL}^i, I_{PL}^R, and I_{PL}^a being the PL intensity for nonirradiated, irradiated, and annealed samples, respectively.
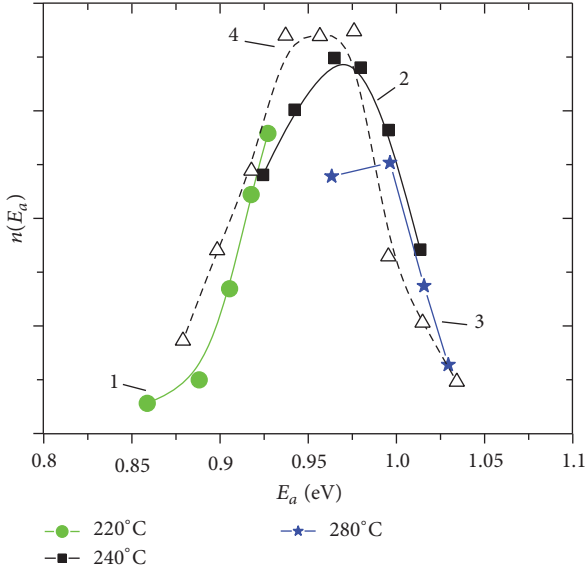
FIGURE 5: Distribution of the activation energy of annealing of radiation damage in nc-Si/SiO$_2$ structure. Segments were obtained from the results of isothermal annealing. Curve 4 was obtained from the results of isochronous annealing. The value of A is 10^7 s^{-1}. Triangles reflect the results obtained from analysis of isochronal annealing, which is described in the caption of Figure 5.

Using the data obtained for the annealing experiments the activation energy E_a and the energy distribution function $n(E_a)$ were calculated from the following expressions [20, 21]:

$$E_a = \tau \ln (At),$$

$$n(E_a) = -\left(\frac{t}{\tau}\right)\left(\frac{dN}{dt}\right) \qquad (2)$$

for the isothermal anneals and

$$\frac{E_a}{\tau} + \ln\left(\frac{E_a}{\tau}\right) = \ln A\Delta t,$$

$$n(E_a) = -\left(\frac{dN}{d\tau}\right)\left(\frac{E_a}{\tau}\right)^{-1} \qquad (3)$$

for isochronous annealing, respectively. In expressions (2) to (3), $\tau = kT$ is the thermal treatment time and A is the frequency factor, respectively.

Using expressions (2) to (3) for each isothermal annealing cycle (Figure 4), the energy distribution functions were built (Figure 5). The value of A ($A = 10^3$–10^{10} s^{-1}) was fitted at the calculations of $n(E_a)$ so as to provide a continuity of distribution between different segments (each of which corresponded to certain isotherm in Figure 4). The best fit corresponded to $A = 10^7$ s^{-1}. It can be seen that the calculated distribution function curves based on different experiments (isothermal and isochronous annealing) have a good agreement. The activation energy of annealing of radiation defects that results in a partial decrease in I_{PL} in the nc-Si/SiO$_2$ structures is distributed in the range of 0.85–1.05 eV with the peak at ~0.96 eV.

The values of E_a and A were compared to those for the radiation defects both in the single crystal Si bulk and in the planar Si/SiO$_2$ systems as well as in the near surface oxidized Si layer [20, 22–25]. It is well known that the primary radiation defects in silicon crystals have rather small values of the activation energy of annealing (in the case of vacancies, for example, the value of E_a amounts the meanings of 0.18 and 0.33 eV for n- and p-Si, resp. [25]). The most part of the secondary radiation defects (in particular, agglomerations of the primary defects with impurity atoms) has E_a values much larger (e.g., complexes of vacancies with oxygen atoms) or much less (e.g., complexes of vacancies with donor impurities). Only some secondary radiation defects in silicon crystals may have the values of E_a similar to that calculated in this study. Thus, for example, Si–P6 defect, which includes Si interstitial atoms, is characterized with $E_a = 0.92$ eV; however it was observed only after neutron irradiation [25]. Vacancy-phosphorous atom complexes (E-centers) have the value of $E_a = 0.95$ eV [25], but the frequency factor of their annealing process is too high ($A = 7 \times 10^8$ s^{-1}), and appearance of such defects in the nc-Si/SiO$_2$ samples seems to be unexpected.

However, the data discussed above are related to the defects which are localized within the bulk of Si crystals. Presence of surface may influence temperature stability of radiation defects. It is known, in particular, that the surface layer (about $2\,\mu$m in depth) of oxidized Si single crystals differs from silicon bulk both by larger sensitivity to ionizing radiation and by parameters of radiation damage annealing [26]. Silicon nanocrystals with the diameter of some nanometers embedded into SiO$_2$ matrix and thus strongly mechanically stressed [27] are more similar to such a surface layer than to silicon bulk. Hence, it seems to be more correct to compare the calculated annealing parameters with those obtained for surface layer of irradiated oxidized silicon crystals. Investigations of the generation life time of the minor carriers in the silicon space charge region under γ-irradiation (with the dose up to 10^6 rad) made it possible to determine the value of the activation energy of annealing of the corresponding surface radiation defects; it amounted (in the maximum of distribution) the meanings of 1.1–1.2 eV depending on the oxidizing technology regimes [26]. This value also differs from that calculated in the present study.

However, it can be easily shown that in our experiments radiation defects, which have been created in silicon nanocrystals, can not influence their light emitting ability. In fact, it is well known that in Si one γ-quant may form about 10^{-3} cm^{-3} of primary radiation defects [28]. Then the number of the point defects, which have been generated in the bulk of all silicon nanocrystals (with the diameter of ~3×10^{-9} m and the concentration of ~10^{18} cm^{-3}) after irradiation with the dose of 2×10^7 rad (or ~3.4×10^{16} γ-quanta), should be about 10^{13} cm^{-3}. On the other hand, a decrease in the PL intensity in the nc-Si/SiO$_2$ structures is known [29] and may be observed if the concentration of the centers of nonradiative recombination amounts the value of 10^{16}–10^{18} cm^{-3}. Thus, one can conclude that in the irradiated nc-Si/SiO$_2$ structures

radiation defects, which influence the PL intensity value, are not localized within the bulk of silicon nanocrystals.

Some decades ago the processes of radiation damage creation and elimination in the MOS structures have been studied intensively. In particular, the values of activation energy of annealing both the centers in the oxide and the fast surface states at the Si–SiO$_2$ interface which were created in the result of different kinds of radiation treatment have been determined [20, 24, 26]. In all the cases activation energy was distributed along the energy axe with the peak position near 0.9 eV, and the meaning of the frequency factor was 10^7 s^{-1}. These values practically coincide with those calculated in the present work. Hence, one may conclude that the radiation defects, which are formed by γ-irradiation of nc-Si/SiO$_2$ structures and lead to partial quenching of luminescence, are located, namely, at the nc-Si–SiO$_2$ interfaces. These defects are the centers of nonradial recombination, their nature and the formation mechanism being identical to those characterizing the radiation surface states in Si/SiO$_2$ planar systems.

The most probable radiation induced defects at the Si–SiO$_2$ interface are the silicon broken bonds Si^{3+} [9]. However, some alternative models exist to explain the mechanism of these defects creation due to active treatments (irradiation, hot carriers injection) [9, 30, 31]. Their main mutual feature is that surface states are the secondary defects; the process of radiation damage generation under irradiation takes place in SiO$_2$ film. In fact, the concentration of the silicon broken bonds, which are created by ionizing radiation within the oxide films at the doses of 10^6–10^7 rad, amounts the value of 10^{18}–10^{19} cm^{-3} [32]. This number is not sufficient to influence the Si–O absorption band, but sufficiently large to affect the PL intensity in the case of nc-Si/SiO$_2$ samples.

4. Conclusions

Gamma irradiation of nc-Si/SiO$_2$ structures with low ($<10^5$ rad) doses both in air and in vacuum resulted in pronounced rise in PL intensity. This effect was explained by radiation assisted passivation of centers of nonradiative recombination at nc-Si–SiO$_2$ interfaces. "Low dose" effect was absent in the case of na-Si/SiO$_y$ and nc-Si/SiO$_2$:N structures, where irradiation only led to monotonous decrease in the PL intensity.

Larger doses of γ-irradiation of nc-Si/SiO$_2$ structures resulted in the formation of radiation induced defects that significantly (twice at 10^7 rad) quench the photoluminescence. These defects are annealed due to heat treatments at the temperatures in the range of $200 \div 300°$C. The activation energy of annealing is distributed within the energy range of 0.85–1.05 eV with the peak at $\sim$0.96 eV. The frequency factor is 10^7 s^{-1}. The values of E_a and A coincide practically with those determined for the fast surface states formed at the Si–SiO$_2$ interface by ionizing radiation. This fact allows one to infer the accumulation of radiation induced defects partially quenching the photoluminescence, at the Si nanocrystal/oxide matrix interface. The nature of these defects and the mechanism of their formation are the most likely similar to those pertinent to the surface states formed by ionizing radiation at the planar Si–SiO$_2$ interfaces.
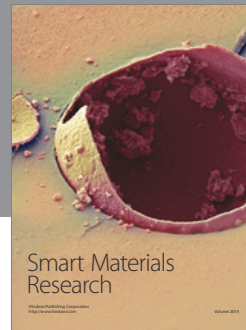
Competing Interests

The authors declare that they have no competing interests.

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