

# Effect of preliminary rapid annealing on the photoluminescence of nanosized silicon inclusions in multilayered $\text{SiO}_x/\text{SiO}_2$ and $\text{SiO}_x/\text{ZrO}_2$ structures

Alexey V. Ershov<sup>1</sup>, Daniil A. Yushkov<sup>1</sup>, Mikhail E. Suvorov<sup>1</sup>,  
Anton V. Zdoroveyshchev<sup>1</sup>, Nikolay V. Baidus<sup>1</sup>

*1 Lobachevsky State University of Nizhny Novgorod, 23 Gagarin Ave., Nizhny Novgorod 603022, Russian Federation*

Corresponding author: Alexey V. Ershov (ershov@phys.unn.ru)

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## Abstract

The effect of rapid thermal annealing (RTA) at 950 and 1000 °C for 3 min on the photoluminescence (PL) in multilayered (~50 layers) nanoperiodical (4/3 nm)  $a\text{-SiO}_x/\text{SiO}_2$  and  $a\text{-SiO}_x/\text{ZrO}_2$  nanostructures (MNS) synthesized using vacuum evaporation has been analyzed in comparison with conventional long-term (~1 h) high-temperature annealing (HTA). It has been shown that the use of sequential anneals (RTA + HTA) at 1000 and 1100 °C, respectively, allows increasing the intensity of the PL band of silicon nanocrystals (at ~800 nm) by 2.5 times as compared with conventional heat treatment. Analysis of the PL spectra suggests that RTA triggers the formation of chain and ring non-phase silicon inclusions and/or small-sized Si nanocrystals (~2 nm) which generate radiation near 600 nm at room temperature. Thus, one can conclude that RTA produces elevated concentrations of the abovementioned silicon nuclei which, upon subsequent HTA, serve as growth centers for larger Si nanocrystals (~3–4 nm). It has been found that  $\text{SiO}_x/\text{ZrO}_2$  MNS exhibit a similar behavior (qualitative) of PL spectra after RTA. However, the PL intensity in this case is 5–10 times lower, due to the chemical interactions between the materials of the MNS layers with the formation of zirconium silicides and silicates.

## Keywords

nanocrystals in dielectric matrix, multilayered nanostructures, rapid thermal annealing, photoluminescence, silicon

## 1. Introduction

In three recent decades, intense work has been done aimed at the synthesis and study of properties of Si nanocrystal (NC) arrays in wide-band dielectric matrices [1] for the development of silicon photonics through the integration of photocells and light emitting devices on one

chip using CMOS compatible technologies. Si NC arrays (Si quantum dots) in silicon dioxide ( $\text{SiO}_2$ ) matrix exhibit intense room temperature luminescence in visible and near-infrared (IR) regions [2, 3]. One of advanced methods to form Si NC arrays in dielectrics is long-term (1–2 h) high-temperature (900–1100 °C) annealing (HTA) [4, 5] of multilayered nanoperiodic structures (MNS) having a “silicon suboxide ( $\text{SiO}_x$ ) / dielectric” structure.

High-temperature heat treatment causes silicon suboxide disproportioning (into the  $\text{SiO}_2$  and Si phases), resulting in the formation of Si nanocrystals (NCs) in dielectrics, the NC sizes being determined by the initial thickness of the  $\text{SiO}_x$  nanolayers [4, 6]. In earlier works [5, 7] the effect of HTA on the structural and luminescent properties of silicon suboxide ( $\text{SiO}_x$ ) / dielectric MNS was studied, where the dielectric nanolayers were  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , or  $\text{ZrO}_2$ . Transmission electron microscopy, X-ray absorption near edge structure, and, photoluminescent spectroscopy methods were used to study the effect of long-term annealing temperature on the morphology of Si NCs, as well as the mechanisms of Si NCs growth and radiative recombination. The effect of varying the dielectric material in MNS ( $\text{SiO}_x/\text{SiO}_2$ ,  $\text{SiO}_x/\text{ZrO}_2$ , and  $\text{SiO}_x/\text{Al}_2\text{O}_3$ ) and the contribution of Si NC emission centers, excitons, dielectrics, and non-radiative Si NC/matrix interface states on the PL spectra were studied [5]. The results showed that dielectric material variation and interface states such as  $P_b$  centers after HTA have the least negative effects for  $\text{SiO}_x/\text{SiO}_2$  and  $\text{SiO}_x/\text{Al}_2\text{O}_3$  MNS [5, 7]. For the best Si NC-containing  $\text{SiO}_2$  specimens, the power efficiency of luminescence was estimated to be 0.2 % [3].

The relatively new rapid thermal annealing (RTA) method was used [8]. The photoluminescent properties were studied for Si NC having controlled size (2–6 nm), grown using different annealing methods in  $\text{SiO}_x/\text{SiO}_2$  superlattice structures. The heat treatment included rapid annealing (duration 10 to 280 s at 900–1100 °C) and HTA in a tube furnace (1 h) at 1100 °C. It was shown that the optimum combination of different heat treatments is preliminary RTA followed by HTA. Then the PL intensity of Si NCs increases by more than an order of magnitude, and additional hydrogen annealing used for the passivation of dangling bond-type defects allows increasing the PL intensity by more than two orders of magnitude. These results are in general agreement with data of earlier works on RTA of Si NC in  $\text{SiO}_2$  structures synthesized using silicon ion implantation in silicon dioxide [9, 10].

The aim of this work is to study the conditions providing for an increase in the room temperature PL of Si NC after rapid annealing combined with subsequent long-term annealing in tube resistance furnace for initially amorphous  $\text{SiO}_x/\text{SiO}_2$  and  $\text{SiO}_x/\text{ZrO}_2$  MNS synthesized by sequential vacuum deposition.

## 2. Experimental

The test specimens for rapid annealing experiments were amorphous  $\alpha\text{-SiO}_x/\text{SiO}_2$  and  $\alpha\text{-SiO}_x/\text{ZrO}_2$  MNS synthesized by sequential vacuum deposition of materials evaporated from different sources under conditions described in detail earlier [5, 7]. The materials were deposited on (100) KDB-12 silicon substrates at  $200 \pm 10$  °C. Silicon suboxide nanolayers in both types of nanoperiodic structures have similar average thicknesses of  $4.1 \pm 0.3$  nm,

and the thicknesses of the  $\text{SiO}_2$  and  $\text{ZrO}_2$  nanolayers were  $2.8 \pm 0.3$  nm and  $2.1 \pm 0.3$  nm, respectively. The  $\alpha\text{-SiO}_x/\text{SiO}_2$  MNS consisted of a total of 62 alternating layers, and the  $\alpha\text{-SiO}_x/\text{ZrO}_2$ , of 54 layers, the overall MNS thicknesses being  $225 \pm 15$  nm and  $160 \pm 15$  nm, respectively. The specimens were cut into  $\sim 7 \times 12$  mm chips and annealed under different conditions in the following scenarios: only HTA, only RTA, and sequential RTA and long-term HTA (RTA + HTA). Long-term (60 min) HTA was carried out in a conventional mode [5, 7] at 1100 °C in a quartz container in a 5N purity nitrogen gas atmosphere, in an SDO-E-261 tube furnace. Rapid annealing was carried out in a JetFirst 100C lamp annealing system (France) in a 6N purity nitrogen gas atmosphere, flowrate  $\sim 50$  l/h. Rapid annealing was conducted at two temperatures, 950 and 1000 °C, for 3 min with a  $\sim 23$  °C/s heating rate to target temperature. After heat treatment, the specimens were cooled down in a nitrogen gas flow after furnace switch-off at a  $\sim 10$  °C/s rate.

The PL spectra of the specimens were recorded at room temperature on a Nanometrics RPM PL Wafer Mapping System (Nanometrics Inc., Canada). Excitation was effected with a 17.5 mW Nd:YAG laser at  $\lambda = 532$  nm, and luminescence was recorded with a CCD512-TE CCD matrix in the 570–1000 nm range. The spectra of all the specimens were recorded at similar optical system settings taking into account their spectral characteristics, in order to ensure correctness of comparison between the results.

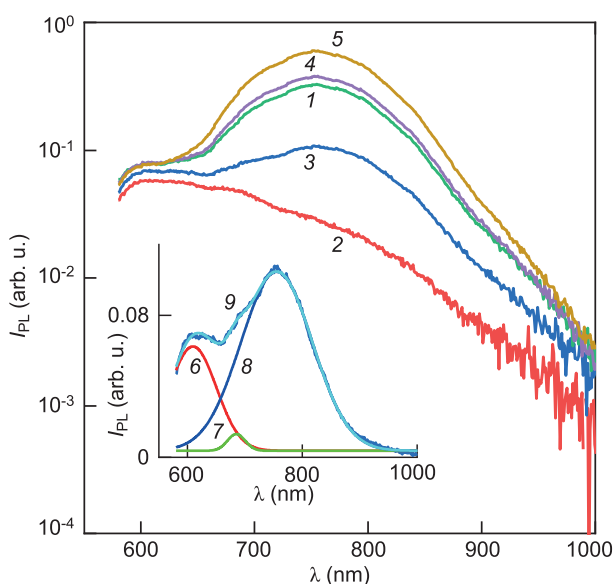
## 3. Results and discussion

The effect of the heat treatment modes used on the PL spectral behavior of the  $\text{SiO}_x/\text{SiO}_2$  and  $\text{SiO}_x/\text{ZrO}_2$  MNS proved to be generally similar. Figure 1 shows semilogarithmic PL spectral intensities of  $\text{SiO}_x/\text{SiO}_2$  annealed using the above-described scenarios, i.e., only HTA at 1100 °C (Spectrum 1), only RTA at 950 and 1000 °C (Spectra 2 and 3, respectively), and RTA + HTA sequentially at 950 + 1100 °C (Spectrum 4) and 1000 + 1100 °C (Spectrum 5). Note that the PL spectra of unannealed specimens with both MNS types did not give any signals resolvable against noise ( $< 3 \cdot 10^{-3}$  arb.u.) and are therefore not shown in Fig. 1. They had no PL maxima, and their intensity decreased monotonically with an increase in wavelength (Table 1).

Variation of annealing modes caused an increase in the PL intensity for both MNS types in the test wavelength range, accompanied with the emergence of a broad maximum in the long-wave region of the spectrum ( $\sim 790$  nm), a clear PL shoulder at  $\sim 700$  nm, and a less intense band at  $\sim 600$  nm. Fitting of the experimental PL spectra by Gaussian line deconvolution was carried out in the assumption of contributions from differently sized Si nanocrystals formed due to thermal disproportioning and growth in  $\text{SiO}_x$  nanolayers and the effect of radiating defects in the dielectric and at the heteroboundaries, as suggested

**Table 1.** PL peak parameters for three bands obtained as a result of deconvolution of experimental spectra, and, by way of comparison, intensities for unannealed specimens for different wavelengths

| Annealing mode<br>(Temperature (°C)) |                         | Band                |                          |                     |                          |                     |                          |
|--------------------------------------|-------------------------|---------------------|--------------------------|---------------------|--------------------------|---------------------|--------------------------|
|                                      |                         | ~600 nm             |                          | ~680 nm             |                          | ~780 nm             |                          |
|                                      |                         | $I_{PL}$ (arb.u.)   | $\lambda_{peak}$ PL (nm) | $I_{PL}$ (arb.u.)   | $\lambda_{peak}$ PL (nm) | $I_{PL}$ (arb.u.)   | $\lambda_{peak}$ PL (nm) |
| $SiO_x/SiO_2$ MNS                    | Unannealed              | $2.3 \cdot 10^{-3}$ | –                        | $1.8 \cdot 10^{-3}$ | –                        | $1.3 \cdot 10^{-3}$ | –                        |
|                                      | HTA (1100)              | $6.8 \cdot 10^{-2}$ | 602.0                    | $1.8 \cdot 10^{-2}$ | 692.5                    | $3.2 \cdot 10^{-1}$ | 754.0                    |
|                                      | RTA (950)               | $4.2 \cdot 10^{-2}$ | 602.7                    | $2.0 \cdot 10^{-2}$ | 670.3                    | $3.1 \cdot 10^{-2}$ | 713.9                    |
|                                      | RTA (1000)              | $6.3 \cdot 10^{-2}$ | 609.4                    | $1.3 \cdot 10^{-2}$ | 685.0                    | $1.1 \cdot 10^{-1}$ | 755.3                    |
|                                      | RTA + HTA (950 + 1100)  | $6.8 \cdot 10^{-2}$ | 602.4                    | $2.0 \cdot 10^{-2}$ | 693.7                    | $3.7 \cdot 10^{-1}$ | 754.8                    |
|                                      | RTA + HTA 1000 + 1100   | $6.9 \cdot 10^{-2}$ | 600.6                    | $3.2 \cdot 10^{-2}$ | 697.0                    | $5.8 \cdot 10^{-1}$ | 756.2                    |
| $SiO_x/ZrO_2$ MNS                    | Unannealed              | $8.1 \cdot 10^{-3}$ | –                        | $4.7 \cdot 10^{-3}$ | –                        | $8.3 \cdot 10^{-4}$ | –                        |
|                                      | HTA (1100)              | $1.9 \cdot 10^{-2}$ | 596.1                    | $6.3 \cdot 10^{-3}$ | 685.5                    | $3.4 \cdot 10^{-2}$ | 785.7                    |
|                                      | RTA (950)               | $1.1 \cdot 10^{-2}$ | 601.5                    | $6.3 \cdot 10^{-3}$ | 673.2                    | $1.6 \cdot 10^{-2}$ | 787.3                    |
|                                      | RTA (1000)              | $1.4 \cdot 10^{-2}$ | 597.0                    | $4.2 \cdot 10^{-3}$ | 685.5                    | $2.1 \cdot 10^{-2}$ | 780.0                    |
|                                      | RTA + HTA (950 + 1100)  | $2.2 \cdot 10^{-2}$ | 596.4                    | $7.1 \cdot 10^{-3}$ | 686.1                    | $3.7 \cdot 10^{-2}$ | 783.5                    |
|                                      | RTA + HTA (1000 + 1100) | $2.2 \cdot 10^{-2}$ | 596.3                    | $7.8 \cdot 10^{-3}$ | 685.1                    | $4.0 \cdot 10^{-2}$ | 784.6                    |

**Figure 1.** PL spectral intensity of  $SiO_x/SiO_2$  MNS annealed using scenarios: (1) only HTA at  $t = 1100$  °C, (2) only RTA at  $t = 950$  °C, (3) only RTA at  $t = 1000$  °C, (4) and (5) RTA + HTA at  $t = 950 + 1100$  °C and  $t = 1000 + 1100$  °C, respectively. Inset: example of Spectrum 3 fitting procedure to Gaussian functions for three PL bands: (6) band at 600 nm, (7) band at 680 nm, (8) band at 780 nm, and (9) fitting result

earlier [3–7]. Fitting revealed 3 main PL bands peaking at 600, 680, and ~780 nm. Example of fitting procedure is shown in Inset of Fig. 1 in linear scale, and numerical data on the parameters of the fitting extrema for both MNS types are summarized in Table 1.

We now consider the effect of conventional HTA on the PL spectrum of  $SiO_x/SiO_2$  MNS with a binary layer thickness of 4/3 nm (Spectrum 1) as well as the PL intensities extracted from the fitting peaks corresponding

to the bands (see Table 1). The PL band peak at ~780 nm recorded after long-term annealing of the  $SiO_x/SiO_2$  MNS at 1100 °C dominates in the test wavelength region. The PL intensity of the as-annealed specimen is more than two orders of magnitude higher than that of the initial unannealed specimen at the same wavelength. This experimental result is well-known in literature: it was observed many times earlier and accounted for by radiative transitions in ~3–4 nm sized Si NCs [2, 4, 8]. The large width of the peak is caused by a Gaussian size distribution of the nanocrystals which was observed earlier, including for the test crystals, using high-resolution transmission microscopy, PL, and Raman scattering [5, 7].

The band near ~680 nm expressed in Spectrum 1 (Fig. 1) in the form of a clear PL intensity shoulder is, on the one hand, close to the 650 nm radiation band generated by radiative non-bridging oxygen hole center (NBOHC) defects [5, 10] and typical of silicon oxide and suboxide films synthesized using different methods. It is well-known that the above radiative centers have at least 2 optical absorption bands: 2.0 and 4.8 eV [11]. For the excitation wavelength used herein, the former absorption band can excite radiation in the NBOHC. On the other hand, this band is close to the radiation wavelength of small-sized silicon nano-inclusions (~2.1–2.5 nm) at ~700 nm [12, 13]. Thus, one can assume the coexistence of the above two origins of the ~680 nm PL band.

The interpretation of the shortest-wave PL peak revealed at ~600 nm after fitting is also controversial. On the one hand, the so-called “yellow” band (Y-band) was first observed [10] during a study of cathodoluminescence (CL) with the radiation band peaking at 580 nm in  $SiO_x$  layers over a wide range of the stoichiometric coefficient ( $x = 0.84$ –2.0), both after annealing at 600–1300 °C and as-synthesized.

Analyzing the data of the cited work, its Authors believe [10] that the origin of the *Y*-radiation are small (3-4-member) ring-shaped silicon clusters forming as a result of reconstruction of stretched (“weak”) Si–O bonds during annealing, e.g. structural transformation of oxygen-deficient centers (Si dimers) to trimers upon interaction with non-bridging oxygen hole centers. According to their model [10, 14–16], 3–6-atomic rings constitute the core of silicon nuclei surrounded by mobile oxygen atoms. It is the surface Si–O shell of those structural aggregates that develops conditions for radiative recombination at the *Y* band wavelength [10]. Seemingly, such ring formations obtained by Monte-Carlo simulation [17] are referred to as percolation chains and clusters, or non-phase inclusions (without phase boundaries) [18]. On the other hand, some authors (e.g. [12, 13]) believe that the 600 nm luminescence band produced after electric or intense (as in this case) optical excitation of as-annealed SiO<sub>x</sub> based specimens can originate from small-sized (about 2 nm) Si NCs. Furthermore, it was shown [9] that 20 ms pulse lamp annealing leads to the formation of nanostructures similar to those generated by Si<sup>+</sup> implantation into SiO<sub>2</sub>.

Thus, earlier studies suggest that the PL band at ~600 nm is caused by nanosized particles of excessive silicon in SiO<sub>2</sub>, or non-phase silicon inclusions of defect origin, or small Si NCs. However, regardless of their nature, these particles can serve as nuclei for further growth of silicon nanocrystals.

The PL pattern obtained after sole rapid annealing of SiO<sub>x</sub>/SiO<sub>2</sub> MNS at 950 and 1000 °C for 3 min is shown in Fig. 1, Spectra 2 and 3. One can see from Fig. 1 the emergence of peaks and the gradual growth of the PL intensity of the ~600, ~700, and ~800 nm bands. The intensity of the short-wave PL maxima at ~600 and ~700 nm is higher (Spectrum 2) or close (3) to that of long-wave radiation. Analysis of the spectrum (see Table 1) suggests that with an increase in the RTA temperature the PL intensity at 600 and 680 nm increases by more than 2 and 3 times as compared to the unannealed SiO<sub>x</sub>/SiO<sub>2</sub> MNS. For long-wave radiation (see Table 1) the PL peak of Si NC (~780 nm) grows by 1 or almost 2 orders of magnitude with an increase in the RTA temperature as compared to the unannealed specimen. Note that the PL band peak emerges initially at 713 nm and then shifts to 755 nm, indicating an increase in the size of the silicon nanoinclusions with an increase in the RTA temperature. The latter trend is demonstrated by both short-wave PL bands and originates from the respective structural transformations of the nanosized formations and NBOHC due to RTA.

Two-stage annealing (RTA + HTA) of SiO<sub>x</sub>/SiO<sub>2</sub> MNS at both RTA temperatures causes a further growth of the Si NC PL intensity. A combination of annealing methods at 1000 + 1100 °C yields the greatest increase in the radiation intensity generated by Si NCs. The intensity grows almost 2-fold as compared to the specimen annealed with only HTA, and exceeds the PL intensity

of the unannealed MNS by 2.5 orders of magnitude for the same wavelength. The PL band peak position remains almost unchanged, close to 755 nm, indicating that the size of the Si NC has reached the effective diameter which is limited by the thickness of the initial SiO<sub>x</sub> layers in the multilayered nanostructure [4, 5, 7].

Importantly, the short-wave 600 nm PL peak intensity changes but slightly after 2-stage annealing, whereas the 680 nm peak intensity grows by several times. The latter peak is associated with the radiation generated by the NBOHC defects and/or small (~2,5 nm) Si NC. Upon RTA + HTA annealing of the SiO<sub>x</sub>/SiO<sub>2</sub> MNS, the concentration of radiating defects increases and/or small NC are generated.

The present experimental data on the SiO<sub>x</sub>/SiO<sub>2</sub> MNS PL behavior due to RTA and combination of RTA + HTA are in agreement with experimental data for Si NC systems in a SiO<sub>2</sub> matrix obtained by Si<sup>+</sup> implantation into SiO<sub>2</sub> [9, 18] and superlattice SiO<sub>x</sub>/SiO<sub>2</sub> structures [8].

The effect of annealing modes on the PL spectra of the SiO<sub>x</sub>/ZrO<sub>2</sub> MNS specimens was generally similar to that for the above nanostructures, indicating the predominant influence of the modification of SiO<sub>x</sub> nanolayer properties due to heat treatment in both MNS types. As a specific feature, one should note that both integral and band-specific PL intensity for SiO<sub>x</sub>/ZrO<sub>2</sub> MNS (see Table 1) is 5–10 times lower as compared to the SiO<sub>x</sub>/SiO<sub>2</sub> MNS specimens. The latter is caused by a decrease in the radiation efficiency due to chemical interactions between silicon and zirconium in the SiO<sub>x</sub>/ZrO<sub>2</sub> MNS layers resulting in the formation of silicate and silicide phases during high-temperature annealing [19].

## Conclusion

Thus, one can conclude that, due to a limited diffusion time, rapid thermal annealing of multilayered nanoperiodic SiO<sub>x</sub>/dielectric structures is only effective at an early stage of Si NC formation, i.e., it provides for the formation of non-phase inclusions and/or nanosized silicon NC having an elevated surface density, which act as nucleation centers. They generate the PL band peaking near ~600 nm. It is the high concentration of silicon nuclei that provides for an increase in the intensity of the 780 nm PL band after additional HTA in a tube furnace as a result of silicon NC growth to ~3–4 nm, provided the heat treatment time is sufficient for excessive silicon drain-diffusion to the nucleation sites. Rapid thermal annealing shows good promise not only for shortening the Si NC specimen processing time at above 1000 °C and reducing the overall heat consumption [8, 9], but also for process optimization. This technology can be the most suitable alternative for conventional long-term high-temperature annealing used to synthesize silicon NC arrays in matrices with relatively low crystallization temperatures, e.g. ZrO<sub>2</sub> [18].

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