# Polymer-nanoparticle coatings on macroporous silicon matrix

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# Abstract

We synthesized light-emitting coatings of CdS nanocrystals in polyethyleneimine and polyethyleneimine with multiwall carbon nanotubes on macroporous silicon. The photoluminescence of CdS nanocrystals in polyethyleneimine on oxidized macroporous Si is more intense in comparison with substrates c-Si, macroporous Si, and macroporous Si with a microporous layer, and is maximal for structures with maximal intensity of the local electric field at the Si-SiO<sub>2</sub> interface. This indicates a significant decrease of non-radiative recombination in the CdS-polyethyleneimine layer due to electron flow from the silicon matrix to the nanocrystal layer. The photoluminescence of polyethyleneimine with carbon multiwall nanotubes on macroporous Si with a microporous layer is about six times more intense in comparison with substrates c-Si, macroporous Si and oxidized macroporous Si. This indicates a non-radiative proton recombination decrease due to hydrogen atoms on the boundary between Si matrix with microporous layer and "polymer-nanoparticles" coating. Copyright © 2017 VBRI Press.

Keywords: CdS nanocrystals, multiwall carbon nanotubes, macroporous silicon.

# Introduction

Generally, the deposition of light-emitting nanocrystals on structured silicon substrates will favor development of new waveguide amplifiers and lasers for silicon microand nanophotonics. Due to their size-tunable physical properties, nanoscale semiconductor materials have promising applications, including the optoelectronic devices such as light-emitting diodes [1] and next generation of quantum dot solar cells [1]. Moreover, nanoscale semiconductors functionalized with biomolecules are promising for molecular fluorescent probes in biological applications [2]. Currently considerable interest exists in the light-emitting semiconductor nanocrystals based on II-VI compounds. This is due to the successes achieved by colloid chemistry in the synthesis of such structures [3]. Ultra-small CdS nanoparticles were prepared by the size-selective photoetching technique [4]. The dependence of photoluminescence of CdSe nanoparticles and CdSe/ZnS on the matrix material was studied on the basis of organic semiconductors and quartz [5]. Quantitative modeling of the role of surface traps in CdSe/CdS/ZnS nanocrystals in photoluminescence decay dynamics demonstrates the sensitivity of photoluminescence interfacial dynamics [6]. The development of methods for obtaining well-defined polymer-QD hybrid materials with tunable optical properties is an active field of research [7]. Narrow-band luminescence has been observed at the short - wavelength

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edge of the luminescence spectrum of polyethylene and polytetrafluoroethylene. The characteristics of this luminescence permit its assignment to the radiation emitted in recombination of ruptured C-C bonds in polymer chains [8].

2D macroporous silicon structures are formed using photoelectrochemical etching on silicon substrates [9] and are promising for photoelectrical [10], optical [11] and biosensors [12]. Light-emitting nanocrystals on macroporous silicon matrix are promising for the development of "white" light sources [3], completely inorganic multicolor LEDs [5]. One of advances of lightemitting coatings of CdS nanocrystals on macroporous silicon is light concentration [1] for silicon solar cells. It is due to the photoluminescence excitation in the visible spectral range after illumination by UV light. This effect is useful to increase the spectral range of light absorption by solar cells [1] due to conversion of UV radiation into visible one. Addition advancement of current work is local electric field at the macropore surface and "macropore-nanocoating" interface [13]. The strength of a local electric field can be determined from the Wannier-Stark effect [14]. The resulted electron flow is from the silicon matrix toward the nanocrystal layer. Such flow might reduce the non-radiative recombination in light-emitting nanocoatings. The deposition of the lightemitting nanocrystals on structured silicon substrates will favor for development of new waveguide amplifiers and lasers for silicon micro- and nanophotonics.

In the present work, light-emitting coatings of CdS nanocrystals in polyethyleneimine [3] and polyethyleneimine with multiwall carbon nanotubes [15] on macroporous silicon were synthesized. Conditions for increase of photoluminescence intensity in the developed structures and the influence of surface local electric fields on photoluminescence of nanocoatings are evaluated and compared. The structures with nanocoatings of polyethyleneimine with CdS nanocrystals on oxidized macroporous silicon and nanocoatings of polyethyleneimine with multiwall carbon nanotubes on macroporous silicon with microporous silicon layers were proposed for fabrication of efficient light-emitting elements due to decrease of the non-radiative recombination on boundary silicon-nanocoating. Owing to the polymer base, the technology makes it possible to increase quantum efficiency of photoluminescence and structure strength as well as to protect the surface from degradation.

# **Experimental**

Macroporous silicon structures with arbitrary distribution of macropores were made of silicon wafers characterized by the [100] orientation and *n*-type of conductivity (the electron concentration  $n_0 = 10^{15} \text{ cm}^{-3}$ ). We used the technique of electrochemical etching at illumination of the backside of a silicon substrate (thickness  $H = 520 \,\mu\text{m}$ ) [16, 17]. The square-lattice periodic structures as well as those with arbitrary distribution of macropores were fabricated. The initial samples are complex microporemacropore silicon structures consisting of 100 nm micropore layers on macropore walls. Addition anisotropic etching in 10% solution of KOH was used to remove the microporous layers from macropore walls. According to the results of optical microscopy (Nu, Carl Zeiss Jena, Germany), macropores with depth  $h_p = 40 \div 120 \ \mu\text{m}$ , diameter  $D_p = 2 \div 5 \ \mu\text{m}$  and concentration  $N_p = (1 \div 6) \cdot 10^6 \text{ cm}^{-2}$  were formed.

CdS nanocrystals 1.8-2 nm in size were obtained in reaction between Cd2+ and HS- in a colloidal solution of polyethyleneimine in water [3]. The samples obtained were washed with distilled water to remove external salts. Carbon multi-walled high purity nanotubes were produced by catalytic pyrolysis of unsaturated hydrocarbons (propylene  $-C_3H_6$ ) [18]. Coatings of CdS nanocrystals and multiwall carbon nanotubes in polyethyleneimine were deposited from a colloidal solution of polyethyleneimine in water on single crystalline silicon, macroporous silicon, oxidized macroporous silicon and macroporous silicon with microporous layer. To reduce the concentration of nonradiative recombination centers (mainly hydrogen), the macroporous surface cleaning was provided by its preliminary oxidation in atmosphere of dry oxygen at 900°C for 15 min (the oxide thickness of 30 nm) and further oxide etching off in 10% aqueous solution of HF. SiO<sub>2</sub> nanocoatings were formed in the diffusion stove after treatment in the nitrogen atmosphere. The oxide layers (thickness of 5÷200 nm) were formed on

macroporous silicon samples in dry oxygen for 40÷60 min at a temperature of 1050°C. The oxide thickness was measured using ellipsometry with 0.2 nm accuracy.

Nanoparticle morphology was investigated by the atomic force microscopy (AFM, NanoScope IIIa Dimension 3000<sup>TM</sup>, Advance Surface Microscopy Inc., USA). The chemical states on the surface of macroporous silicon structures with nanocoatings and the electric field at the "Si-SiO<sub>2</sub>" boundary were identified by IR absorption spectra using a PerkinElmer Spectrum BXII IR Fourier spectrometer (Germany) in the spectral range of 300÷8000 cm<sup>-1</sup>. The optical absorption spectra were measured at normal incidence of IR radiation on the sample (along the main axis of cylindrical macropores). The spectral measurement error was about 2 cm<sup>-1</sup>. Raman spectra of macroporous silicon structures with nanocoatings of polyethyleneimine with multiwall carbon nanotubes were measured using a Horiba Jobin-Yvon T64000 spectrometer (France). The photoluminescence spectra of the nanocoatings on macroporous silicon samples were obtained in the 1.8-3.3 eV range of photon energy. The excitation radiation with photon energy of 0.34 eV falls on the sample through an optical fiber; and photoluminescence emission of the test sample falls on the sensor and the optical fiber through a slit with width of 2.5 nm. The angle between the excitation radiation and photoluminescence emission is 5°. IR absorption, Raman and photoluminescence spectra measurements were carried out in air at room temperature.

## **Results and discussion**

**Fig. 1** (a) shows morphology of CdS nanocrystals in polyethyleneimine according to the AFM data, which confirmed that the sizes of CdS nanocrystals are 1.8-2 nm. A fragment of the macroporous silicon structure with an arbitrary distribution of macropores with nanocoatings is presented in **Fig. 1** (b).



Fig. 1. (a) Morphology of CdS nanocrystals in polyethyleneimine according to the AFM data; (b) a fragment of the macroporous silicon structure with an arbitrary distribution of macropores with silicon oxide and "CdS nanocrystal–polyethyleneimine" (PEI) layers.

The IR absorption spectra of macroporous silicon structures after cleaning and next surface oxidation (oxide thickness of  $5\div 200$  nm) are shown in Fig. 2 (a). The IR absorption of macroporous silicon with surface oxide thickness of 5 nm is 1.5 times bigger than that without surface oxidation. Both the nature and intensity of absorption peaks are almost identical. The IR spectrum of the macroporous silicon sample with surface oxide

thickness of 10 nm changes dramatically. We measured a 364 cm<sup>-1</sup> peak of one-phonon absorption and 465 cm<sup>-1</sup> peak associated with Si-O-Si rotation [**12**].



**Fig. 2.** (a) IR absorption spectra of macroporous silicon structures after surface cleaning and with the surface oxide thickness of 0 -200 nm, (b) IR absorption spectra of macroporous silicon having surface oxide 200 nm thick, with and without surface cleaning.

There is a strong growth of the Si-O-Si oxide peak (1095 cm<sup>-1</sup>) with further oxidation of macroporous silicon to surface oxide thickness of 20-200 nm (curves 4–7). This indicates increase of the concentration of bridge-like oxygen atoms in Si-O-Si (TO phonons) due to reduction of passivation of silicon and oxygen dangling bonds in the absence of hydrogen [12]. In addition to the TO phonon peaks (1086-1095 cm<sup>-1</sup>), LO phonon absorption peaks (1250-1256 cm<sup>-1</sup>) appear due to radiation incidence along the surface of cylindrical macropores (geometry of frustrated total internal reflection [19].

New absorption peaks appear in the spectral regions of TO and LO phonons. A series of light absorption bands at  $\omega \ge \omega_{LO}$  can be explained by formation of multi-phonon states  $(\omega_S^+)_N = \omega_{TO}[N(\epsilon_0/\epsilon_\infty)^{1/2} - (N-1)]$  as a result of interaction of phonons of the SiO<sub>2</sub> film with waveguide modes in the silicon matrix. In our case, the guided and quasi-guided modes are formed on a silicon matrix, with parameters of modes equal to the distance between the macropores. As a result, absorption increases as the frequency  $(\omega_S^+)_N$  of the *N*-th mode of surface phonon-polariton coincides with the frequencies of the waveguide modes.

**Fig. 2 (b)** shows the IR spectra of macroporous silicon samples having surface oxide 200 nm thick, with and without surface cleaning. Absorption of macroporous silicon samples without oxidation and oxide removal exceeds by 4–20 times that of macroporous silicon samples with preliminary cleaning. The

oscillations of IR absorption result from electron resonance scattering in a strong electric field by impurity states on the surface of macropores, with the difference between two resonance energies  $\Delta E = Fa = 8 \div 20 \text{ cm}^{-1}$  equal to the Wannier–Stark step [14]. The oscillations have small amplitudes of IR absorption and nearly the same period for samples with surface cleaning and those of macroporous silicon without preliminary surface cleaning. A sharp decrease of the oscillation amplitude in the electro-optical effects is determined by an increase of the broadening parameter  $\Gamma$  [20].



**Fig. 3.** (a) The results of calculation (curve) of the  $\Delta A/\Delta A_0$  dependence on *Fa*/ $\Gamma$  by Eq. (1). The symbols indicate experimental data from **Fig. 2** (a) for IR absorption of Si-O-Si surface states; (b) dependencies of oscillation amplitudes on absorption for local surface states of oxidized macroporous silicon after preliminary surface cleaning (3/2 law) and without surface cleaning (linear law).

The calculations of the broadened electro-optical function for the Franz–Keldysh effect were performed in [21]. By analogy with this approach, we determined the effect of broadening on the amplitude of oscillations in IR absorption spectra ( $\Delta A$ ) in the form of convolution of the "non-broadened" oscillation amplitude ( $\Delta A_0$ ) with the Lorentz distribution:

$$\Delta A / \Delta A_0 = \frac{\Gamma}{\pi} \int \frac{d\omega'}{(\omega' - \omega)^2 + \Gamma^2} = \arctan(\Delta \omega / \Gamma) / \pi; \quad (1)$$

where  $\Delta \omega = Fa$ . Fig. 3 (a) shows the results of calculation (curve) of the  $\Delta A/\Delta A_0$  dependence on  $Fa/\Gamma$  by Eq. (1). Low values  $\Gamma = 0.1 \div 10 \text{ cm}^{-1}$  confirm the same period of oscillations (see Fig. 2 (b). The obtained values of the broadening parameter  $\Gamma$  correspond to those for

surface phonon polaritons measured in thin films of II–VI semiconductors [14].

Fig. 3 (b) shows the dependencies of oscillation amplitudes  $\Delta A$  on absorption A for local surface states of macroporous silicon samples without surface cleaning and with surface oxide 7-200 nm thick that are linear. These dependencies obey the "3/2 law" for the samples after surface cleaning and with surface oxide thicknesses of 10-200 nm. The obtained dependencies of oscillation amplitudes  $\Delta A$  on absorption A correspond to  $\Gamma^{-1} \sim \tau \sim E$ or  $E^{3/2}$  (where  $\tau = \hbar/\Gamma$  is electron scattering lifetime and E is electron energy). Thus, the resonance electron scattering perpendicularly to Si-SiO<sub>2</sub> border for oxidized macroporous silicon samples without preliminary surface cleaning with  $\tau_r \sim E$  [13] transforms into ordinary electron scattering by ionized impurities at all directions with law  $\tau_I \sim E^{3/2}$  [22] for the samples with surface cleaning. The effect of broadening on the amplitude of oscillations in the IR absorption spectra is due to interaction of the surface multi-phonon polaritons with scattered electrons [14]. This interaction transforms the resonance electron scattering  $(\tau_r)$  in the samples without preliminary surface cleaning into ordinary scattering by ionized impurities ( $\tau_l$ ) for the samples with preliminary surface cleaning. The counter flow of electrons from the silicon matrix toward the Si-SiO<sub>2</sub> interface growth.

Fig. 4 (a) shows the infrared absorption spectra of oxidized macroporous silicon structures without and with a nanolayer of CdS nanoparticles.



**Fig. 4.** (a) IR absorption spectra of oxidized macroporous silicon structures (the oxide thickness of 10 nm) without and with a nanolayer of CdS nanoparticles (30 nm thick); (b) dependencies of oscillation amplitudes ( $\Delta A$ ) on absorption (A) for macroporous silicon samples with CdS nanocrystals and with the silicon oxide thicknesses 5 nm ( $\blacksquare$ ), 10 nm (O) and 20 nm ( $\blacktriangle$ ) for surface Si-O bonds.

IR absorption of oxidized macroporous silicon structures with a layer of CdS nanoparticles increases as compared with IR absorption of oxidized macroporous silicon structures without a layer of CdS nanoparticles. Deposition of a nanolayer of CdS increases local electric field on the Si-SiO<sub>2</sub> boundary too. The corresponding electric field strength  $F = \Delta E/a$  is determined from the energy  $\Delta E$  between the oscillation maxima and the silicon lattice parameter a [13]. The obtained electric field  $F = 4.5 \cdot 10^4 \text{ V/cm}$ strength data vary from to  $F = 6.8 \cdot 10^4 \text{ V/cm}.$ 

Photoluminescence was measured on oxidized macroporous silicon structures with oxide thickness of 5-20 nm and CdS nanocrystal with thickness of 10-30 nm. Fig. 5 (a) shows the spectral dependence of photoluminescence intensity in macroporous silicon structures with CdS nanocrystals (thickness of 30 nm), the oxide thickness of 5, 10 and 20 nm. The maximum of photoluminescence spectra coincides with the corresponding data for aqueous colloidal solution of CdS in polyethyleneimine (2.6-2.7 eV) [3]. The maximal photoluminescence intensity was measured for the structure of macroporous silicon with CdS nanocrystal layer (thickness of 30 nm) and oxide thickness of 10 nm, with the maximal electric field intensity  $F_s = 6.8 \cdot 10^5$  V/cm at the Si-SiO<sub>2</sub> border.



Fig. 5. (a) Spectral dependence of photoluminescence intensity for the macroporous silicon structures with oxide thickness of 20 nm and (b) dependence of the photoluminescence quantum yield and electric field strength on SiO<sub>2</sub> thickness (CdS nanocrystal thickness of 30 nm).

The photoluminescence of CdS nanocrystals is determined by the exciton generation and electron-hole radiative recombination [23]. The maximal intensity of

the CdS nanoparticle photoluminescence was measured for structures with the maximum strength of the local electric field at the Si-SiO<sub>2</sub> interface (**Fig. 5 (b**)). This indicates a significant decrease of non-radiative recombination of electrons generated on CdS nanocrystals due to the counter flow of electrons from the silicon matrix toward the CdS nanocrystal layer. This process is enhanced by the electron scattering on ionized impurities at all directions for oxidized macroporous silicon samples without preliminary surface cleaning instead the resonance electron scattering perpendicular to Si-SiO<sub>2</sub> border for oxidized macroporous silicon samples without preliminary surface cleaning.



**Fig. 6.** (a) AFM of the multiwall carbon tube; (b) IR (1) and Raman (2) spectra of macroporous silicon structures with nanocoatings of polyethyleneimine with carbon multiwall nanotubes; (c) photoluminescence spectra of polyethyleneimine with multiwall carbon tubes on macroporous silicon with microporous layer, on macroporous silicon and on silicon single crystal.

The photoluminescence quantum yield does not change for macroporous silicon structures with optimal oxide thickness of 10 and 20 nm and decreases for macroporous silicon structures without silicon oxide layer. Photoluminescence spectra and photoluminescence quantum yield were measured over 7 and 14 days after the sample preparation. The photoluminescence spectral maximum increases by 4-6 times through 7 days after the sample preparation. This indicates a decrease in the rate of non-radiative recombination at the nanocoating because of decrease in the concentration of recombination centers in this area of structures.

The photoluminescence quantum yield of CdS nanoparticles on the surface of oxidized macroporous silicon with optimum thickness of SiO<sub>2</sub> layer increases by  $1.5 \div 2.3$  times and reaches 28%. With further storage of samples, the range and photoluminescence quantum yield almost did not change. The quantum yield of photoluminescence for such structures increases with time due to evaporation of water molecules from the CdS-polyethilenimine layer [23]. The obtained value of the photoluminescence quantum yield for investigated nanostructures is comparable with that for CdS quantum dots of  $2 \div 3$  nm in size (26-27%) [3] and after an annealing process (29%) [24].

Carbon multiwall nanotubes 50 nm in diameter (Fig. 6 (a)) were deposited from a colloidal solution of polyethyleneimine in water onto silicon substrate, macroporous silicon, oxidized macroporous silicon and macroporous silicon with microporous silicon layer. The Raman spectra of carbon multiwall nanotubes (Fig. 6 (b)) in polymer have peaks similar to IR spectra (sp<sup>2</sup>- and orbitals  $CH_2$ sp<sup>3</sup>-hybrid and bonds). The photoluminescence of polymer is determined by exciton generation and electron-hole radiative recombination [25]. The photoluminescence intensity of polyethyleneimine with carbon multiwall nanotubes on macroporous silicon with microporous layer (Fig. 6 (c)) is 5.6 times higher than photoluminescence intensity of polyethyleneimine with carbon multiwall nanotubes on single crystalline silicon at the photon energy range 2.2-2.7 eV. The microporous layer includes hydrogen atoms [12] and decreases a non-radiative proton recombination on boundary microporous layer and "polymer-nanoparticles" nanocoating.

## Conclusion

The conditions for increase of photoluminescence intensity were evaluated and compared for substrates of c-Si, macroporous Si, oxidized macroporous Si and macroporous Si with microporous layer. The photoluminescence of CdS nanocrystals in polyethyleneimine on oxidized macroporous silicon is more intense in comparison with photoluminescence of CdS nanocrystals on c-Si, macroporous Si, oxidized macroporous Si and macroporous Si with microporous layer. It was found that the photoluminescence intensity is maximal for CdS nanocrystals on oxidized macroporous silicon, with maximal intensity of the local electric field at the Si-SiO<sub>2</sub> interface, indicating a significant decrease of non-radiative recombination in CdS-polyethyleneimine nanocoating due to flow of electrons from the silicon matrix toward the nanocrystal layer. The photoluminescence of polyethyleneimine with carbon multiwall nanotubes on macroporous silicon with microporous layer is about six times more intense than

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photoluminescence of polyethyleneimine with carbon multiwall nanotubes on single crystalline silicon, macroporous silicon and oxidized macroporous silicon. This indicates a non-radiative proton recombination decrease due to hydrogen atoms on boundary silicon matrix with microporous layer "polymerand nanoparticles" nanocoating.

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### Author's contributions

Conceived the plan: lk, mk, ol, vo, kp, os; Performed the expeirments: lk, ol, vo, kp; Data analysis: lk, mk, vo, os; Wrote the paper: lk, mk, ol, vo, kp, os. Authors have no competing financial interests.

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