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INFLUENCE OF LOCAL ELECTRIC FIELDS ON THE PHOTOLUMINESCENCE OF CdS NANOCRYSTALS ON THE OXIDIZED MACROPOROUS SILICON SURFACE

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Oxidized macroporous silicon structures with CdS surface nanocrystals have been proposed to enhance the photoluminescence of CdS nanoparticles due to reducing the electron recombination outside the nanoparticle layer. It has been found that the resonance electron scattering on the Si–SiO₂ interface for samples with low concentration of Si–O–Si states transforms into scattering on ionized surface states for samples with high concentration of Si–O–Si in the structured oxide. The maximum intensity of photoluminescence was measured for a structure with the maximal strength of the local electric field at the interface of silicon matrix with the structured oxide. It indicates a significant decrease of non-radiative recombination of electrons generated in CdS nanocrystal layer due to the counter flow of electrons from the silicon matrix towards the nanocrystals layer. The quantum yield of photoluminescence increases with time due to evaporation of water molecules.

Keywords: local electric fields, CdS nanocrystals, photoluminescence, oxidized macroporous silicon

INTRODUCTION

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 [1]. The capability to control the optical properties by varying the nanocrystal size allows the development of «white» light sources, radiation converters and inorganic LEDs. The researches in this area are aimed at the development of nanocrystals in a dielectric matrix to reduce non-radiative recombination. The dependence of photoluminescence of CdSe and CdSe/ZnS nanoparticles on the matrix material (substrate) was studied on the basis of organic semiconductors and quartz [2].

The deposition of the light-emitting nanocrystals on structured silicon substrates will favor to develop new waveguide amplifiers and lasers for silicon micro- and nanophotonics. For example, modification of structure of macroporous silicon nanocoatings will provide millions of built-in functional channels which have a great potential for the design of optical components in microdevices.

In this paper, oxidized macroporous silicon structures with CdS surface nanocrystals were proposed to reduce the flow of electrons and recombination outside the nanoparticles. In particular, an analysis of oxidized macroporous silicon structures with CdS nanoparticles was made on the basis of measurements of IR absorption spectra. Macroporous silicon is a promising material for the development of 2D photonic structures with the required geometry and large effective surface [3, 4]. This determines the optical and electro-optical characteristics of macroporous silicon structures [5, 6]. Thus the local electric field at the «macroporous silicon–nanocoating» interface was determined. And the influence of electric field on the photoluminescence intensity of CdS nanoparticles on oxidized silicon macroporous structures was evaluated.

PROCEDURE

CdS nanocrystals were deposited onto silicon substrates from colloidal solution with polyethylenimine (PEI). The CdS nanocrystal size values were obtained from the data given by atomic force microscopy. The layer of CdS nanoparticles 1.8–2 nm in size (Fig. 1 a) was

deposited from a colloidal solution with polyethylenimine onto the oxidized macroporous silicon structure.

Macroporous silicon structures with arbitrary distribution of macropores (Fig. 1 b) were made of *n*-silicon wafers of [100] orientation (the electron concentration $n_0 = 10^{15} \text{ cm}^{-3}$) using photo-electrochemical etching [4]. The structures with macropore depth $h_p = 40 \div 80 \text{ }\mu\text{m}$, diameter $D_p = 2 \div 5 \text{ }\mu\text{m}$ and concentration $N_p = (1 \div 6) \times 10^6 \text{ cm}^{-2}$ were etched.

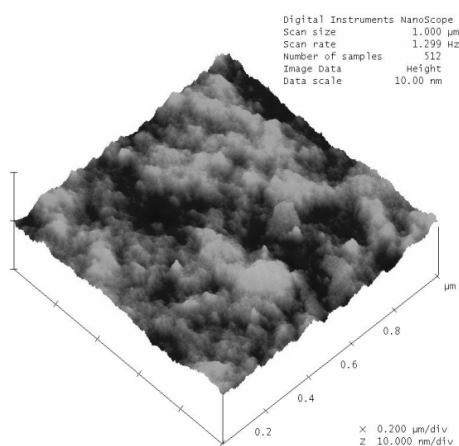


Fig. 1 a. Morphology of CdS nanocrystals in polyethylenimine according to the data of atomic force microscopy

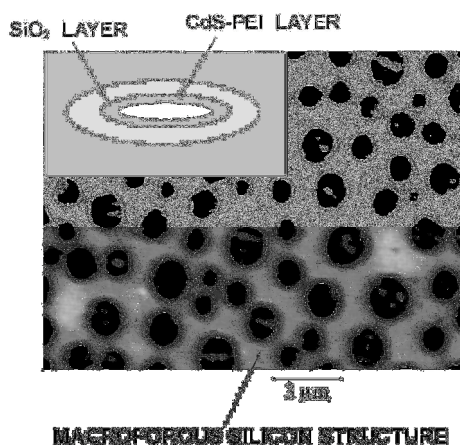


Fig. 1 b. A fragment of the macroporous silicon structure with arbitrary distribution of macropores and direction of light incidence on the sample (along the main axis of cylindrical macropore)

SiO_2 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 the

temperature of 1050 °C. The oxide thickness was measured using ellipsometry.

The chemical states on the surface of macroporous silicon structures with nanocoatings and the electric field at the Si– SiO_2 boundary were identified by IR absorption spectra using a PerkinElmer Spectrum BXII IR Fourier spectrometer in the spectral range 300–8000 cm^{-1} . The optical absorption spectra were recorded at normal incidence of IR radiation on the sample (along the main axis of cylindrical macropores – see Fig. 1 b). The experiments were carried out in air at room temperature.

The photoluminescence spectra of the macroporous silicon samples with a silicon oxide layer and CdS nanoparticles with PEI 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°.

EXPERIMENTAL

Macroporous silicon structures with SiO_2 nanocoatings. Preliminary surface cleaning of macroporous silicon with oxidation and oxide etching off reduces concentration of organic compounds, CH_2 and OH bonds. The IR absorption spectra of macroporous silicon structures after cleaning and next surface oxidation (oxide thickness of 5–200 nm) are shown in Fig. 2 a. The IR absorption of macroporous silicon with surface oxide thickness of 5 nm (curve 2) is 1.5 times greater than that without surface oxidation (curve 1). The nature and intensity of absorption peaks are almost identical for curves 1 and 2.

The IR spectrum of the macroporous silicon sample with surface oxide thickness of 10 nm (curve 3) changes dramatically. We measured a 364 cm^{-1} peak of one-phonon absorption and 465 cm^{-1} peak associated with Si–O–Si rotation [7]. There is a strong growth of the Si–O–Si oxide peak (1095 cm^{-1}) with further oxidation of macroporous silicon (curves 4–7). This indicates an increase in 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 [8]. In addition to the TO phonon peaks (1086–1095 cm^{-1}), LO phonon

absorption peaks ($1250\text{--}1256\text{ cm}^{-1}$) are formed due to radiation incidence along the surface of cylindrical macropores (geometry of frustrated total internal reflection [9]).

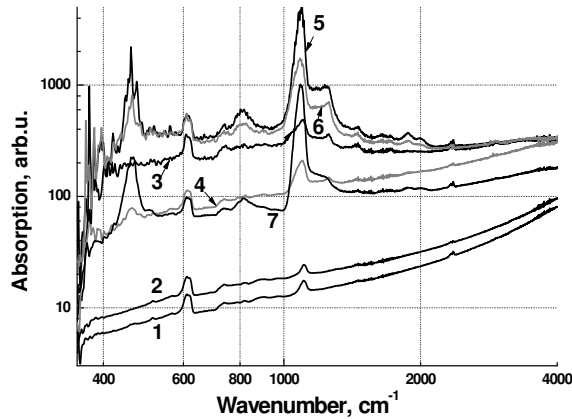


Fig. 2a. The IR spectra of macroporous silicon structures after surface cleaning and: without surface oxidation (curve 1), with the surface oxide thickness of 5 nm (curve 2), 10 nm (curve 3), 20 nm (curve 4), 50 nm (curve 5), 100 nm (curve 6) and 200 nm (curve 7)

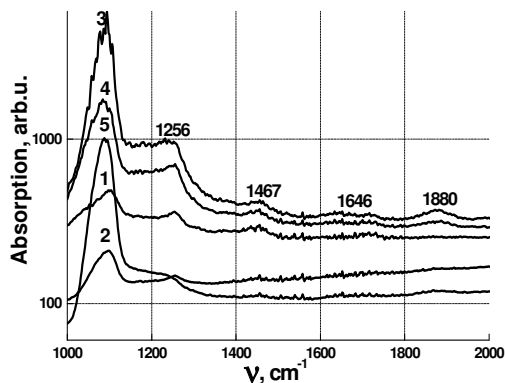


Fig. 2b. IR absorption spectra of macroporous silicon with surface oxide thickness of 10 nm (curve 1), 20 nm (curve 2), 50 nm (curve 3), 100 nm (curve 4) and 200 nm (curve 5) in the spectral region of TO and LO phonons

In addition, new absorption peaks are formed in the spectral regions of TO and LO phonons at $\omega = 1467\text{ cm}^{-1} = \omega_{\text{LO}} + (\omega_{\text{LO}} - \omega_{\text{TO}})$, $1646\text{ cm}^{-1} = \omega_{\text{LO}} + 2(\omega_{\text{LO}} - \omega_{\text{TO}})$ and $1880\text{ cm}^{-1} = \omega_{\text{LO}} + 3(\omega_{\text{LO}} - \omega_{\text{TO}})$, that are presented separately in Fig. 2a. A series of light absorption bands at $\omega \geq \omega_{\text{LO}}$ can be explained by the formation of multi-phonon states [10] as a result of the interaction of phonons of the SiO_2 layer with waveguide modes in the silicon matrix. It is known that the surface LO phonons are phonon-polaritons

[11], so the waveguide modes increase the density of the polariton multi-phonon states [12]. As a result, absorption increases as the frequency $(\omega_{\text{S}}^+)_N$ of the N -th mode of surface phonon-polariton coincides with the frequencies of the waveguide modes. In our case, the guided and quasi-guided modes are formed on a silicon matrix with the parameters of modes equal to the spacing between the macropores [13, 14]. According to Fig. 2, the resonance frequency N -th mode of surface phonon polaritons is

$$(\omega_{\text{S}}^+)_N = \omega_{\text{TO}}[N(\epsilon_0/\epsilon_\infty)^{1/2} - (N-1)], \quad (1)$$

taking into account that the frequency of the longitudinal optical phonon is $\omega_{\text{LO}} = \omega_{\text{TO}}(\epsilon_0/\epsilon_\infty)^{1/2}$ for p-polarized surface mode [10]. It should be noted that impurities increase the density of the polariton states too.

Comparison of the IR spectra of oxidized macroporous silicon with and without surface cleaning. Fig. 3a shows the IR spectra of macroporous silicon samples having surface oxide 50 and 200 nm thick, with (curves 1, 2) and without (curves 3, 4) surface cleaning.

Absorption of macroporous silicon samples without oxidation and oxide removal exceeds by 4–20 times that of macroporous silicon samples with previous cleaning. The oscillations of IR absorption (Fig. 3a, b) result from the electron resonance scattering in a strong electric field by impurity states on the surface of macropores, with the difference between two resonance energies $\Delta E = Fa = 8\text{--}20\text{ cm}^{-1}$ equal to the Wannier–Stark step [14, 15]. The oscillations (Fig. 3a) have small amplitudes of IR absorption and nearly the same period for samples with surface cleaning and those of macroporous silicon without previous surface cleaning (Fig. 3b).

Sharp decrease in the oscillation amplitude in the electro-optical effects is determined by an increase in the broadening parameter Γ [16]. The calculations of the broadened electro-optical function for the Franz–Keldysh effect were performed in [16, 17]. By analogy with this approach, we determined the effect of broadening on the amplitude of oscillations in IR absorption spectra (ΔA) in the form of convolution of the «non-broadened» oscillation amplitude (ΔA_0) with the Lorentz distribution:

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

where $\Delta\omega = \omega' - \omega$ is the energy of the Wannier–Stark step Fa .

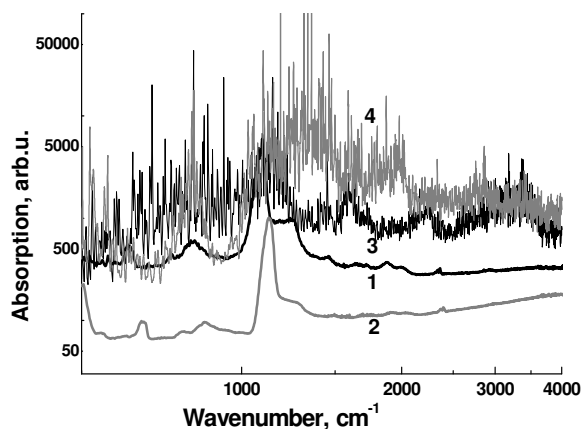


Fig. 3 a. IR absorption spectra of macroporous silicon having surface oxide 50 and 200 nm thick, with (curves 1, 2) and without (curves 3, 4) surface cleaning

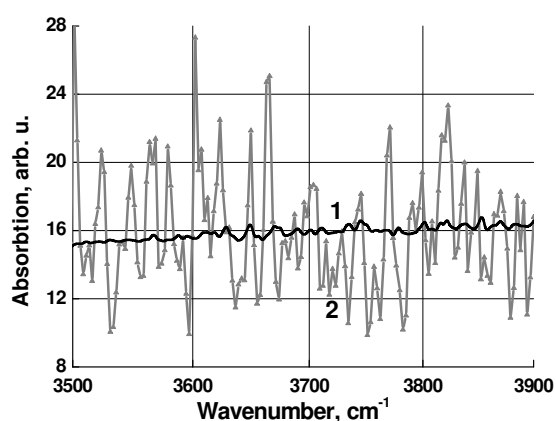


Fig. 3 b. Fragment of IR absorption spectra of macroporous silicon having surface oxide 200 nm thick, with (curve 1) and without (curve 2) surface cleaning

The results of calculation with Eq. (2) are shown in Fig. 4 a, from which it follows that the oscillation amplitude increases linearly with $Fa\Gamma$ up to $Fa\Gamma \approx 1$. The $Fa\Gamma$ values lie within $1 < Fa\Gamma < 100$ for the experimental data on IR absorption in macroporous silicon structures with oxide thicknesses of 50 and 200 nm in the spectral region of the Si–O surface states (Fig. 2 b) and $\Delta A/\Delta A_0 = 0.2-0.5$ (Fig. 3 b). The same period of oscillations (see Fig. 2 b) confirmed low values $\Gamma = 0.1-10 \text{ cm}^{-1}$. The obtained Γ values correspond to this parameter for surface phonon polaritons measured in thin films of II–VI semiconductors [14, 18].

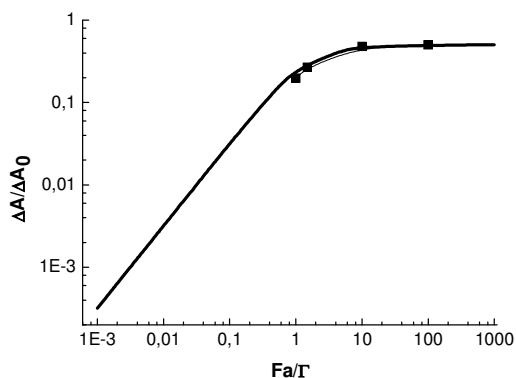


Fig. 4 a. The results of calculation (curve) of the $\Delta A/\Delta A_0$ dependence on $Fa\Gamma$ by Eq. (2). The symbols are experimental data from Fig. 3 a on IR absorption by macroporous silicon structures with oxide thicknesses of 50 and 200 nm in the spectral region of Si–O–Si surface states

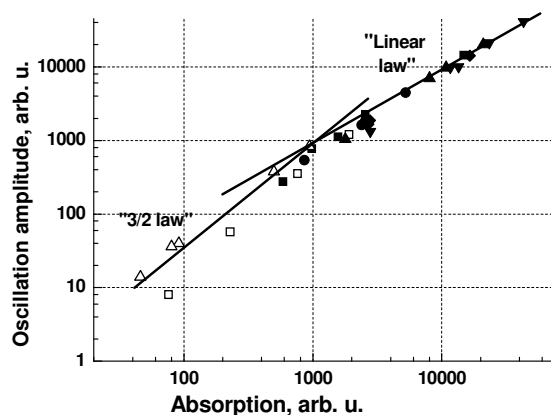


Fig. 4 b. Dependences of oscillation amplitudes on absorption for local surface states of macroporous silicon samples with SiO₂ nanocoatings after preliminary surface cleaning (open symbols) and without surface cleaning (full symbols)

Let us analyze the dependences of oscillation amplitudes ΔA on absorption A for local surface states of macroporous silicon structures (Fig. 4 b). The dependence $\Delta A(A)$ for macroporous silicon samples without surface cleaning are linear. And the dependences $\Delta A(A)$ obey the «3/2 law» for the samples after surface cleaning. The obtained dependences of oscillation amplitudes ΔA 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). The resonance scattering of oxidized macroporous silicon samples without

preliminary surface cleaning with $\tau_i \sim E$ transforms into an ordinary electron scattering by ionized impurities with lifetime $\tau_i \sim E^{3/2}$ for the samples with surface cleaning.

Thus, the effect of broadening on the amplitude of oscillations in the IR absorption spectra is due to interaction of the surface multiphonon polaritons with scattered electrons. This interaction transforms the resonance electron scattering (τ_r) in the samples without preliminary surface cleaning into scattering on ionized impurities (τ_i) for the samples with preliminary surface cleaning and the structured oxide formation.

Photoluminescence spectra. IR spectra and photoluminescence were investigated on oxidized macroporous silicon structures with preliminary surface cleaning and the layer of CdS nanocrystals with thickness of 10–30 nm and oxide thickness of 5–20 nm. Figure 5 shows the infrared absorption spectra of oxidized macroporous silicon structures without (1, 2) and with (1', 2') a nanolayer of CdS nanoparticles 30 nm thick; the oxide thickness: 1, 1' – 10 nm, 2, 2' – 20 nm. IR absorption of oxidized macroporous silicon structures with a layer of CdS nanoparticles increases in comparison with IR absorption of those without such a layer (1, 2).

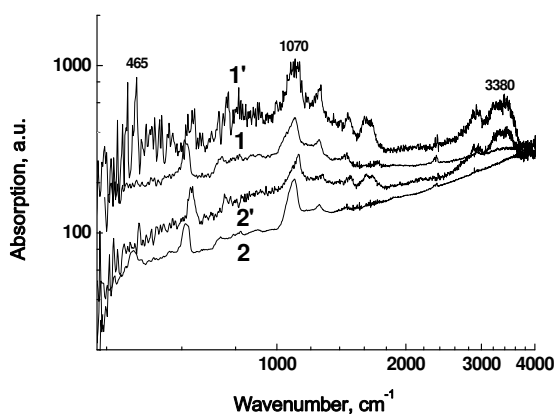


Fig. 5. The infrared absorption spectra of oxidized macroporous silicon structures without (1, 2) and with (1', 2') a nanolayer of CdS nanoparticles 30 nm thick; the oxide thickness: 1, 1' – 10 nm, 2, 2' – 20 nm

The obtained dependences of oscillation amplitudes ΔA on absorption A (Fig. 6) correspond to scattering of electrons by ionized impurities with $\tau_i \sim E^{3/2}$. The electron scattering by ionized impurities increases the flow of electrons from the silicon matrix towards the CdS nanocrystal layer in comparison with the resonance scattering with

$\tau_r \sim E$. Thus, the deposition of a nanolayer of CdS increases IR absorption in comparison with IR absorption of oxidized macroporous silicon structures without a layer of CdS nanoparticles and save the mechanism of the electron scattering on ionized surface states.

The dependences of the spectral position of oscillation maxima (Fig. 5) in macroporous silicon with CdS nanoparticles are linear. The corresponding electric field strength ($F = \Delta E/a$) varies from $4.5 \cdot 10^4$ V/cm to $6.8 \cdot 10^4$ V/cm (Table).

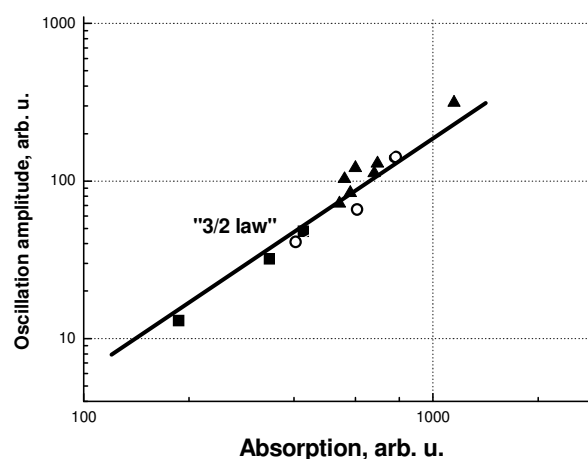


Fig. 6. Dependences of oscillation amplitudes (ΔA) on absorption (A) for macroporous silicon samples with CdS nanocrystals and with the silicon oxide thicknesses 5 nm (■), 10 nm (○) and 20 nm (▲) for surface Si-O bonds

Table. The electric field intensity for oxidized macroporous silicon structures with CdS nanoparticles

d_{SiO_2} , nm	d_{CdS} , nm		
	10	20	30
	$F_S \cdot 10^{-4}$, V/cm	$F_S \cdot 10^{-4}$, V/cm	$F_S \cdot 10^{-4}$, V/cm
10	4.5	5.8	6.8
20	4.9	5.7	6.4

Fig. 7 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. For structures of macroporous silicon with oxide thickness of 5 and 10 nm and CdS nanocrystals, the maximum of photoluminescence spectra (Fig. 7, curves 1, 2) coincides with the corresponding data for aqueous colloidal solution of CdS – PEI (2.7 eV). This indicates a decrease in the distance

between the pairs of opposite charges localized in the «deep» traps [19].

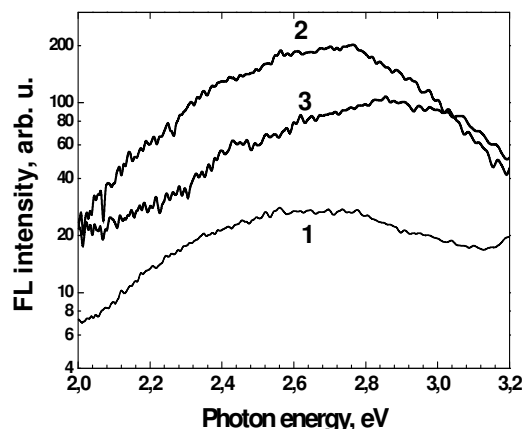


Fig. 7. Spectral dependence of photoluminescence intensity in the macroporous silicon structures with CdS nanocrystal with thickness of 30 nm, oxide thickness 5(1), 10 (2) and 20 nm (3)

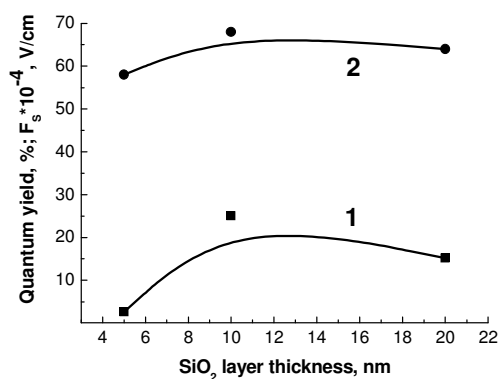


Fig. 8. The dependence of the photoluminescence quantum yield (1) and electric field strength (2) versus the SiO₂ thickness in macroporous silicon structures with CdS nanocrystal layer of 30 nm thickness

The maximum intensity of the CdS nanoparticle photoluminescence was measured for structures with the maximum strength of the local electric field at the Si–SiO₂ interface (Fig. 8). It indicates a significant decrease in non-radiative recombination of electrons generated on CdS nanocrystals due to the counter flow of electrons from the silicon matrix towards the CdS nanocrystal layer. The photoluminescence intensity maximum was measured for the structure of macroporous silicon with CdS nanocrystal layer with thickness of 30 nm and oxide thickness of 10 nm. This structure has the maximal electric field intensity $F_S = 6.8 \cdot 10^5$ V/cm (Table) at the Si–SiO₂ border.

Fig. 9 shows the dependence of the photoluminescence quantum yield versus the CdS nanocrystal thickness for macroporous silicon structures without silicon oxide (1), with oxide thickness of 10 nm (2) and 20 nm (3). 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.

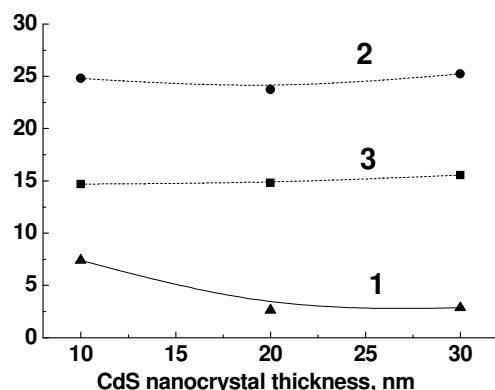


Fig. 9. The dependence of the photoluminescence quantum yield versus the CdS nanocrystal thickness in macroporous silicon structures without silicon oxide (1), with oxide thickness of 10 nm (2) and 20 nm (3)

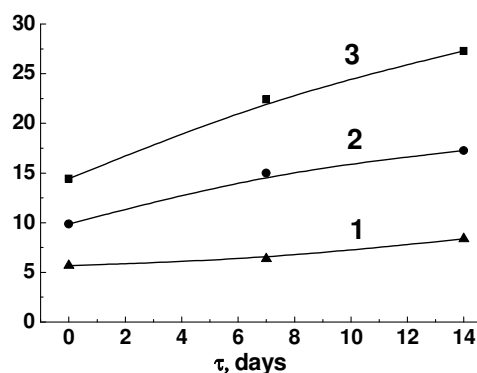


Fig. 10. Time dependence of the photoluminescence quantum yield in macroporous silicon structures with oxide thickness of 10 nm and the thickness of CdS nanocoating 10 (1), 20 (2) and 30 nm (3)

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. That indicates a decrease in the rate of non-radiative recombination at the

nanocoating as a result of decrease in the concentration of recombination centers in this area of structures. In addition, the spectral maximum shifts due to redistribution with time the density of nanoparticles in the CdS–polyethylenimine nanocoating.

The photoluminescence quantum yield of CdS nanoparticles on the surface of oxidized macroporous silicon with optimum thickness of SiO₂ layer increases by 1.5–2.3 times (Fig. 10) and reaches 28 %. With further storage of samples range and photoluminescence quantum yield almost do not change. The quantum yield of photoluminescence for such structures increases with time due to evaporation of water molecules from the CdS–polyethylenimine layer [20].

CONCLUSIONS

Macroporous silicon structures with SiO₂ nanolayers and CdS nanocrystals are proposed to enhance the photoluminescence of CdS nanoparticles due to reducing the electron recombination outside the nanoparticle layer.

It has been found that the resonance electron scattering on the Si–SiO₂ interface for samples with low concentration of Si–O–Si states in oxide layer transforms into scattering on ionized surface states for samples with the high concentration of

Si–O–Si states in the structured oxide. The deposition of a nanolayer of CdS on oxidized macroporous silicon structures (1) increases IR absorption in comparison with that on the structures without a layer of CdS nanoparticles and (2) saves the mechanism of the electron scattering on ionized surface states. That increases the flow of electrons from the silicon matrix towards the CdS nanocrystal layer.

The maximal intensity of the CdS nanoparticle photoluminescence was measured for structures with the maximum strength of the local electric field at the Si–SiO₂ interface with the structured oxide. It indicates a significant decrease of non-radiative recombination of the electrons generated on CdS nanocrystals due to the counter flow of electrons from the silicon matrix towards the CdS nanocrystal layer.

The photoluminescence quantum yield of CdS nanoparticles on the surface of oxidized macroporous silicon with optimum thickness of SiO₂ layer increases of 1.5÷2.3 times during the first 2 weeks due to evaporation of water molecules from the nanoparticles in the polymer layer and reaches 28 %. With further storage of samples range and photoluminescence quantum yield almost do not change.

Вплив локального електричного поля на фотолюмінесценцію нанокристалів CdS на поверхні окиснених структур макропористого кремнію

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Для підсилення фотолюмінесценції наночастинок CdS завдяки зменшенню безвипромінювальної рекомбінації електронів за межами шару наночастинок запропоновані окиснені структури макропористого кремнію з нанокристалом CdS на поверхні макропор. Встановлено, що резонансне розсіяння електронів на межі Si–SiO₂ для зразків з низькою концентрацією Si–O–Si станів в оксиді трансформується в розсіяння електронів на іонізованих поверхневих станах для зразків з високою концентрацією Si–O–Si в структурованому оксиді. Максимальна інтенсивність фотолюмінесценції наночастинок CdS була виміряна для структур з максимальною напруженістю електричного поля на межі кремнієвої матриці з структурованим оксидом. Це свідчить про суттєве зменшення безвипромінювальної рекомбінації електронів, генерованих на нанокристалі CdS, завдяки зустрічному потоку електронів з кремнієвої матриці в напрямку шару нанокристалів. В таких структурах в результаті випаровування молекул води квантовий вихід фотолюмінесценції з часом збільшується.

Ключові слова: *локальне електричне поле, нанокристали CdS, фотолюмінесценція, окиснений макропористий кремній*

Влияние локального электрического поля на фотолюминесценцию нанокристаллов CdS на поверхности окисленных структур макропористого кремния

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Для усиления фотолюминесценции наночастиц CdS благодаря уменьшению безызлучательной рекомбинации электронов за пределами слоя наночастиц предложены окисленные структуры макропористого кремния с нанокристаллами CdS на поверхности макропор. Обнаружено, что резонансное рассеяние электронов на границе Si-SiO₂ для образцов с низкой концентрацией Si-O-Si состояний в оксиде трансформируется в рассеяние электронов на ионизированных поверхностных состояниях для образцов с высокой концентрацией Si-O-Si в структурированном оксиде. Максимальная интенсивность фотолюминесценции наночастиц CdS была измерена для структур с максимальной напряженностью электрического поля на границе кремниевой матрицы и структурированного оксида. Это свидетельствует о существенном уменьшении безызлучательной рекомбинации электронов, генерированных на нанокристаллах CdS, благодаря встречному потоку электронов из кремниевой матрицы в направлении слоя нанокристаллов. В таких структурах в результате испарения молекул воды квантовый выход фотолюминесценции со временем увеличивается.

Ключевые слова: локальное электрическое поле, нанокристаллы CdS, фотолюминесценция, окисленный макропористый кремний

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