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ROOM TEMPERATURE WANNIER-STARK EFFECT IN 2D MACROPOROUS SILICON STRUCTURES WITH NANOCOATINGS

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The IR light absorption oscillations in 2D macroporous silicon structures with microporous silicon, SiO₂ nanocoatings, and ZnO nanocrystals have been studied at room temperature. Oscillations with giant amplitude are observed in the spectral ranges of absorption by surface levels on "silicon – nanocoating" boundary. The electro-optical effect has been considered within the strong electric field approximation due to resonance electron scattering on the surface impurity states with the difference between two resonance energies equal to the Wannier-Stark ladder. Resonant electron scattering gives rise to the resonances of the permittivity and a corresponding change in the absorption. In this case, the Wannier-Stark effect is realized as a result of large-time electron scattering as compared with the period of its oscillations in the strong electric field of illuminated "silicon – nanocoating" boundary.

INTRODUCTION

One of the promising materials for the development of 2D photonic structures is macroporous silicon that can be obtained using the photoanodic etching. It is connected with formation of the structures of necessary geometry and high ratio between the cylindrical macropore depth and diameter [1, 2]. Presence of periodically located cylindrical pores divided by silicon columns provides large effective surface area of the samples. This determines optical and photo-physical characteristics of macroporous silicon structures [3–5]. For wavelengths below the optical period of structures, a reduction of light absorption is observed owing to the guided and radiation optical modes formed by macroporous silicon as a short waveguide. According to [6], the absolute absorption maxima are determined by the guided optical mode position. The results obtained were explained by specificity of a macroporous silicon surface. The existence of an intrinsic electric field $F_S = (5 \div 9) \cdot 10^5$ V/cm is confirmed in [7] by an electroreflectance study of macroporous silicon surfaces. In view of the potential barrier on a macropore surface, one should take into account recharging of the local surface centres at energies below that of the indirect interband transition.

The near-IR optical absorption in 2D photonic macroporous silicon structures was investigated in [8] with allowance made for the linear electro-optical effect. The spectral dependence of optical absorption of macroporous silicon structures in the near-IR spectral part has an oscillating structure and varies under the "3/2" law at long wavelengths. This correlates with frequency dependence of the imaginary part of permittivity for optical transitions between impurity levels and the allowed bands of a crystal in an electric field (the impurity Franz-Keldysh effect). The electric field of the reflected electromagnetic wave at the grazing angle of light incidence onto macropore surface changes effectively the local electric field on the macropore surface. The experimental absorption spectra of macroporous silicon agree well with the corresponding spectral dependences of the electro-optical energy and the imaginary part of permittivity in the weak electric field approximation, thus confirming realization of the linear impurity Franz-Keldysh effect.

In this paper, the near-IR light absorption oscillations of 2D macroporous silicon structures with microporous silicon, SiO₂ nanocoatings, and ZnO nanocrystals were investigated at room temperature taking into account the electro-optical effect within the strong electric field approxima-

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tion. An analysis of the experimental absorption spectra is carried out within the model of the resonant electron scattering on impurity states in strong electric field with difference between two resonant energies equal to the step of the Wannier-Stark ladder. An additional growth of electric field intensity at illuminated "silicon – nanocoating" boundary was considered.

METHODOLOGY

The samples to be studied were made of silicon wafers characterized by the $\langle 100 \rangle$ 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 back side of a silicon substrate (thickness $H = 520 \mu\text{m}$) [6]. Macropores with the depth $h_p = 80 \div 100 \mu\text{m}$, diameter $D_p = 2 \div 5 \mu\text{m}$ and period $a_p = 4 \div 7 \mu\text{m}$ were formed. The square-lattice periodic structures, as well as those with arbitrary distribution of macropores, were fabricated. The initial specimens are combined micropore-macropore silicon structures consisting of 100 nm micropore layers on macropore walls. Addition anisotropy etching in 10% solution of KOH was used to remove the microporous layers from macropore walls.

ZnO nanocrystals 5–6 nm in size were synthesized in a colloidal solution of $\text{Zn}(\text{CH}_3\text{COO})_2$ in ethanol [9]. A colloidal solution of ZnO nanocrystals was deposited onto a layer of microporous silicon on the surface of macropores, and the sample was kept until complete evaporation of the solvent. The sample obtained was washed with distilled water to remove residual electrolytes. The SiO_2 nanocoatings (thickness of $70 \div 200 \text{ nm}$) have been formed on macroporous silicon samples in dry oxygen during $40 \div 60$ minutes at the temperature of $1050 \div 1200^\circ\text{C}$. The 800 nm oxide layer was formed for 50 minutes at the temperature of 1100°C in wet oxygen using a steam generator with deionized water. The oxide layer thickness was measured using ellipsometry.

We performed optical investigations in the $1.3 \div 25 \mu\text{m}$ spectral range using "Perkin Elmer" Spectrum BXII IR Fourier spectrometer. The optical absorption spectra were recorded at normal incidence of IR radiation on the sample (along the main axis of cylindrical macropores). The experiments were carried out in air at room temperature. The spectral measurement error was about 2 cm^{-1} .

EXPERIMENTAL

Surface nanocrystals. For macroporous silicon structures with microporous layers and surface nanocrystals, the light absorption increases, and an oscillating structure appears (Fig. 1).

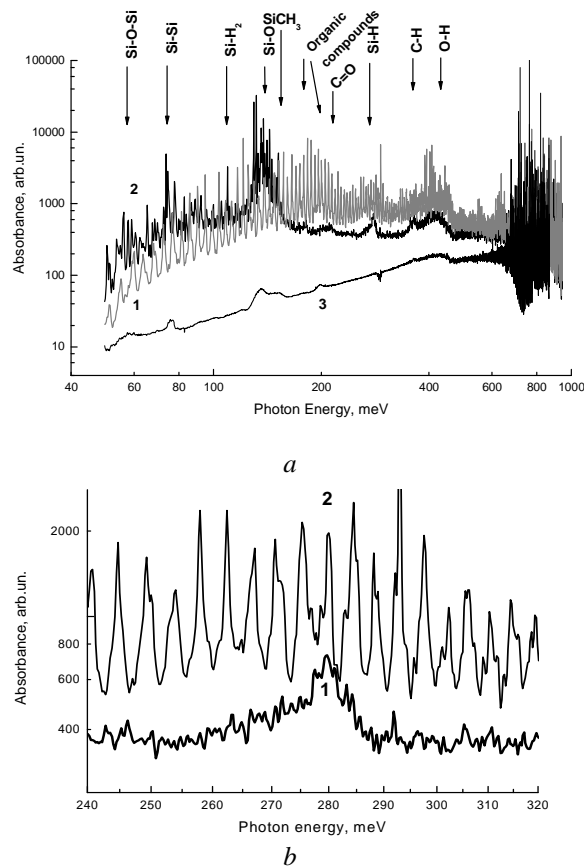


Fig. 1. Absorption spectra (a) and their fragments in the vicinity of Si–H bonds (b) of macroporous silicon structures with microporous layers (1), ZnO surface nanocrystals (2) and without coatings (3) in the vicinity of Si–H bonds

The absorption spectra of macroporous silicon structures with ZnO surface nanocrystals (Fig. 1a, curve 1) and those with initial microporous layers (Fig. 1a, curve 2) have similar shapes at photon energies over 200 meV.

The amplitude of oscillations is maximum at spectral ranges of surface levels (organic species, Si–H, C–H, and O–H bond vibrations [10, 11]) in the absorption spectra of macroporous silicon structures with ZnO surface nanocrystals. And Si–O–Si, Si–Si, Si–H₂, Si–O, SiCH₃, C=O bonds were observed only in absorption spectra of macroporous silicon structures with the microporous silicon layer (Fig. 1a, curve 1) or without nanocoatings (Fig. 1a, curve 3). The form of oscillations (Fig. 1b) indicates their resonant character.

The spectral positions of oscillation maxima of macroporous silicon structures with surface nanocrystals and microporous layers vs oscillation number curves (Fig. 2) are straight lines, and the oscillation period is almost constant (Fig. 3).

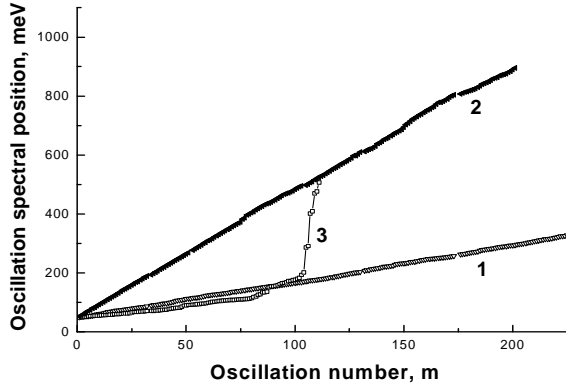


Fig. 2. The spectral position of oscillation maxima of macroporous silicon structures with microporous layers (1), ZnO (2) and without coatings (3) as function of oscillation number

The oscillation energies ΔE of macroporous silicon structures with microporous layers lie between 0.7–2 meV. The oscillations of small amplitude (Fig. 2, curve 3) have been investigated in [8] for macroporous silicon structures without nanocoatings. The experimental absorption spectra of macroporous silicon agree well with the corresponding spectral dependences of the electro-optical energy and the imaginary part of permittivity in the weak electric field approximation, thus confirming realization of the impurity Franz-Keldysh effect.

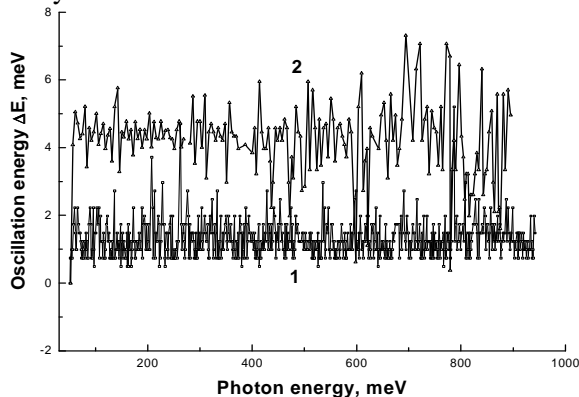


Fig. 3. Spectral dependences of the oscillation period of macroporous silicon structures with microporous layers (1), ZnO nanocrystals (2)

SiO₂ nanocoatings. For macroporous silicon structures with SiO₂ nanocoatings, the light absorption increases and an oscillating structure occurs (Fig. 4a, curves 1 and 2). We observed the essential absorption growth in spectral area

of Si–O, Si–H, O–H bonds and organic compounds. The amplitude of oscillations is maximal in the spectral ranges of surface level absorption (Fig. 4b).

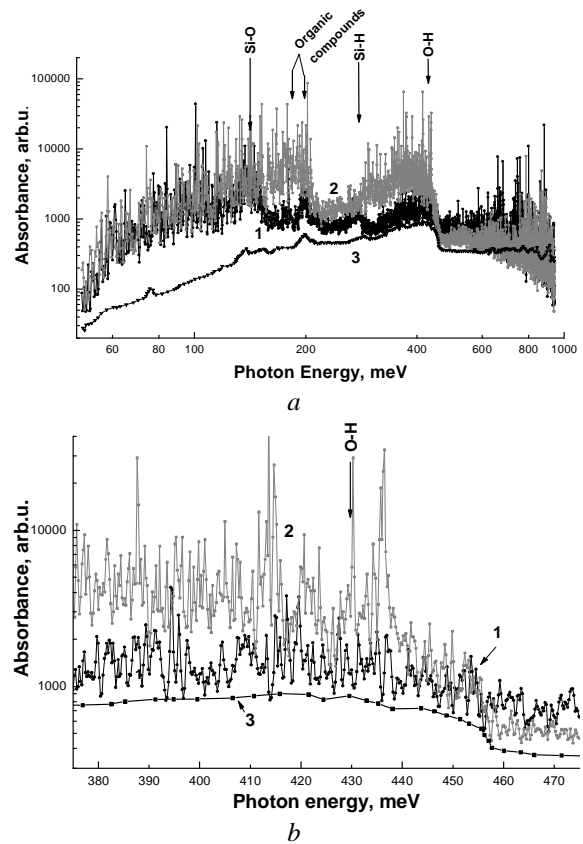


Fig. 4. Absorption spectra (a) and their fragments in the vicinity of O–H bonds (b) of macroporous silicon structures with SiO₂ nanocoatings 70 nm (1) and 800 nm (2) thick and without coating (3)

The spectral positions of oscillation maxima in the macroporous silicon structures with SiO₂ nanocoatings and without coating are rather different (Fig. 2, curve 3 and Fig. 5).

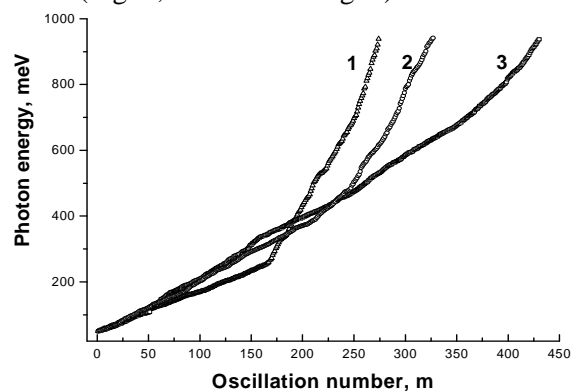


Fig. 5. The spectral position of oscillation maxima in the macroporous silicon structures with SiO₂ nanocoatings of 70 nm (1), 200 nm (2) and 800 nm (3) thickness

The dependence of oscillation maxima of macroporous silicon structures with SiO₂ nano-coating on oscillation number reveals spectral features at the energies of 0.25 eV, 0.4 eV, and 0.7 eV. In addition, the oscillation period fluctuates around a constant value at low spectral energies and becomes quadratic in photon energy depending on SiO₂ nano-coating thickness (Fig. 6).

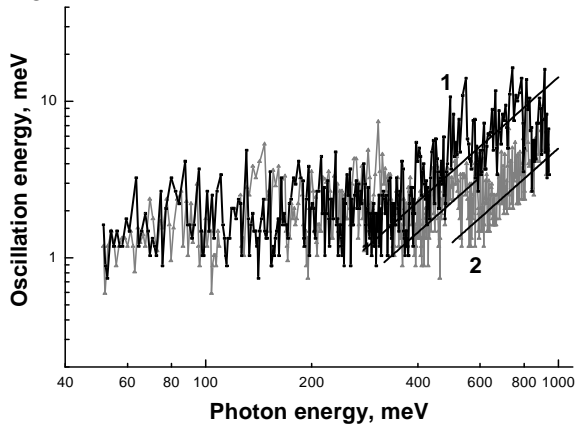


Fig. 6. Spectral dependencies of the oscillation energy in the macroporous silicon structures with SiO₂ nano-coatings of 200 nm (1) and 800 nm (2) thickness

DISCUSSION

Resonant electron scattering. We observed the oscillating structure in the absorption spectra of macroporous silicon structures with microporous silicon, SiO₂ nano-coatings, and ZnO nanocrystals at room temperature. The amplitude of oscillations is maximum at spectral ranges of organic species, Si-H, C-H and O-H bonds. The results obtained indicate strong effect of impurity states on the surface of macroporous silicon structures with nano-coatings. This may result from scattering of both electromagnetic radiation and electrons on the impurity states. The form of oscillations (Fig. 1b) indicates resonant character of scattering.

The oscillations of small amplitude in macroporous silicon structures without nano-coatings [8] correspond to the weak electric field approximation. The macroporous silicon structures with surface nanocrystals described in this paper have higher surface potential of the "silicon – nano-coating" boundary. Therefore, the onset of oscillations with giant amplitude can be attributed to the electro-optical processes in strong electric field. Moreover, the constant oscillation period (Fig. 3) may specify the realization of the

Wannier-Stark effect on randomly distributed surface bonds on nanocrystals.

A method of experimental observation of Wannier-Stark ladder was proposed in [12]. It was shown that the scattering amplitude had resonant character in the case of electron scattering on impurities. If the electric field is directed along the *x*-axis of the crystal, then electron scattering occurs in the plane (*y*, *z*), and the difference between two resonant energies is approximately equal to the Wannier-Stark ladder. In our case, an electric field of "silicon – nano-coating" heterojunctions on the macropore surface is also directed normally to the surface too (Fig. 7), and the surface states that scatter electrons are concentrated perpendicularly to the *x*-direction in the plane (*y*, *z*) what is the plane of resonant scattering.

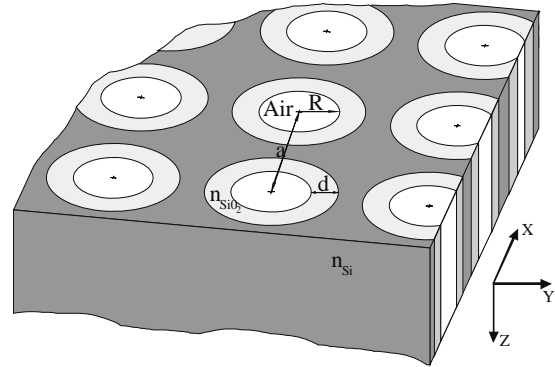


Fig. 7. A fragment of system considered

Let us consider a semiconductor with the dispersion law $E(k) = E_0 - \Delta(\cos k_y a + \cos k_z a)$ where *k* is a quasi-momentum with components *k_y*, *k_z*, *E*₀ the energy corresponding to the midgap, Δ the energy equal to 1/6 the band gap, *a* the lattice parameter. The wave function in the Wannier representation was written as [12]

$$\langle j | \psi_E \rangle = \langle j | \Phi_E \rangle + \frac{\langle j | \hat{G}_0(E) | 0 \rangle V_0 \langle 0 | \Phi_E \rangle}{1 - V_0 \langle 0 | \hat{G}_0(E) | 0 \rangle}. \quad (1)$$

Here the first (second) term describes the incident wave (scattered waves); *j* numbers the lattice site, $\hat{G}_0(E)$ is the Green operator, *V*₀ is the impurity potential. The complex energies for which the denominator of the second term becomes zero correspond to the resonances in electron scattering $1/V_0 = \langle 0 | \hat{G}_0(E) | 0 \rangle$ at $E = \varepsilon - i\Gamma$ ($\Gamma > 0$). The difference of two neighbouring resonance energies is approximately equal to

the value of the step in the Wannier-Stark ladder. Resonant electron scattering gives rise to the resonances of the permittivity at room temperature and a corresponding change in the absorption.

The Wannier-Stark effect realization. The fact is that the levels of the Wannier-Stark ladder have a certain width Γ while its detection requires this width to be lower than the difference between the energies of adjacent levels, $\Gamma < Fd$. The contributions to the width Γ come from the interband interaction, electron-phonon interaction, and interaction with impurity atoms.

The interband interaction was studied in [13], the electron-phonon interaction was considered in [14]. It has been demonstrated that it does not break the Wannier-Stark ladder. The paper [15] deals with the influence of impurities on the Wannier-Stark ladder and with calculation of the width of the Wannier-Stark ladder levels ΔE due to scattering on impurities. The Wannier-Stark ladder is not broken by impurities if the intervals between the transitions due to scattering from impurity atoms with lifetime τ are greater than the period of electron oscillations in external field, T_B ($\tau/T_B > 1$) where $T_B = 2\pi\hbar/\Delta E$, τ is equal to $1/W$ (W is the probability for an electron to leave the state per time unit due to scattering from an impurity atom at lattice site). In [19], the following estimate of the probability W for an electron to leave the state per time unit due to scattering from an impurity atom at lattice site was obtained: $W < 2V_0N_i/(Nh)$ where V_0 is the impurity potential, N_i the impurity concentration and $N \approx (a^2)^{-1}$ the density of states. As a result, the inequality $\tau/T_B > 1$ passes to $N_i < \Delta E/(4\pi a^2 V_0)$. Using the later inequality, we find a numerical estimation of impurity concentration.

We obtained the surface impurity concentration in macroporous silicon structures by the method of the photoconductivity dependence on the distance between macropores [16] and the temperature dependences of the photocarrier lifetime in macroporous silicon [17]. From the experimental temperature dependences of the photocarrier lifetime in 2D macroporous silicon structures the dimensionless surface potential γ_0 is about 12 at room temperature corresponding to the equi-

librium surface band bending of about 0.31 eV and to the surface impurity concentration $N_i = 5 \cdot 10^{10} \text{ cm}^{-2}$ for the electron concentration $n_0 = 10^{15} \text{ cm}^{-3}$ of investigated macroporous silicon samples.

The Bloch oscillation time is equal to $T_B \approx (4 \div 8) \cdot 10^{-12} \text{ s}$ for macroporous silicon structures with ZnO nanocrystals and $T_B \approx (1 \div 4) \cdot 10^{-11} \text{ s}$ for macroporous silicon structures with microporous layers. The lifetime relation is $\tau/T_B > 1$ in the whole spectral region studied for macroporous silicon structures with surface nanocrystals (Fig. 8). Relatively long electron scattering time in 2D macroporous silicon structures is due to the small surface impurity concentration on macropore surface ($N_i \leq 5 \cdot 10^{10} \text{ cm}^{-2}$) [16].

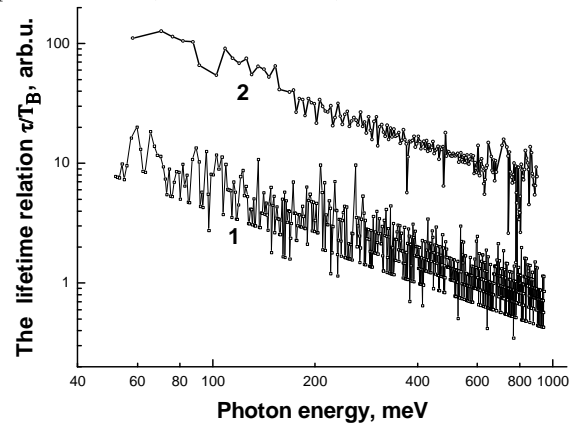


Fig. 8. Spectral dependence of the lifetime relation τ/T_B for macroporous silicon structures with microporous layers (1) and ZnO (2) surface nanocrystals

The inequality $\tau/T_B > 1$ for the lifetime ratio is satisfied (Fig. 9a) in the whole investigated spectral region for macroporous silicon structures with SiO_2 nanocoatings taking into account that the surface impurity concentration for macroporous silicon structures, N_i , is lower than 10^{11} cm^{-2} . Thus, the Wannier-Stark ladder is preserved in the whole spectral range for macroporous silicon structures with microporous silicon, SiO_2 nanocoatings, and ZnO nanocrystals.

Electric field intensity on "silicon – nanocoating" boundary. Usually, the basic sources of external electric field at a semiconductor surface are the charge of the electron levels and the built-in charge in the semiconductor surface oxide [7]. The oscillation period and electric field intensity of macroporous silicon structures with SiO_2 nanocoatings

fluctuate about a constant value at low spectral light energies and become quadratic in the photon energy depending on the geometrical parameters of silicon matrix and SiO₂ nano-coatings (see Fig. 9b and Table). The electric field intensity growth corresponds to the quasi-guided mode formation [19] in the silicon matrix (minimum distance between macropores) with $2\rho_{Si} = a_p - (D_p + d_{SiO_2})$ and in the silicon column with $2\rho_{Si} = 1.4 \cdot [a_p - (D_p + d_{SiO_2})]$ (see Table). The mode parameter $Q_{Si} \sim k\rho_{Si}$ is determined by the beginning of the photon energy quadratic growth (Fig. 9b, curves 1–4).

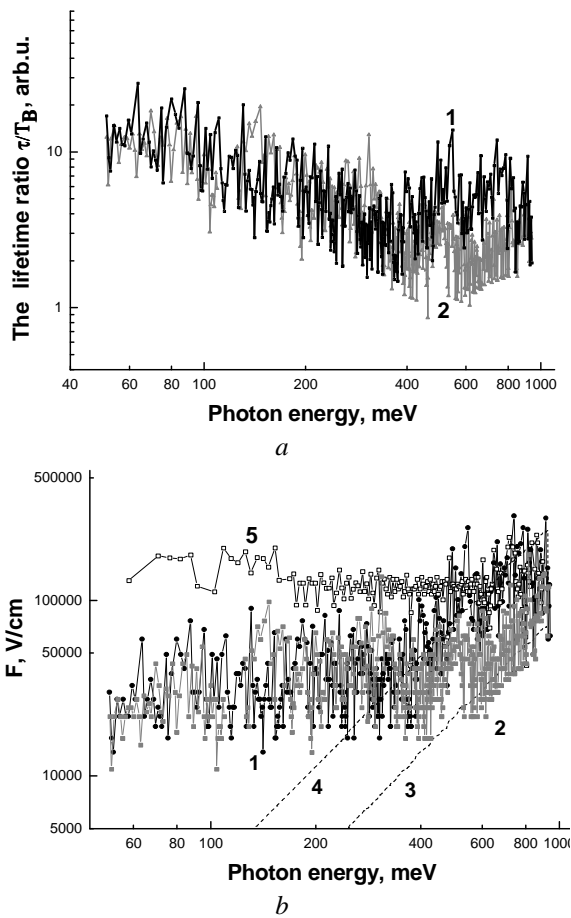


Fig. 9. Spectral dependencies of the lifetime ratio τ/T_B (a) and those of the electric field intensity F on macroporous silicon surface (b) for structures with SiO₂ nano-coatings of 200 nm (1) and 800 nm (2) thickness; spectral dependencies of the electric field intensity as a result of quasi-guided mode formation in the silicon matrix (3, 4); spectral dependencies of the electric field intensity in the macroporous silicon structures with ZnO surface nanocrystals(5)

In general, at grazing angle of light incidence, the electric field of the reflected electromagnetic wave changes the local electric field in the near-surface region of the macropore walls with thickness $d \approx 0.1\lambda$ for wavelength λ [20]. Let us consider that d is determined by the electric component of electromagnetic wave with $\hbar\omega$ and by the change of built-in electric field ΔF_s ($d = \hbar\omega / (e \cdot \Delta F_s)$). Indeed, under our experimental condition of the grazing angle of light incidence onto the macropore surface, the electric field intensity on macroporous silicon surface for the structures with SiO₂ nano-coatings is about $F_s + \Delta F_s$ with $\Delta F_s \approx \hbar\omega / (0.1\lambda_1) \sim \hbar\omega^2$ according to the experiment (Fig. 9b). Light wavelength is equal to $\lambda_1 = \lambda/n_i$ (n_i is effective refractive index of pores with SiO₂ nano-coatings or refractive index of SiO₂ nano-coatings). The electric component of electromagnetic wave changes from $E = \hbar\omega$ to zero when going from the silicon matrix to the macropore. This corresponds to reverse bias at the surface barrier and its band bending growing. The effect is strong for light energy corresponding to spectral range of the quasi-guided (leakage) mode formation in the silicon matrix (see Fig. 9b and Table).

Table. The sample and optical mode characteristics

d_{SiO_2} nm	$D_p + d_{SiO_2}$ μm	$a_p - (D_p + d_{SiO_2})$ μm	Mode type	$2\rho_{Si}$, μm
70	2.07	2.4	quasi-guided modes in the silicon matrix	$2[a_p - (D_p + d_{SiO_2})] = 4.8$
			quasi-guided modes in the silicon matrix	$[a_p - (D_p + d_{SiO_2})] = 2.2$
200	2.2	2.27	quasi-guided modes in the silicon matrix	$2[a_p - (D_p + d_{SiO_2})] = 4.1$
			quasi-guided modes in the silicon column	$1.4[a_p - (D_p + d_{SiO_2})] = 3.65$
800	2.8	1.67	quasi-guided modes in the silicon matrix	$2[a_p - (D_p + d_{SiO_2})] = 3.25$
			quasi-guided modes in the silicon column	$1.4[a_p - (D_p + d_{SiO_2})] = 2.34$

This result differs from that in [8] for macroporous silicon structures without nanocoatings where the ΔF_s change was observed for all the spectral range investigated due to the low surface barrier. On the other hand, the electric field intensity in the macroporous silicon structures with microporous layer and surface nanocrystals does not change in the short wavelength spectral range (Fig. 3 and Fig. 9b, curve 5).

Constant oscillation period (and electric field intensity) in macroporous silicon structures with nanocrystals may be attributed to absence of the quasi-guided mode formation in the silicon matrix due to strong light scattering by nanocrystals. As a result, the local electric field variation in the heterojunction area became negligible for microporous silicon nanocoatings and ZnO nanocrystals in comparison with macroporous silicon structures with SiO₂ nanocoating. In addition, the nanocrystals form local contact with silicon surface, thus increasing the constant local electric field intensity and decreasing surface level concentration. That is why harmonic oscillations with low period deviations were measured in absorption spectra of the macroporous silicon structures with the contacted nanocrystals (Fig. 1b, curve 2).

CONCLUSIONS

We observed well-separated oscillations in absorption spectra of macroporous silicon structures with surface nanocrystals and with SiO₂ nanocoatings. The amplitude of oscillations is maximal in the spectral ranges of organic species, Si-H, C-H and O-H bonds. It results from resonant electron scattering on the impurity states in an electric field of "silicon-nanocoating" heterojunction on macropore surface. Resonant electron scattering gives rise to the resonances of the permittivity at room temperature and to corresponding changes in the absorption.

The constant oscillation period specifies the realization of the Wannier-Stark effect on the randomly distributed surface bonds. The amplitude of scattering has resonant behavior in the case of electron scattering on impurity and the difference between two resonant energies is equal to Wannier-Stark ladder. The

oscillation periods ΔE of macroporous silicon structures with surface nanocrystals and microporous layers depend mainly on the band gap of the nanocrystal material. The electric field intensity F is of $10^4 \div 10^5$ V/cm for $F = \Delta E/a$, thus indicating validity of the model of electron oscillations in the atomic lattice.

The Wannier-Stark ladder is not broken by impurities, if the intervals between the transitions due to scattering from impurity atoms with lifetime τ are greater than the period of electron oscillations in external field, T_B . The lifetime relation is $\tau/T_B > 1$ in all spectral regions considered for macroporous silicon structures with microporous silicon layers and ZnO surface nanocrystals, taking into account that the surface impurity concentration for macroporous silicon structures is lower than $N_i \approx 5 \cdot 10^{10}$ cm⁻². Also, the inequality $\tau/T_B > 1$ holds for the entire spectral regions considered for macroporous silicon structures with SiO₂ nanocoatings, taking into account that the surface impurity concentration N_i for macroporous silicon structures is lower than $5 \cdot 10^{11}$ cm⁻². Thus, the Wannier-Stark ladder is preserved in the whole spectral range for macroporous silicon structures with considered nanocoatings. The observation of coherent electronic wavepacket oscillations in a semiconductor heterostructure at room temperature was also reported in [21].

The oscillation period and electric field intensity in the macroporous silicon structures with SiO₂ nanocoatings fluctuate around a constant value at low photon energies and become quadratic in photon energy depending on the geometrical sizes of silicon matrix and SiO₂ nanocoatings. The relevant electric field intensity growth corresponds to the quasi-guided mode formation in the silicon matrix (minimum distance between the macropores) and in the silicon column. The electric field intensity in the macroporous silicon structures with microporous layer and surface ZnO nanocrystals does not change due to the high surface barrier, strong light scattering by nanocrystals, and absence of the quasi-guided mode formation in the silicon matrix. That is resulted in the negligible local electric field variation in macroporous silicon structures with SiO₂ nanocoating.

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**Ефект Ваньє-Штарка при кімнатній температурі
в двовимірних структурах макропористого кремнію
з нанопокриттями**

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Досліджено при кімнатній температурі осциляції оптичного ІЧ поглинання в двовимірних структурах макропористого кремнію з нанопокриттями мікропористого кремнію, SiO₂ та нанокристалів ZnO. Осциляції з великою амплітудою спостерігались у спектральному діапазоні смуг поглинання поверхневими рівнями на межі поділу "кремній – нанопокриття". В наближенні сильного електричного поля розглянуто електрооптичний ефект, пов'язаний з резонансним характером розсіювання електронів поверхневими рівнями з різницею енергій між двома резонансами, яка дорівнює енергії сходинок Ваньє-Штарка. Резонансне розсіювання електронів визначає резонанси діелектричної проникності і відповідну зміну поглинання. При цьому ефект Ваньє-Штарка реалізується в результаті більшого часу розсіювання електронів у порівнянні з періодом їх осциляцій у сильному електричному полі освітленої межі "кремній – нанопокриття".

**Эффект Ваньє-Штарка при комнатной температуре
в двумерных структурах макропористого кремния
с нанопокртиями**

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Исследованы при комнатной температуре осцилляции оптического ИК поглощения в двумерных структурах макропористого кремния с нанопокртиями микропористого кремния, SiO₂ и нанокристаллов ZnO. Осцилляции с большой амплитудой наблюдались в спектральном диапазоне полос поглощения поверхностными уровнями на границе "кремний – нанопокртие". В приближении сильного электрического поля рассмотрен электрооптический эффект, связанный с резонансным характером рассеяния электронов поверхностными уровнями с разностью энергий между двумя резонансами, равной энергии лестницы Ваньє-Штарка. Резонансное рассеяние электронов определяет резонансы диелектрической проницаемости и соответствующее изменение поглощения. При этом эффект Ваньє-Штарка реализуется в результате большего времени рассеяния электронов по сравнению с периодом их осцилляций в сильном электрическом поле освещенной границы "кремний – нанопокртие".