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Wannier–Stark electro-optical effect, quasi-guided and photonic modes in 2D macroporous silicon structures with SiO_2 coatings

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ABSTRACT

Opportunities to enhance the properties of structured surfaces were demonstrated on 2D macroporous silicon structures with SiO_2 coatings. We investigated the IR light absorption oscillations in macroporous silicon structures with SiO2 coatings 0–800 nm thick. The Wannier–Stark electro-optical effect due to strong electric field on Si-SiO_2boundary and an additional electric field of quasi-guided optical modes were taken into account. The photonic modes and band gaps were also considered as peculiarities in absorbance spectra of macroporous silicon structures with a thick SiO_2 coating. The photonic modes do not coincide with the quasi-guided modes in the silicon matrix and do not appear in absorption spectra of 2D macroporous silicon structures with surface nanocrystals.

1. Introduction

The macroporous silicon structures were actively investigated during the last two decades. A material with periodic arrangement of cylindrical macropores is a photonic crystal and has a photonic band gap that reacts to variation of permittivity in macropores [1]. Macroporous silicon is a promising material for development of 2D photonic structures with the required geometry and large effective surface [1,2]. This determines optical and electro-optical characteristics of macroporous silicon structures [3–7]. In view of a potential barrier on macropore surface, one should take into account recharging of the local surface centers at energies below that of the indirect interband transition.

Macroporous silicon has found application and further development in nano- and microelectronics, optics and optoelectronics due to the easy manufacturing, structural and physical properties and the possibility of integration in the chips. Intermolecular Van der Waals forces cause physical adsorption, regardless of the substances nature. This allows the development of sensors based on measurements of optical, electric, photovoltaic and photoluminescence characteristics of macroporous silicon. Thus, macroporous silicon-based optical biosensors were designed to detect low concentrations of DNA [8]. The capacitive humidity sensors [9], gas and biosensors of CMOS-compatible manufacturing, solar cells with efficiency up to 13% [10] and coating with less than 0.1% reflection have been developed [11].

The near-IR optical absorption in 2D photonic macroporous silicon structures was investigated in [6], with allowance made for the linear electro-optical effect. The experimental absorption spectra of macroporous silicon agreed well with the corresponding spectral dependencies 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. In [7], we investigated the near-IR light absorption oscillations in 2D macroporous silicon structures with microporous silicon layers and CdTe, ZnO, CdS surface nanocrystals taking into account the electro-optical effect within the strong electric field approximation. The model [12,13] of the resonance electron scattering on impurity states in an electric field of "silicon-nanocoating" heterojunction on macropore surface and realization of the Wannier–Stark effect on randomly distributed surface bonds were confirmed. In this case, the Wannier–Stark effect was measured due to a large-time electron scattering as compared with the period of its oscillations in the strong electric field of "silicon-nanocoating" interface.

The contribution of the electron-phonon interaction to the broadening parameter Γ of the Wannier–Stark ladder levels was investigated in oxidized macroporous silicon structures with arbitrary macropore distribution [14]. The obtained value of the

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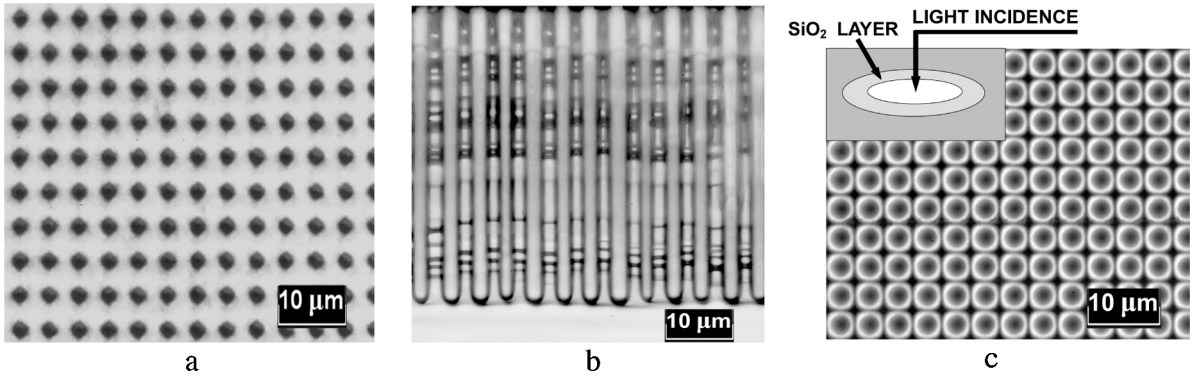

Fig. 1. Surface (a) and profile (b) of 2D macroporous silicon square-lattice structure with period $a = 4$ μm. The oxidized macroporous silicon square-lattice structure (c) with a fragment (insertion) of the cylindrical macropore with SiO_2 nanocoating and direction of light incidence on the sample (along the main axis of cylindrical macropore).

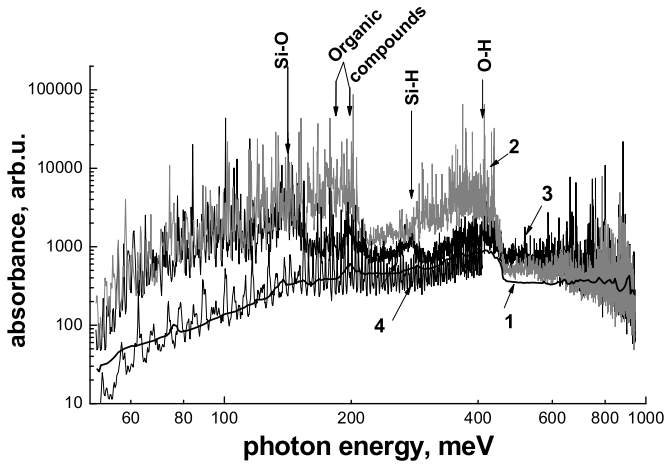

Fig. 2. Absorption spectra of periodic macroporous silicon structures: without coating (1), with 54 nm (2) and 805 nm (3) thick SiO_2 coatings and CdTe nanocrystals (4) from Ref. [7].

Wannier–Stark ladder parameter Γ is much less than the adjacent level energy evaluated from the giant oscillations of resonance electron scattering on the surface states. The growth of the concentration of bridge-like oxygen atoms in Si-O-Si (TO phonons) after oxidation of macroporous silicon is due to reduction of the dangling bond passivation in the absence of hydrogen.

In this paper, the near-IR light absorption oscillations of 2D periodic macroporous silicon structures with 50–800 nm thick SiO_2 coatings were investigated taking into account the Wannier–Stark electro-optical effect due to a strong electric field on Si-SiO_2 interface and an additional electric field of quasi-guided modes. The photonic modes and photonic band gaps were evaluated as peculiarities in absorbance spectra of macroporous silicon structures with a thick SiO_2 coating. A comparison of results obtained for SiO_2 coatings and II–VI (CdTe [7]) surface nanocrystals on 2D periodic macroporous silicon structures was made.

2. Procedure

The samples to be studied were made of silicon wafers characterized by the [100] orientation and *n*-type of conduction (the electron concentration $n_0 = 10^{15}$ cm^{-3}). We used the technique of electrochemical etching at illumination of the back side of silicon substrate (thickness $H = 520$ μm) [3]. The 2D macroporous silicon square-lattice periodic structures (Fig. 1a and b) with period 4 μm, the macropore diameter $D_p = 2$ μm, the macropore depth $h_p = 90$–100 μm and the macropore concentration

Table 1
Oxidation regimes and thickness of SiO_2 coatings formed on macroporous silicon structures.

No.	Oxidation regimes			SiO_2 thickness (nm)
	T (°C)	Atmosphere	Duration (min)	
1	1050	$N_2 + O_2$ (dry)	16	54
2	1200	$N_2 + O_2$ (dry)	50	203
3	1100	$N_2 + O_2$ (wet)	50	805

$N_p = 6.25 \times 10^6$ cm^{-2} were formed. The initial samples are complex micropore-macropore silicon structures consisting of 100 nm micropore layers on macropore walls. An additional anisotropic etching for 30 s in 10% water solution of KOH was used to remove the microporous layers from macropore walls.

SiO_2 coatings on macroporous silicon structures were formed in the diffusion stove in the nitrogen atmosphere (Table 1). The oxide layers with 50 ± 5°nm and 200 ± 5°nm thick have been formed on macroporous silicon samples in dry oxygen during 15–50 min at a temperature of 1050–1200 °C. The 800 ± 5°nm oxide was formed for 50 min at a temperature of 1100°C in wet oxygen using a steam generator with deionized water. The oxide thickness was measured with accuracy of 0.2 nm using ellipsometry.

CdTe nanocrystals 20 nm in size were grown using "a hot wall" molecular epitaxy on periodic macroporous silicon substrates [7] for comparison with results obtained for SiO_2 coatings. The thickness of the deposited films (200 nm) was determined by time of structure staying above the source of evaporation.

The chemical states on the surface of macroporous silicon structures with coatings and the electric field at the "Si—SiO_2" boundary were identified by IR absorption using a PerkinElmer Spectrum BXII IR Fourier spectrometer. The optical absorption spectra are recorded at normal incidence of IR radiation on the sample (along the main axis of cylindrical macropores—see Fig. 1c, insertion). The experiments are carried out at room temperature in air. The error of spectral measurements is about 2 cm^{-1}. The sensitivity of the light intensity is 0.5%.

We compared the "air-to-sample" FTIR spectra to evaluate the possible influence of the interference phenomena on the resulting spectra. No additional processing (smoothing) of the measured spectra was not conducted. We confirmed the reproducibility of FTIR spectra by repeated measurements.

3. Experimental

For macroporous silicon structures with SiO_2 coatings 54 nm and 805 nm thick, the light absorption increases and an oscillating structure occurs (Fig. 2, curves 1 and 2), similarly to the macroporous silicon structures with CdTe surface nanocrystals

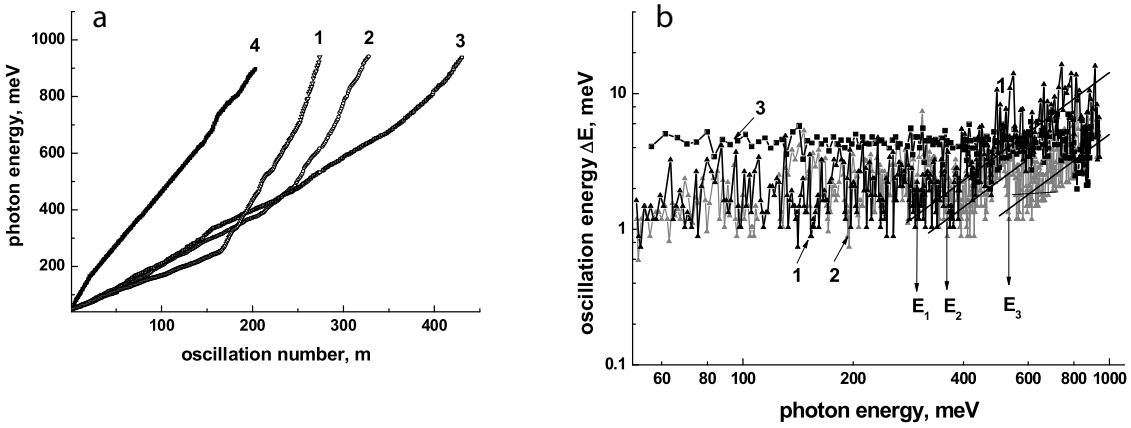

Fig. 3. (a) Spectral position of oscillation maxima in the macroporous silicon structures with 54 nm (1), 203 nm (2) and 805 nm (3) thick SiO_2 coatings and CdTe surface nanocrystals (4). (b) Spectral dependencies of the oscillation energy ΔE in the macroporous silicon structures with 203 nm (1) and 805 nm (2) thick SiO_2 coatings and CdTe surface nanocrystals (3); E_1–E_3—minimal photon energies of the oscillation energy ΔE quadratic dependencies.

(Fig. 2, curve 3) from [7]. We observed the essential absorption growth in the spectral region 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. The form of oscillations indicates their resonant character. In general, such peculiarities take place for macroporous silicon with SiO_2 coatings with thickness above 10 nm [14].

The dependences of oscillation maxima of macroporous silicon structures with 54 nm, 203 nm and 805 nm thick SiO_2 coatings on oscillation number have bends (Fig. 3a, curves 1–3) at photon energies from 200 meV to 650 meV. The spectral position of oscillation maxima of macroporous silicon structure with CdTe surface nanocrystals vs oscillation number (Fig. 3a, curve 4) is straight line.

The oscillation period $\Delta E = 1$–4 meV fluctuates about a constant value at low spectral energies and becomes quadratic at photon energies depending on SiO_2 coating thickness (Fig. 3b, curves 1 and 2). The oscillation period of macroporous silicon structures with CdTe surface nanocrystals is almost constant (Fig. 3b, curve 3) with oscillation energy $\Delta E = 4.5$ meV. Minimal photon energies of the oscillation period quadratic dependencies vs photon energy change from 300 meV to 600 meV for the 203 nm and 805 nm thick SiO_2 coatings (Fig. 3b).

In addition, we observed some peculiarities at photon energies of 110–200 meV and 220–480 meV (Fig. 4) in the absorption spectra of macroporous silicon structures with 805 nm thick SiO_2 coating. The peculiarities are similar to those observed in reflectance spectra of macroporous silicon photonic crystals at near-normal (5°) light incidence [15]. The critical points $E_{01} = 110$ meV and $E_{02} = 220$ meV in absorbance may be related to singularities in diffracted intensity $D_i(\omega) = C_i(h\omega - E_{0i})^{-1/2}$ due to a photonic mode excitation as an "absorption" process that includes intensity of diffracted beam: $A = A_{0i} + (D_i)^{-1}$. Excitation of photonic modes in reflectance spectra of macroporous silicon structures was explained in [15] by the diffracted beam too.

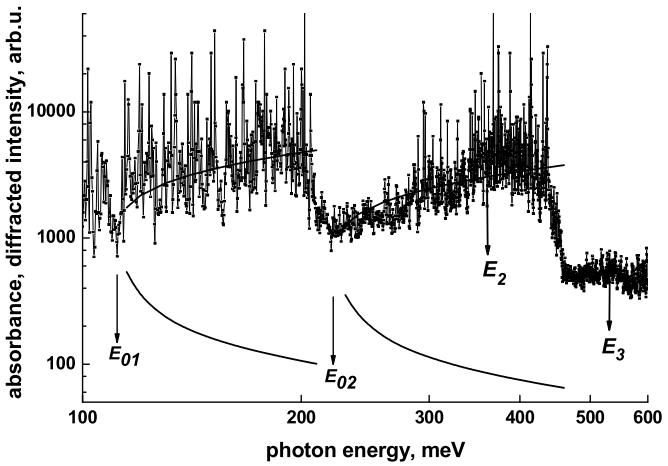

Fig. 4. IR absorption spectra of macroporous silicon structures with 805 nm thick SiO_2 coating: E_2 and E_3—the minimal energies of the oscillation period quadratic dependencies vs photon energy from Fig. 3b; E_{01} and E_{02}—critical points of peculiarities in absorption spectra of macroporous silicon structure. Below: spectral dependence of the diffracted beam intensity $D_i(\omega) \sim (h\omega - E_{0i})^{-1/2}$.

4. Discussion of results

4.1. Wannier–Stark effect

We observed the oscillating structure in the absorption spectra of macroporous silicon with 50–800 nm thick SiO_2 coatings. The amplitude of oscillations is maximal in spectral ranges of the surface state absorption (Si—O, Si—H, O—H bonds and organic compounds). The results obtained indicate strong effect of local states on the boundary "macroporous silicon-SiO_2 coating". This may result from scattering of electromagnetic radiation and electrons on local surface states. The form of oscillations (Fig. 2) indicates their resonant character.

The oscillations of small amplitude in macroporous silicon structures without coatings [6] correspond to the weak electric field approximation. The macroporous silicon structures with surface coatings investigated in this paper have higher surface potential. And the onset of oscillations with giant amplitude may be explained by the electro-optical process in strong electric fields. Really, well-separated oscillations in the spectral ranges of the surface bond absorption that we observed in the absorption spectra of macroporous silicon structures with surface nanocrystals [7] are explained by realization of the Wannier–Stark effect on randomly distributed surface bonds.

We analyzed a method of experimental observation of Wannier–Stark ladders proposed in [12,13] to confirm the realization of the resonant scattering of electrons on local surface states. In [12], a semiconductor with lattice constant a and dispersion law $E(k) = E_0 - \Delta(\cos k_y a + \cos k_z a)$ was considered; here k is a quasi-momentum with components k_y and k_z, E_0 the

energy corresponding to the midgap, and Δ the energy equal to 1/6 the band gap. The wave function in the Wannier representation is

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

Here the first (second) term describes the incident wave (scattered waves); j is the number of the lattice site, $\hat{G}_0(E)$—the Green operator, V_0 the impurity potential. The complex energies for which the denominator of the second term becomes zero correspond to the resonances in electron scattering:

$$1/V_0 = \langle 0|\hat{G}_0(E)|0\rangle,$$

where $E = \varepsilon - i\Gamma$ ($\Gamma > 0$). The difference of two neighboring resonance energies ΔE is approximately equal to the value of the step Fa in the Wannier–Stark ladder for electric field strength F. If the electric field is directed along the x-axis of the crystal, then electron scattering occurs in the perpendicular plane (y, z), and the difference between two resonant energies is approximately equal to Wannier–Stark ladder.

In our case, an electric field of "macroporous silicon-coating" boundary is directed at a normal to the macropore surface too, and the surface states that scatter electrons are concentrated perpendicularly to the x-direction in the plane (y, z) that is the plane of resonant scattering.

Detection of the Wannier–Stark ladder requires that this width Γ would be less than the difference of energies of adjacent levels $\Delta E = Fa$ ($\Gamma < Fa$) [13]. And the Wannier–Stark ladder is not broken by local surface states if the intervals between the transitions due to scattering from the local state with lifetime τ are bigger than the period of electron oscillations in external electric field or the Bloch oscillation time, T_B ($\tau/T_B > 1$). The Bloch oscillation time is equal to $T_B = 2\pi h/Fa$, and the scattering time is equal to $\tau = 1/W$ (W is the probability for an electron to leave the state per unit time due to scattering from an impurity atom at a lattice site). The probability W for an electron to leave the local state per unit time due to scattering from this local state was obtained in [13] as $W < 2V_0 Ni/(Nh)$, 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 last inequality, we find a numerical estimate of the local surface state concentration.

The surface state concentration in macroporous silicon structures changes from $10^{10}\,cm^{-2}$ to $10^{11}\,cm^{-2}$ [16], and $N_i^{max} > 10^{11}\,cm^{-2}$ for the spectral range studied. Spectral dependencies of the lifetime ratio τ/T_B for macroporous silicon structures with SiO_2 coatings of 203 nm (1) and 805 nm (2) thickness are presented in Fig. 5.

According to Fig. 5, the inequality $\tau/T_B > 1$ for the lifetime ratio is satisfied over the whole spectral region studied for macroporous silicon structures with SiO_2 coatings taking into account that the surface impurity concentration for macroporous silicon structures, N_i, is less than $10^{11}\,cm^{-2}$, and the Bloch oscillation time is $T_B \approx 10^{-11}$ s. The Wannier–Stark ladder is preserved in the same spectral range for macroporous silicon structures with surface nanocrystals [7] and the Bloch oscillation time $T_B \approx (4–8) \times 10^{-12}$ s.

4.2. Quasi-guided modes

The dependence of oscillation maxima in macroporous silicon structures with SiO_2 coatings on oscillation number has bends (Fig. 3a). The oscillation period fluctuates about a constant value at low spectral energies and becomes quadratic in photon energy, depending on SiO_2 coating thickness (Fig. 3b). The oscillation period ΔE is the Wannier–Stark ladder and depends on the electric field strength: $\Delta E = Fa$. Usually, the basic sources of external electric field

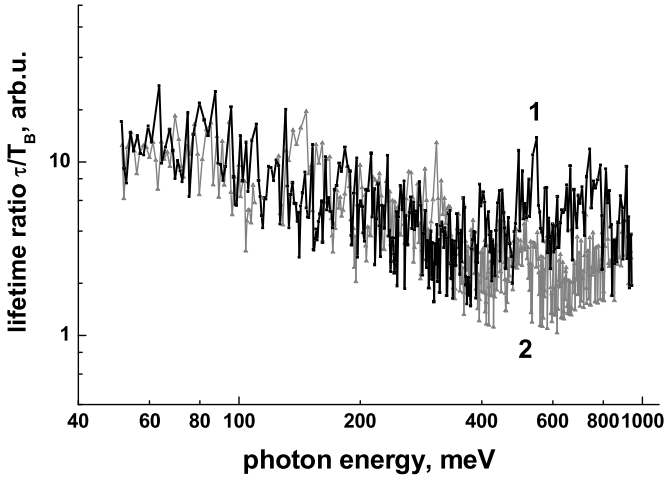

Fig. 5. Spectral dependencies of lifetime ratio τ/T_B for macroporous silicon structures with 203 nm (1) and 805 nm (2) thick SiO_2 coatings.

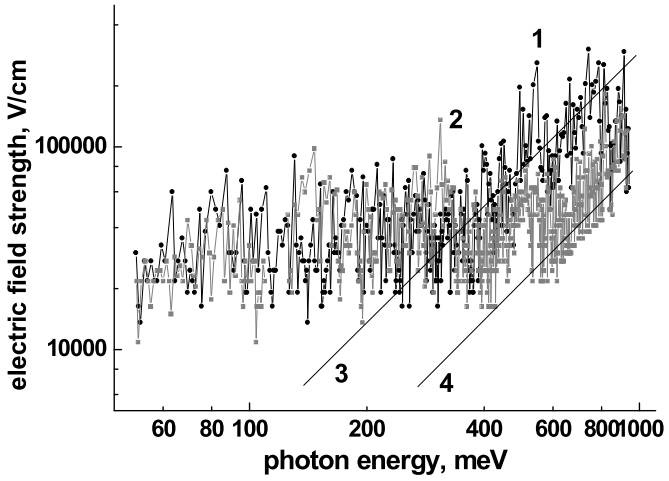

Fig. 6. Spectral dependence of electric field strength F_s on "macroporous silicon-coating" boundary for structures with 203 nm thick (curve 1) and 805 nm thick (curve 2) SiO_2 coatings; quadratic spectral dependencies of the electric field strength (lines 3 and 4) are a result of quasi-guided mode formation in the silicon matrix.

at a semiconductor surface are the charge of surface states and the built-in charge in the semiconductor surface oxide [17].

Spectral dependencies of the electric field strength F on "macroporous silicon-coating" boundary for structures with 203 nm and 805 nm thick SiO_2 coatings are presented in Fig. 6 (curves 1 and 2). The oscillation period and electric field strength of macroporous silicon structures with SiO2 coatings fluctuate about a constant value at low spectral light energies and follow quadratic low at high spectral light energies (Fig. 6, curves 3 and 4).

The electric field strength F (Fig. 6) became quadratic at some photon energies. 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 [18]. 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 conditions of the grazing angle of light incidence onto the macropore surface, the electric field strength on macroporous silicon surface for structures with SiO_2 nanocoatings is about $F_s + \Delta F_s$, with $\Delta F_s \approx \hbar\omega/(0.1\lambda_1) \sim \hbar\omega^2$ (Fig. 6, lines 3 and 4) according to the experiment (Fig. 6, curves 1 and 2). Light wavelength is equal to $\lambda_1 = \lambda/n_i$

Table 2
Structure characteristics and quasi-guided mode parameters.

Silicon matrix and silicon column sizes			Minimal photon energy (wavelength) of ΔE quadratic dependence		Mode type	$2\rho_{Si}$, (μm)
d_{SiO2}, (nm)	$a-(D_p+2d_{SiO2})$, (μm)	$1.4a-(D_p+2d_{SiO2})$, (μm)	Photon energy, (meV)	Wave-length, (μm)		
54	1.9	2.7	205	**6.05**	Quasi-guided modes in the silicon column	$2[1.4a-(D_p+2d_{SiO2})]$= **=5.4**
			493	**2.5**	Quasi-guided modes in the silicon column	$1.4a-(D_p+2d_{SiO2})$= **=2.7**
203	1.6	2.4	305	**4.1**	Quasi-guided modes in the silicon column	$2[1.4a-(D_p+2d_{SiO2})]$= **=4.8**
			595	**2.2**	Quasi-guided modes in the silicon column	$1.4a-(D_p+2d_{SiO2})$= **=2.4**
805	0.4	2.0	338	**3.67**	Quasi-guided modes in the silicon column	$2[1.4a-(D_p+2d_{SiO2})]$= **=4.0**
			607	**2.04**	Quasi-guided modes in the silicon column	$1.4a-(D_p+2d_{SiO2})$=**2.0**

(n_i is effective refractive index of pores with SiO_2 coatings or refractive index of SiO_2 coatings).

The silicon matrix sizes for the structures studied as well as the minimal photon energy of $\Delta E(F)$ quadratic spectral dependencies from Figs. 2b and 6 are presented in Table 2.

It is evident from Table 2 that the minimal energy ΔE quadratic spectral dependence is equal to light wavelengths corresponded to geometrical sizes of macroporous silicon matrix and to the quasi-guided mode formation [19]. Quasi-guided modes have the complex value of the mode propagation β, and its mode parameter $Q \sim k\rho$ is determined by the size of the waveguide core [19]. Thus, for our case the mode parameter might be proportional to the silicon matrix size (minimal distance between macropores) with $2\rho_{Si} = a-(D_p+2d_{SiO2})$ and to the silicon column size (diagonal distance between macropores) with $2\rho_{Si} = 1.4a-(D_p+2d_{SiO2})$ (Fig. 1). According to Fig. 3b and Table 2, the relevant photon energy ΔE quadratic growth corresponds to the quasi-guided mode formation in the silicon column for investigated macroporous silicon structures with 54 nm, 203 nm and 805 nm thick SiO_2 coatings.

4.3. Photonic modes

The peculiarities at photon energies 110–200 meV and 220–480 meV in absorption spectra of macroporous silicon structures with 805 nm thick SiO_2 coating (Fig. 4) are similar to those observed in reflectance spectra of macroporous silicon photonic crystals at the near-normal (5°) light incidence [15]. Such peculiarities in absorbance of macroporous silicon structures are due to matching of the external field with a Bloch state. Such a state behaves like a one-dimensional (1D) critical point. The density of states is determined by photon dispersion in the vertical direction along macropores only. The in-plane momentum and the parallel vector k are conserved. Onset of a diffracted beam corresponds to a complex wave-vector component q that passes through zero and becomes real [20]. The out-of-plane dispersion of all bands is quadratic in q around q = 0 [21], with a threshold at $E(\omega,q) = E_0$ and a diffracted intensity $D(\omega) \sim (h\omega - E_0)^{-1/2}$. It corresponds to 1D density of states. The peculiarities in absorption spectra (Fig. 4) mark the onset of a photonic mode that is excited and remains propagating for higher frequencies [15].

The diffracted intensity $D_i(\omega)$ is removed from the absorbance (Fig. 4) and has the form of square root at $E(\omega,q) > E_0$. This proves the 1D critical point measurement in absorbance [22]. Thus the calculated diffracted intensities of allowed modes at normal incidence have the form of the inverse square root close to thresholds E_{01} = 110 meV and E_{02} = 220 meV. Such bands do not appear in absorption curves of macroporous silicon structures with SiO_2 layers of less thickness (Fig. 2b) due to very weak spectral strength.

The threshold energies E_{01} and E_{02} (Fig. 4) correspond to normalized frequencies $\omega a/2\pi c = 0.36$ and 0.71. The obtained results are similar to calculations for 2D square lattice composed of circular air-rods in dielectric material with dielectric constant 2.1 and filling factor 0.25 [23]. There are two band gaps between the first and the second 1D eigenmodes (*A* modes) and between the second and the third *A* modes. Our structure is three-component one, with averaged refractive index 1.4, and structure from [23] is two-component, with averaged refractive index 1.3. And pronounced structure that was observed in experimental reflectance spectra at near-normal (5°) light incidence on macroporous silicon structure with averaged refractive index 3 around higher normalized frequency 0.47 [15] corresponded to the allowed band with symmetry Γ^{-5}.

The quasi-guided mode formation in the silicon matrix at energies 200–600 meV (Table 2) does not appear in absorption spectra (Figs. 2 and 4) and does not coincide with the photonic modes as peculiarities in absorbance (and reflectance) spectra of macroporous silicon structures. In addition, the quasi-guided and photonic modes do not appear in absorption spectra of 2D macroporous silicon structures with microporous silicon layers and CdTe, ZnO surface nanocrystals [7] as well as in oxidized macroporous silicon with arbitrary macropore distribution [14].

5. Conclusions

The near-IR light absorption oscillations in the 2D macroporous silicon structures with 50–800 nm thick SiO_2 coatings are investigated taking into account the Wannier–Stark electro-optical effect within the strong electric field approximation. We observed the oscillating structure in the absorption spectra of macroporous silicon structures with SiO_2 coatings. The amplitude of oscillations is maximal in the spectral ranges of surface level absorption as a result of the resonance electron scattering on impurity states, with the difference between two resonance energies equal to the Wannier–Stark ladder.

The oscillation period ΔE = 1–4 meV in the macroporous silicon structures with 50–800 nm thick SiO_2 coatings fluctuates about a constant value at low photon energies and becomes quadratic in photon energy. The oscillation period of macroporous silicon structures with CdTe surface nanocrystals is almost constant with oscillation energy ΔE = 4.5 meV. A comparison of the silicon matrix sizes and minimal energy of oscillation period ΔE quadratic dependence confirmed the quasi-guided mode formation in the silicon column for structures with 50–800 nm thick SiO_2 coatings.

The peculiarities in absorption spectra of periodic macroporous silicon structures with thick SiO_2 coating are similar to those observed in reflectance spectra of macroporous silicon photonic crystals at near-normal light incidence. The critical points in absorbance are related to photonic modes excitation as an "absorption" process that includes intensity of the diffracted beam. The photonic modes do not coincide with quasi-guided modes in the silicon matrix and do not appear in absorption spectra of 2D macroporous silicon structures with surface nanocrystals and

in oxidized macroporous silicon with arbitrary macropore distribution.

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