Quantum-size effects in semiconductor heterosystems

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Abstract. Created on the basis of Si, GaAs and C_{60} fullerenes were low-dimensional heterostructures with a surface quantum-size effect at the film-substrate interface. There have been defined technological conditions of its appearance. Using modulation electroreflectance spectroscopy, calculated were spectral broadening parameters, the energy relaxation time of excited light charge carriers, the energy of quantized levels and the width of the quantum wells.

Keywords: heterostructures, interface, electroreflectance, electronic parameters, quantum-size effect.

1. Introduction

Physical properties of heterosystems are not a simple sum of the contacting materials. They acquire new electronic and optical properties due to appearance of the surface potential, internal mechanical stresses, built-in electric fields and quantum size effects at the film-substrate interface. When the size of the region of free charge carrier localization becomes comparable with the free length, their movement is restricted to one, two or three directions. Low-dimensional heterosystems have a different energy dependence of the electron density of states in the band structure, which depends on their dimensions. Decreasing localization of free charge carriers reduces heterosystem dimensionality. These heterosystems can be two-dimensional with quantum wells, one-dimensional in the case of quantum wires and zero-dimensional when quantum dots are available. The energy dependence of the density of electron states in these low-dimensional structures is different. Monotonous quadratic energy dependence, which is typical for bulk sample, becomes discrete. For two-dimensional structures, it is monotonous and changes stepwise with increasing the energy of charge carriers. For one-dimensional structures, it is continuous and sawtooth, and is set of dashed lines for zero-dimensional structures [1]. It is necessary to distinguish between the bulk quantum-size effect in heterosystems with a rectangular quantum well, where quantized are both types of charge carriers, from the surface quantum-size effect, when quantized is only one their type in the triangular quantum well. Transformation of the band structure in the low-dimensional heterosystems leads to changes in their electronic and optical properties [2].

Silicon is still the main material of semiconductor electronics. Gallium arsenide ranks second after the silicon and is also widely used in optoelectronics. On its basis, there are LEDs, lasers, photodetectors and solar cells. Sulfidation of the GaAs surface decreases the density of surface electronic states and increases the intensity of photoluminescence [3]. Development of technology for production of fullerenes in the form of film material on different substrates expands the use of nanostructures with C_{60} fullerenes [4], including solar cells [5, 6] and sensors of physical quantities [7].

Modulation electroreflectance spectroscopy has considerable advantages in comparison with classical reflectance and transmittance spectroscopy. When investigating the band structure of semiconductors, their electronic and optical properties, the sensitivity of this method is several orders of magnitude more sensitive than the classical optical methods. The advantage of the modulation spectra is that they reveal the thin structure usually hidden unstructured background. The electroreflectance method is more sensitive to the band structure of the semiconductors, because its signal is the

third derivative of the optical constants. The signal exists at the critical points of the Brillouin zone only in the region of direct transitions, and it disappears at some distance from the critical point. The electroreflectance signal is determined by the optical and electronic properties of the surface under study [8-10].

The purpose of this study was to find and study the surface quantum-size effect in semiconductor heterosystems based on silicon, gallium arsenide, and C_{60} fullerene by using modulation electroreflectance spectroscopy.

2. Experiment

Measurements of electroreflectance spectra in the heterosystems were carried out with the electrolytic method. Essence of the method is to register changes in the reflectivity of the semiconductor surface $\Delta R/R$ upon application of a weak modulating electric field. Measurements were made using the setup based on the DMR-4 monochromator with automatic registration of spectra on the screen in a linear energy scale. The spectral resolution was 3 meV. The sensitivity of this apparatus for $\Delta R/R$ signal measurement reached 10^{-6}, and the accuracy of measuring intensity was 2%. The signal was recorded within the spectral range 1.5 to 3.5 eV at room temperature in a weak-field measuring regime. The modulating voltage did not exceed 0.7 V.

The samples were placed into a silica electrolytic cell with 0.1 N water KCl solution. From the photomultiplier, the signal was fed to the selective amplifier U2-6 and registered using a synchronous detector. This method allowed us to obtain information about the band structure of heterosystem on its surface and in the region of the film-substrate interface, as well as to detect its change depending on the technological conditions of heterosystems manufacturing and influence on them caused by external actions.

In order to obtain heterosystems with the quantum size effect, we used anisotropic chemical etching of Si [11] and GaAs [12] surfaces, sulfide passivation of GaAs surface by chemical etching [13], neutron irradiation and annealing of Si wafers [14], as well as thermal vacuum deposition of C_{60} fullerenes on Si and the cover glass substrate [15]. An analysis of the electroreflectance spectra in a specific region of the *k*-space in the Brillouin zone for a direct transition makes it possible to determine the transition energy E_g, the phenomenological Lorentz broadening parameter Γ, which allows for dissipation processes in the electron transition, and the energy relaxation time τ of the light excited charge carriers. These parameters were determined using the three-point method by Aspnes [16].

3. Results and discussion

The results of measurement of the electroreflectance spectra of the initial *n*-Si (100) surface and

anisotropically etched in HF:HNO$_3$ = 20:1 mixture in the course of 20…30 min depicted in Fig. 1. The electroreflectance signal of the initial surface Si was recorded in the region of 3.2 to 3.55 eV (curve *1*, Fig. 1). According to the electron band structure of silicon, this electroreflectance signal corresponds to direct transitions that occur between the valence and conduction bands at the center of the Brillouin zone. In the process of anisotropic chemical etching, the surface of the Si(100) plate dimmer, and microrelief appears on the surface. The SiO$_2$ layer of crystal modification of β-crystoballite forms at the plate surface [11]. The electroreflectance spectrum of anisotropically etched silicon surface is shown in Fig. 1, curve *2*. There are inversion of the polarity signal, the value of the phenomenological parameter Γ decreases, and peaks are separated by 40 meV.

At the initial surface of the silicon substrate, and also after the oxide layer is removed from the microrelief surface, the bands undergo depletion bending. The surface is depleted of electrons, and the band are bent upward (Fig. 1, spectrum 1). On the Si surface under a layer of SiO$_2$, the potential is of enrichment type, and the bands are bent downward (Fig. 1, spectrum 2). The substrate surface is enriched with electrons, and this leads to the change in the electroreflectance signal phase at the interface SiO$_2$–Si.

The doublet nature of the peaks of electroreflectance in anisotropically etched silicon can be explained by the effect of surface quantization of the electron energy in the enriched surface layer of silicon at the boundary. Between the SiO$_x$ layer and silicon, there is a quantum well. Availability of 2D electrons in the conduction band leads to a situation when, in addition to electron transitions between the main bands, there are transitions between the valence band and the quantized level e_1 in the 2D quantum well. The presence of two transitions with energies E_g and E_g+e_1 splits the peaks in the electroreflectance spectrum.
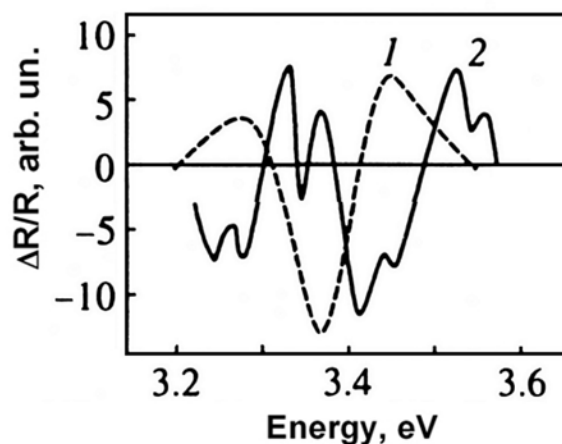

Fig. 1. Electroreflectance spectra of initial (*1*) and anisotropically etched (*2*) silicon surfaces in the E_0 transition region.

The energy of optical transitions in the quantum-sized system for the first quantization level is given by the expression

$$e = E_g + \hbar^2 \pi^2 / 2\, m^* L^2, \qquad (1)$$

where E_g is the band-to-band transition energy, m^* – the band-to-band effective mass, and L – width of the quantum well. The value $e_1 = 40$ meV found from the experimental electroreflectance spectra under the assumption that the effective hole mass is $m_p = 0.49 m_0$ at the point Γ'_{25} C and that the effective electron mass is $m_e = 0.156 m_0$ (there m_0 is the free electron mass). With allowance for the expression (1), the width of the quantum well L is 4 nm.

Using the electroreflecance spectroscopy method, we also examined the microrelief As_2O_3–pnn^+-GaAs heterostructures [12]. On the Te-doped n^+-GaAs(100) substrate with electron concentration of $8 \cdot 10^{18} \dots 2 \cdot 10^{19}$ cm^{-3} and thickness of 300 μm, we used the CVD method to deposit the 10…15-μm Si-doped (n-type) and the 10…15-μm Zn-doped (p-type) GaAs layers with the carriers concentration of about $8 \cdot 10^{16} \dots 10^{17}$ cm^{-3}. The microrelief heterostructures were formed on the frontal surface of the pnn^+-GaAs substrates by chemical etching in a 10…15 N HNO$_3$ solution for 10 s at room temperature.

Fig. 2 depicts electroreflectance of E_1 and $E_1+\Delta_1$ transition for initial pnn^+-GaAs substrate (curve *1*) and microrelief As_2O_3–pnn^+-GaAs heterostructure obtained using 10-s anisotropic etching (curve *2*). Table 1 illustrates the electron zone parameters of pnn^+-GaAs substrate and relief As_2O_3–pnn^+-GaAs heterostructure: the E_1 and $E_1+\Delta_1$ energy transitions, the value of spin-orbit splitting Δ_1, the spectral broadening parameters Γ_1 and $\Gamma_{1+\Delta 1}$. The decease of Γ parameters for the substrate in heterosystem caused by a good quality of the interface As_2O_3–pnn^+-GaAs. Such a feature is observed repeatedly, for example, at the SiO_2–Si microrelief interface [11].
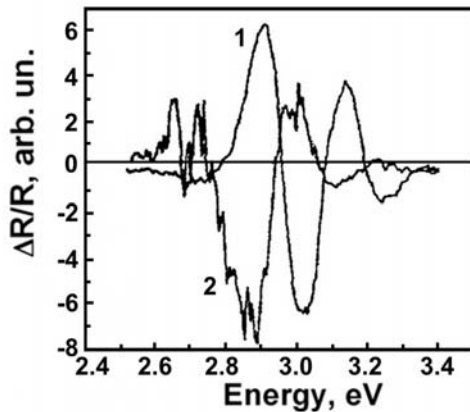
Table 1. Effect of anisotropic chemical etching on zone parameters of pnn^+-GaAs ($t_{ACE} = 10$ s).

Surface type	Zone parameters, eV				
	E_1	$E_1+\Delta_1$	Δ_1	Γ_1	$\Gamma_{1+\Delta l}$
Non-patterned	2.969	3.161	0.192	0.119	0.104
Patterned	2.895	3.105	0.210	0.090	0.089

In Fig. 2, there are two other remarkable features in the electroreflerctance spectrum of the microrelief sample: 1) inversion of signal polarity and 2) formation of a fine peaked structure, which is seen most clearly on the short-energy shoulder of the ER spectrum. These features may result from: 1) inversion of substrate conductivity type from p to n under formation of the inversion layer on As_2O_3–pnn^+-GaAs interface and 2) decrease in the heterosystem dimensionality from 3D to 2D for the carrier motion in the direction perpendicular to the interface. At the interface, the triangular quantum well (QW) appears with quantized levels (QL) in the two-dimensional electron gas channel. In Fig. 2, one can see 3 quantum levels.

Table 2 illustrates the energy of quantized levels E_n and the width of quantum well L_n at the As_2O_3–pnn^+-GaAs interface. In accordance with Table 2, the inversion layer is presented by the triangular quantum well with energy quantum levels of 36, 64 and 93 meV on its width from 14 to 26 nm.

It was applied GaAs surface passivation with sulphur to improve its structural perfection and electronic properties. Using the electroreflectance method, we investigated the real and chemically sulfided gallium arsenide surface within the spectral regions of E_0 and E_0+E_1 transitions. Natural oxide was removed from real surface of substrate by etching for 10 min in the mixture of H_2SO_4:H_2O_2:H_2O = 3:1:2 and by washing in distilled water. Then, the samples were placed for 5 to 20 min in saturated solution of $Na_2S \cdot 9H_2O$ and illuminated with an incandescent lamp 500 W. After washing with distilled water, the samples were dried in a stream of warm air. Under the action of photochemical reaction, the bonds Ga–As were broken and the bonds Ga–S appeared. Investigations of GaAs surface before and after its sulfidation were carried out in the spectral range of 1.3…1.6 eV for E_0 transition and 2.6…3.4 eV for E_1 transition. Fig. 3a shows electroreflectance spectra of initial surface (curve 1) and after its sulfidation (curve *2*) for E_0 transition. Fig. 3b shows the corresponding spectra for E_1 transition. In contrast to the initial surface of GaAs, the electroreflectance signal from sulphided surface also appeared in the region 2.5…2.65 eV. Its appearance can be explained by formation of Ga_2S_3 sulphide coating with $E_g = 2.55$ eV band [17]. The coating had a p-type conductivity, and $\Gamma = 55$ meV.

Fig. 2. Electroreflectance spectra of pnn^+-GaAs with flat (*1*) and microrelief As_2O_3–GaAs (*2*) surfaces.

Table 2. Energy position of quantum level E_n on the width of quantum well L_n.

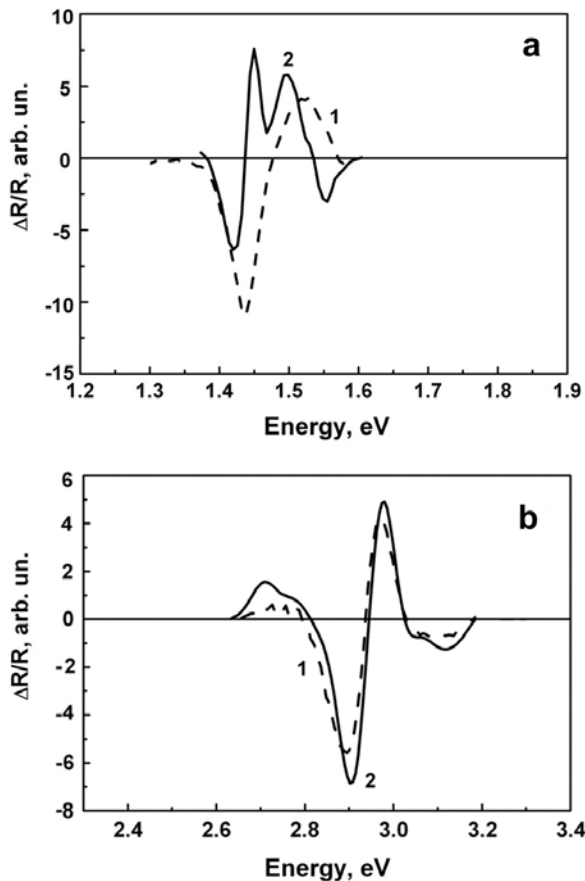| The position of QL E_n, eV	Width of QW L, nm
0.036	14.08
0.064	21.11
0.093	25.7

Fig. 3. Electroreflectance spectra of initial (*1*) and sulphurized (*2*) GaAs surfaces in the E_0, $E_0+\Delta_0$ (a) and E_1, $E_1+\Delta_1$ (b) transition regions.

Presented in Table 3 are electronic parameters of the electroreflectance spectra of GaAs surface for different duration of its sulphidation [18]. The decrease of Γ value and increase of the energy relaxation time inherent to the excited light charge carriers ($\tau = \hbar/\Gamma$) for both transitions is an evidence of electronic passivation of gallium arsenide surface after its sulphidation. Decreasing E_g by 14 meV for the transition E_0 and 7 meV for E_1 is caused by occurrence of internal mechanical stresses ($1.3 \cdot 10^8$ Pa) in Ga$_2$S$_3$–GaAs heterosystems [19]. Splitting the electroreflectance signal is associated with appearance of the surface quantum size effect in the heterosystem. In this case, the parameter Γ is decreased and τ is increased at the interface, which lead to an increase in the free path of charge carriers and increases their mobility μ.

Table 3. Electronic parameters of GaAs surface determined from electroreflectance spectra (region E_0) at different sulfidation time.

t, min	E_0, eV	ΔE_0, eV	Γ, meV	τ, 10^{-14} s	μ, cm/(V·s)
0	1.451		80	0.82	2000
5	1.437	−0.014	32	2.05	3200
10	1.397	−0.054	36	1.82	3000
20	1.390	−0.061	51	1.29	2400

The method of light electroreflectance spectroscopy was used to study crystals of *p*-type silicon, grown by the Czochralski method (specific resistivity was 10 Ohm·cm, oxygen concentration – $8 \cdot 10^{17}$ cm^{-3}). Measurements of electroreflectance spectra were carried out in the spectral region of 3 to 3.8 eV (direct transition E_0 in the centre of the Brillouin zone of Si). The samples were irradiated with fast neutron fluence $10^{15}...10^{18}$ n/cm^2 at 70 °C [14]. Particular attention was given to preparation of its surface to measurements [20]. It was used isothermal annealing (800 °C, 700 hours) in order to create the oxygen phase in crystals. The layer disrupted by mechanical polishing was removed by polishing etching in the mixture HF:HNO$_3$:H$_2$O = 1:3:1.

The shape of electroreflectance signal depended on irradiation conditions and thermal treatment of the samples (the presence of oxygen-silicon precipitates, radiation defects). The electroreflectance spectrum of the initial sample was bipolar, $\Gamma = 138$ meV (Fig. 4a, curve *1*). After annealing of the initial sample (800 °C, 700 hours), the broadening parameter of the spectrum decreased down to 68 meV. The spectrum has shifted to the high-energy region. The annealed sample (Fig. 4a, curve *2*) *p*-type conductivity changed to *n*-type. Inversion of the signal due to formation of high-temperature thermal donors occurred in the process of annealing [21]. E_g increase and decrease of the parameter Γ is associated with a decrease of mechanical stresses and improvement of the structural quality of the sample surface after chemical treatment.

Neutron irradiation of the samples before annealing led to transformation of the structural defects in the crystal. Radiation defects on the surface of the sample formed at lower doses than in the bulk. Therefore, the surface layer of 15-µm thickness was removed by chemical etching. At low doses, plate precipitates of crystalline phase (kristobalit) appear. With increasing of the neutron fluence, there occurred colony of microprecipitates (amorphous SiO$_2$) [22]. Fig. 4b shows the electroreflectance spectra of silicon irradiated with the fluence 10^{18} n/cm^2 before annealing (curve *1*) and after annealing at 800 °C (curve *2*). On the curve *1*, two maxima are clearly seen at 3.325 and 3.403 eV. The splitting of the signal (78 meV) in annealed silicon is caused by appearance of different areas of disorder in it (accumulation of divacancies or interstitial defects).
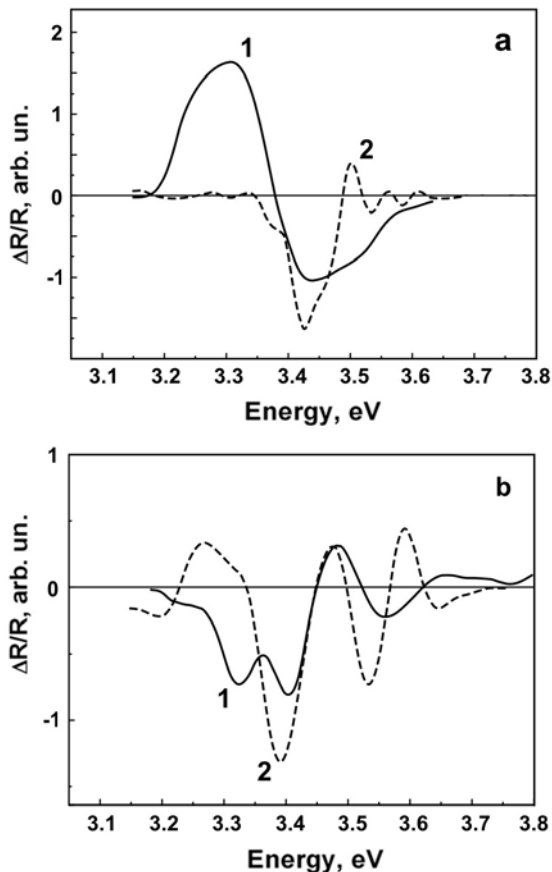
Fig. 4. Electroreflectance spectra of monocrystalline mechanically polished and chemically etched silicon: a) non-annealed before irradiation (*1*) and annealed (*2*); b) irradiation fluence 10^{18} n/cm^2 non-annealed (*1*) and annealed (*2*).

Mechanical stresses change semiconductor band gap [23]. Vacancy type disordering in Si leads to appearance of tensile stresses and an increase of E_g from 3.348 up to 3.403 eV. Interstitial defects create compressive stresses, and E_g value decreases down to 3.325 eV. The local increase and decrease of E_g in the sample splits of the electroreflectance signal.

After annealing at 800 °C, the regions of both types of disorder and point radiation defects are annealed, mechanical stresses of different sign compensate one another, the defect structure of the sample improves, and there is a quantum-size effect (Fig. 4b, curve *2*). During annealing of the irradiated samples, there appear thermodonors and electrically active particles of a new phase SiO$_x$. In the annealed sample, influence of the interface SiO$_x$/Si on the band structure of heterosystem and electronic processes in it increases with growth of the neutron fluence. The negative charge compensating positive charge in the oxide accumulates during the annealing process at the interface with silicon. Bands are curved down, and charge carriers are quantized in a triangular potential well at the interface. With increasing the irradiation dose, the energy of quantized level increases from 60 up to 85 meV, and the width of the quantum well decreases from 3 down to 2 nm [14].

The discovery of new carbon molecules C$_n$ called fullerenes has led to the appearance of a new class of carbon based solids and heterostructures [24, 25]. Among the famous fullerenes, C$_{60}$ molecules are most symmetrical and stable. The carbon atoms in them have a covalent type of bonding, and the C$_{60}$ molecules in the crystal have the weak bond corresponding to van der Waals interaction.

The C$_{60}$ films were obtained using thermal evaporation of C$_{60}$ powder in vacuum at pressure 10^{-4} Pa from an effusion tantalum cell at the temperature of 650 K on non-heated *n*-Si and cover glass substrates [15]. The thickness of films was 0.2 μm. Substrate material does not effect on the films structure at the deposition rate 5…10 nm/s. According to [26], C$_{60}$ fullerene films are a new direct band semiconductor with E_g band gap value near 1.6 eV.

Fig. 5 presents the electroreflectance spectra of C$_{60}$/*n*-Si heterosystem from the films surface (Fig. 5a) and from the interface (Fig. 5b). The band gap E_g was 1.7 eV for C$_{60}$ film and 3.59 eV for Si substrate. The C$_{60}$ films on *n*-Si substrate have *p*-type conductivity and the weak splitting of the electroreflectance signal. The C$_{60}$ film surface on *n*-Si substrate is depleted on electrons, and the bands are bent upward (Fig. 5a). Splitting of the electroreflectance signal indicates appearance of weak quantum-size effect in the film.
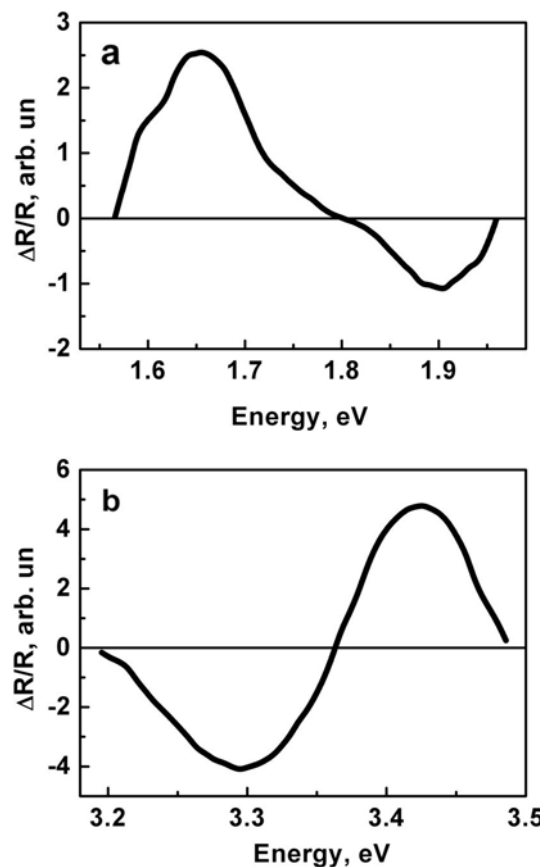

Fig. 5. Electroreflectance spectra of the film (a) and interface (b) in C$_{60}$/Si heterosystem.
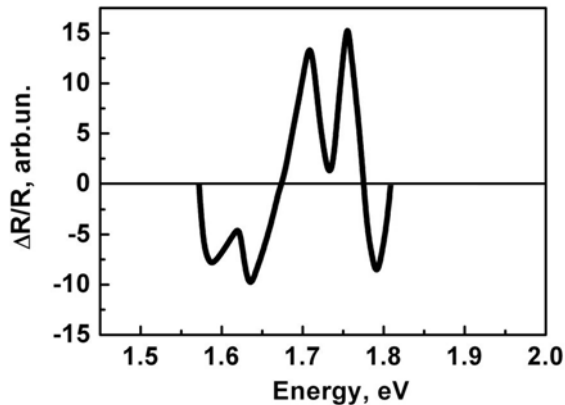
Fig. 6. Electroreflectance spectrum of C_{60} film on glass substrate.

The conductivity type of C_{60} film on the insulating glass substrate changed from *p* to *n*, and the signal splitting increased (Fig. 6). At the interface, the film C_{60} is enriched with electrons, and the bands are bent downwards. The enrichment of the films with electrons contributes to appearance of interfacial electronic states. It causes a phase change of the electroreflectance signal in the C_{60} film on cover glass substrate [27]. The doublet nature of the electroreflectance peaks in C_{60} films on insulating substrate can be explained by the influence of surface quantization of electron energy in the enriched layer of C_{60} film on the interface (Fig. 6). The presence of two transitions with the energies $E_g = 1.65$ eV and $E_g + e_1 = 1.7$ eV splintered peaks in the spectrum. The energy of quantum level $e_1 = 50$ meV. For E_0 transition in C_{60} film, the hole effective mass m_p is $1.3m_0$, the electron effective mass m_e is $1.5m_0$. The reduced effective mass m^* is $0.697m_0$. For 50 meV quantum level, the width of the quantum well is 2 nm.

4. Conclusion

To obtain heterosystems with quantum size effect, the complex of technological methods was used. It included the anisotropic chemical etching of *p*-Si and pnn^+-GaAs surface, different doses of neutron irradiation and thermal annealing of *p*-Si crystals with the concentration of dissolved oxygen $8 \cdot 10^{-17}$ cm^{-3}, the chemical sulfide passivation of *n*-GaAs (100) surface, C_{60} fullerene thermal deposition on *n*-Si and cover glass substrates. Electrolytic method of modulation spectroscopy of light electroreflectance is a most sensitive to the band structure of semiconductors. Therefore, it was used for investigations of the band structure and electronic parameters of the surface and interface of the heterosystems with the quantum size effect.

The quantum size effect in heterosystems prepared using different methods appears as a result of inversion of semiconductor conductivity type (from *p* to *n*) at the film-substrate interface. It leads to appearance of additional electronic transitions at the interface and splitting of the electroreflectance signal. Depending on the heterosystems manufacturing conditions and external influences, energies of the main electronic transition and quantized levels in a triangular potential well and the width of the well for each level, as well as broadening parameters of the spectra, the energy relaxation time of the excited light volume and quantized carriers were identified. Found in the patterned As_2O_3–pnn^+-GaAs heterosystems were 3 quantized levels with the energies 36, 64 and 93 meV for the widths of the quantum well 14.08, 21.11 and 25.7 nm, respectively.

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