In situ ellipsometric monitoring of composition and temperature of HgCdTe layers during their growth

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An ellipsometric technique has been developed which makes it possible to observe changes in the composition and temperature of cadmium–mercury–tellurium layers during their growing by molecular beam epitaxy. The technique was tested for diagnostics the variations of the composition and the temperature during layer growth in the mode of constant power of the substrate heater and with its sharp change. It was found that the drop in power and a following decrease in the growth temperature were also accompanied by a monotonic decrease in the composition. In the case of a constant heater power, a slight increase in the sample temperature was observed with an almost unchanged composition of the growing layer.

Keywords: ellipsometry, mercury–cadmium–telluride, *in situ* control, epitaxial growth, temperature.

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1. Introduction

The synthesis of epitaxial layers of the ternary $Hg_{1-x}Cd_xTe$ (CMT) solution by molecular beam epitaxy (MBE) requires continuous monitoring of the parameters of the growing structure and the growth conditions. The composition (i.e., fraction x of CdTe in the CdTe−HgTe solid solution) is one of the key parameters affecting the electrooptic properties of CMT. The spectral ellipsometry method [1–4] is used widely to determine the composition and monitor it in the process of growth. We have developed a technique that involves the use of *in situ* laser ellipsometry [5,6]. This technique relies on the dependence of optical constants of CMT on the composition [7,8]. Fortuitously, the wavelength of He−Ne lasers often used in laser ellipsometers is near the spectral maximum of this dependence. This makes ellipsometric characterization highly sensitive to the composition. A high-stability ellipsometer, which was designed specifically for these studies, was mounted on the CMT epitaxy module and provided a composition measurement accuracy of several thousandths of x [9].

Temperature is another parameter affecting the growth conditions and the crystalline quality of the produced layers. Even an insignificant deviation of temperature from the optimum value may give rise to imperfections of the crystal structure: formation of threading defects [10], disengagement of phases of different composition [11], development of roughness of the growth surface [6], and other unwanted effects. Temperature monitoring in the process of growth is a highly topical problem in CMT MBE technology and attracts considerable attention [12–18]. In addition to affecting the epitaxy process, uncontrolled temperature variation makes it difficult to determine the composition accurately using the ellipsometry method. Owing to the thermo-optic effect, such variations result in alteration of the optical constants, and these alterations may be interpreted erroneously as changes in the composition. It is very hard to measure the substrate temperature directly with a thermocouple. The difficulties arising in these measurements are engineering in nature: the processing units are designed so that a reliable thermal contact between a thermocouple junction and the substrate is hard to establish. Therefore, one is forced to use alternative techniques, such as band edge thermometry [12–14], Fourier transform infrared spectroscopy (FTIR) [16], and low-temperature pyrometry [17]. The equipment used in these methods applies certain requirements to the design of vacuum setups, which are not always acceptable.

Using spectral ellipsometry, we have partially solved the problem of temperature monitoring in [19,20]. The experimental results and methodological approaches detailed in these studies demonstrate the possibility of temperature measurement before the onset of CMT growth. The techniques proposed in [19,20] rely on the temperature dependences of ellipsometric spectra of the buffer CdTe layer. With the start of CMT epitaxy, the problem of interpretation of ellipsometric spectra gets much more complicated: in addition to being temperature-dependent, they become dependent on composition and on the continuously varying thickness of the growing CMT layer. At the same time, the issue of temperature monitoring is particularly acute at the initial growth stage, since the forming CMT layer alters the emissivity of the sample and violates the steady-state thermal balance conditions, thus possibly leading to temperature variations.

When the thickness exceeds the penetration depth of light, the contribution of the buffer CdTe layer to the analyzed reflected light vanishes, and the measured spectra become dependent only on temperature and the CMT composition. In contrast to the laser ellipsometry method, spectral examinations of such layers provide an opportunity to distinguish between the contributions of composition and temperature, since different regions of the spectrum have different sensitivities to these parameters.

The aim of the present study is to investigate the possibility of independent determination of the temperature and composition of growing CMT layers based on the results of ellipsometric monitoring and develop the a proper algorithm for interpreting the measured ellipsometric spectra. Experiments on the growth of CMT layers in different modes with continuous measurement of spectra of ellipsometric parameters in the process of growth were performed for this purpose. The set goal was achieved by comparing the measured spectra with the spectra of sensitivity of ellipsometric parameters to composition and temperature.

2. Experimental setup and experimental conditions

Si/ZnTe/CdTe/$Hg_{1-x}Cd_xTe$ heterostructures were synthesized in a multichamber setup with separate growth of buffer layers and CMT [21]. CMT layers were grown at a temperature close to 170°C. The substrate was heated by thermal radiation from a heater supplied with stabilized voltage, which was measured throughout the entire growth process. Indirect data on temperature were provided by a chromel/alumel thermocouple in thermal contact with the heater. The mercury vapor pressure and the temperature of sources of molecular Te and Cd beams were measured in the process of growth. The parameters of growth of all layers of the structure (growth rate, thickness, crystalline quality) were monitored with laser ellipsometers mounted on each module. The CMT epitaxy module was also fitted with a spectral ellipsometer designed for thermometry applications.

The spectral ellipsometer operated in the static photometric mode [22] had the following parameters: wavelength range $\lambda = 350-1000$ nm; spectral resolution — 3 nm; complete spectrum measurement time — 30 s. The stability of ellipsometric measurements is crucial for solving the problem at hand. The stability of parameter Ψ of the used instrument was $\pm 0.01°$ within 1 h of operation, and the corresponding value for Δ was 2 times higher. The sensitivity threshold for ellipsometric angles Ψ and Δ (the smallest detectable variation) was 0.002°. Scanning over the spectrum with a pitch of 1 nm was performed in the process of measurements.

Two test CMT layers of compositions with $x = 0.24$ (sample 1) and 0.34 (sample 2) were grown for measurements. The correspondence of the composition to the given x value was checked by measuring parameter Ψ with a laser ellipsometer [9]; a constant Ψ value was maintained by adjusting slightly the flow of Te. Sample 1 was grown under varying temperature conditions: after 32 min of growth, when the layer thickness became several times higher than the depth of short-wavelength light penetration, the voltage applied to the substrate heater was reduced by 17%. According to the thermocouple readings, this induced a monotonic temperature reduction at a rate that decreased slowly with time. Test sample 2 was grown under a constant heater voltage in steady-state conditions. In both cases, the growth of CMT commenced after stabilization of the substrate temperature at the stage of its pre-epitaxial thermal processing. Temperature values were monitored at this stage with the spectral ellipsometer in accordance with the procedure detailed in [20].

3. Sensitivity of ellipsometric spectra to temperature and composition

It follows from the analysis of ellipsometric spectra for test sample 1 that the ellipsometric parameters changed insignificantly after the heater was switched to a different mode: the variation does not exceed 0.1° for Ψ and 0.2° for Δ. In view of this, we applied the difference method of comparison of spectra measured immediately prior to the heater voltage adjustment (reference spectrum $\Psi_0(\lambda)$) and a certain time t after (spectrum $\Psi_t(\lambda)$). Figure 1 presents the difference spectra of parameter Ψ: $\delta\Psi(\lambda) = \Psi_t(\lambda) - \Psi_0(\lambda)$. The greatest variations of Ψ are seen near critical points E_1 and $E_1 + \Delta_1$ and are manifested in the form of well-marked peaks. In CMT of the studied composition, these critical points correspond to a wavelength of 550 and 420 nm. The amplitudes of peaks increase monotonically with time

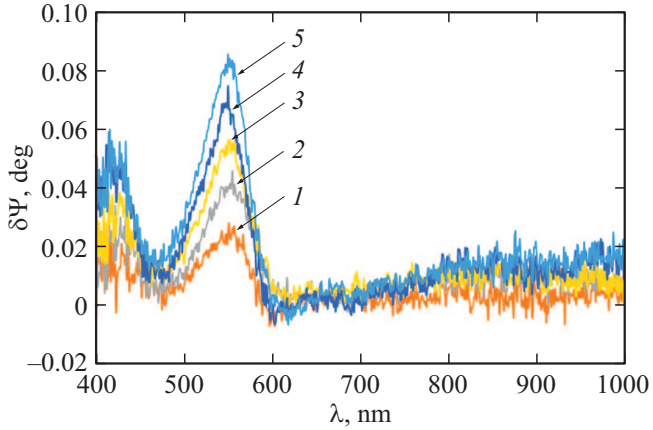

Figure 1. Difference spectra of parameter Ψ measured after the voltage applied to the substrate heater was reduced. Reference spectrum $\Psi_0(\lambda)$ was measured immediately prior to the voltage drop. The time of measurement of the studied spectra determined relative to the moment of voltage drop, s: *1* — 800, *2* — 1200, *3* — 1600, *4* — 2000, *5* — 3350. (A color version of the figure is provided in the online version of the paper).

between measurements of the reference spectrum and the studied one and tend to saturation.

All this is indicative of a relation between the presented results and the temperature variation. In addition to temperature, difference spectra depend on the composition variation that may also occur in the process of growth. In order to distinguish between the contributions of these components to $\delta\Psi$, one needs to determine the functions of spectral sensitivity of parameter Ψ to variations of temperature, $\alpha_T(\lambda, x)$, and composition, $\alpha_x(\lambda, x)$. The parametric model of optical CMT constants presented in [23] allows one to calculate easily the sensitivity to composition. This model was obtained by interpolating the data of *in situ* experiments performed in the process of growth of a series of samples of various compositions. It provides an opportunity to calculate analytically the spectra of ellipsometric parameters $\Psi(\lambda, x)$ and $\Delta(\lambda, x)$ for any composition with x varying from 0.2 to 0.4. The sensitivity function is defined as derivative $\alpha_x(\lambda, x) = d\Psi(\lambda, x)/dx$.

It turned out to be much harder to determine the sensitivity of ellipsometric spectra of Ψ and Δ to temperature. Two series of temperature experiments were performed for this purpose: *ex situ* measurements of ellipsometric spectra near room temperature under thermal cycling and *in situ* measurements in the process of passive cooling of the grown samples. The key issue complicating the ellipsometric examination of the temperature dependence of optical CMT constants is the instability of the surface after blockage of the molecular tellurium flow.

Figure 2 shows the kinetics of variation of ellipsometric parameters at wavelength $\lambda = 500$ nm observed after termination of the growth process (blockage of the molecular Te flow and simultaneous disconnection of voltage from the substrate heater). The mercury vapor pressure remained unchanged in these measurements. What stands out immediately is the sudden (almost jump-like) Ψ and Δ drop within the first seconds after growth termination.

This drop is not associated with variations of the substrate temperature, since the thermocouple readings vary more smoothly. Similar jumps of ellipsometric parameters occurring after blockage of the Te flow were examined in [24]. It was found that they are related to a complex of different surface processes: desorption of the adsorbed Te layer, formation of the CdTe layer, and diffusion of mercury atoms from the near-surface region to the surface.

Judging by the Δ variation rate relaxation, surface processes remain active for several minutes; following their decay, the ellipsometric parameters decrease smoothly, and their reduction is associated primarily with the temperature variation. The same estimate of the duration of surface processes follows from the data presented in [24]. This is the reason why the initial stage of the cooling process is not suitable for the determination of the temperature sensitivity of parameters Ψ and Δ. In order to exclude (or reduce considerably) the influence of surface effects on the determination of $\alpha_T(\lambda, x)$, the reference spectrum was measured 10 min after the onset of cooling, and the

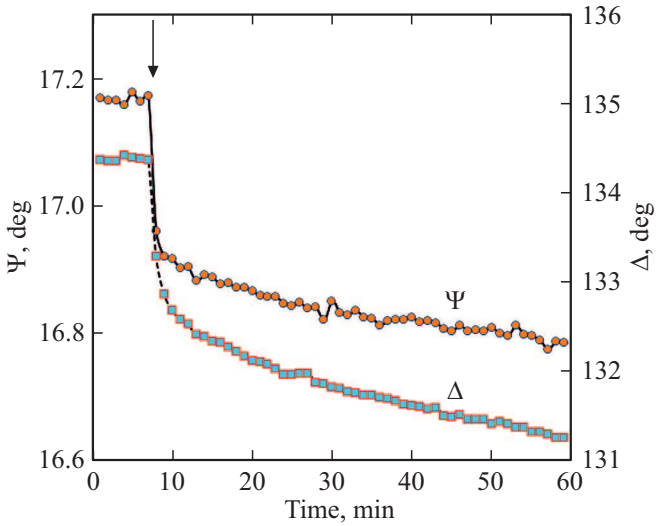

Figure 2. Kinetics of variation of ellipsometric parameters Ψ and Δ of the grown CMT sample in the process of its cooling in a vacuum chamber. The moment of termination of growth is indicated by a vertical arrow.

second spectrum was measured after another 50 min had passed. According to the estimates based on the shift of the $\Psi(\lambda)$ spectrum maximum near critical point E_1, the temperature variation within this time interval was $\sim 40^\circ$C. Figure 3 shows difference spectra $\delta\Psi$ measured this way for three samples of different composition: $x = 0.160$, 0.235, and 0.327.

Similar *ex situ* measurements were performed for the same samples subjected to repeated heating by 40°C relative to room temperature. The results of these measurements are also shown in Fig. 3. A small-sized heater [19] mounted on the ellipsometer sample stage was used in *ex situ* experiments. Temperature values were monitored using a chromel/alumel thermocouple. Repeated cycles of sample heating and cooling demonstrated the reproducibility of spectra upon relaxation to room temperature. This suggests that the temperature rise induces no irreversible changes on the surface. The comparison of curves obtained in these experiments with the data of *in situ* measurements reveals a shift along the wavelength axis. This shift is to be expected and emerges due the difference in temperature ranges (20–60°C for *ex situ* measurements and around 150°C for *in situ* measurements).

The sensitivity of ellipsometric parameters to temperature for an arbitrary value of composition parameter x was modeled based on the presented results. The data of *ex situ* measurements were deemed more reliable and were thus taken as the basis. The dependence of the sensitivity on composition at a constant wavelength was determined by quadratic approximation of the values of $\delta\Psi_j$ $(j = 1, 2, 3)$ measured for the three samples used. The $\delta\Psi$ functions obtained this way were then corrected for the growth temperature by shifting them toward higher wavelengths. Comparing the positions of extrema of *ex situ*

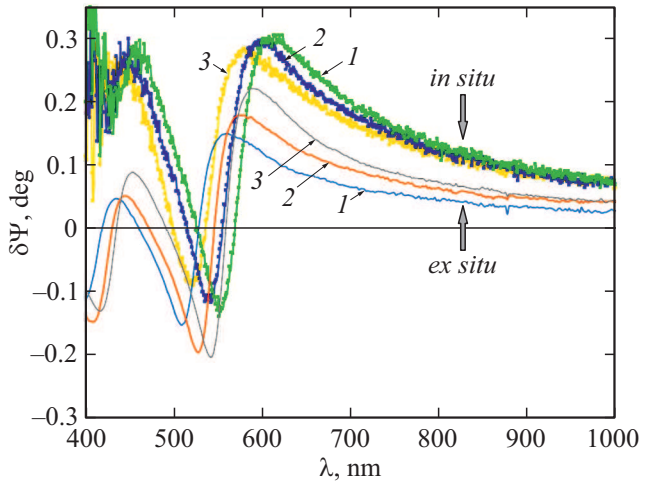

Figure 3. Difference spectra $\delta\Psi(\lambda)$ for samples of different compositions measured *in situ* and *ex situ*. $x = 0.160$ (*1*), 0.235 (*2*), 0.327 (*3*).

and *in situ* curves in Fig. 3, one finds that the shift magnitude is nonuniform and increases appreciably with wavelength. This was also taken into account in the model. The resulting $\delta\Psi(\lambda, x)$ dependence normalized to 1°C is a parametric model for calculation of the temperature sensitivity of parameter Ψ: $\alpha_T(\lambda, x) = \delta\Psi(\lambda, x)/40°\text{C}$. It is based on the experimentally measured $\delta\Psi(\lambda)$ sets for three samples of different compositions and provides an opportunity to calculate $\alpha_T(\lambda, x)$ analytically for CMT of an arbitrary composition. A parametric model for the temperature dependence of Δ was obtained in a similar fashion.

4. Determination of temperature and composition variations in the process of growth

Figure 4 presents the sensitivities of parameter Ψ to composition (normalized to $\delta x = 0.001$) and temperature (normalized to 1°C) calculated for the composition with $x = 0.24$ using the corresponding parametric models. The curves are symmetric in shape, but have a significant difference. The $\alpha_x(\lambda)$ curve is of constant sign, while $\alpha_T(\lambda)$ is sign-variable: the sensitivity to temperature is negative around critical points E_1 and $E_1 + \Delta_1$ and positive in the remaining region of the spectrum; therefore, the considered functions are linearly independent: composition and temperature cannot cancel out the $\Psi(\lambda)$ spectrum variations. This is crucial in the context of the problem to solve and indicates the possibility to distinguish between the contributions of temperature and composition to the experimentally measured spectra of parameter Ψ. In mathematics, a problem of this kind is called a well-posed one.

Let us formulate it in the following way. Suppose the $\Psi_0(\lambda)$ reference spectrum was measured at a certain point in the process of growth of a CMT layer. The values of the composition parameter on the surface and temperature at this point are x_0 and T_0. The second $\Psi_t(\lambda)$ spectrum measured at a certain later time t corresponds to different values of temperature T and composition x, which are close to the initial ones. The task is to determine composition $\delta x = x - x_0$ and temperature $\delta T = T - T_0$ variations using the $\delta\Psi(\lambda) = \Psi(\lambda) - \Psi_0(\lambda)$ difference spectrum. This problem is solved by minimizing functional

$$S_\Psi(\delta x, \delta T) = \sum_\lambda \left[\delta\Psi_m(\lambda) - \delta\Psi_c(\lambda, \delta x, \Delta T)\right]^2, \qquad (1)$$

where $\delta\Psi_m$ and $\delta\Psi_c$ are the measured and calculated values of the difference spectra of parameter Ψ. Owing to the smallness of the sought-for quantities, the calculated spectrum may be determined within the linear approximation in δx and δT. Then,

$$\delta\Psi_c = \frac{\partial\Psi}{\partial T}\delta T + \frac{\partial\Psi}{\partial x}\delta x. \qquad (2)$$

The partial derivatives with respect to temperature and composition in Eq. (2) are the functions of sensitivity of parameter Ψ shown in Fig. 4.

Figure 5 shows one of the $\delta\Psi$ difference spectra of test sample 1 (measured 1600 s after the measurement of the reference spectrum) and the calculated curve plotted in accordance with formula (2) for the optimum values of $\delta T = -8°\text{C}$ and $\delta x = -0.0035$ that minimize functional (1). The calculated curve agrees fairly closely with the experimental data. This example indicates that the variation of sample temperature, which is induced by the drop in voltage applied to the heater, is accompanied by a change in

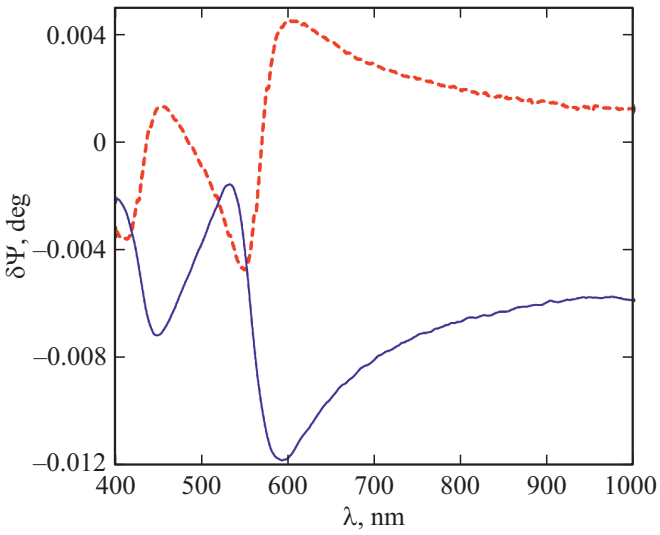

Figure 4. Sensitivity of parameter Ψ to composition $\alpha_x(\lambda)$ (solid curve) and temperature $\alpha_T(\lambda)$ (dashed curve) for CMT composition with $x = 0.24$. The curves are normalized to 0.001 of x variation ($\alpha_x(\lambda)$) and to 1°C ($\alpha_T(\lambda)$).

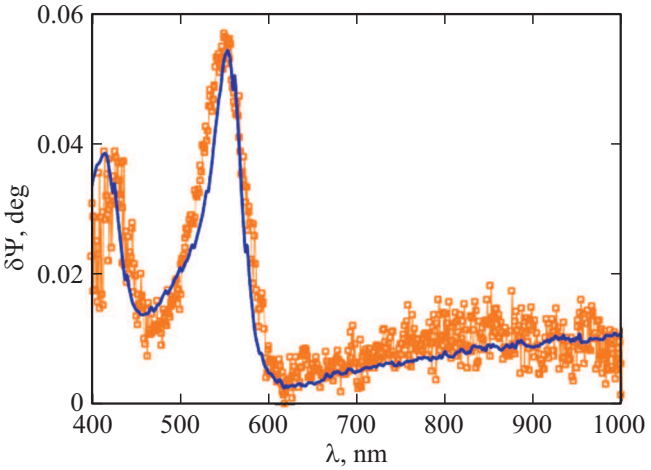

Figure 5. Measured $\delta\Psi$ spectrum (dots) and calculated curve for optimized values of $\delta T = -8^\circ$C and $\delta x = -0.0035$ (solid curve).

composition of the growing CMT layer, and changes in both parameters may be monitored using ellipsometric spectra. It becomes evident that both temperature and composition should be varied in the process of optimization if one remembers that the measured $\delta\Psi(\lambda)$ spectrum remains positive throughout the entire spectral range and cannot be characterized by sign-variable function $\alpha_T(\lambda)$ alone.

The fitting of all difference spectra for rest sample 1 was performed this way; the dependences of temperature and composition variations on the growth time obtained as a result are shown in Fig. 6. The values of $\delta x, \delta T$ decrease with increasing time and tend to saturation. The reduction of composition parameter x is likely to be attributable to the variation of tellurium flow, which grew continuously to maintain a constant value of parameter Ψ. However, it is also possible that temperature affects directly the growth kinetics. The concentration of mercury on the surface increases at lower temperatures [15], and this may result in a shift of the kinetic equilibrium of reactions toward lower values of the composition parameter.

The obtained temperature variations agree qualitatively with the readings of the thermocouple measuring the heater temperature. In the course of the experiment, the thermocouple output voltage dropped by ~ 1 mV, which corresponds to 25°C. The time constant for the thermocouple turns out to be smaller than the one for the sample.

The analysis of difference spectra of parameter Δ yields similar results. However, it should be noted that co-minimization of functional $S_\Psi(\delta x, \delta T)$ and functional $S_\Delta(\delta x, \delta T)$, which is constructed in a similar fashion, does not always yield the same solutions for δx and δT. This is attributable to the fact that parameter Δ is much more sensitive to the surface microrelief. Therefore, the use of $\Psi(\lambda)$ spectra in the analysis of experimental data is preferable. The second possible cause of divergence of results is the slight drift of the incidence angle within the period of time between the measurements of the reference spectrum and the studied one. If angle variation $\delta\varphi$ is included into functional (1) as an additional fitting parameter, its convergence does indeed improve slightly, but the determined value of $\delta\varphi$ in most cases remains within $\pm 0.01^\circ$.

Thus, it was established that the composition of the growing CMT layer varies appreciably ($\delta x \approx 0.006$) in the process of growth of the studied sample. Note that the Te flow increased slightly within the examined time interval. The flow was adjusted in the process of growth by the operator to maintain a constant value of parameter Ψ measured by the laser ellipsometer. According to the sensitivity functions in Fig. 4, a temperature variation of 1°C exerts the same effect on parameter Ψ at wavelength

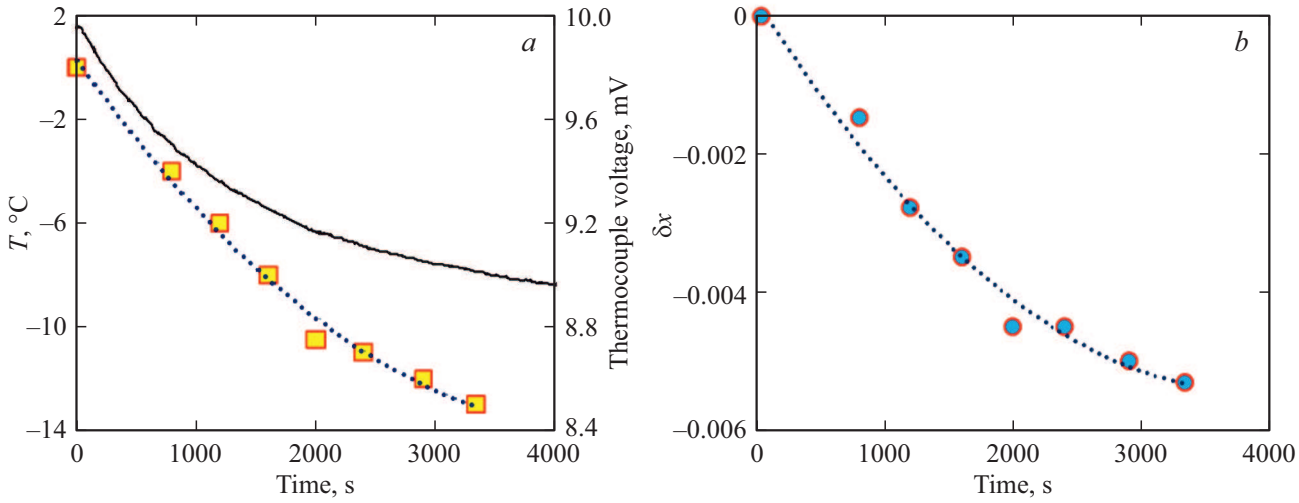

Figure 6. Variations of temperature (a) and composition (b) in the process of growth of a test sample after the heater voltage reduction. Dots represent the data obtained by minimizing functional (1); dotted curves are the trend lines for experimental data. The solid curve in a corresponds to thermocouple readings.

$\lambda = 632.8$ nm as a composition parameter (x) variation of 0.0004. Therefore, Ψ may be maintained at a fixed level if temperature and composition are varied simultaneously and in a coordinated fashion, and parameter Ψ may be used as a criterion for precision composition monitoring only if the growth temperature is stable.

In view of this, one should note the wavelength at which the $\alpha_T(\lambda)$ sensitivity curve crosses the horizontal axis and vanishes (this wavelength for the composition with $x = 0.24$ is $\lambda = 568$ nm). At this wavelength, parameter Ψ is insensitive to small temperature variations, while the $\alpha_x(\lambda)$ composition sensitivity remains high. Therefore, the ambiguity of interpretation of experimental data may be resolved if one monitors the composition using the value of Ψ at this wavelength.

With the heater power being constant, the temperature of the sample should stabilize with the passage of time provided that its emissivity and the surrounding background remain unchanged. With the onset of CMT growth and the subsequent formation of the variband layer, the emissivity and absorbance of the sample change, thus altering the thermal balance. In the steady-state scenario, the sample temperature is indeed determined based on the condition of equality of absorbed and emitted radiation fluxes. If the heat flux from the surrounding background is neglected, only the heater radiation, which is represented fairly accurately by blackbody radiation, reaches the sample. In contrast to the heater, the sample features selective emissivity that depends on the CMT composition and the thickness of CMT and the buffer CdTe layer. Since the heater temperature is higher than the sample temperature, the Planck distribution of thermal flux incident on the sample is shifted toward shorter wavelengths relative to the distribution of the sample radiation. When composition parameter x decreases, the non-transparency interval in the infrared range broadens, and both the absorbed and the radiated fluxes increase. However, owing to the difference in temperature between the heater and the sample, the radiated flux increases more significantly. This should lead to a reduction in equilibrium temperature of the sample.

The second cause of thermal imbalance is related to the process procedures that alter the background thermal flux. Specifically, the CMT grows starts after the baffle, which screens the sample from heated molecular sources, is opened; the resulting additional incident radiation flux may induce heating of the sample.

The calculation of the resulting temperature change caused by the above-mentioned mechanisms is a rather complicated problem with a great number of unknown parameters. We obtained the needed data experimentally by analyzing the difference spectra of test sample 2 grown at a stabilized heater temperature. Just as the spectra of sample 1, the difference spectra for sample 2 have a salient feature around critical point E_1 that is indicative of a change in temperature. However, in contrast to the spectra of sample 1 where this feature was the maximum of function $\delta\Psi(\lambda)$, the data for sample 2 reveal the minimum of this

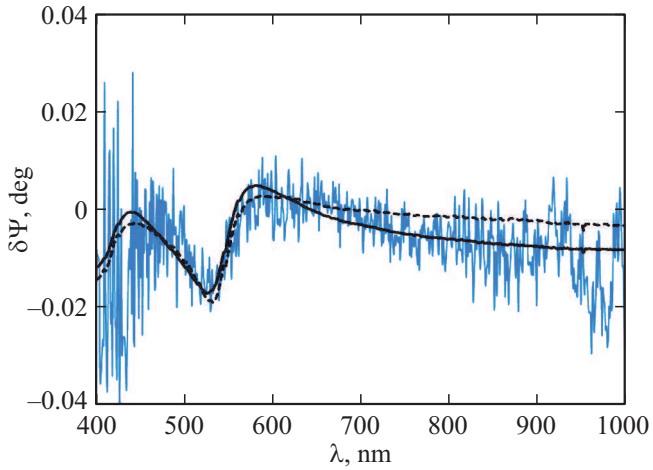

Figure 7. Difference spectrum of test sample 2 (noisy curve) and the result of its simulation with adjustment of temperature, composition, and incidence angle φ: dashed curve — $\delta T = 4°\text{C}$, $\delta x = 0.001$, $\delta\varphi = 0$; solid curve — $\delta T = 2.5°\text{C}$, $\delta x = -0.0006$, $\delta\varphi = 0.01$.

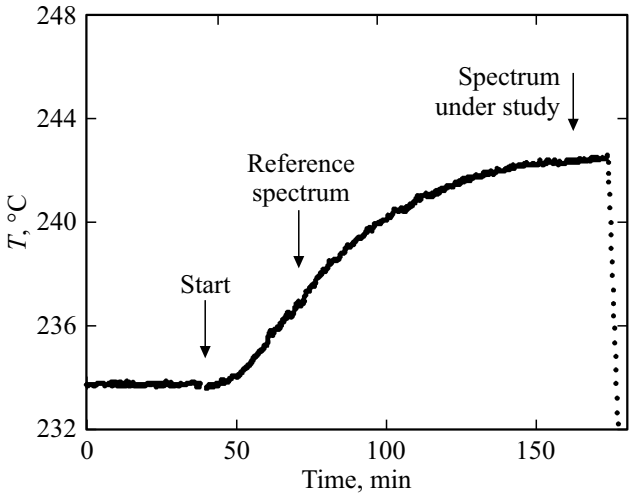

Figure 8. Variation of the heater temperature in the process of CMT layer growth. Arrows denote the moments of actuation of sources (onset of growth) and measurement of the reference spectrum and the studied one.

function that becomes more and more apparent with the passage of time and corresponds to a temperature increase.

Figure 7 shows one of these difference spectra: reference spectrum $\Psi_0(\lambda)$ was measured 30 min after the onset of growth, while the studied $\Psi_t(\lambda)$ spectrum was measured 1.5 h later, at the end of growth. Simulations were performed for two scenarios: with variation of temperature and composition at a constant incidence angle (dashed curve) and with variation of all three parameters: temperature, composition, and incidence angle (solid curve). In the latter case, the data agree more closely with the experimental ones. It should be noted that the amplitude of variation of the difference spectrum in this experiment is just several

times higher than the experimental scatter. Therefore, the obtained composition values are within the error of method. However, a 2−4°C temperature increment follows directly from the shape of the measured spectrum and agrees with the heater temperature dynamics measured by the thermocouple (see Fig. 8).

This slight increase in temperature is apparently attributable to the thermal radiation of molecular sources that penetrates the sample due to its transparency in the infrared range. Thus, the proposed ellipsometric technique allows one to detect small temperature variations in the process of growth.

5. Conclusion

The issue of spectral ellipsometric monitoring of temperature and composition in the process of growth of CMT layers by MBE was considered. A numerical technique for quantitative processing of experimental spectra was developed based on the measured temperature dependence of ellipsometric CMT spectra and the dependence of these spectra on composition, which has been established earlier. This technique allowed us to distinguish between the contributions of temperature and composition and measure independently the variations of these parameters in the process of growth. Experiments with reference samples grown for the purpose demonstrated that the sensitivity threshold of the method in question is on the order of one degree Celsius for temperature variations and on the order of thousandths of parameter x for composition variations. The developed technique was used to demonstrate that the sample temperature increases slightly in the process of growth of a CMT layer with a stabilized substrate heater temperature. This temperature increment is attributable to the influence of the thermal flux of molecular sources.

Conflict of interest

The authors declare that they have no conflict of interest.

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