

Epitaxial growth of highly stressed InGaAs/InAlAs layers on InP substrates by molecular-beam epitaxy

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In this paper we present the study of the features of epitaxial growth highly stressed superlattices based on highly stressed thin InGaAs/InAlAs layers on InP substrates by the molecular beam epitaxy. It was shown that the growth rates of the InGaAs and InAlAs bulk layers lattice-matched to InP substrates do not allow us to precisely determine the growth rates of thin highly stressed $\text{In}_{0.36}\text{Al}_{0.64}\text{As}/\text{In}_{0.67}\text{Ga}_{0.33}\text{As}$ strain compensated superlattices and the error is about 10 percent. The effect is related to the difference in the growth temperatures of InGaAs and InAlAs bulk layers, which affects the intensity of indium evaporation from the growth surface.

Keywords: molecular-beam epitaxy, superlattice, quantum-cascade lasers.

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Introduction

Epitaxial growth of InGaAs and InAlAs type layers on InP substrate requires strict matching of the ratio of indium, aluminum, gallium and arsenic fluxes to ensure consistency in the lattice constant of the resulting materials, which is achieved only for the case of layers $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ and $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ respectively. With other compositions, InGaAs and InAlAs layers turn out to be highly stressed due to the difference in the lattice constant with InP substrate, and their volumetric growth is impossible [1]. Semiconductor thin layers of InGaAs and InAlAs (about 10 nm), grown by molecular beam epitaxy (MBE) are widely used in epitaxial heterostructures for the manufacture of optoelectronic devices [2–6], including heterostructures of quantum cascade lasers (QCLs) of middle infrared range [6–12]. To fabricate such QCL heterostructures the heteropairs $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}/\text{In}_{0.52}\text{Al}_{0.48}\text{As}$, lattice-matched with InP substrate, are used. However, as was shown in the paper [13], matched InGaAs/InAlAs heteropairs limit the efficiency of QCL in the spectral range 4.4–4.8 μm due to the thermal release of charge carriers from the upper level of size quantization into the continuous spectrum, caused by insufficient discontinuity of the conduction band at the heterointerface InGaAs/InAlAs. To solve this problem, thin, highly stressed layers of InGaAs/InAlAs are used [14,15]. The main difficulty in the manufacture of heterostructures with thin highly stressed InGaAs/InAlAs semiconductor layers by MBE is the need to simultaneously fulfill the conditions for exact correspondence of the indium mole fraction in the ternary solution and the layer thickness to the specified values during a long-term growth process to

eliminate the occurrence of structural defects and surface roughness [16]. Calibration of growth parameters, such as material flow rates, temperature of material sources, and substrate temperature during epitaxial growth, is an integral part of preparation for the growth process of the final heterostructure. However, the calibration of these parameters in the general case is carried out using the epitaxial growth of bulk layers with a thickness of several hundred nanometers, which makes it possible to make accurate calculations and measurements using the methods of defectometry, photoluminescence and X-ray diffraction analysis [17]. As practice shows, when fully calibrating the growth parameters using heterostructures of bulk InGaAs and InAlAs layers, during the epitaxial growth of thin, highly stressed layers of these solid solutions, a discrepancy between the mole fraction and thickness arises, and, consequently, there is need in further adjustment of the parameters.

This paper presents the results of a study of the features of epitaxial growth using the molecular beam epitaxy method of superlattices (SL) based on highly stressed thin InGaAs/InAlAs layers on InP substrate.

1. Experimental results and discussion

To carry out epitaxial growth experiments a Riber49 MBE setup was used. The Riber49 setup was equipped with three types of sources — ABN160 (Si source), ABN700 (Al source) and ABI1000 (Ga, In source). The ABN700 source has two heating elements. Two heaters are used to precisely maintain the temperature of the main

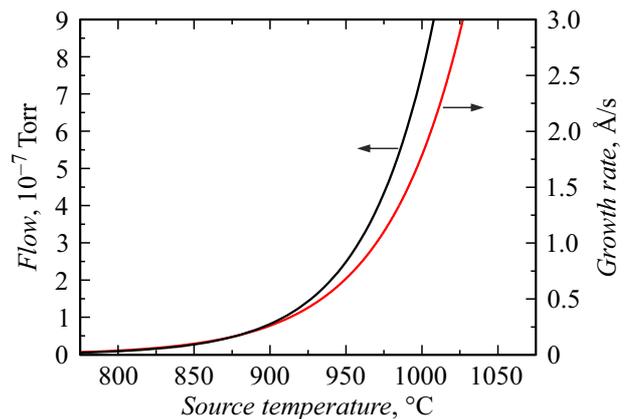
Table 1. Results of preliminary calibration of growth rates

Layer	GaAs			AlAs			InAs		
	$V, \text{Å/s}$	$F, \cdot 10^{-7} \text{Torr}$	$T, ^\circ\text{C}$	$V, \text{Å/s}$	$F, \cdot 10^{-7} \text{Torr}$	$T, ^\circ\text{C}$	$V, \text{Å/s}$	$F, \cdot 10^{-7} \text{Torr}$	$T, ^\circ\text{C}$
$\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$	0.88	3.90	904	—	—	—	1.00	7.80	823
$\text{In}_{0.52}\text{Al}_{0.48}\text{As}$	—	—	—	0.92	1.50	1094	1.00	7.80	823

crucible volume and the crucible aperture. The crucible itself has a conical shape. The ABI1000 source has a similar design, but unlike the ABN700 it is equipped with a cylindrical crucible consisting of three parts: the crucible itself, a conical neck and a neck ring covering the fasteners. All parts of the crucible are made of pyrolytic boron nitride. The use of such a crucible makes it possible to significantly increase the stability of element flows during long-term epitaxial growth [18,19].

To study the growth modes, undoped semi-insulating InP substrates of „epi-ready“ orientation quality (100) were used. The molybdenum substrate holder was loaded into the Riber49 loading module and then transferred to the pre-annealing module. In the pre-annealing module the substrate was heated for half an hour at a temperature of 300°C to desorb atmospheric gases from its surface. After this, the substrate holder was transferred to the growth chamber of the Riber49 setup. The surface oxide was desorbed by heating the substrate to 540°C for 5–10 min [20] in an arsenic flow with overpressure $2 \cdot 10^{-8} \text{Torr}$ (vapor pressure were measured with an ion sensor). Upon reaching the given temperature, a change in the RHEED pattern was observed, associated with a change in the surface reconstruction. The striped (4×1) RHEED reconstruction of the surface indicated an atomically smooth and clean surface of the substrate, without the oxide presence on its surface. During the experiments, it was discovered that a characteristic surface reconstruction is observed only if semi-insulating InP substrates are used. When using heavily doped InP ($N \sim (1-3) \cdot 10^{18} \text{cm}^{-3}$) substrates, the fast electron diffraction pattern did not change significantly in the substrate temperature range from 300 to 540°C . Substrate temperatures were evaluated by an Iron optical pyrometer, and high-energy electron diffraction equipment was used to observe the RHEED pattern. Epitaxial growth began after oxide complete removal and setting the growth temperature of the substrate required for this experiment.

For precision growth of quantum-sized layers of a given chemical composition, it is necessary to carry out preliminary calibrations of the fluxes of metals Ga, Al and In using a standard flux meter (ionization sensor). The results of flux measurements of Group III elements make it possible to estimate the growth rates of the corresponding binary compounds using the available calibration dependencies. The appearance of such a calibration curve for Ga05 source is presented in Fig. 1. At the chosen growth rate of the InAs compound (in our case 0.1nm/s), the

**Figure 1.** Flux and growth rate vs. temperature for Ga05 source.

growth rate of GaAs should be 0.089nm/s to obtain layer $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ with lattice parameter-matched to InP substrate. Knowing the growth rate of GaAs, it is possible to determine the flux of Ga05 element, which shall be obtained by changing the temperature of the corresponding source.

Immediately before the growth of test heterostructures, a preliminary calibration of the growth rates was carried out according to the flux meter readings. The source temperatures were adjusted to obtain the required growth rates of binary compounds. Results of preliminary calibration are given in Table 1. The Table uses the following designations for parameters: V — growth rate, F — flux, T — temperature of the lower heater of the source according to thermocouple readings. For InGaAs, InAlAs layers the required growth rates and the corresponding fluxes obtained by proportional increasing or decreasing the fluxes of materials are given. The Table also shows the source temperatures corresponding to the obtained fluxes.

Test heterostructures S1 and S2 were grown, nominally consisting of a layer of $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ and $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$, respectively. The thickness of each layer was 400nm . The thicknesses of the test layers were determined by measuring and analyzing the oscillation period on the X-ray diffraction curves. The measurements were carried out on PANalytical x'PertPro setup with proprietary software in the geometry $\Omega-2\Theta$. The composition of the layers was determined by modeling the experimental rocking curve using standard software. The measurement results and approximation data of the experimental curves are presented in Fig. 2 for

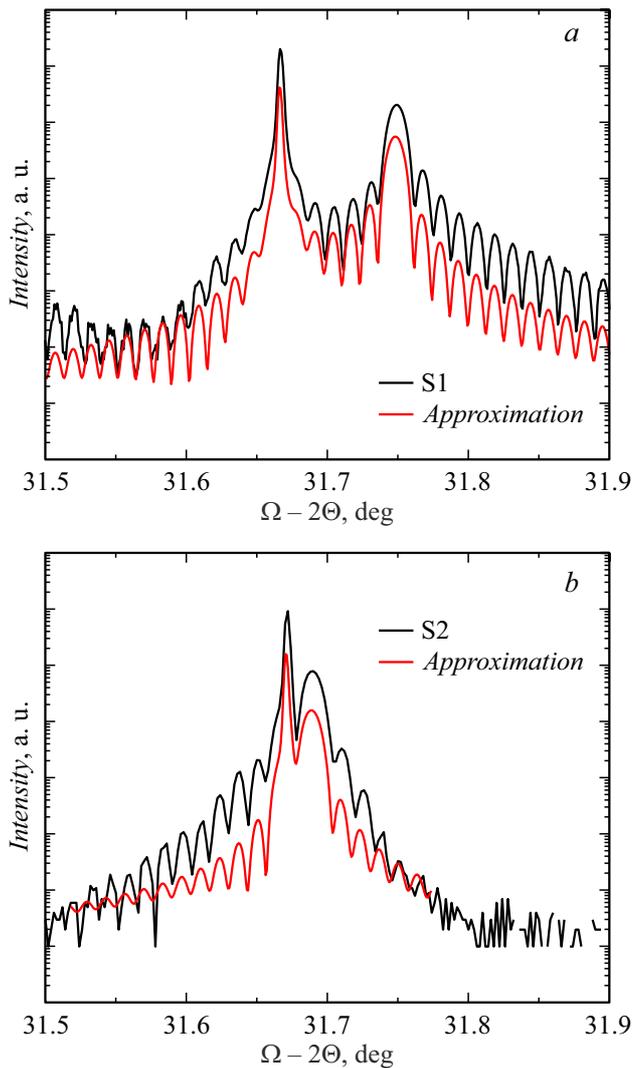


Figure 2. X-ray diffraction curve and the results of its approximation of samples: *a* — S1, *b* — S2.

Table 2. Results of X-ray diffraction analysis

Sample	Parameter	As per growth design	Result approximation
S1	Thickness, nm	400	407
	Composition	In _{0.53} Ga _{0.47} As	In _{0.513} Ga _{0.487} As
S2	Thickness, nm	400	390
	Composition	In _{0.52} Al _{0.48} As	In _{0.517} Al _{0.483} As

structures S1 and S2, respectively. The thicknesses and composition of the test layers are given in Table 2.

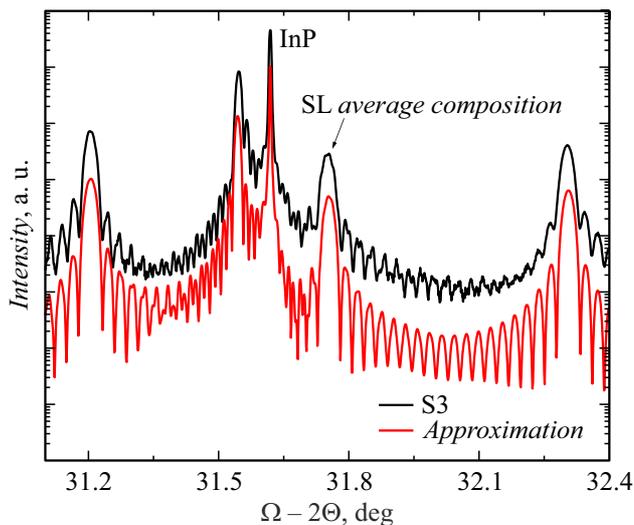
After determining the growth rates of all binary compounds, a heterostructure S3 was grown, consisting of 20 periods of the superlattice (SL) of stressed layers In_{0.36}Al_{0.64}As/In_{0.67}Ga_{0.33}As with thickness of 5 nm each. Equal thicknesses of the superlattice layers en-

sured that the average composition of SL was consistent with the InP substrate. The growth of such SL allows one to calibrate the growth rates of layers In_{0.36}Al_{0.64}As and In_{0.67}Ga_{0.33}As that are not matched by lattice constant with the substrate InP and cannot be grown separately by analogy with samples S1 and S2. To determine the exact composition and thickness of the layers of test SR In_{0.36}Al_{0.64}As/In_{0.67}Ga_{0.33}As, the X-ray diffraction method was also used. Periodic oscillations in the X-ray diffraction curve correspond to the periodic structure of the superlattice (Fig. 3).

Data on the thickness of the SL layers and their composition (Table 4) were determined by approximating of the measured X-ray diffraction curve. The measurements were carried out at position (0,0). The results obtained indicate that both layers in the structure turned out to be less than the nominal value in terms of mole fraction InAs. The average composition of SL S3 is far from the substrate peak. The composition of InGaAs layers turned out to be by 2.9%, and the composition of InAlAs layers by 6.2% were less than required. The deviation in thickness was also downward: 3.3 and 8.7% for the InGaAs and InAlAs layers, respectively. It is noteworthy that the data obtained indicated a difference in the growth rate of InAs in the InGaAs and InAlAs layers. Based on the results obtained, the difference in growth rates was 10%. We also observed this effect during the production of less stressed layers, in which the growth rates differed by 8%. For matched layers, the difference in growth rates ranged from 2 to 4% depending on the temperature difference between Al and Ga sources. It was suggested that the effect is associated with the difference in the growth temperature of InGaAs and InAlAs layers, which affects the intensity of indium evaporation from the growth surface. Additional heating of the growth surface can occur from Ga and Al sources when the shutter is opened due to thermal radiation of the molten material. Since the sources are heated to different temperatures, the influence of radiation during the growth of different layers is different. The difference in the temperature of Ga and Al sources in the previous case was 80°C. In our case, the Ga source at the chosen growth rates was heated to temperature of about 920°C, while the Al source was heated to 1140°C, i.e. the difference increased by 40°C (up to 120°C), which led to a stronger effect. To evaluate the effect of the difference in substrate temperatures on the growth rate of InAs during the growth of InGaAs and InAlAs layers, a test heterostructure S4 was fabricated, consisting of two InGaAs layers lattice-matched with InP substrate, and formed at the same temperatures of effusion sources, but differing by 20° Substrate temperatures (500 520°C). According to the X-ray diffraction analysis of the resulting heterostructure S4 (Fig. 4, *a*), the mole fraction of InAs in InGaAs layer grown at temperature of 500°C was 53.1%. For InGaAs layer formed at an elevated substrate temperature (520°C), the mole fraction of InAs decreased to 52.0%, which was equivalent to a decrease in the growth rate

Table 3. Results of growth rates correction

Sample	GaAs			AlAs			InAs		
	$V, \text{ \AA/s}$	$F, \cdot 10^{-7} \text{ Torr}$	$T, \text{ }^\circ\text{C}$	$V, \text{ \AA/s}$	$F, \cdot 10^{-7} \text{ Torr}$	$T, \text{ }^\circ\text{C}$	$V, \text{ \AA/s}$	$F, \cdot 10^{-7} \text{ Torr}$	$T, \text{ }^\circ\text{C}$
S1	0.93	3.90	904	—	—	—	0.98	7.80	823
Corrected values	0.89	3.73	902	—	—	—	1.00	7.96	824
S2	—	—	—	0.91	1.50	1094	0.98	7.80	823
Corrected values	—	—	—	0.92	1.52	1094.5	1.00	7.96	824

**Figure 3.** X-ray diffraction curve of test sample S3 with SL composition $\text{In}_{0.36}\text{Al}_{0.64}\text{As}/\text{In}_{0.67}\text{Ga}_{0.33}\text{As}$.

of InAs by 4.4%. Thus, from the experiment performed it followed that in a linear approximation the change in the growth rate of InAs can be estimated as 0.22% for each degree of change in the substrate temperature. Note that the data obtained contradict the established ideas, according to which in MBE the desorption of indium atoms from the growth surface becomes significant for substrate temperatures above 550–560°C. However, it was previously shown experimentally that a change in the substrate temperature in the range 500–550°C also affects the growth rate of InGaAs [21,22]. Thus, the data presented in the paper [21] indicate a difference of up to 10% in the growth rates of InGaAs layers grown at substrate temperature of 510 and 550°C.

To analyze the actual temperature changes during our heterostructures growth, we measured the substrate temperature using a pyrometer directly during the growth of highly stressed InGaAs and InAlAs layers. The pyrometer was calibrated by the temperature at which natural oxide began to flow from the surface of the GaAs substrate (590°C) and before the start of the growth process its readings corresponded to 500°C (Fig. 4, b). Measurements of the substrate temperature after the start of growth showed

Table 4. Results of modeling the experimental rocking curves of structures S3 and S5

Sample	Mole fraction of InAs, %		Thickness, nm	
	InGaAs	InAlAs	InGaAs	InAlAs
S3	65.1	33.9	4.84	4.60
S5	67.6	37.1	5.05	5.07

that the pyrometer readings are strongly influenced by illumination from effusion sources when their shutter is opened: a sharp jump in temperature occurs. Then a stage of gradual temperature rise is observed, which we associate with heating of the substrate surface by radiation from the sources. Moreover, the change in the substrate temperature at the stage of gradual rise is greater for the hottest aluminum source, for which it reached 31°C, and decreases during the growth process, which is most likely due to the gradual heating of the substrate and the weakening of effect of thermal radiation from the sources [23]. According to the measurement results, the final heating of the substrate during the experiment was approximately 50°C: from 500°C before the start of growth to 550°C at the end of growth with the closed shutters of the metal sources. The obtained result experimentally confirms our assumption about the additional heating presence of the growth surface when the shutters of effusion sources of metals are opened due to thermal radiation of the molten material and may be the reason for the difference in the growth rates of InAs in InGaAs and InAlAs layers.

It is shown that the deviation in the thickness and composition of InGaAs test layer is 2 and 3%, respectively. For S2 structure containing InAlAs test layer, the deviation in the thickness and composition of the test layer is 2.5 and 0.6%, respectively. As a result, the growth rates were calculated, which for binary compounds were as follows: 0.98 Å/s — for InAs, 0.93 Å/s — for GaAs, 0.90 Å/s — for AlAs. These deviations were taken into account, and the growth rates of binary compounds were adjusted using temperatures from the corresponding sources according to the flux meter data. The results of calculations and adjustments are given in Table 3.

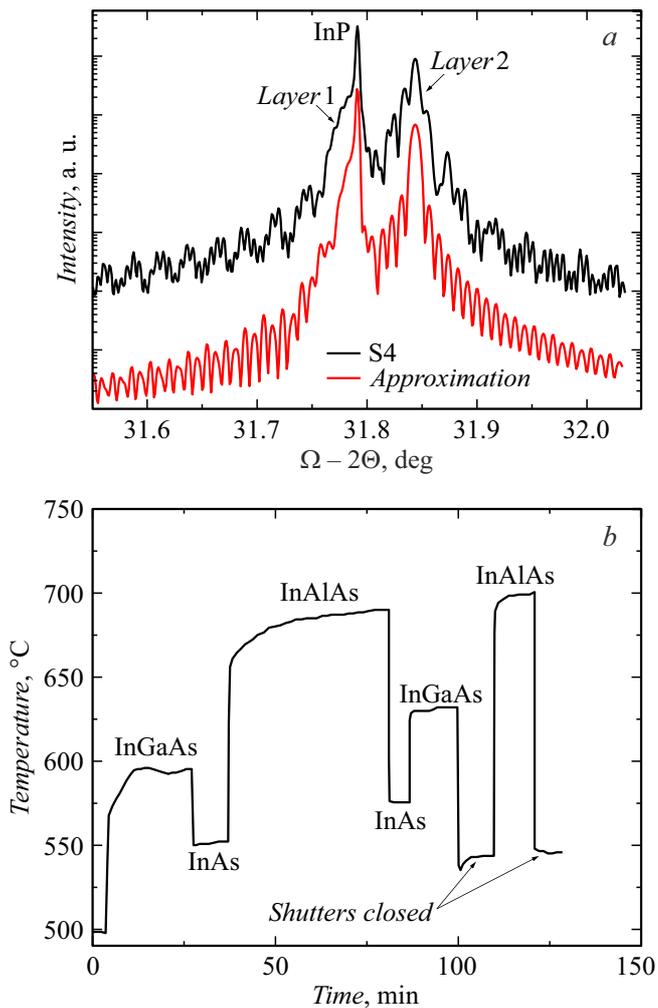


Figure 4. X-ray diffraction curve of the test sample S4 (a) and experimental data of the pyrometer when the shutters of effusion sources are opened (b).

Such a significant difference in growth rates had to be taken into account in growth programs. At that, it was necessary to keep in mind that InGaAs and InAlAs layers are deposited using the same In source and there is no other way to specify different growth rates for these layers except by changing the growth rate of AlAs or GaAs. A decrease in the AlAs growth rate specified in the growth program by 10% relative to the actual growth rate was used, which made it possible to compensate for the difference. The changes made led to a positive result, since the following test sample with SL $\text{In}_{0.36}\text{Al}_{0.64}\text{As}/\text{In}_{0.67}\text{Ga}_{0.33}\text{As}$ (S5) demonstrated a significantly more accurate correspondence of compositions and thicknesses to the required values (Fig. 5).

To adjust the composition of the layers in S5 structure, the temperatures of Ga05 and Al03 sources were additionally adjusted (decreased) relative to the values used during the growth of S3 structure. As a result, a slight excess of layer thicknesses relative to the nominal value was obtained

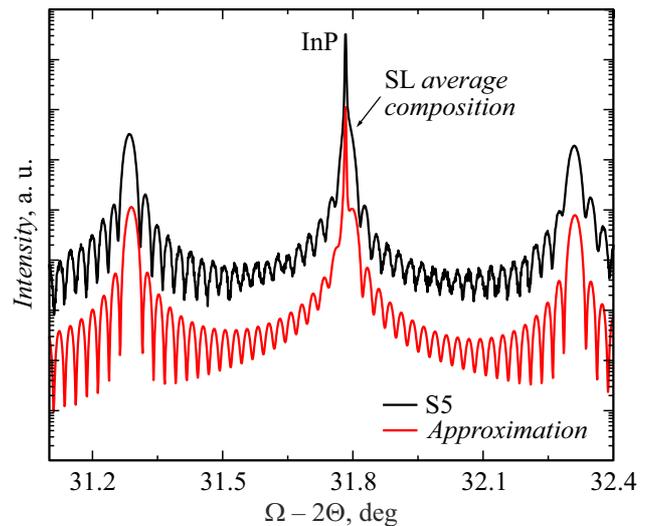


Figure 5. X-ray diffraction curve of test sample S5 with SL composition $\text{In}_{0.36}\text{Al}_{0.64}\text{As}/\text{In}_{0.67}\text{Ga}_{0.33}\text{As}$.

(maximum 1.5%). The average composition of SL almost fell within the agreed value and was poorly distinguishable in the rocking curves against the background of the peak from the substrate. The compositions of the layers differed by 1% for InAlAs layer and by 0.9% for InGaAs layer, both in the direction of higher indium content. The grown test samples and the data obtained from them made it possible to adjust the source temperatures for subsequent growth studies. Taking into account the exceeding of the thicknesses and compositions, it was decided to reduce the temperature of the source In06.

Conclusion

The performed study showed that knowledge of the growth rate of bulk layers of InGaAs and InAlAs does not allow us to accurately determine the growth rate of thin stressed superlattices of type $\text{In}_{0.36}\text{Al}_{0.64}\text{As}/\text{In}_{0.67}\text{Ga}_{0.33}\text{As}$, and the error is about 10%. The effect is associated with the difference in the growth temperature of InGaAs and InAlAs layers, which affects the intensity of indium evaporation from the growth surface. Experiments showed that additional heating of the growth surface can occur from Ga and Al sources when the shutter is opened due to thermal radiation of the molten material. The difference in temperature of the Ga and Al sources in our experiments was 80–120°C, which determined the different influence of thermal radiation on the growth of different layers. This effect can be universal and shall be taken into account in various MBE installations.

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Conflict of interest

The authors declare that they have no conflict of interest.

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