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Application of the X-ray Reflectometry method for determination of composition gradients at the quantum well boundary in laser structures based on the AlGaAs/GaAs system

© A.V. Berezutskiy, G.T. Mikayelyan, S.D. Tariverdiev

LLC „Lassard“,
249032 Obninsk, Russia

E-mail: tekton1321@gmail.com

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Devoted to the application of X-ray reflectometry to determine composition gradients at the boundaries of a quantum well in heterostructures based on AlGaAs/GaAs obtained by the MOCVD method. It is shown that the method is applicable at the stage of working out the active area. The parameters for recording reflectometric curves have been selected. The composition gradients in the experimental sample have been measured.

Keywords: X-ray reflectometry, quantum well, heterojunction, transition layer, GaAs.

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1. Introduction

Semiconductor injection lasers (also called diode lasers) are currently one of the most sought-after devices in quantum electronics and photonics. One of the main stages of the diode laser manufacturing technology is the growth of a semiconductor heterostructure on a monocrystalline substrate. At the current level of technology development, growth is performed by the metalorganic vapour phase epitaxy method (MOCVD) or molecular beam epitaxy (MBE). Each method has its advantages and disadvantages. One of the disadvantages of the MOCVD method is the formation of inhomogeneities at the boundaries of the quantum-dimensional layers of the laser heterostructure, which can have a significant impact on the parameters of the final heterostructure [1]. Currently, there are no non-destructive and precise techniques for testing the transition layers formed in this way for gallium arsenide-based structures. X-ray reflectometry is a well-known non-destructive method for determining the thicknesses of thin films [2,3].

The X-ray reflectometry method is based on the phenomenon of total external reflection of X-ray radiation from a solid (for which the refractive index for X-ray radiation is always less than 1) at lower angles of incidence (in reflectometry, the angle of incidence is called the angle between the beam and the surface of a solid) than the critical one (for GaAs $2\theta_{cr} = 0.31^\circ$). This method makes it possible to determine the thickness of thin films on the surface of single crystals. It was used in [4–6] to study various oxide coatings, silicon-based heterostructures (Si/Ge) and to determine the thicknesses of quantum wells in structures $\mathrm{A^{III}B^{V}}$. However, it was not used to determine composition gradients in quantum-dimensional layers of laser heterostructures.

Three systems of interference maxima with different periods superimposed on each other are observed on a reflectometric curve from a two-layer epitaxial structure (see Figure 1, curve *4*) grown on a single-crystal substrate: two systems correspond to the thicknesses of individual layers (curves *1* and *2*), and the third (with minimum period) — the total thickness of the structure (curve *3*) [7,8].

The interpretation of the reflectometric curve is as follows:

1) the compositions (according to the positions of the main peaks) and layer thicknesses (according to the positions of satellite peaks) are determined by X-ray diffractometry;

2) a calculated reflectometric curve is constructed from the structure defined in the previous step in the X-pert reflectivity program;

3) thin layers (up to 8) of intermediate composition are added at the boundaries of the quantum well. The thicknesses are selected to fully match the position of the maxima of the calculated and experimental curves. It is assumed that the composition gradients on both sides of the quantum well are identical.

The reflectometric curve from the N-layered structure contains $N(N+1)/2$ periods and can be correctly deciphered when comparing the experimental curve with the calculated one for structures containing no more than 10 layers [7,8].

The purpose of this work is to apply the above-mentioned technique to determine and control composition gradients in quantum-dimensional layers of heterostructures for its subsequent use in technological processes for the production of semiconductor lasers based on solid solutions $\mathrm{A^{III}B^{V}}$.

2. Technique and experimental results

When X-ray radiation falls on the surface of a real one (Table 1) laser heterostructure at large angles, in order to fulfill the condition of complete external reflection, its

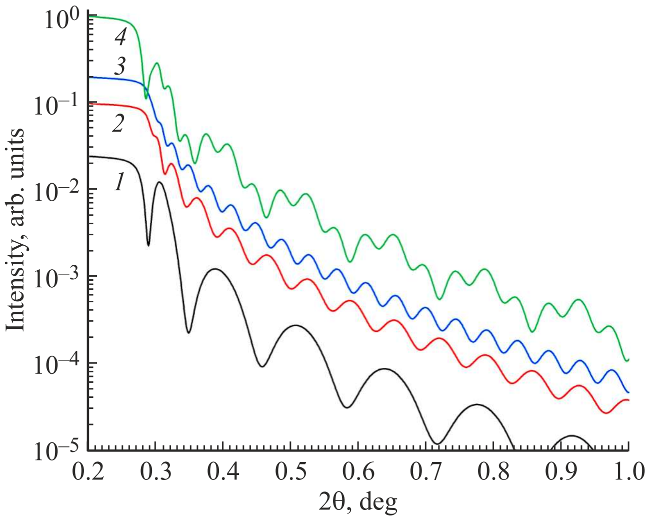

Figure 1. Curves *1* and *2* correspond to the interference maxima of individual layers; *3* — total thickness of the structure; *4* — reflectometric curve of a two-layer structure — addition of curves *1, 2* and *3*.

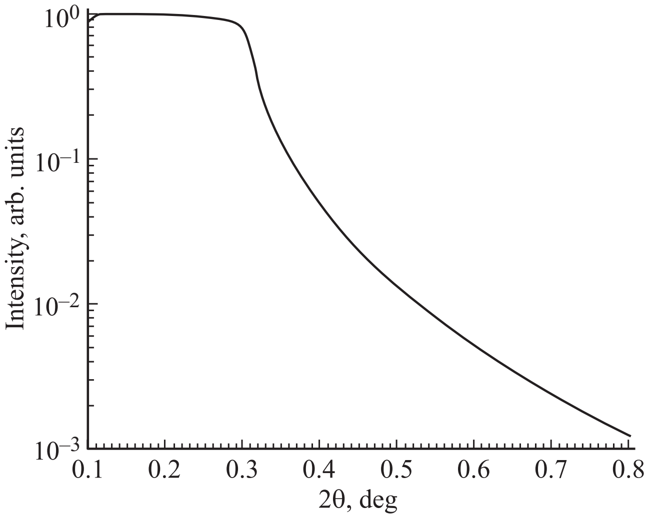

Figure 2. The calculated reflectometric curve of the laser structure presented in Table 1.

Table 1. The structure of the classical laser heterostructure

Layer	Composition	Thickness, μm
Contact layer	GaAs	50
p-injector	$Al_{0.55}Ga_{0.45}As$	1.5
Waveguide	$Al_{0.35}Ga_{0.65}As$	0.2
Quantum well	GaAs	0.013
Waveguide	$Al_{0.35}Ga_{0.65}As$	0.2
n-injector	$Al_{0.55}Ga_{0.45}As$	1.5
Substrate	GaAs	450

Table 2. The structure of the experimental sample obtained by the method MOCVD*

Layer	Composition	Thickness, μm
Waveguide	$Al_{0.25}Ga_{0.75}As$	0.054
Quantum well	GaAs	0.013
Waveguide	$Al_{0.25}Ga_{0.75}As$	0.5
Substrate	GaAs	450

Note. * Composition and thicknesses were determined by X-ray diffractometry.

complete absorption occurs in the upper layers of the heterostructure. In this regard, X-ray radiation reflected from the boundaries of the quantum well is not recorded. This leads to the absence of a system of reflectometric peaks on the curve (Figure 2), therefore it is necessary to use specially prepared samples with thin upper layers.

Special heterostructures consisting of a waveguide layer, a quantum well and a thin cover layer were grown on n-GaAs substrates to study the composition gradients in a quantum well, in this work (Table 2). In such structures, the X-ray reflectometry method is applicable due to the possibility of achieving X-ray radiation of the lower boundary of the quantum well. The exact data on the composition and thickness were determined using X-ray diffractometry (Figure 3).

Three systems of interference patterns corresponding to reflections from the following boundaries should be observed in a structure consisting of a thin quantum well ($\approx 5-12$ nm) and a cover layer on top of the well ($\approx 50-100$ nm):

a) the surface of the heterostructure — the upper bound of the quantum well;

b) the surface of the heterostructure — the lower boundary of the quantum well;

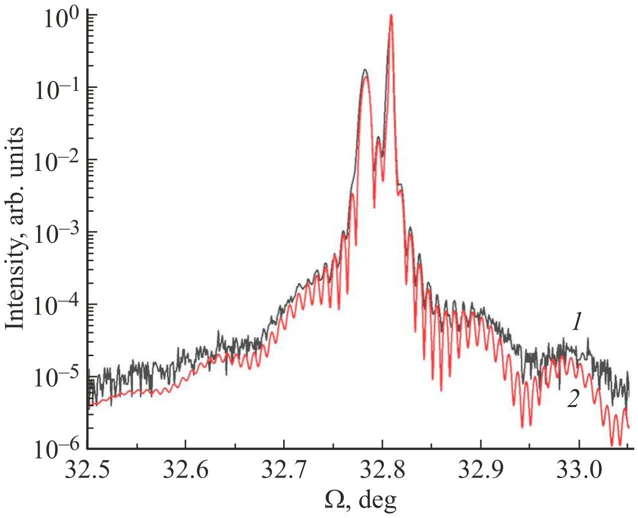

Figure 3. X-ray diffractometry of the experimental sample. *1* — experimental curve, *2* — calculated curve adapted to the experiment. (The colored version of the figure is available on-line).

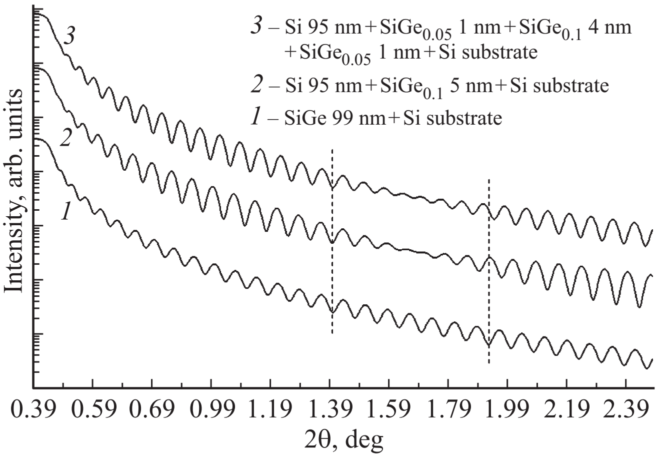

Figure 4. Calculated curves for a silicon-based structure [8].

c) the upper bound of the quantum well — the lower bound of the quantum well.

However, due to the small thickness of the quantum well, the system of maxima corresponding to the two boundaries of the quantum well in the considered range of incidence angles is not observed, due to the long period.

A wave reflected in antiphase from the interface of the quantum well and a less optically dense layer of the heterostructure is superimposed on the system of reflectometric maxima corresponding to reflections from the surface of the heterostructure and the upper boundary of the quantum well. This leads to a decrease in the intensity of interference peaks on the reflectometric curve, which is consistent with the data of the work [7,8].

Figure 4 shows the calculated reflectometric curves for the structure containing the quantum well SiGe [8]. On the curve *2* before attenuation from a 5 nm thick quantum well, the phase of the reflectometric minima coincided with the phase on the curve *1* from a single SiGe layer (the left vertical dashed line in Figure 4). After attenuation, the maximum on the curve *2* roughly corresponds to the minimum on the curve *1*. The appearance of inhomogeneities at the boundaries of the quantum well leads to an additional phase shift (curve *3* in Figure 4). This effect is used to identify the transition layers that arise during the growth of the heterostructure. Using this method, it is possible to determine the thickness of layers with an accuracy of 0.1 nm [8].

In this work, experimental measurements were carried out using the Panalytical X'pert 3 MRD X-ray diffractometer, the diagram of which is shown in Figure 5. A structure with a GaAs quantum well with a thickness of 13 nm was used as a sample, located between two wide-band layers $Al_{0.35}Ga_{0.65}As$ with a thickness of 50 nm and 1 μm (Table. 1) grown on a substrate *n*-GaAs by the MOCVD method on an AIX installation 2800G4-TM (IC2).

A detector *1* was used for measurements, in front of which two parallel germanium plates are located, from which the incident X-ray radiation is reflected three times before entering the detector. This configuration leads to the elimination of diffuse scattering in the region of the critical angle, but also significantly reduces the signal intensity. Therefore, the parameters of the diffractometer were selected from maximizing the signal-to-noise ratio. The following parameters of the X-ray unit were set for recording experimental reflectometric curves: signal accumulation time — 0.6 s; slit width after the monochromator — 1 mm; slit width in front of the detector — 0.5 mm. The signal-to-noise ratio was $6.4 \cdot 10^4$ with these values.

The obtained experimental reflectometric curve, as well as the simulation results, are shown in Figure 6. Three regions with different amplitudes of the interference pattern are observed. The positions of the maxima of all five curves coincide from the angle $2\theta = 0.31^\circ$ to the range of angles corresponding to the reflection from the lower boundary of the quantum well. However, after it, there is a slight shift in the simulated curve without blurring, which clearly indicates the presence of inhomogeneous layers in the quantum well. The characteristics of the gradients of the composition of the quantum-dimensional layer are obtained by adjusting the calculated curve to the experimental one. There are no noticeable changes in the calculation results

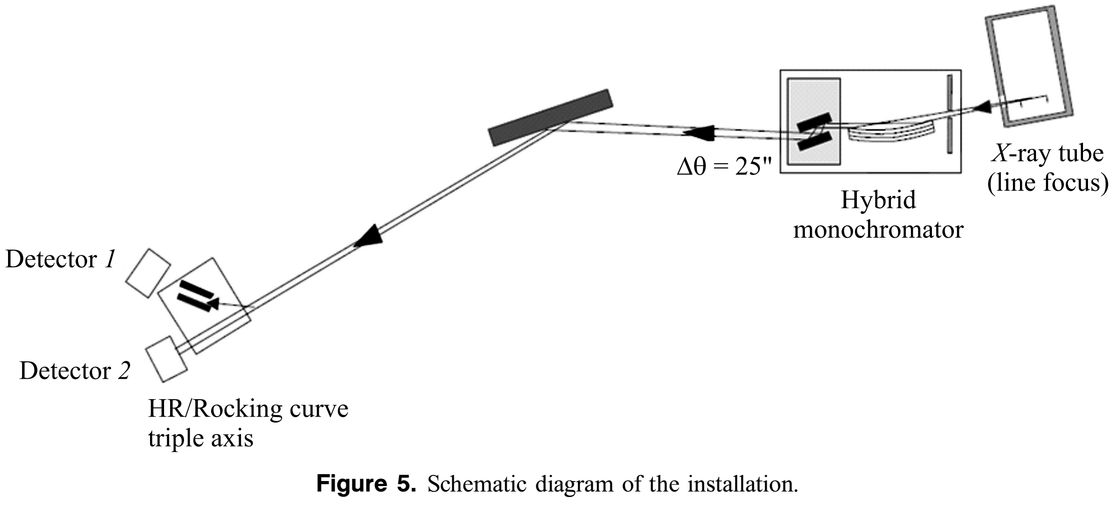

Figure 5. Schematic diagram of the installation.

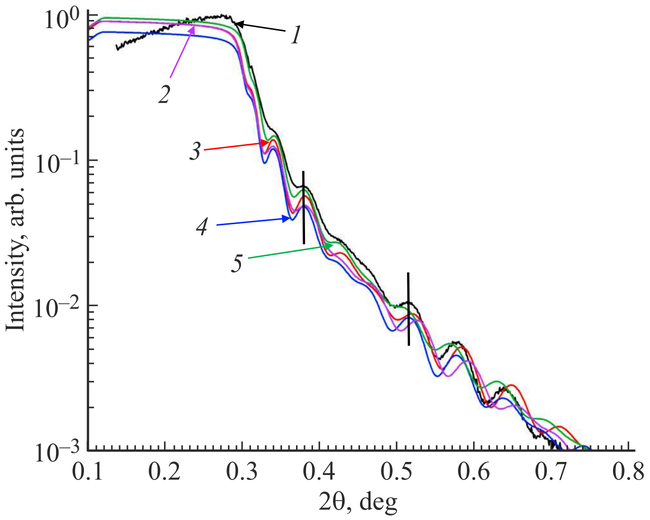

Figure 6. Comparison of the experimental reflectometric curve with the simulated ones. *1* (black curve) — experimental, *2* (purple) — modeled without taking into account inhomogeneities, *3* (red) — modeled taking into account the presence of inhomogeneities with a thickness of 1 nm, *4* (blue) — modeled taking into account the presence of inhomogeneities with a thickness of 1.2 nm, *5* (green) — modeled taking into account the presence of inhomogeneities with a thickness of 1.5 nm. (A color version of the figure is provided in the online version of the paper).

with an increase of the number of fitting layers of over 2. Therefore, two pairs (in total 4) of transitional layers of intermediate composition were used for the adjustment of the calculated curve to the experimental one. Thus, it was assumed that the inhomogeneity of the quantum-dimensional layer is represented as two pairs of thin layers of different (intermediate) composition. The criterion χ^2 was calculated for each of the four calculated curves. For the curve *2* it was $1.7 \cdot 10^{-3}$, for the curve *3* —$4.5 \cdot 10^{-4}$, for the curve *4* — $2.2 \cdot 10^{-6}$ and for the curve *5* — $4.9 \cdot 10^{-4}$. Thus, it can be concluded that the curve *4* is most consistent with the experimental curve.

Table 3. Structure composition. The thicknesses of the quantum well and the gradients of the composition are determined by adjusting the calculated reflectometric curves to the experimental curve

Layer	Composition	Thickness, μm
Top layer	$Al_{0.25}Ga_{0.75}As$	0.054
Transition layer	$Al_{0.2}Ga_{0.8}As$	0.0002
Transition layer	$Al_{0.1}Ga_{0.9}As$	0.001
Quantum well	GaAs	0.0106
Transition layer	$Al_{0.1}Ga_{0.9}As$	0.001
Transition layer	$Al_{0.2}Ga_{0.8}As$	0.0002
Top layer	$Al_{0.25}Ga_{0.75}As$	0.5
Substrate	GaAs	450

The final configuration of the quantum well (Table 3) contains two transition layers with a total thickness of ~ 1.2 nm, located presumably at the boundaries of the pit with a thickness of 10.6 nm.

3. Conclusion

A technique for determining the thickness of gradient layers at the boundaries of quantum wells of semiconductor quantum-dimensional heterostructures using X-ray reflectometry has been applied to GaAlAs/GaAs heterostructures. The proposed method for determining the characteristics of transition layers can be used at the stage of testing the technology for manufacturing semiconductor heterostructures, to set optimal parameters and adjust the conditions of the growth process. The technique makes it possible to control, within the technological capabilities of the growth setting, the thickness and gradients of the composition of quantum-dimensional layers in the manufacture of complex multilayer quantum-dimensional structures, including superlattices.

The technique was applied on experimental GaAlAs/GaAs samples obtained by the MOCVD method on the AIX 2800G4-TM (IC2) installation. The thickness of the gradient layers at the boundary between $Al_{0.35}Ga_{0.65}As$ and the GaAs quantum well was determined, which was 1.2 nm.

Conflict of interest

The authors declare that they have no conflict of interest.

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