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INFLUENCE OF THE In/Ga RELATION IN THE GAS PHASE ON THE CHARACTERISTICS OF THE $In_XGa_{1-X}P$ EPITAXIAL LAYERS OF CASCADE SOLAR CELLS

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The modern solar arrays for the most spacecrafts consists of solar cells which are formed by the thirty nano- and micro-dimensional epitaxial layers based on AIIIBV materials forming triple junction InGaP / InGaAs / Ge. This article presents the results of a study of experimental samples of thin single-crystal epitaxial $In_xGa_{1-x}P$ layers with different indium and gallium concentrations (x = 38 to 53 %) that were grown on Ge – substrate by MOCVD industrial equipment. The theme of present investigation is the influence of epitaxial growth parameters on the crystal structure characteristics.

The ratio of the components of the third group in the gas phase were calculated from the specified technological parameters. The rocking curves obtained by high-resolution two-crystal X-ray diffractometry were investigated. The lattice parameter and the ratio of indium to gallium in the solid phase were calculated. A high perfection of a single-crystal structure with an insignificant broadening of the X-ray diffraction peaks was observed in the range from 45 to 53 %. It is shown that the broadening of the diffraction peak of the structure can be the criterion of estimation of the quality of the grown structure in addition to the mismatch of diffraction maximum. Also the In / (In + Ga) ratio in the solid phase was calculated using the method of photoluminescence effect measuring. It was shown in comparison of data of x-ray diffraction with photoluminescence method the composition determination by photoluminescence method should be considered only as estimated.

Keywords: solar cell, solar battery, epitaxial layer, gas-phase epitaxy, photoelectric converter, X-ray diffractometry, photoluminescence, AIIIBV, semiconductor structure.

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ВЛИЯНИЕ СООТНОШЕНИЯ In / Ga В ГАЗОВОЙ ФАЗЕ НА ХАРАКТЕРИСТИКИ ЭПИТАКСИАЛЬНЫХ СЛОЕВ $In_XGa_{1-X}P$ КАСКАДНЫХ СОЛНЕЧНЫХ ЭЛЕМЕНТОВ

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На сегодняшний день для энергообеспечения подавляющего большинства космических аппаратов используются солнечные батареи, состоящие из солнечных элементов, структура которых образована тремя десятками нано- и микроразмерных эпитаксиальных слоев на основе материалов AIIIBV, формирующих каскады InGaP / InGaAs / Ge. Приведены результаты исследования экспериментальных образцов тонких монокристаллических эпитаксиальных слоев типа $In_xGa_{1-x}P$ с различным содержанием индия и галлия (x = от 38 до 53 %), выращенных методом газофазной эпитаксии из металлоорганических и гидридных соединений в установке промышленного типа на германиевой подложке. Предметом исследования является влияние параметров эпитаксиального роста на характеристики кристаллической структуры.

Расчетным методом получено соотношение компонентов третьей группы в газовой фазе из заданных технологических параметров. Исследованы кривые качания, полученные с помощью высокоразрешающей двухкристальной рентгеновской дифрактометрии, рассчитан параметр решетки и соотношение индия и галлия в твердой фазе. В диапазоне от 45 до 53 % наблюдается высокое совершенство монокристаллической структуры с незначительным уширением дифракционных рентгеновских пиков. Показано, что критерием оценки качества выращенной структуры наряду с рассогласованием дифракционных максимумов может служить уширение дифракционного пика структуры. Также соотношение In / (In + Ga) в твердой фазе получено *посредством метода измерения эффекта фотолюминесценции. При сравнении данных, полученных с помощью рентгеновской дифрактометрии и метода измерения эффекта фотолюминесценции, показано, что определение состава с помощью метода измерения фотолюминесценции следует рассматривать только оценочно.*

Ключевые слова: солнечный элемент, солнечная батарея, эпитаксиальный слой, газофазная эпитаксия, фотоэлектрический преобразователь, рентгеновская дифрактометрия, фотолюминесцения, AIIIBV, полупроводниковая структура.

Introduction. Due to the increased requirements for onboard spacecraft systems, there is a need to create solar array (SA) with high performance and energy characteristics and increased service life (more than 15 years). The SA will convert sunlight directly into electricity with high efficiency, creating almost constant power at low operating costs [1]. The most promising elements for a modern SA for operating as a part of the spacecraft and satellites are cascade solar cells (SC) based on AIIIBV materials.

Modern triple-junction CS is a complex planar device. Generating semiconductor part of such a solar cell, produced by Metalorganic Chemical Vapour Deposition (MOCVD), consists of three dozen nano- and micro-sized functional layers forming triple junction InGaP / InGaAs / Ge. Due to the relatively large surface of the device – about 30 cm^2 [2; 3], in order to achieve high characteristics of the structure, it is particularly important to comply with the requirements for uniformity of properties of all layers of the structure. To create a multi-layer SC, using different materials, a large number of technological operations are necessary, each of which requires careful monitoring of the process parameters and properties of the product, since the deviation from the norm at each stage can affect the properties of the finished structure and the output characteristics of the SC [3]. Intermediate control of properties allows detecting defects at each stage (epitaxial growth of semiconductor structure, post-growth processes: photolithography, metallization, deposition of anti-reflection coating, the formation of overall dimensions, etc. [2]) and reject defective wafers. To identify the causes of possible defects, it is necessary to determine the dependences between the properties of the structure and the technological parameters of the process [3].

One of the main factors for improving the efficiency of SC is the perfection of growth of semiconductor layers, namely: matching the lattice parameters, the minimum concentration of defects in the crystal structure and achieving high homogeneity of the composition on the entire surface of the wafer. The mismatch of lattice parameters is of the first importance, as it is in itself a measure of deformation in the layer [4]. Therefore, for the development of special epitaxial layers with precise matching of the lattice parameter in the process of growth, it is essential to have full data about the ratio in the gas stream of the elements of the third group In / Ga of the periodic system of chemical elements for obtaining a target composition in the structure of the epitaxial layer $In_xGa_{1-x}P$ (the main material not only of modern triple junction solar cell, but other promising photovoltaic devices), i. e. it is necessary to study the dependence of the ratio of the composition in the gas and solid phase.

Experimental Details. For a detailed study of the epitaxial layer of $In_xGa_{1-x}P$ on the subject of the influence of the epitaxial growth parameters on the characteristics of the crystal structure model samples of simplified structure of more than 60 pieces with different content of indium and gallium (x = from 38 to 53 %) doped with silicon and tellurium, on the germanium substrate with a diameter of 100.0 ± 0.4 mm were manufactured. The samples were grown by the MOCVD method on the installation of an industrial type Veeco E450. Schematic images of the structures assigned are shown in fig. 1, as well as the technological parameters for each layer assigned in the calculations, were obtained applying the program for the analysis and comparison of recipes Veeco RCPAnalysis [5; 6].

Method of gas-phase epitaxy of metal-organic and hybrid compounds. The feature of MOCVD method lies in the fact that in the epitaxial reactor creates a high temperature area, which receives a gas mixture containing decomposable compounds [7]. In the reactor, the release and deposition of the substance on the substrate occurs, and gaseous reaction products are carried out by the flow of hydrogen carrier gas. For the preparation of compounds AIIIBV, as the source of III group element is used metal-organic compounds, for example, trimethylgallium (TMGa) and trimethylindium (TMIn) for the synthesis of InGaP. As a source of group V elements such gas as phosphine (PH_3) is used.

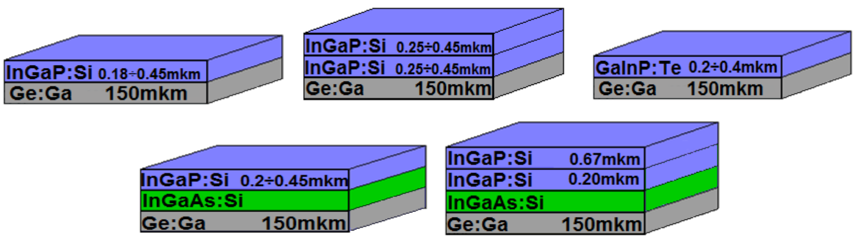

Fig. 1. Schematic representations of the structure of model samples
(for epitaxial layers $In_xGa_{1-x}P$ values of thickness are indicated)

Рис. 1. Схематические изображения структуры модельных образцов
(для эпитаксиальных слоев $In_xGa_{1-x}P$ указаны значения толщины)

Since the structure of the SC consists of many semiconductor layers, with different chemical composition and different levels of alloying and doping, before the process of creating the entire semiconductor structure in one cycle of epitaxial growth it is necessary to use the software installation epitaxial growth recipe.The recipe is a table in which the technological parameters for each metal-organic and hydride compound at each time of the epitaxial growth process are specified, as well as the temperature in different zones of the growth chamber, etc. [3; 8].

To obtain an epitaxial layer with specified properties, such as the crystal lattice parameter, the band gap width, it is necessary to control the chemical composition of the growing layers, determined by the composition of the gas mixture and the distribution of metal-organic compounds in the reactor.

The composition of the gas mixture is set by such technological parameters as the speed of flow of substances, the pressure in babbler and temperature [9; 10]. The schematic design of the bubbler for dosing liquid volatile alkyl TMGa and bubbler for TMIn into the reactor is shown in fig. 2.

Physico-chemical calculation of the ratio of elements of the third group In / Ga in the gas phase. In order to calculate the flow of the component in the gas phase for each metal-organic compound, it is necessary to use the following formula (1):

$$V^{\text{газ}}_{\text{TMGa,TMIn}} = \frac{S \cdot P_v}{(P_b - P_v) \cdot V}, \tag{1}$$

where S is the flow of substance through the bubbler, cm^3/min; P_b – pressure in the bubbler, Pa; P_v – the partial pressure of metal-organic compounds at a given temperature, Pa; V – the molar volume of ideal gas (under normal conditions), cm^3/mol.

The saturated steam pressure is determined by individual equations, depending on the temperature (T) according to the tabular data [11]. For TMGa according to the expression (2), and for TMIn according to the expression (3):

$$\log_{10} P_{v(\text{TMGa})} = 8.070 - \frac{1703}{T}, \tag{2}$$

$$\log_{10} P_{v(\text{TMIn})} = 10.52 - \frac{3014}{T}. \tag{3}$$

Thus, having considered the equations (1), (2), (3) it can be concluded that the velocity of the molar flow at the outlet of the bubbler can be controlled by changing the velocity of the hydrogen flow, the pressure in the bubbler and the bubbler temperature. The increase in pressure in the bubbler (P_b) reduces the velocity, temperature rise (equivalent to an increase in P_v), as well as the flow of hydrogen – increases the molar velocity. Thus, the given values are technological parameters that can be changed to achieve the required composition of the layer.

Despite the perfection of the MOCVD method in the technology of semiconductor production, the periodic inspection of key parameters of epitaxial structures is required [3], in addition, such a procedure is necessary for the synthesis of new layers. One of the main methods is a method of x-ray diffraction (XRD).

Methods of measurement using high-resolution two-crystal x-ray diffraction. To determine the lattice parameter of separate compounds on the XRD Vector measurements were carried out, which resulted in a rocking curve.

It should be noticed that, the maximum contribution to the intensity, gives the thickest layer of the structure, namely Ge substrate. The presence of broadening and any other stand-alone peaks indicates the presence of layers mismatched from the lattice parameters [12].

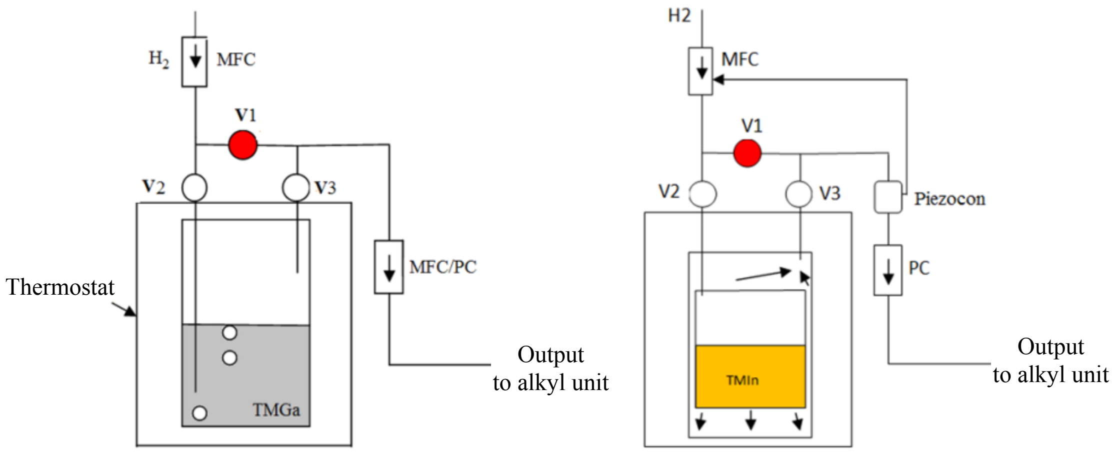

Fig. 2. Construction of bubbler: $V1$, $V2$, $V3$ – pneumatic valves; MFC – device for supplying a given flow of hydrogen; MFC/PC (PC) – pressure controller – device for maintaining the preset pressure in the bubbler; Piezocon – the device that determines the molar concentration of TMIn at the outlet of the bubbler

Рис. 2. Конструкция барботёров: $V1$, $V2$, $V3$ – пневматические клапаны; MFC – прибор подачи заданного потока водорода; MFC/PC (PC) – контроллер давления – прибор поддержания заданного давления в барботёре; Piezocon – прибор, определяющий мольную концентрацию TMIn на выходе из барботёра

A classic example of the rocking curve for the epitaxial layer of InGaP is shown in fig. 3. In structures with different number of misaligned layers, the rocking curves are characterized by the presence of one narrow peak with high intensity and spaced from it by a certain number of angular seconds of the peak with lower intensity. When the peak is located with lower intensity to the left of the main, there is an increase in the lattice parameter relative to the substrate, while the location to the right of the main one is a decrease:

$$a^2 = \frac{d^2}{n} \cdot (H^2 + K^2 + L^2), \tag{4}$$

where a is the lattice parameter, Å; d is the inter-plane distance between the reflecting planes, Å; n is an integer describing the diffraction order of the reflection; H, K, L – the indices of the interference;

$$a_{\mathrm{In}_x\mathrm{Ga}_{1-x}\mathrm{P}} = x \cdot a_{\mathrm{InP}} + (1-x) \cdot a_{\mathrm{GaP}}, \tag{5}$$

where x is the content of In in the solid solution.

The obtained experimental data are well placed on the line based on the table data on the Vegards rule, but there are some deviations near the table value of the Ge lattice parameter. Since in this case it is difficult to separate the peaks of the substrate and the epitaxial layer, there is an error in determining the distance between them. An example of such a rocking curve is shown in fig. 4, *a*.

For samples with a significant deviation of the lattice parameter, the rocking curves are characterized by a large value of the broadening of the peak is about 800 arcseconds (fig. 4, *c*). For the classical rocking curve of the faultless structure, the distance between the peaks of the order of 200–400 arcseconds and the broadening is less than 200 (fig. 4, *b*).

The XRD survey, the data processing of the rocking curves of experimental samples of epitaxial structures, as well as data for calculating the composition of solid solution from the gas phase composition, a diagram of dependence of the lattice parameter (the primary axis) and the broadening of the x-ray peak (secondary axis) the ratio of In / Ga in the epitaxial layer of $\mathrm{In}_x\mathrm{Ga}_{1-x}\mathrm{P}$ was made (fig. 5).

The lattice parameter increases with the increase in the percentage of indium in $\mathrm{In}_x\mathrm{Ga}_{1-x}\mathrm{P}$. Basically, all experimental data are well placed on the straight line, the misalignment of the lattice parameter is 0.1–0.26 %. In the range of 45 to 53 % monocrystalline structure is characterized by high perfection. When the ratio of In and Ga is 1:1 the epitaxial layer is characterized by the best properties with the most similar value of the lattice parameter in accordance with the Ge. The deviation from the composition for these samples ranges from 0.5 to 3 %.

Additionally, studies have been carried out to determine the homogeneity of the solid solution throughout the surface of the sample. Due to the center symmetry of the growth chamber, single rocking curves are measured along the sample radius. The comparison of measurement results shown in fig. 6 indicates a homogeneous growth of the epitaxial structure in composition according to table, which makes it reasonable to survey at the central point with multiple measurements and confirms the reliability of the data obtained.

Thus, according to the results of studies on the high-resolution two-crystal XRD it is possible to conclude about the state of samples of SC, the presence of misaligned layers, defects and the possibility of further work with the resulting epitaxial structures, as well as if necessary to make adjustments to the recipe for epitaxial growth.

Method for measuring the effect of photoluminescence. To determine the composition of the solid solution by using the method of measuring the effect of photoluminescence (PL) at 2000 RPM equipment. This method is easier to implement, the equipment allows to obtain the maps to analyze the uniformity of properties, but the method is indirect and it is necessary to take into account the contribution of other factors (alloying).

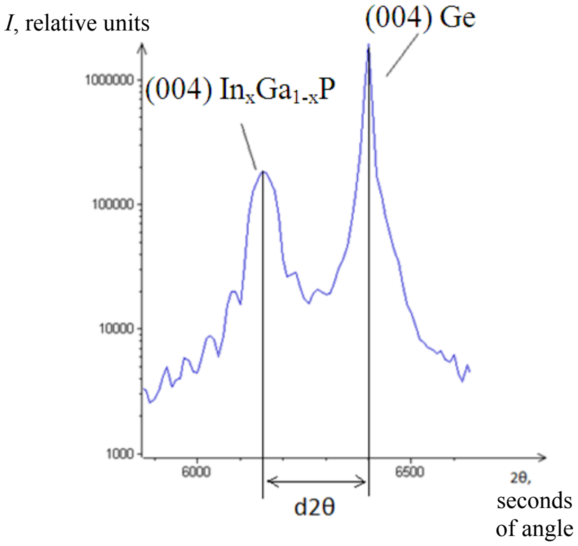

Fig. 3. Experimentally obtained classical rocking curve for an epitaxial layer InGaP

Рис. 3. Экспериментально полученная классическая кривая качания для эпитаксиального слоя InGaP

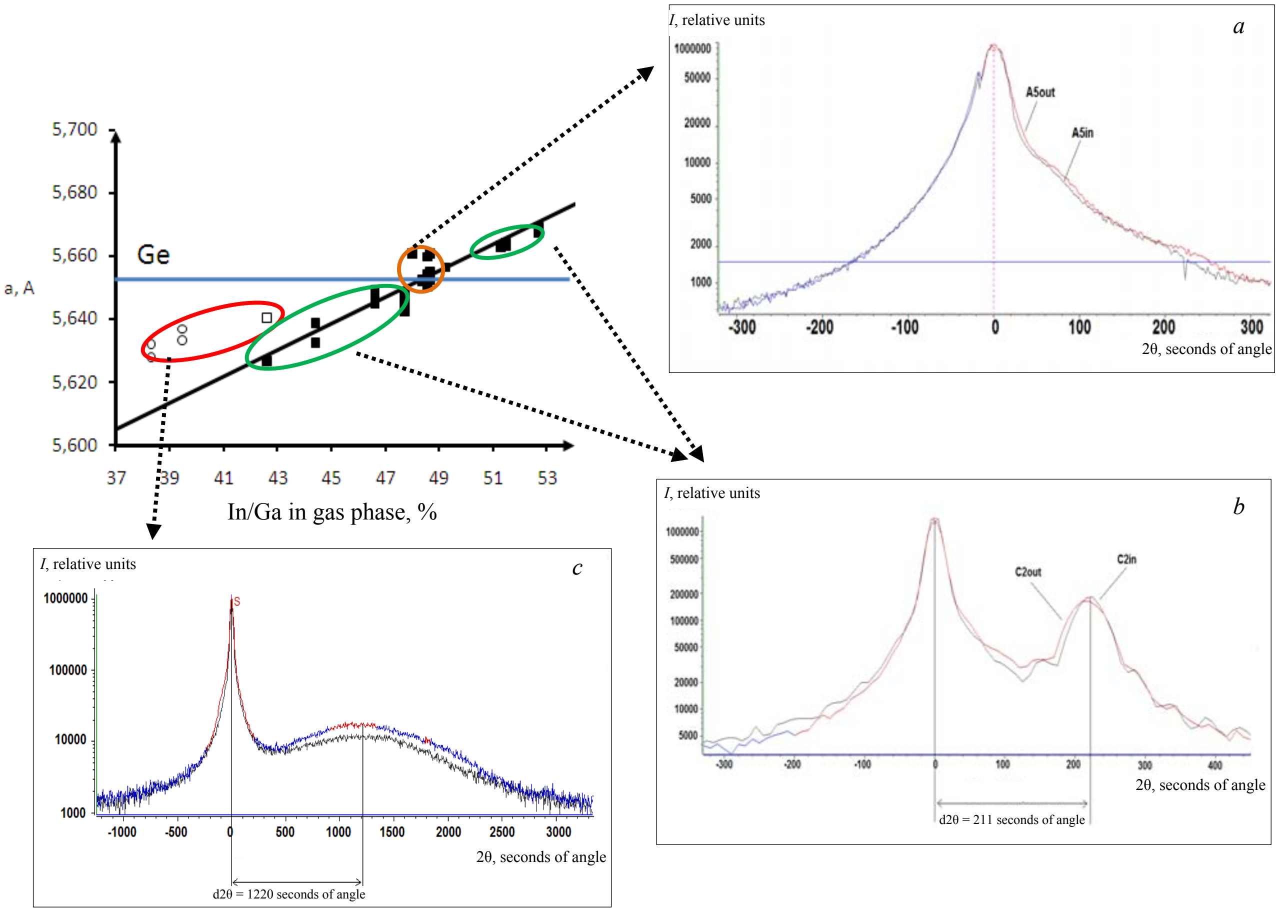

Fig. 4. Rocking curves for a different ratio In / Ga in the solid phase

Рис. 4. Кривые качания для различного соотношения In / Ga в твердой фазе

The results values calculation of the of the lattice parameter and the composition of the solid solution of epitaxial layer $In_xGa_{1-x}P$ for 5 points

№ survey point	*d*2θ, arcsecond	θ, °	*a*, Å	Δ*a*/*a*$_{Ge}$, %	Ga, %	In, %
1	290.4	33.03	5.6518	0.108	51.87	48.13
2	295.8	33.03	5.6517	0.110	51.90	48.10
3	300.3	33.04	5.6516	0.112	51.92	48.08
4	296.1	33.03	5.6517	0.111	51.90	48.10
5	282.3	33.03	5.6519	0.105	51.83	48.17

According to the data illustrated in fig. 7, we can judge about the uniformity in thickness and composition of the epitaxial structure at the stage of growth, because the PL signal depends on the width of the band gap of the material given by the chemical composition.

Spectral maps show the distribution of the integral intensity of PL, wavelength peak, intensity peak and full width at half maximum of the spectrum on the planar epitaxial structure [3; 10]. Spectral maps (fig. 7) stored in a format that allows determining the spectrum data at each point of the epitaxial structure. In particular, the data is processed by the elements of the technology support system [13; 14].

Using the results obtained, it is possible to determine the homogeneity of the studied layer and calculate the value of the band gap width using the formula (6):

$$E_g = \frac{h \cdot c}{\lambda}, \quad (6)$$

where E_g is photon energy, eV; λ is photon wavelength (characteristic wavelength of photoluminescence spectrum), nm; h – Planck's constant, eV/sec; c – is the speed of light, m/sec.

The obtained results show the uniform distribution of peak spectra of PL (electron-optical properties) over the entire surface of the sample, while the average deviation of the maximum spectra of PL is 2.8 %.

When comparing the data of the ratio of In / Ga in the solid phase measured by the two methods for samples dopped with Si and Te, it is seen that the composition data obtained by PL measurement should be considered as estimated. The results are presented in fig. 8.

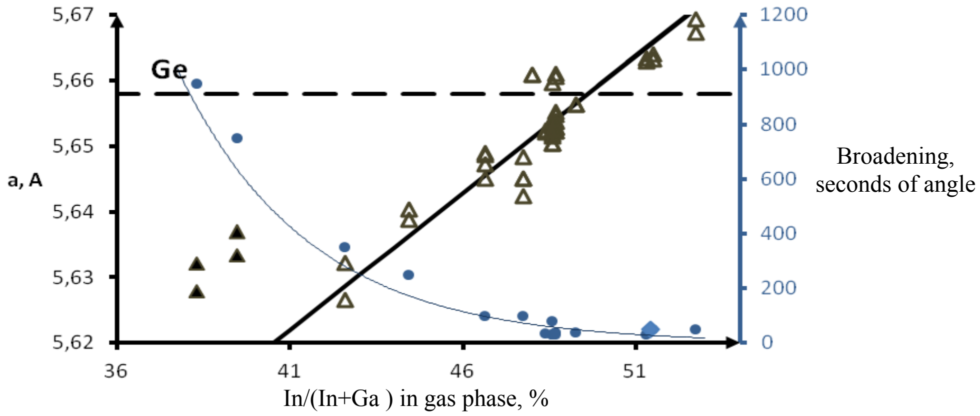

Fig. 5. The graph of the dependence of the lattice parameter and the broadening of the x-ray peak on the ratio In / Ga in the epitaxial layer $In_xGa_{1-x}P$: triangular points – the experimental data of the lattice parameter; points – the broadening of the diffraction peaks of some samples; the dotted line – the value of the lattice parameter Ge; direct line – data constructed according to Vegard's rule

Рис. 5. График зависимости параметра решетки и уширения рентгеновского пика от соотношения In / Ga в эпитаксиальном слое $In_xGa_{1-x}P$: треугольные точки – полученные экспериментальные данные параметра решетки; круглые точки – значения уширения дифракционных пиков некоторых образцов; пунктирная линия – значение параметра решетки Ge; прямая линия – данные, построенные по правилу Вегарда

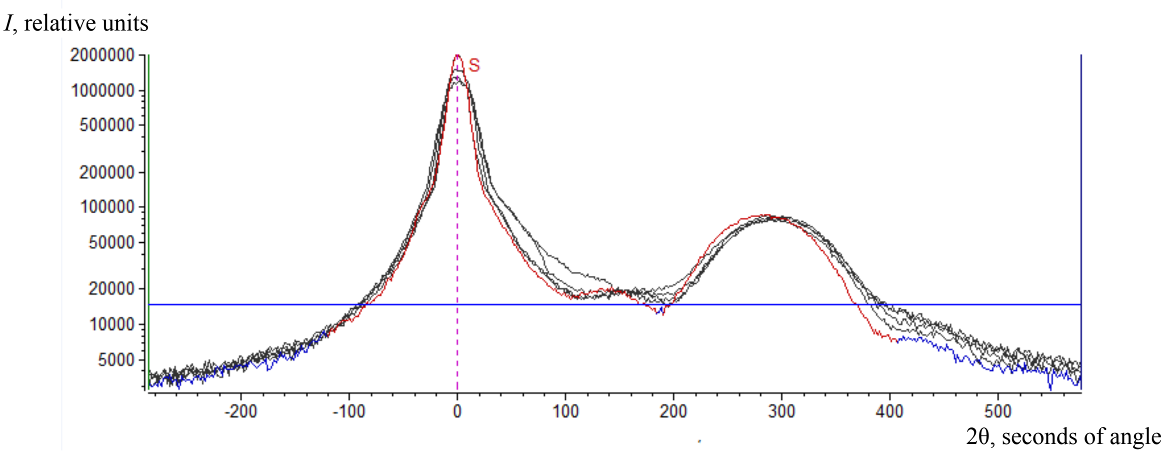

Fig. 6. A set of rocking curves obtained along the radius of the sample

Рис. 6. Набор кривых качания, полученных вдоль радиуса образца

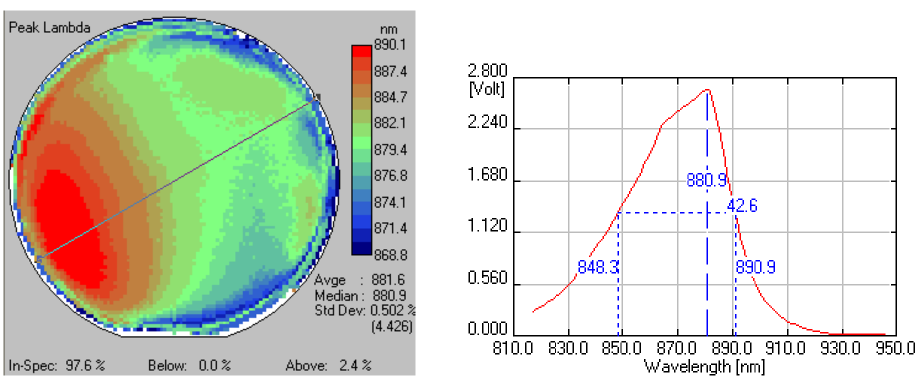

Fig. 7. Spectral map and single photoluminescence spectrum for one sample

Рис. 7. Спектральная карта и единичный спектр ФЛ для одного образца

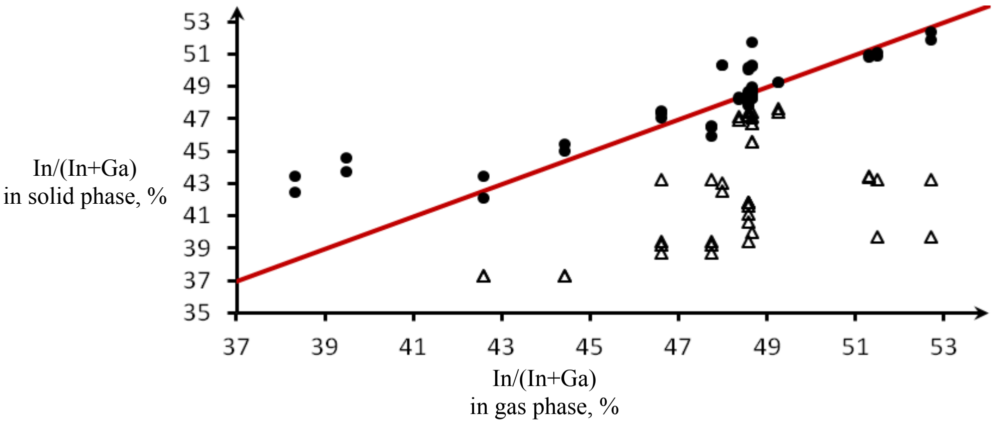

Fig. 8. Graphs of the ratio In / Ga of the epitaxial layer $In_xGa_{1-x}P$ in the solid phase, obtained by means of X-ray diffractometry and photoluminescence from the ratio in the gas phase: round shaded points – the results obtained with the X-ray diffractometry method; triangular unpainted dots – results obtained using the photoluminescence method; a straight line – the line of consistency

Рис. 8. Графики зависимости соотношения In / Ga эпитаксиального слоя $In_xGa_{1-x}P$ в твердой фазе, полученные с помощью методов РД и ФЛ от соотношения в газовой фазе: круглые закрашенные точки – результаты, полученные с помощью метода РД; треугольные незакрашеные точки – результаты, полученные с помощью метода ФЛ, прямая линия – линия соответствия данных

For $x < 0.63$ $Ga_xIn_{x-1}P$ material, the following expression [15] is true to calculate the composition of the epitaxial layer:

$$E_g = 1.34 + 0.69 \cdot x + 0.48 \cdot x^2. \quad (7)$$

Conclusion. The research, the results of which are presented in this article, was completed as a part of the determining of the conditions of a highly homogeneous, defect-free growth of epitaxial layers. The general scheme of such studies was tested mainly on InGaAs layers [16; 17].

In the course of the work, the following important results were obtained:

– the optimal technological parameters of epitaxial growth in the composition of the solid solution $In_xGa_{1-x}P$ are determined;

– it is revealed that for the solid solution $In_xGa_{1-x}P$ in the range from 45 to 53 % there is a high perfection of single crystalline structure, a slight broadening of diffractional x-ray peaks (less than 200 acrsecond);

– it is shown that the criterion for assessing the quality of the grown structure, along with the misalignment of diffraction maximum, can be the broadening of the diffraction peak of the structure.

Additionally, it is shown that:

– the plotted rocking curves along the radius of the sample indicate a high homogeneity of the solid solution over the entire surface of the sample (deviation of the lattice parameters 0.01 %), which makes it reasonable to detect at the central point in multiple measurements and confirms the reliability of the data obtained;

– the composition of the solid solution determined using the method of measuring the effect of photoluminescence, should be considered as evaluative.

The obtained results allow us to move to the next stage of work to determine the optimal technological parameters for the growth of perfect epitaxial layers of $In_xGa_{1-x}P$ on a substrate of Ge in the structure of the modern and perspective high-performance cascade SC for space applications, namely achieving the required electrical characteristics using precision dopping [17; 18]. It is also possible to use the created and tested on the compound $In_xGa_{1-x}P$ algorithm for similar studies of other materials.

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