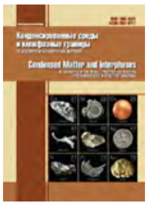

ISSN 1606-867X (Print)
ISSN 2687-0711 (Online)
Condensed Matter and Interphases
Kondensirovannye Sredy i Mezhfaznye Granitsy
https://journals.vsu.ru/kcmf/

Original articles

Research article
https://doi.org/10.17308/kcmf.2021.23/3430

Growing epitaxial layers of InP/InGaAsP heterostructures on the profiled InP surfaces by liquid-phase epitaxy

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Abstract

The effect of various planes was studied when growing epitaxial layers by liquid-phase epitaxy (LPE) on the profiled InP substrates. The studies allowed obtaining buried heterostructures in the InP/InGaAsP system and creating highly efficient laser diodes and image sensors.

It was found that protruding mesa strips or in-depth mesa strips in the form of channels formed by the {111}A, {111}B, {110}, {112}A, or {221}A family of planes can be obtained with the corresponding selection of an etching agent, strip orientation, and a method of obtaining a masking coating. It was noted that in the case of the polarity of axes being in the direction of <111>, the cut of mesa strips was conducted along the most densely packaged planes. This cut led to the difference in rates of both chemical etching and epitaxial burying of profiled surfaces.

The cut was made along the planes at a low dissolution rate {111}A for a sphalerite lattice, to which the studied material, indium phosphide, belongs. Analysis of planes {110} and $\{\bar{1}10\}$ showed that the location of the most densely packaged planes {111}A and {111}B relative to them is different.

Keywords: Heterostructures, Laser diodes, Indium phosphide, Buried heterostructures, Channel in the substrate

Acknowledgements: the work was supported by the Ministry of Science and Higher Education of Russia as part of the state assignment of Kurnakov Institute of General and Inorganic Chemistry of the Russian Academy of Sciences.

For citation: Vasil'ev M. G., Vasil'ev A. M., Izotov A. D., Kostin Yu. O., Shelyakin A. A. Growing epitaxial layers of InP/InGaAsP heterostructures on the profiled InP surfaces by liquid-phase epitaxy. *Kondensirovannye sredy i mezhfaznye granitsy = Condensed Matter and Interphases*. 2021;23(2): 204–211. https://doi.org/10.17308/kcmf.2021.23/3430

Для цитирования: Васильев М. Г., Васильев А. М., Изотов А. Д., Костин Ю. О., Шелякин А. А. Выращивание эпитаксиальных слоев гетероструктур InP/InGaAsP на профилированных поверхностях InP методом жидкофазной эпитаксии. *Конденсированные среды и межфазные границы*. 2021;23(2): 204–211. https://doi.org/10.17308/kcmf.2021.23/3430

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1. Introduction

InP/InGaAsP heterostructures are the main type of structures used for obtaining quantum electronics devices, such as semiconductor lasers, superluminescent radiation sources, and photodiodes with a wavelength range from 1.20 to 1.65 μm which is the most important for the systems of fibre optic communication channels and fibre optic sensors [1–6]. The uniqueness of these structures is that InGaAsP solid solutions are isoperiodic to indium phosphide, which allows creating “perfect” heterojunctions suitable for wide application in engineering [7–9].

There are various methods for obtaining such heterostructures: liquid-phase epitaxy (LPE) [10,11], molecular-beam epitaxy (MBE) [12, 13], and metalorganic chemical vapour deposition (MOCVD) [14–16]. All these methods are aimed at the creation of heterostructures forming the basis of chips for quantum electronics devices. Creating chips from the structures that are grown using MBE and MOCVD methods requires foreign equipment and a unique post-growth technology for processing structures, which makes the technological cycle of the production of devices more complicated and expensive. It also requires high-precision and expensive technological equipment and is conducted under conditions of nonequilibrium growth, which complicates their creation on profiled surfaces.

At the same time, there are methods of obtaining epitaxial heterostructures on profiled surfaces [17, 18] that allow simplifying the technological cycle and creating chips for quantum electronics devices from solutions-melts in growth modes close to equilibrium using a rather simple and cheap LPE method and Russian equipment. These growth modes allow creating device structures with a high level of perfection of layers by simple doping of different layers of the structure in a wide range of concentrations of the dopant. Forming an initial profiled surface of substrates, it is possible to use a selective growth mode on various sides, thus creating the specified electrical and optical limitations for light transmission in laser diodes. The aim of this work was to study the modes of formation of profiled surfaces of indium phosphide as well as the growth of InP/InGaAsP heterostructures on profiled surfaces and production of laser diodes with a spectral range of wavelengths 1300–1650 nm.

2. Experimental

2.1. Creating and burying a “fishtail” laser pinned structure

To determine the modes of formation of the profiled surface of indium phosphide and rates of epitaxial growth of InP layers and solid solutions InGaAsP, the samples of substrates InP with the orientation of planes {100} were used. The orientation accuracy was +/- 0.5 degrees.

The substrates with *n*-type conductivity were doped with Sn or S to a concentration of 10^{18}–10^{19} cm^{-3}, the substrates with p-type conductivity were doped with Zn to the concentration of $(4–5)\cdot10^{18}$ cm^{-3}. The substrates were mechanically polished to a thickness of 320±10 μm. Then, after the mechanical polish, the samples were washed three times in organic solvents and etched in a polishing etching agent Br : CH_3COOH to eliminate the damaged surface layer of ~ 10 μm. The masking coating for the further formation of a “fishtail” mesa was a SiO_2 film deposited using the method of pyrolytic reaction of SiH_4 and O_2 with the temperature of the InP substrate being 450 °C. The thickness of the oxide film was 0.15 μm. The processes of liquid-phase epitaxy were conducted in the hydrogen atmosphere with the dew point of –80 °C in a plate made of high-purity MPG-7 graphite with a limited growth cell volume [19].

The profile of a strip was formed along the cleavage in the directions of [110] or [$\bar{1}$10]. The direction of the strip in relation to the plane of the substrate was determined by etching an InP

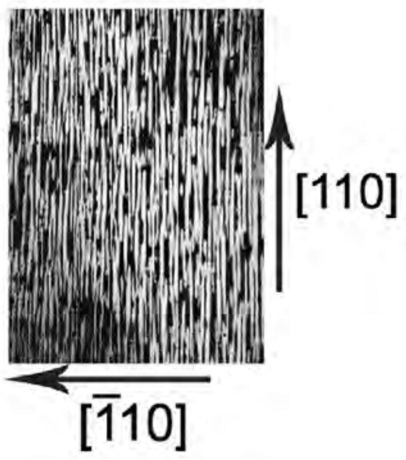

Fig. 1. Etching figures on plane (100)

substrate fragment ~ 1 mm wide with further etching in concentrated HCl for 2 minutes. Characteristic “straws” were formed on the surface of the InP substrate fragment, and their elongated part indicated the direction of [110]. The direction of [Ī10] was perpendicular to the elongated part of the “straws”.

Mesa strips were formed using a standard photolithography method. The width of a strip was 10 µm with the 400 µm interval between the strips. Further etching for the creation of a mesa strip structure was conducted at room temperature with normal lighting conditions and slight stirring of the solution. There are different chemical etching agents for indium phosphide [20]. The composition of the Br : CH_3COOH etching agent was used in this work as the most preferable for InP [21]. Geometric sizes of the profile were determined by the measurements in the “Epival” optical microscope with an amplification of ×450. The cleavage for the determination of geometric sizes was made parallel to the planes {110}. The etching rate was calculated by the etching depth defined by the cleavage using an optical microscope. After the formation of the strips, the etched surface between them was mirror-smooth without any visible defects. Various volume ratios of this etching agent were used (1:160; 1:80; 1:25; 1:9). With the specified direction of the strip, the shape of the mesa strip structure did not change with different ratios of the etching agent. In our opinion, the most suitable ratio for Br : CH_3COOH was 1:9. The etching rate of this etching agent was 2 µm per minute on the samples without a masking coating. Further experiments on the formation of mesa strips were conducted by chemical etching in the Br : CH_3COOH mixture with the 1:9 ratio. Depending on the orientation of masking strips, two types of mesa structures were obtained, see Fig. 2.

This etching agent was also used for test multi-layer InP/InGaAsP heterostructures without any deviations from the selected modes.

Analysis of planes (110) and (Ī10) showed [22] that the position of the most densely packaged planes {111}A in relation to them is different, see Fig. 3.

During the etching of the InP layer with the strip orientation along the direction of 110], the mesa strip had a shape of a fishtail limited by planes (ĪĪI)A and (Ī1Ī)A which were located at an angle of 125°16′ to (001) and by planes (1Ī2) and (Ī12) (angle 35°16′). However, when the etching was conducted along the direction of [Ī10], the mesa strip had a shape of a hill limited by planes (111)A and (I Ī Ī)A which were located at an angle of 54°44′ to (001). We studied the etching rates of indium phosphide substrates with different crystallographic orientations (111)A; (111)B; (100); (001). The changes in the etching rate of the substrates with different orientation are shown in Fig. 4.

We can see that $V_{(111)B} > V_{(100)} > V_{(111)A}$. It should be noted that the slowly etched plane (111) A always remains matt while planes (111)B, (100), and (001) are mirror-like. Such significant differences in the etching rates for various crystallographic orientations are apparently related to polar properties of the sphalerite lattice in the direction of <111>.

Fig. 5 shows the etching rates of the mesa strip structure in the strip direction of <110>. We can see that the etching rate of mesa strips in the Br : CH_3COOH etching agent greatly depends

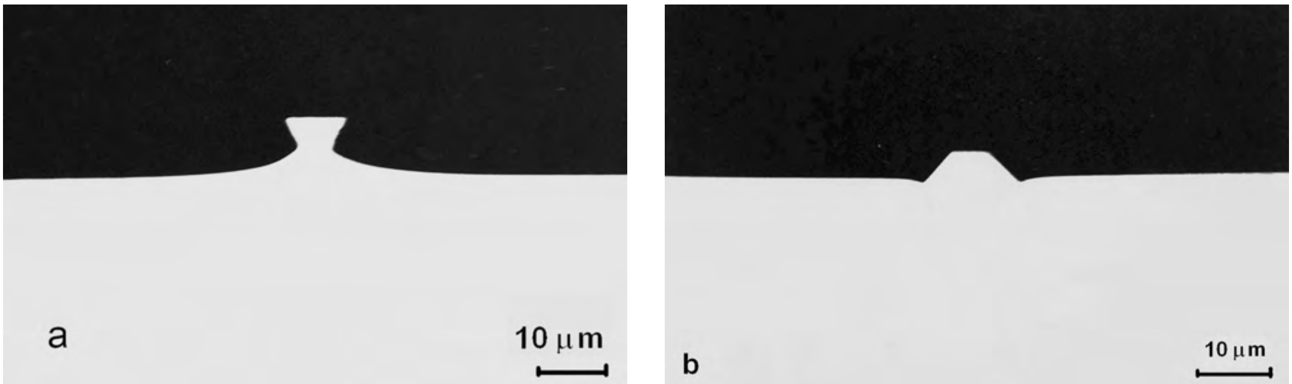

Fig. 2. Types of mesa strips depending on the orientation of the masking strips: a-[110], b-[Ī10]

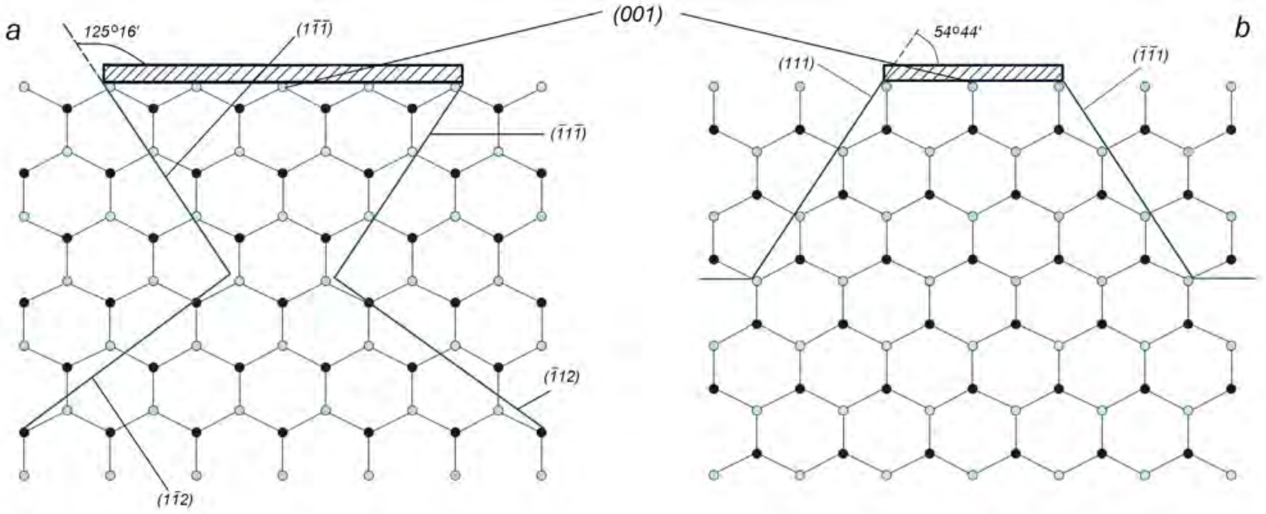

Fig. 3. Projection of the InP crystal structure on plane (110) – a and (Ī10) – b

on the mutual combination of planes cutting the mesa strip. It was also noted that the absolute values of the etching rates of the mesa strips changed their values when the etching agent was stirred, while the relative value of the rates remained constant.

Diffusion is important for determining the etching rate and can conceal the difference in the absolute rates of some low-index planes, although it cannot hide the insufficient reactivity properties of planes {111}A. We can also assume that masking coatings are highly significant both for diffusion processes during the formation of mesa strips and for their burying using the method of liquid-phase epitaxy.

A laser heterostructure was grown on the substrates of indium phosphide with the p-type conductivity. At first, an initial heterostructure was grown, an InP layer with the p-type conductivity, then an undoped layer of the GaInAsP solid solution and an InP layer with the *n*-type conductivity doped to the concentration of 1×10×19 cm^{-3}. After that, a mesa strip in the form of a fishtail was formed. A protruding mesa strip was buried with two layers of InP of the *n*-type and p-type conductivity respectively. These layers were “blocking layers” that limited the the flow of the current outside the mesa strip, so the pumping current of a laser diode flows only along the mesa strip at the lowest value outside the strip. Such limitation of current allows obtaining a laser diode with low operating current and good optical limitation of light emitted by the diode. The use

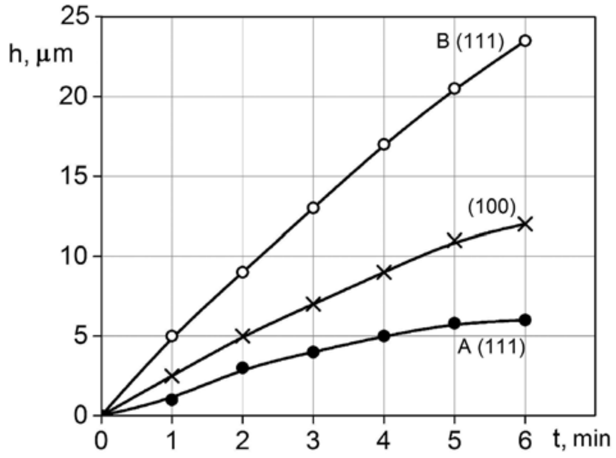

Fig. 4. Changing the rate of InP etching depending on the orientation of the substrate

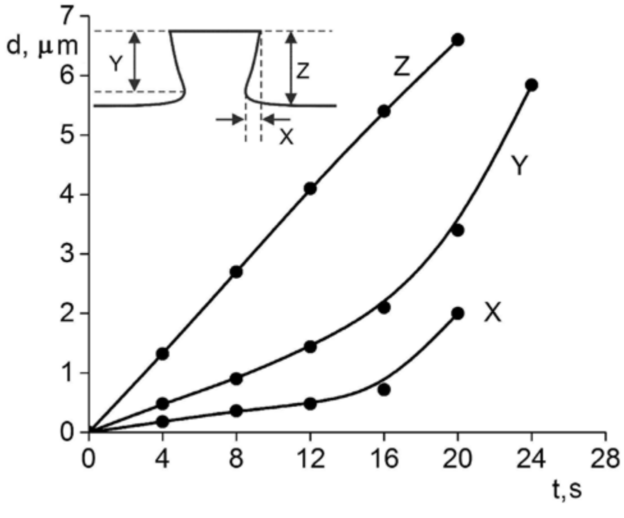

Fig. 5. Etching rate for mesa strips along the direction of [110] in the etching agent Br : CH_3COOH

of the InP substrates with the p-type conductivity allowed reducing the leakage currents outside the mesa strip and avoid using an additional contact layer of the GaInAsP solid solution required for the creation of an ohmic contact of the laser diode [21]. Burying was conducted in the temperature range of 620–600 °C with different degrees of supercooling of the solution-melt. A disadvantage of this structure is low reproducibility of the narrow part of the “fishtail” (active area of the laser diode) due to adsorption of the Br : CH_3COOH etching agent on the surface of the masking coating and its further diffusion into the etching area free from the masking coating. The same adsorption is also observed in the form of “excrescences” of crystals along the mesa strip during the process of growth from the solution-melt. These excrescences had a negative effect on the further formation of chips of laser diodes.

3. Results and discussion

During the experiments, it was shown that the masking coating must be removed from the surface of the mesa strip and epitaxial burying must be conducted without a masking coating in order to eliminate the adsorption.

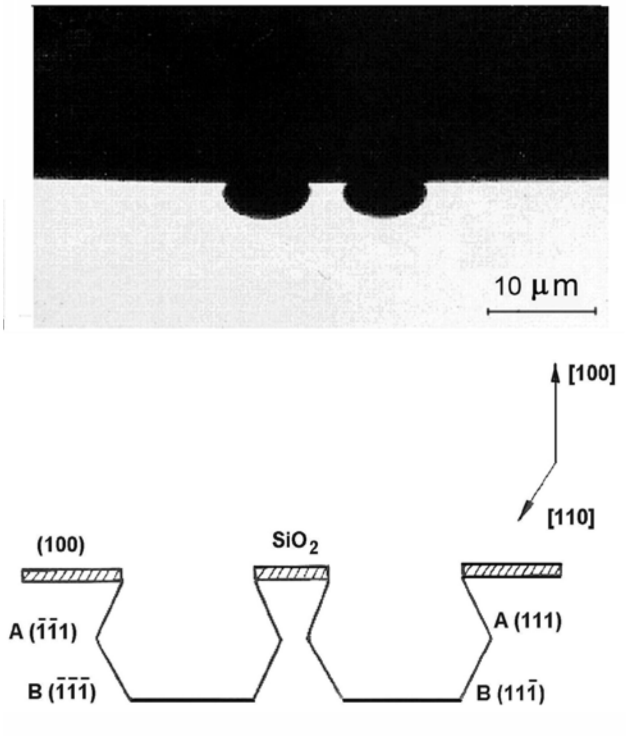

Fig. 6a Two-channel mesa strip

The experiment was conducted on the InP substrates with the p-type conductivity. Two barrier layers *n*-InP and p-InP were grown sequentially. It was noted that there was no growth on the surface when the mesa strip without a masking coating was less than 6 µm wide. This effect can presumably be explained by the fact that the growth of a more densely packaged plane (111)B requires a larger amount of InP as compared to plane (100). Therefore, InP actively moves towards plane (111)B in the solution-melt and is completely weakened above the surface of the mesa strip with the specified growth modes.

3.1. Creating and burying a two-channel laser strip structure

From a practical perspective, mesa strips with the minimum sizes of the radiation area of a future device are of special interest. For this reason, we considered an option of a mesa strip limited by two etched channels, see Fig. 6 (a, b). This structure of a mesa strip allows creating the “top” of a strip with a size less than 1–2 µm, which is highly important for the future laser structure, as this size is responsible for the size of the active area of the laser diode, and the size of 1.5–2.5 µm is considered optimal for lasers with a spectral range of wavelengths 1210–1650 nm.

A photoresist was used to create a two-channel mesa-strip structure, which considerably simplified the process of formation of the two-channel structure. A photoresist, placed using a standard photolithography method, was left on the surface after the formation of a pattern.

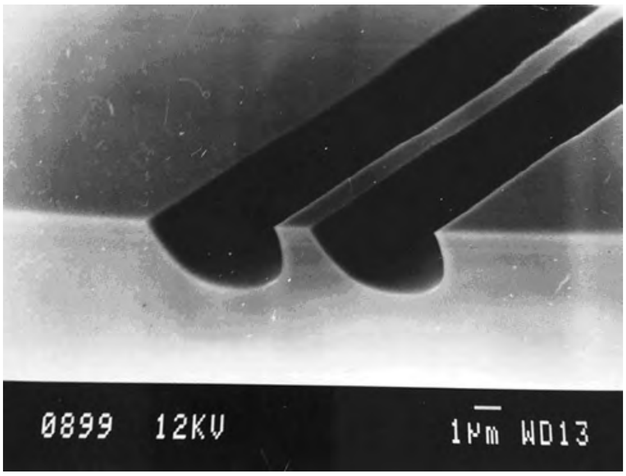

Fig. 6b Two-channel mesa strip structure after etching in the Br : CH_3COOH = 1:9 mixture

A two-channel mesa strip structure was formed on the InP (100) substrate with the direction of the strips [110] by chemical etching in the Br : CH_3COOH = 1 : 9 mixture. After the formation of the two-channel mesa strip, the photoresist was removed from the profiled surface (Fig. 6b).

In this case, there is no classic cut with planes (111)A; $(1\bar{1}\bar{1})$A; $(\bar{1}\bar{1}\bar{1})$B; $(11\bar{1})$B, and (100) , and the etching profile of the two-channel structure has a smooth transition between planes $(\bar{1}\bar{1}\bar{1})$B; $(11\bar{1})$ B, and (100). This smooth transition allowed levelling the growth rates of indium phosphide and GaInAsP solid solutions between the planes with a high index of the cut of the channels.

The buried two-channel laser structure on the InP/InGaAsP heterojunction was created in three stages: the first stage was growing the initial laser heterostructure InP/GaInAsP, the second stage was etching the mesa strips and removing the protective coating from the photoresist, and the third stage was growing blocking layers on the mesa strip structure.

At the first stage of LPE, the following layers were grown on the *n*-InP (001) substrate.

- An emitter layer of *n*-InP doped with Sn, $n = 2\cdot10^{18}$ cm^{-3}, 7 μm thick.

– An active layer of InGaAsP, undoped, 0.15 μm thick.

– An emitter layer of *p*-InP doped with Zn, $p = 3\cdot10^{17}$ cm^{-3}, 0.5 μm thick.

- A technological layer of InGaAsP, undoped, 0.5 μm thick.

At the second stage, a mesa strip was formed under the mask of the photoresist along direction <110> using photolithography. The geometry of the strip before burying had the following sizes: width – 2.0–2.5 μm, height and width of side channels 7 μm each – 3 μm. After that, the photoresist was removed, and the profiled structure was sent to burying (Fig. 7b).

At the third stage of LPE, the mesa strip without a coating was buried with the blocking layers of *p*-InP and the layer of *n*-InP. Due to the set orientation, the selected geometry of mesa strips, and the controlled technological mode of growth from the liquid phase in the quasi-equilibrium mode, the blocking layers were grown only in the channels and on the planar section of the surface between the channels, but there was no growth on the mesa strip itself. Then the next blocking layer of *p*-InP and a contact layer of *p*+ InGaAsP were grown, which made the surface of the two-channel buried heterostructure almost planar. The buried InP/GaInAsP two-channel laser heterostructure is presented in Figs. 7a and b.

After that, a part of the substrate was removed by chemical-dynamic polishing [22] and ohmic contacts were deposited: Au + Sn on the *n*-side, Au + Zn on the p-side. The planar structure was cleaved into separate chips 400 μm wide and 350 μm long. The chips were mounted on a copper cold conductor using an indium solder, and the parameters of laser diodes were measured.

Laser diodes made from the chips of the buried two-channel structure had the following parameters:

– The threshold current was ~10÷15 mA at 25 °C.

– The differential quantum efficiency was ~ 50 %.

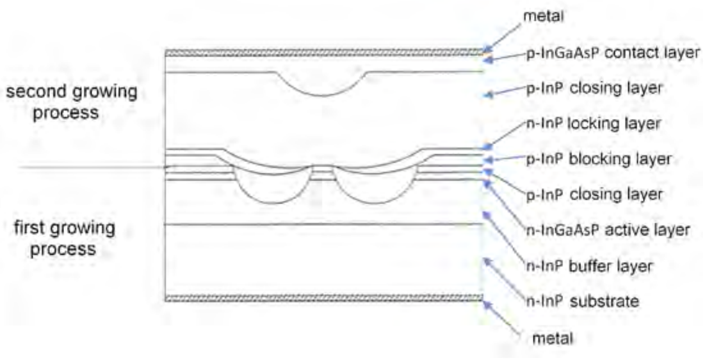

Fig. 7a A schematic representation of the buried two-channel laser heterostructure InP/GaInAsP

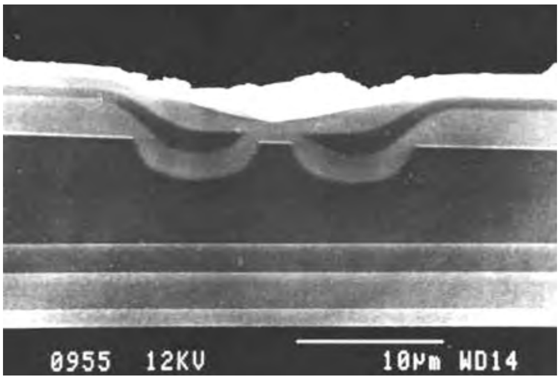

Fig. 7b Microphoto of the buried two-channel laser heterostructure InP/GaInAsP

– The linearity of watt-ampere characteristics was observed up to a power of ~ 20 MW.

– The radiation wavelength was 1320 nm.

– The yield ratio was ~ 30 %.

4. Conclusions

We studied the mechanisms of the formation of profiled surfaces of indium phosphide with different orientations of a substrate and mesa strips. We determined the modes of creation of profiled surfaces for obtaining laser diodes in the wavelength range of 1210–1650 nm. The modes of growing laser heterostructures on the InP profiled surfaces were also studied. We proposed a Russian technology for the creation of laser diode chips using Russian equipment. It was shown that the conditions of nonequilibrium growth of thin layers from the liquid phase on a profiled surface allowed conducting unique processes of local growth.

Based on the buried two-channel laser heterostructures we obtained highly efficient Russian laser diode chips working in a continuous mode of generation. The suggested technology allows obtaining laser diodes in the wavelength range from 1210 to 1650 nm. The diode chips can be used in fibre optic communication channels, environmental monitoring systems, and other quantum electronics devices.

We studied the processes of creation of buried laser mesa structures InP/GaInAsP on the profiled surfaces of indium phosphide.

It was shown that the set orientation of mesa strips along the directions of <110> with the orientation of the substrates on plane (001) is highly important for obtaining these structures. We determined optimal etching agents and etching modes for creation of the profiled surface of indium phosphide. We discovered masking coatings for the creation of a complex profiled relief of the surface of indium phosphide. We described an original technology for the creation of an InP/GaInAsP two-channel laser heterostructure on the profiled surface without a protective growth coating with the width of the active area of 1.5-2.5 μm, which allows obtaining single-mode laser diodes with high quantum efficiency.

Contribution of the authors

All authors have contributed equally to the publication.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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Received 16 February 2021; Approved after reviewing 30 March 2021; Accepted for publication 15 May 2021; Published online 25 June 2021.

Translated by Valentina Mittova
Edited and proofread by Simon Cox