Для можливості формування наноструктурованих шарів на поверхні напівпровідників із регульованими властивостями розроблено морфологічний критерій якості. Отримано шари низькопоруватого фосфіду індію з мезопоруватою структурою. Поруваті шари формувалися методом електрохімічного травлення у розчині соляної кислоти при постійній щільності струму. За розробленим критерієм проаналізовано якість синтезованих зразків por-InP. Це дозволить виготовляти структури з поруватими шарами на поверхні у промислових масштабах. Представлений критерій може бути застосованим для інших режимів обробки фосфіду індію, або для інших напівпровідників. Це дозволяє розглядати його як універсальний морфологічний критерій якості поруватих структур. Встановлено кореляцію між морфологічними властивостями поруватих структур на поверхні фосфіду індію та умовами травлення. Для цього було проаналізовано поруваті структури, які формувалися у інтервалі часу травлення від 10 до 20 хв при різній концентрації кислоти у електроліті. У результаті встановлено, що форма пор наноструктурованих шарів на поверхні напівпровідників залежить не лише від параметрів кристалу, а й від умов травлення, зокрема від часу травлення та складу електроліту. Застосування насичених електролітів призводить до формування масивних пор, які мають форму канавок – витягнуті еліпси. Отримані кореляції є корисними з практичної точки зору, тому що дозволяють обґрунтовано підходити до визначення режимів електрохімічної обробки напівпровідників. Крім того, це відкриває нові перспективи у побудові моделі самоорганізації поруватої структури на поверхні напівпровідників. Представлено методику розрахунку основних статистичних характеристик ряду розподілу пор за розміром, зокрема розмах варіації, дисперсію,середньоквадратичне відхилення, коефіцієнти варіації та асиметрії. Це дозволяє більш детально оцінювати морфологічні показники поруватих структур та просунутися у розумінні механізмів, що лежать в основі пороутворення на поверхні напівпровідників під час електрохімічної обробки

Ключові слова: фосфід індію, електрохімічне травлення, морфологічні показники, поруваті напівпровідники, критерій якості

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FORMING THE LOW-POROUS LAYERS OF INDIUM PHOSPHIDE WITH THE PREDEFINED QUALITY LEVEL

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1. Introduction

Nanostructured semiconductors are of interest [1, 2] due to the possibility of their application in photonics and microelectronics [3, 4]. Thin films [5], nanowhiskers [6], quantum spots [7], nanograins [8], etc. are widely used nowadays. A variety of forms and types of nanostructures gives rise to the problem of establishing a unified approach to their classification and determining criteria to assess nanomaterials. One of the promising directions is nanostructuring semiconductor surfaces with the view to forming a porous layer [9, 10]. Porous structures are obtained on the surface of indium phosphide [11, 12], gallium phosphide [13, 14], gallium arsenide [15, 16], silicon [17], germanium [18], etc. Nanostructures, formed on the surface of these semiconductors, demonstrate a variety of shapes, dimensions, and number of nanoobjects. On the one hand, it extends the limits of application, on the other hand, leads to difficulties associated with the development of the criterion apparatus of evaluation of nanostructures quality indicators. The interest in these structures was caused primarily by an increase in the area of effective surface [19]. This makes it possible to use these structures as material for creating photoelectric energy transducers [20]. The search for ways of unifying approaches to determining morphological indicators of porous structures, which will enable standardization of the requirements for nanomaterials, is relevant. In addition, existence of quantum-dimensional effects is observed in porous structures [21]. This property causes a shift of photoluminescence peaks to the short-wave part of the spectrum [22]. This effect becomes useful for application of porous layers in the laser technology [23, 24]. However, no general mechanism of pores formation on the surface of semiconductors has been determined up to now. The influence of the factors that determine the surface micromorphology of the obtained structures has not been studied enough either.

Another feature of porous structures is the ease of synthesis [25, 26]. As a rule, such structures are synthesized by the chemical [27], electrochemical [28] or lithographic [29] technologies. Today, usual electrochemical etching remains the most common method [30]. This method is well-researched and enables getting porous layers of various configurations [31, 32]. The main problem with the synthesis by the electrochemical technology is obtaining structures with adjustable properties [33]. First of all, it concerns morphological characteristics of porous structures, as they determine the functional purpose of nanomaterial. That is why research that is related to establishing quality criteria of porous nanomaterials and finding conditions, under which synthesis of porous structures with specific properties becomes possible, is relevant. In addition, it is extremely important to determine the correlations between conditions of synthesis of nanostructures and basic morphological indicators. Such studies are necessary, above all, to create standards and regulations that will make it possible to regulate the properties of nanostructures at the stage of their synthesis.

2. Literature review and problem statement

In paper [34], the authors examine the growth of a porous structure towards the depth of monocrystalline indium phosphide. It was shown that the orientation of pores in the volume of a crystal depends on etching rate and crystallographic orientation of InP. However, in paper [34], the influence of etching rate on the cross section of porous holes was not studied. In the paper [35], the authors proposed to obtain high-quality samples of porous indium phosphide with adjustable properties using photolithography. This approach provides a regular porous layer in the established areas of the surface. In paper [36], it was also proposed to use a photolithographic window for the formation of a regular porous structure. The only drawback of the proposed technology can be related to its cost and complexity of the technological operations. The authors of article [37] observed the effect of the applied potential on the morphology of porous layers, formed on the surface of monocrystalline indium phosphide. However, it is known from works [38, 39] that not only applied potential determined a microrelief of a porous surface. Thus, paper [38] studies boundary strain at the beginning of pore formation on the surface of semiconductors. However, it is not entirely clear, which factors are responsible for the value of this magnitude. Article [39] shows that dependence of morphological characteristics of porous indium phosphide on the type of electrolyte, which takes part in dissolving a semiconductor surface. However, the dependence on the concentration of acid in a solution of electrolyte was not determined. In research [40], it is reported that dimensions of pores are influenced by various factors, including etching condition and characteristics of the original crystal. However, these data are insufficient to reveal common mechanisms of pore formation and establish the conditions, under which formation of porous structures with specified properties becomes possible. In paper [41], it was shown that it is necessary to perform quality control of nanomaterials primarily for industrial applications. However, no major morphological criteria of the quality of porous structures on the surface of semiconductors were determined. In research [43], porous semiconductors were used to create photosensors, based on them. However, no criteria of quality of porous materials, which will make it possible to use them in the industrial scale, have been determined so far.

That is why the problems of the criterion apparatus of quality of porous semiconductors, adjustability of their properties and establishing the modes, under which synthesis of structures with specific characteristics becomes possible, remain unresolved. Insufficiently determined correlations between etching conditions and morphological characteristics of nanostructures cause problems in creation of the materials with adjustable properties. This largely inhibits the industrial application of porous semiconductors and causes the need for research into the quality control of nanostructured materials in the process of synthesis.

3. The aim and objectives of the study

The aim of this study is to develop a morphological criterion of quality of porous structures and to obtain porous layers of indium phosphide of the specified quality level.

To accomplish the aim, the following tasks have been set:

– to develop the morphological criterion of the quality of porous layers, synthesized on the surface of semiconductors;

– to assess morphological properties of por-InP by the quality criterion;

– to establish the correlation between etching conditions and the quality of the obtained structures.

4. Materials and methods, used for the synthesis and morphological analysis of properties of por-InP

4. 1. Examined materials and equipment, used in the experiment

Porous structures of por-InP were formed using the technology of usual electrochemical etching of monocrystalline Indium phosphide in hydrogen solution of hydrochloric acid. Before the experiment, the samples were cleaned in order to remove the mechanical and chemical pollutants. Then, the plates were immersed in the electrochemical cell with platinum on the cathode. Etching occurred at a constant current density of 150 mA/cm^2. Conditions of the experiment are shown in Table 1.

After the experiment, the samples were exposed to annealing in an ammonia solution with the aim of stabilization of their properties. The morphology of the obtained structures was studied on the raster electronic microscope JEOL-6490. Analysis of the main morphological characteristics was carried out using software ImageJ (USA) and OriginPro (USA).

Table 1

Conditions of synthesis of porous layers on the surface of indium phosphide

Sample number	Etching time	Electrolyte
1	10	$10H_2O$+1HCl
2	15	$10H_2O$+1HCl
3	20	$10H_2O$+1HCl
4	10	$10H_2O$+3HCl
5	15	$10H_2O$+3HCl
6	20	$10H_2O$+3HCl
7	10	$10H_2O$+5HCl
8	15	$10H_2O$+5HCl
9	20	$10H_2O$+5HCl

4. 2. Procedure for determining the indicators of quality of the synthesized porous layers at the surface of indium phosphide

To optimize the process of synthesis of nanostructures with specified parameters, it is advisable to use the criterial approach [44]. The general quality criterion should contain all the analyzed characteristics of samples – partial quality criteria. Convolution of quality criteria will be carried out by the linear law:

$$K=a_1k_1+a_2k_2+a_3k_3, \tag{1}$$

where a_1, a_2, a_3 are the weight coefficients; k_1, k_2, k_3 are the partial quality criteria.

We well consider the following quality condition:

$$K \to \max. \tag{2}$$

Among a number of morphological characteristics of porous nanostructures, we will select those that most accurately describe the surface micromorphology of samples. These characteristics include:

– surface porosity;
– diameter of pores;
– shape of pores.

By surface porosity of samples, we will imply the ratio of the area, occupied by
pores to the total area of the sample:

$$P=\frac{s_p}{s}, \tag{3}$$

where S_p is the total area of the surface, occupied by pores; S is the total area of the sample.

By the diameter of pores, we will assume arithmetic mean value of all the pores in the sight of the microscope.

The shape of pores will be characterized by the magnitude that is called the shape factor, or the pour roundness:

$$F=\frac{4\pi s_p}{p^2}, \tag{4}$$

where s_p is the area of a pore; p is the perimeter of a pore.

The value of shape factor F_{sh}=1 indicates that cross section of a pore is an ideal circle. The closer to 0 the value of roundness is, the more elongated or deformed the cross section of a pore.

The formula for calculations of partial quality criteria are shown in Table 2.

Table 2

Indicators that characterize partial criteria of quality of porous structures, formed on a surface of indium phosphide

Indicator	Calculation formula	Note
Indicator, which characterizes surface porosity	$k_1=\frac{P}{P_{st}}$, if P is smaller than standard value; if P is larger than standard value	P – surface porosity of samples, %; P_{st} – standard porosity, %
Indicator that characterizes the shape of pores	$k_2=\frac{F}{F_{st}}$	F – factor of the pore shape; F_{st} – standard value of shape factor
Indicator that characterizes the shape of pores	k_3=1 if d gets into standard range; $k_3=\frac{\lvert d-d_{\min}\rvert}{\lvert d-d_{\max}\rvert}$, is d is smaller than the lower boundary of standard range; $k_3=\frac{\lvert d-d_{\max}\rvert}{\lvert d-d_{\min}\rvert}$, if d is larger than the upper boundary of standard range	d – average diameter, μm; $d_{\min}$ – minimum permissible value of diameter of pores, μm; $d_{\max}$ – maximum permissible value of diameter of pores, μm

The values that correspond to structures with low density of pores (surface porosity) and to micropores of round cross sections will be accepted as the standard values of morphological characteristics of the porous layer, formed on the surface of indium phosphide (Table 3)

Table 3

Standard quality indicators nanostructures, formed on the surface of indium phosphide

Indicator	Standard values
Porosity	30 %
Diameter of pores	50...100 μm
Shape factor	1

The value of weight coefficients must satisfy the requirements:

$$a_1+a_2+a_3=1. \tag{5}$$

The weight coefficients were determined from considerations that for industrial use of porous structures, values of surface porosity are most important; the shape and dimensions of pores are less important. That is why we will accept:

$$a_1=0,4;\ a_2=0,3;\ a_3=0,3. \tag{6}$$

Provided we use the values of weight coefficients (6), condition (5) is met.

5. Results of research into morphological characteristics of porous layers of InP

5. 1. Results of research into porous layers at the surface of indium phosphide and separation of reference sample

Based on the results of raster electronic microscopy, it was established that all the studied samples after electrochemical treatment in the solution of hydrochloric acid had a porous layer on the surface. Fig. 1 shows the morphology of one of the examined samples (sample No. 5).

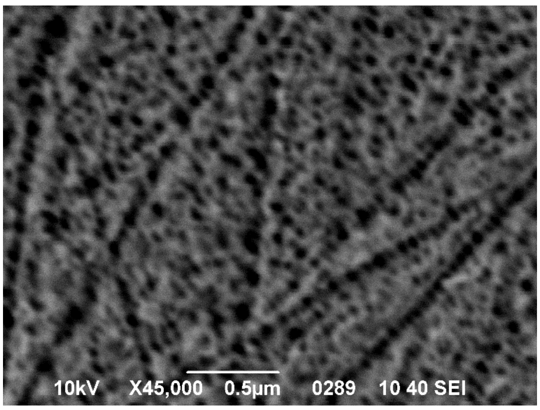


Fig. 1. REM-image of morphology of por-InP (sample No. 5)

Visual analysis of Fig. 1 makes it possible to see that an orderly assemble of pores was formed on the surface of monocrystalline indium phosphide under specified etching conditions. Elongated chains of pores are a consequence of existence of defects, which became the source of primary etching pits, on the surface of the original sample. The pores on these sections are more massive than on the faultless areas. In general, such porous layer can be considered conditionally qualitative. To give a more detailed description of morphological characteristics, it is necessary to make analysis in the ImageJ program. This program makes it possible to determine the number of pores and their main characteristics. Fig. 2 shows the histogram of distribution of pores by diameter. Table 4 shows the basic statistical characteristics of a series of pores distribution according to dimensions.

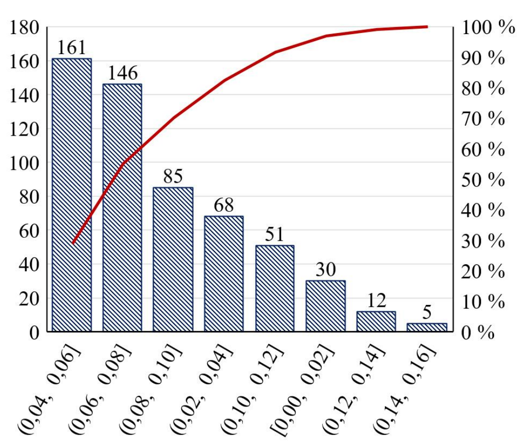


Fig. 2. Histogram of pores distribution according to dimensions of sample no. 5, plotted in program Origin based of the data, obtained with the help of ImageJ

Table 4

Basic statistical data of a series of pores distribution according to dimensions for sample No.5 (based on microphotography, shown in Fig. 1)

Parameter	Value	Notes
Number of pores	558	Within the sight of a microscope
Arithmetic mean value	0,0683 μm	Averaged value of diameter of all pores in the sight of a microscope
Mode	0.071364995 μm	Value of diameter of pores, most often found in this series of values
Median	0.071364995 μm	Value of diameter that divides the series into two
Variation span	0.156407458 μm	Difference between maximum and minimum values of diameters of pores of a series
Dispersion	0.000821	Measure of spread near its mean value (measure of dispersion, i.e. deviation from the mean)
Root-mean-square deviation	0.0286	It shows how much each value of the series is different from the mean value
Variation factor	41.92 %	Measure of relative spread of totality values: shows what part of mean value of diameter is made up by its mean spread
Moment asymmetry coefficient	4,254	Characterizes the degree of asymmetry of the series

The data, shown in Fig. 2 and in Table 4, make it possible to see that mode and the median of the series of pores distribution by the diameter converge and exceed the mean value (arithmetic mean). This can indicate the right-side asymmetry of the series. To prove this hypothesis, moment coefficient of the series was calculated from formula:

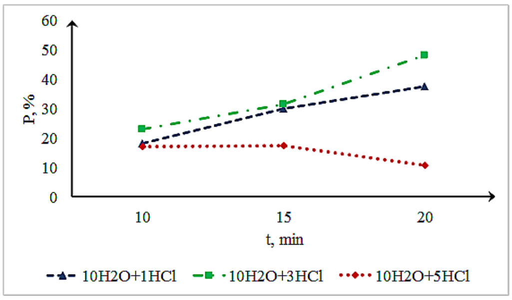
$$A_s = \frac{M_3}{s^3}, \qquad (7)$$

where M_3 is the central moment of the third order; s is the root-mean-square deviation.

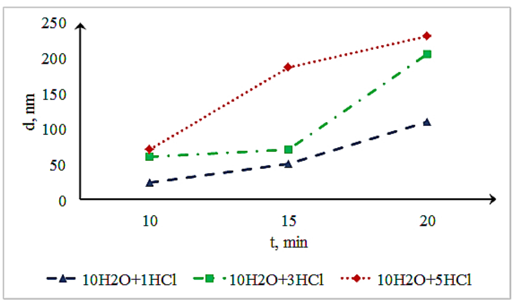
The positive magnitude of moment asymmetry coefficient indicates the right-side asymmetry and proves our hypothesis. This means that there are more pores with the diameter that is higher than the average one than with the value below the mean value. This result indicates that the etching process is not at the initial stage, all pores have been formed up to this moment, and nucleus pores reached their mean values. Analysis of all samples was conducted by the same principle.

Fig. 3, *a–c* demonstrates the value of porosity, average diameter of the pores and shape factor for all the studied samples at different composition of the electrolyte.

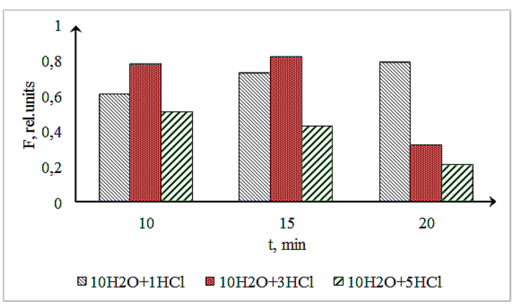
Analysis of Fig. 3, *a–c* makes it possible to see the correlation between morphological characteristics of the synthesized porous layers and etching conditions. The time of etching causes an increase in transverse diameter of a pore. In addition, the time of etching causes an increase in surface porosity. It is necessary to pay attention to the fact that under too severe conditions (electrolyte $10H_2O+5HCl$), starting at minute 15 of etching, porosity does not increase but decreases. This can be explained by the effect of separation the porous space from the substrate surface and its scattering into a solution of electrolyte. Thus, the sample surface is polished. Porous layers with pores of almost round shape are formed in moderate solutions of electrolyte ($10H_2O$+1HCl and $10H_2O$+3HCl), while an increase in content of hydrochloric acid in the electrolyte solution leads to the formation of massive pores of irregular shape. This proves etching of surface defects and their proliferation on the surface of the samples.



a



b



c

Fig. 3. Dependence of the basic morphological characteristics on etching time for different compositions of electrolyte: *a* is the value of surface porosity; b is the value of average diameter of pores; *c* is the value of shape factor

5. 2. Results of determining the morphological criterion of quality of por-InP samples

Based on the data, shown in Fig. 3, *a–c*, we will calculate the value of partial criteria of quality of the samples of indium phosphide with a porous layer on the surface (Table 5). Table 6 presents calculation of morphological criterion of quality.

Based on the results of Tables 4, 5, it can be argued that the sample that corresponds to the specified quality level is sample No. 5. From this, we can infer that porous layers of indium phosphide with specified characteristics (Table 3) should be formed within 15 min in a solution of electrolyte $10H_2O$+3HCl. The results, obtained for samples No. 2 and No. 4, are also considered admissible.

Table 5

Partial criteria of por-InP samples

Sample number	P, %	*d*, µm	*F*	k_1	k_2	k_3
1	18.1	24	0.61	0.6	0.34	0.61
2	29.8	51	0.73	0.99	1	0.73
3	37.5	109	0.79	0.8	0.15	0.79
4	22.8	61	0.78	0.76	1	0.78
5	31.2	71	0.82	0.96	1	0.82
6	47.9	205	0.32	0.63	0.68	0.32
7	16.9	70	0.51	0.56	1	0.51
8	17.2	187	0.43	0.57	0.64	0.43
9	10.5	231	0.21	0.35	0.72	0.21

Table 6

Calculation of morphological criterion of the quality of the studied samples

Sample number	a_1k_1	a_2k_2	a_3k_3	$K=a_1k_1+a_2k_2+a_3k_3$
1	0.24	0.102	0.183	0.525
2	0.396	0.3	0.219	0.915
3	0.32	0.045	0.237	0.602
4	0.304	0.3	0.234	0.838
5	0.384	0.3	0.246	0.93
6	0.252	0.204	0.096	0.552
7	0.224	0.3	0.153	0.677
8	0.228	0.192	0.129	0.549
9	0.14	0.216	0.063	0.419

6. Discussion of results of studying the morphological criterion of quality of nanostructures

Development of morphological criterion of quality of nanostructured porous layers of indium phosphide was based on the assumption about the dependence of the functional purpose of nanostructures on micromorphological properties of the surface. The presented criterion can be applied to other modes of treatment of indium phosphide or for other semiconductors. This makes it possible to treat it as a universal morphological criterion of quality of porous structures. However, we can but note that this criterion contains only three basic surface characteristics and does not take into account the others. In addition, for the industrial use of nanostructured semiconductors, it is often necessary to take into consideration not only morphological indicators of quality, but also chemical, mechanical, and radiation ones. Not taking into account these indicators could be interpreted as a drawback of this work. However, this opens up the prospects for further research into development of the generalized criterion of quality of nanostructures on the surface of semiconductors.

In addition, during the development and determining the morphological criterion of por-InP quality, interesting findings on correlations of morphological properties of InP-por and etching time were obtained. Such studies are not new, in particular, similar results were demonstrated in paper [42]. However, unlike the research results, obtained in [42], the obtained correlations make it possible to trace dependences not only of surface porosity and dimensions of pores, but also of an important indicator of the quality, such as pores shape factor.

The obtained data on the influence of the time of electrochemical treatment on the shape of pores make it possible to state the following:

– the shape of pores of the nanostructured layers on the surface of semiconductors depends not only on parameters of a crystal, but also on etching conditions, specifically, time of etching and the composition of the electrolyte;

– application of saturated electrolytes leads to the formation of massive pores, which have the shape of grooves – elongated ellipses.

The following conclusions may be feasible from the practical point of view because it makes it possible to approach reasonably determining the modes of electrochemical treatment of semiconductors. From the theoretical point of view, they open new prospects in the construction of models of self-organization of a porous structure on the surface of semiconductors.

However, we cannot but note that the results of determining the correlation between etching time and the basic morphological indicators have an ambiguous impact, it is the case of autocorrelation. That is, we can conclude that micromorphology of porous layers on the surface of semiconductors is influenced by many factors, taking into consideration of which allows controlling the processes of structure formation on the surface of semiconductors.

7. Conclusions

1. The morphological criterion of quality of porous layers on the surface of semiconductors was developed. It was shown that this indicator should include assessment of the quantity, dimensions and shape of pores. This approach makes it possible to select from the lot of samples the ones that satisfy the established quality level. The standard indicators of quality of mesoporous indium phosphide were established, specifically: porosity – 30 %, diameter of pores – 50...100 μm, shape factor – 1. The quality factor should tend to 1.

2. The assessment of the porous layers of indium phosphides, formed in a solution of hydrochloric acid, was carried out by the developed morphological quality criterion. Calculation of the morphological criterion of quality of the studied por-InP samples showed that the structures, formed within 15 min in a solution of electrolyte $10H_2O+3HCl$, corresponds to the established quality level (low-porous surface with mesopores of a round shape). For them: porosity P=31.2 %, diameter of pores d=71 μm, shape factor F=0.82, quality coefficient K=0.93.

3. The correlation between morphological properties of synthesized porous layers of indium phosphide and etching conditions was studied. It was shown that the shape, dimensions and number of pores depend on etching time and electrolyte composition. In this case, there are critical values of etching time and the electrolyte concentration, at which a porous layer is separated from the substrate and electrochemical polishing of a crystal occurs. When using the solution of electrolyte $10H_2O+5HCl$, beginning at minute 15, a porous layer is separated from the monocrystalline substrate and scatters in the electrolyte solution. The obtained results indicate a possibility of synthesis of nanostructures with adjustable properties of the specified quality level.

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