

RESEARCH INTO REGULARITIES OF PORE FORMATION ON THE SURFACE OF SEMICONDUCTORS

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Розроблено схему керування процесом формування поруватих шарів на поверхні напівпровідників методом електрохімічного травлення. Показано, що побудована схема може бути застосована для різних випадків синтезу наноструктурованих напівпровідників. Досліджено процеси, що лежать в основі пороутворення і визначають морфологічні властивості наноструктур. Досліджено відношення падіння потенціалу в шарі Гельмгольца. Виділено основні морфологічні критерії якості поруватих наноструктур для застосування їх у сонячних батареях. З урахуванням цих критеріїв було отримано поруваті простори на поверхні напівпровідників АЗВ5

Ключові слова: якість наноструктур, електрохімічне травлення, поруваті напівпровідники, шар Гельмгольца, морфологія, напівпровідники

Разработана схема управления процессом формирования пористых слоев на поверхности полупроводников методом электрохимического травления. Показано, что построенная схема может быть применена для различных случаев синтеза наноструктурированных полупроводников. Исследованы процессы, лежащие в основе порообразования, которые определяют морфологические свойства наноструктур. Исследовано относительное падение потенциала в слое Гельмгольца. Выделены основные морфологические критерии качества пористых наноструктур для применения их в солнечных батареях. С учетом этих критериев были получены пористые пространства на поверхности полупроводников АЗВ5

Ключевые слова: качество наноструктур, система управления, электрохимическое травление, пористые полупроводники, слой Гельмгольца, морфология, полупроводники

1. Introduction

Interest in nanostructures is associated with the possibility of substantial modification of the properties of known substances, as well as new opportunities opened up by nanotechnologies in creating materials and products from the structural elements of nanometer range. Current state of the Ukrainian economy and industry in the global market requires fundamental improvement of production processes at Ukrainian enterprises. Ukrainian market is constantly supplied with imported goods. Only high-quality Ukrainian products are capable of competing with them at decent level.

However, this necessitates a change in the underlying principles in the production management processes at Ukrainian enterprises, as well as creation of quality control systems, similar to those that exist in developed countries.

Ukraine possesses potential in the field of renewable energy sources, in particular, solar energy. In this context, particularly relevant are the tasks on improving the quality of raw materials for solar cells [1–3]. Increasingly, for these purposes, nanostructured materials have been employed [4, 5]. In addition, relevant and timely is the development of electronic and micro-electronic technology. Modern electronic devices are created based on promising advanced

materials that are used in many of the most important production sectors, such as aviation, space, atomic, energy engineering, etc. Development of the technology for manufacturing microelectronic devices is aimed, above all, at reducing geometric dimensions of microcircuits. Modern means for the execution of technological operations make it possible to receive elements with the size in submicron range. Control over geometric dimensions of materials and components correlates with the development of nanotechnological methods for obtaining new materials.

At present, there are different types of nanostructures: quantum wire, quantum dots, nanotubes, fullerenes, graphenes, thin films, etc [6, 7]. A special place in this case is occupied by porous semiconductors, which have been employed as:

- raw materials for the fabrication of solar cells;
- buffer layers for the epitaxial growth of heterostructures;
- material for the manufacture of gas sensors;
- material for creating light-emitting structures, etc.

Expediency of choosing porous semiconductors as the object of modification is predetermined by the variety of morphological types [8]. That is why formation of electron nano-objects based on the modified semiconductor structures is a relevant task.

2. Literature review and problem statement

There are various techniques to obtain porous structure at the surface of semiconductors. However, the most widely applied is the method of electrochemical etching. This is predetermined by the ease and affordability of the method. Porous layers at the surface of silicon [9, 10], germanium [11], gallium arsenide [12, 13], gallium phosphide [14], indium phosphide [15, 16], etc, were formed by the method of electrochemical etching.

Modifications of this method have been actively developing at present. Thus, in article [17], indium nitride was created using additional mode of etching – illumination of samples during anodizing. It resulted in the obtained porous layers that had a redshift of the edge of photoabsorption. The observed phenomenon is accurately explained by the Franz-Keldysh effect. Authors of paper [18] controlled a change in the size and shape of pores using a photolithographic window. Nanomembranes and nanowires formed at the surface of indium phosphide. It was shown that under potentiostatic conditions of etching morphology of the etched samples was highly dependent on the applied voltage. It was discovered that anodizing at 5...7 V leads to the creation of highly porous layers made of mechanically stable skeletons that demonstrate percolations. At the same time, dominant formation of nanowires was observed while increasing the applied voltage to 15 V. Membranes from nanoporous InP were formed for the purpose of growing based on nanowires of Co [19]. The membranes were formed by the method of electrochemical etching in four stages, each of which involved different electrolytes and modes of etching. Grown polycrystalline cobalt nanowires are characterized by a very small size of grain. Studies show a narrow hysteresis loop with dominant orientation in the direction of magnetization along the long axis of a nanowire. Because of this, there occurs anisotropy of cobalt nanowires. Mechanisms that occur at the border “semiconductor – electrolyte” were in-

vestigated in articles [20–23]. A multitude of approaches to the description of processes of pore formation during electrochemical treatment of materials necessitates systematization of this knowledge. That is why there is a need to establish principles and patterns that underlie control over the process of pore formation at the surface of semiconductors.

3. The aim and objectives of the study

The goal of present study is to establish physical and technological regularities in the formation of porous surface based on the semiconductors A3V5.

To accomplish the set goal, the following tasks had to be solved:

- to develop a scheme of control over the process of electrochemical dissolution of crystal as a part of the system of quality control of nanostructures;
- to explore the processes that take place at the border of the contact “semiconductor – electrolyte”;
- to establish physical and technological regularities of pore formation on the surface of semiconductors.

4. Materials and methods for examining the process of control over pore formation on the surface of semiconductors

4. 1. Examined materials and equipment used in the experiment of electrochemical treatment of crystals

For the given experiment we selected sets of semiconducting plates of gallium arsenide, gallium phosphide, and indium phosphide. Before the experiment the samples were polished from both sides and cleaned in a stream of atomic nitrogen. Nanostructures were formed by the method of electrochemical etching in the solutions of acids. Schematic of experimental setup is shown in Fig. 1.

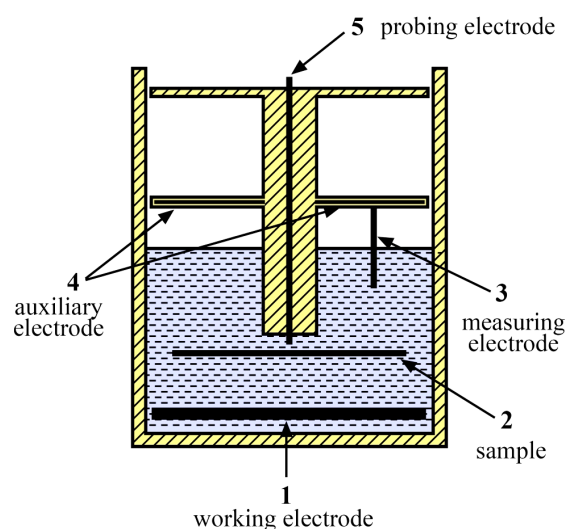


Fig. 1. Schematic of setup for electrochemical etching of semiconductors

Setup for etching consists of an indivisible base with a working electrode at the bottom and movable rod that is equipped with auxiliary, measuring and probing electrodes.

A semiconductor plate (examined sample) is pressed from one side to the working electrode, and from the other – to the probing one. Thus, the probing electrode performs dual function – it measures the potential in the centre of the sample and it presses it to the working electrode, thereby reducing the magnitude of resistance at transition sample/working electrode.

In order to establish the value of boundary voltage of the early pore formation, we applied the mode of gradual increase in the voltage of anodizing. Etching was carried out in the solution of hydrofluoric acid. To study morphological properties of nanostructures, we used a method of scanning electron microscopy.

4. 2. Construction of scheme of control over pore formation on the surface of semiconductors

When we deal with control over the process of electrochemical pore formation on the surface of semiconductors, we shall consider a general scheme of control over the process of electrochemical dissolution of crystal and its component – “semiconductor – electrolyte”. As the system “semiconductor – electrolyte” is exposed to many external factors, it is open.

The subject is the process of electrochemical treatment of crystals, and the object, that is, a controlled system, is considered to be the subsystem “semiconductor – electrolyte”.

The state of the controlled system depends on external influences, impacts from controlling element and the action of the controlled system itself (Fig. 2). The actions of controlled system will be understood as the processes of self-organization of nanostructures formation on the surface of semiconductors.

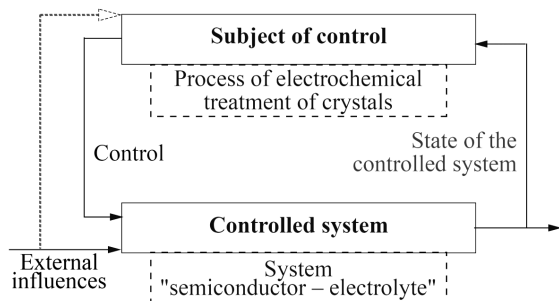


Fig. 2. General scheme of control over the process of electrochemical dissolution of crystal

The main task when controlling the process of electrochemical dissolution of crystal (CPEDC) is to execute such actions that will make it possible to provide the required state of the controlled system. In addition, in this case, we shall take into account information on external actions.

Detailed scheme with explanation of structural components and their interaction is shown in Fig. 3.

External environment should be understood as the totality of all objects/subjects that are not included in this system, and objects/subjects whose properties vary depending on the state of the system. A change in their properties affects the examined system. In our case, external influences include:

- illumination of the room (since under the action of photons the speed of pore formation increases);
- temperature of the electrolyte solution (depending on the type of anion, which takes part in the process of dissolving a crystal, the electrolyte is heated or cooled);

– purity of the experiment (the surface of the crystal typically contains active recombination centers that easily enter reaction with ions contained in the air), etc.

Thus, when controlling the process of pore formation on the surface of crystal, it is necessary to consider:

- pore formation conditions under which we understand the modes of electrochemical treatment of crystals;
- requirements put forward to the quality of received nanostructures; in this case, it is necessary to clearly define the main and the secondary criteria;
- mechanisms that underlie the process of pore formation.

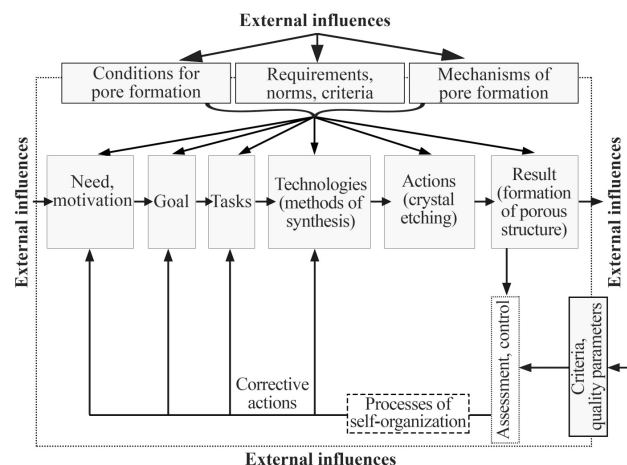


Fig. 3. Structural components of the detailed scheme of control over technological process of electrochemical dissolution of crystal

During etching of crystals, there start to manifest themselves the processes of self-organization of pore growth that occur at the border of the system “electrolyte – semiconductor”. In addition, according to a defect-dislocation mechanism, pore formation begins in the point of the surface where the point or dislocation defects exist.

Control over the process of electrochemical dissolution of crystal starts with a need that is caused by the necessity to create nanomaterials with required properties. This becomes possible only under condition of manageability of the process and understanding of the basic mechanisms underlying pore formation.

To satisfy the need, it is necessary to formulate a goal, which in this case is the formation of porous space on the surface of a semiconductor plate. Given the conditions, requirements, norms and operating principles, the goal is transformed into a set of tasks. Tasks are the establishment of such technological modes under which it becomes possible to obtain nanostructures with predictable and programmable properties.

Technology is a system of conditions, criteria and means of solving the set tasks. In this context, first of all, it is necessary to decide on the methods of synthesis of nanostructures, which include taking into account additional conditions and effects of external factors.

Next, one selects a certain action (or a set of actions), which, taking into account the influence of the external environment, leads to a specific result of the activity. The choice of actions is based on determining the stages in the formation of nanostructures and is predetermined by manufacturing as it is.

To evaluate the result, the received nanostructures are compared with the reference by the criteria defined in advance. In this case, selection of criteria is usually dictated by the goals of evaluation. Evaluation is made based on an analysis of examining the surface of the crystal. Desirable is the use non-destructive methods of control, which may include:

- scanning electron microscopy (SEM);
 - method of chemical analysis of the surface of the crystals (EDAX);
 - x-ray spectroscopy;
 - photoluminescence (FL), etc.
- Corrective actions include:
- heating/cooling of the electrolyte;
 - agitation of electrolyte;
 - illumination of samples during etching, etc.

5. Results of examining regularities that underlie pore formation on the surface of semiconductors

To understand the processes that underlie the pore formation of crystals, it is necessary to examine the boundary “semiconductor – electrolyte”. When controlling the process of electrochemical etching of crystals, it is necessary to determine the voltage, which triggers the processes of pore formation. Manageability of the process of pore formation is possible only under condition of determining the required criteria of structure quality. Criteria are selected for each case separately based on data on the resulting purpose of the nanostructure.

5. 1. Selection of criteria of nanomaterial quality and the conditions of pore formation

To control the process of pore formation on the surface of semiconductors, it is necessary to determine:

- desired parameters of nanostructures to be obtained;
- basic mechanisms that underlie pore formation;
- conditions of pore formation on the surface of crystal.

Desirable parameters of nanostructures to be obtained should be understood as unique properties that predetermine the choice of this material for the application with certain purposes. For example, solar panels are advisable to fabricate not from the mono-crystalline phase of a semiconductor but rather from plates that have developed surface morphology. This is caused by an increase in the effective area of semiconductor by hundreds of times. In addition, it is expedient to form on the surface relatively large (60...200 nm) and deep pores that can greatly enhance the absorbing properties of material. Porous semiconductor in certain approximation could be considered an absolutely black body, as the rays of light get stuck in the porous space (Fig. 4).

Thus, in this case, we shall accept the following basic criteria of porous surface quality:

- mean diameter of pores in the range of 20...250 nm;
- length of pore 20...40 μm ;
- surface porosity 40...80 %.

In order to predict the possibility of formation of porous surface with required quality, it is necessary to choose the modes/conditions for pore formation taking into account basic mechanisms that underlie the formation of pores on the surface of semiconductor.

It should be noted that today there is no a single mechanism of pore formation. However, it is possible to highlight some of the patterns of the course of this process. As

mentioned above, during anode dissolution of crystal, there occur the processes of self-organization, predetermined by a number of factors, including:

- orientation of the surface of semiconductor (defines the shape of pores);
- type of conductivity (typically, pores of satisfactory quality form only on the surface of semiconductors of the n-type);
- type and concentration of doping additive, etc.

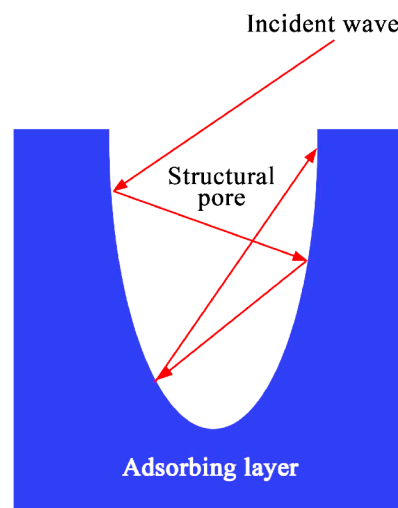


Fig. 4. Diagram of light absorption by a pore

In addition, quality of the porous layers is greatly affected by condition of the surface of semiconductor – number of surface defects and dislocations.

Consideration of these factors makes it possible to select basic modes for electrochemical process, which include:

- concentration of the electrolyte;
- type of anion that participates in the electrochemical dissolution of crystal;
- current density and voltage of pore formation;
- time of etching, etc.

It should be noted that the modes of electrochemical treatment are selected for each particular case individually.

5. 2. Examination of the boundary voltage in early pore formation

Conditions of pore formation are always limited by a more or less narrow range of polarization voltage [24]. The sharpest, clearly defined boundary of this range, is the minimal threshold voltage magnitude, which is essential for the early origin of the pores, the so-called early pore formation voltage – U_n .

Boundary voltage of early pore formation (BVPF) can serve as a quantitative characteristic of the process of pore formation that occurs in a particular system “semiconductor/electrolyte”. U_n depends on the formulation of electrolyte and the original surface of the crystal, which is why it is determined for each case individually.

BVPF increases with increasing pH of the environment. Under equal conditions (identical crystals, the same charge and concentration of anions in solution), boundary voltage depends on the type of anion that participates in the reaction. Electrolytes are divided into strong and weak by the ability to dissociate into ions when dissolved. A part of the molecules of weak electrolytes splits into ions under the action of solvent. The process of their dissociation is reverse

since when collisions occur, the ions are easily associated [25]. In the solutions of weak electrolytes, dynamic equilibrium sets in between the ions and non-dissociated molecules. While dissolving strong electrolytes, dissociation proceeds almost completely, ionic crystals or molecules break up with the formation of hydrated (solvated) ions. Among the essential acids, strong electrolytes include HNO_3 , H_2SO_4 , HClO_4 , HCl , HBr . Weak electrolytes include most of the inorganic compounds H_2CO_3 , H_2S , HCN , HF .

Voltage of the early pore formation was determined in the following way. Rate of change in voltage was 1 V/min. In this case, current density (to the critical value of voltage) remained within 20 mA/cm². Starting with a certain value of voltage, the current density increases dramatically. This is the boundary voltage of early pore formation. Dramatic growth of current density over time can be explained by the gradual increase in the number of input openings of pores and their branching beneath the surface. After some time, current ceases to grow. Fig. 5 shows volt-ampere characteristics to determine the boundary voltage of the early pore formation of crystals of indium phosphide, gallium phosphide, and gallium arsenide. To preserve purity of the experiment, conditions for pore formation in all three cases were similar: electrolyte $\text{HF}:\text{C}_2\text{H}_5\text{OH}:\text{H}_2\text{O}=1:2:1$; etching time is 15 min.

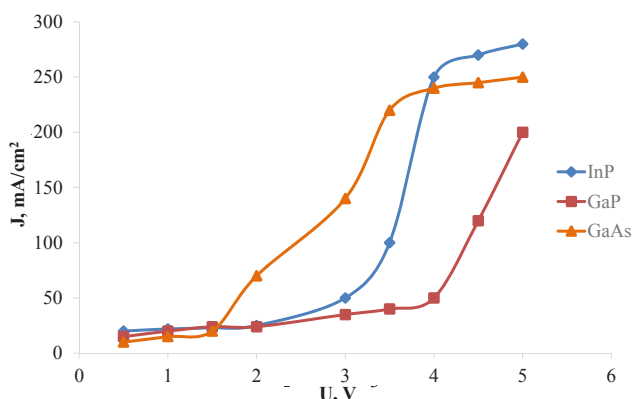


Fig. 5. Dependence of current density on the value of applied voltage during electrochemical treatment of crystals of indium phosphide, gallium phosphide, and gallium arsenide (electrolyte $\text{HF}:\text{C}_2\text{H}_5\text{OH}:\text{H}_2\text{O}=1:2:1$; etching time is 15 min)

The value of boundary voltage of the early pore formation for selected crystals under identical conditions is given in Table 1.

Table 1
Value of BVPF for semiconductors of group A3V5 during electrochemical treatment in the alcohol solution of hydrofluoric acid

Semiconductor	U_n , V
InP	3
GaP	4.5
GaAs	3.5

We can conclude that at selected conditions of etching the largest ability to pore formation is displayed by crystals of indium phosphide. The process of dissolving the crystal starts at a voltage of 3 V. The least active is gallium arsenide. To form pores on its surface, it is necessary to apply high voltage, which in this case is 4.5 V.

5.3. Thermodynamic analysis of processes at the boundary of contact “semiconductor-electrolyte”

The boundary of the system “semiconductor – electrolyte” has its own peculiarities. Different character of conductivity (electron/hole) and aggregate states (solid body/liquid) contribute to specific physical-chemical properties of the interphase boundary [26]. Electrolytic layer at the border of the system “semiconductor – electrolyte” can be conditionally divided into three regions:

- regions of spatial charge of semiconductor;
- Helmholtz layer;
- Gooney layer (region of spatial charge of the electrolyte) (Fig. 6).

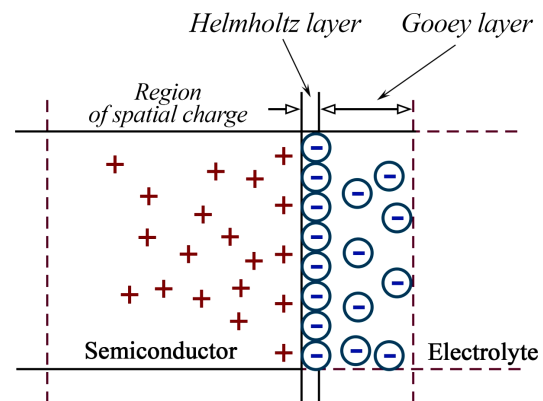


Fig. 6. Diagram of the structure of double electric layer at the boundary section semiconductor/electrolyte

Energy levels of these states occur as a consequence of the following factors [27, 28]:

- adsorption of impurities on the surface;
- formation of polar bonds between the atoms of crystal, which are on the surface, and oxygen atoms, etc.

Galvani potential $\phi_{1,2}$ (difference in electrical potentials between two points in the phases semiconductor/electrolyte is determined by formula (1) [29]:

$$\phi_{1,2} = \phi_1 + \phi_0 + \psi, \quad (1)$$

where ϕ_1 , ϕ_0 , and ψ are the fall of potential in the region of spatial charge of semiconductor, in the Helmholtz layer and region of spatial charge of the electrolyte, respectively.

Relation (2) makes it possible to evaluate a relative drop in the potential in the Helmholtz layer [30, 31]:

$$\phi_1/\phi_0 = L_1 \epsilon_1 / (d_0 \epsilon_1), \quad (2)$$

$$\phi_1/\psi = L_1 \epsilon_2 / (L_2 \epsilon_1), \quad (3)$$

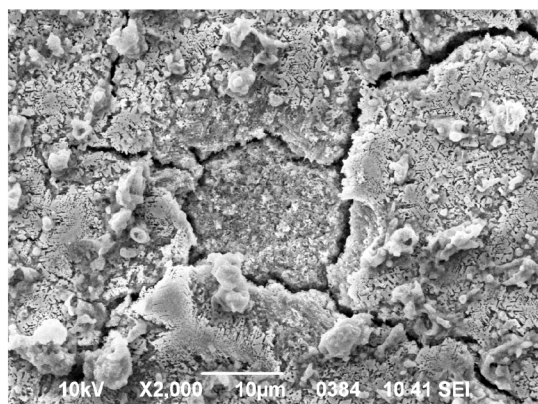
where L_1 is the thickness of the region of spatial charge; L_2 is the thickness of the Gooney layer; d_0 is the thickness of the Helmholtz layer; ϵ_1 is the relative dielectric permittivity of semiconductor; ϵ_2 is the dielectric permittivity of electrolyte.

For this case (semiconductor is indium phosphide, electrolyte is $\text{HF}:\text{C}_2\text{H}_5\text{OH}:\text{H}_2\text{O}=1:2:1$), the fall of potential in the region of spatial charge of semiconductor is 253 times larger than that in the Helmholtz layer, and is 233 times larger than that in the Gooney layer. We can conclude that the main part of the Galvani-potential falls in the region of spatial charge of semiconductor.

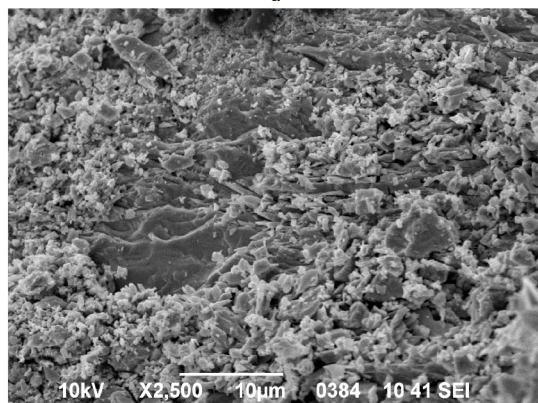
6. Discussion of results of examining control over the processes of pore formation during electrochemical treatment of crystals

In the course of examining control over the processes of pore formation, we established basic regularities that affect dissolution of the surface of crystal during electrochemical treatment. It is shown that in order to build a scheme of CPEDC, it is necessary to establish in advance criteria of quality of the nanostructures to be obtained.

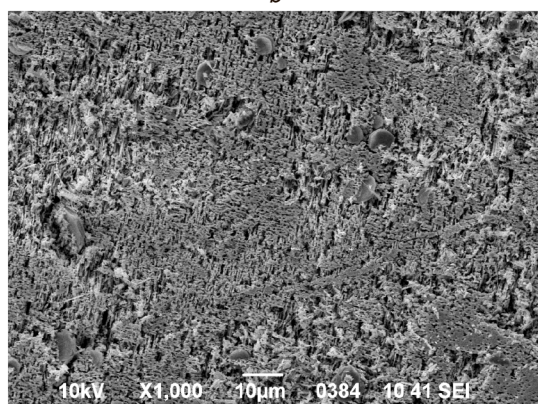
Fig. 7, *a–c* shows microphotographs of surface morphology of the crystals of group A3V5 that were treated under similar conditions in the solution of hydrofluoric acid. Basic characteristics of porous layers are given in Table 2.



a



b



c

Fig. 7. Morphology of porous surfaces of semiconductors of group A3V5, obtained by the method of electrochemical etching in the solution of hydrofluoric acid for 15 min:

a – InP; *b* – GaP; *c* – GaAs

Table 2

Characteristics of porous layers, obtained on the surface of semiconductors of group A3V5 by the method of electrochemical etching in the solution of hydrofluoric acid

Semiconductor	Mean pore diameter, nm	Mean pore length, μm	Porosity, %
InP	80 nm	35	55
GaP	260 nm	15	40
GaAs	140 nm	25	60

Under similar conditions of etching, semiconductors possess different capacity to pore formation. Quality porous structure among the presented cases is demonstrated by indium phosphide and gallium arsenide. Chosen conditions for pore formation do not appear optimal for gallium phosphide.

Presented scheme of control over the process of pore formation on the surface of semiconductors could be used for other needs as well, such as the formation of textured surfaces, superlattices, clusters, fractal structures, etc. [32–34].

However, this system is quite general and needs further clarification. In particular, it is necessary to devise a criterial apparatus of nanostructure quality. In addition, further research is to address the processes of self-organization in the formation of porous spaces on the surface of semiconductors. These processes underlie behavior of the subsystem “semiconductor – electrolyte”. This predetermines morphology of the nanostructured surfaces.

7. Conclusions

1. We devised a procedure to control the process of electrochemical dissolution of crystal as part of the control system over quality of nanostructures. The main task of control over the process of electrochemical dissolution of crystal is to perform such actions that would make it possible to provide the required state of the controlled system. When controlling the of process pore formation on the surface of crystal, it is necessary to consider conditions for pore formation, requirements that are put forward to quality of the obtained nanostructures and mechanisms that underlie the process of pore formation.

2. We investigated the processes that take place at the boundary of contact “semiconductor – electrolyte”. A thermodynamic analysis of the processes at the boundary of the contact was conducted. We established a relative fall of potential in the Helmholtz layer. The main part of the Galvani-potential falls in the region of spatial charge of semiconductor. Major morphological criteria of quality of porous nanostructures are selected for use in solar batteries, which include diameter and depth of the pore, as well as the degree of porosity of the surface of nanostructured crystal. Taking into account these criteria, we received porous spaces on the surface of semiconductors A3V5 that can be used for solar cells.

3. We examined physical and technological regularities of pore formation on the surface of semiconductors. Morphological properties are determined by the conditions of pore forma-

tion, as well as by processes of self-organization, which occur during etching. We determined the value of boundary voltage in the early pore formation for semiconductors of group A3V5 during etching in the electrolyte $\text{HF}:\text{C}_2\text{H}_5\text{OH}:\text{H}_2\text{O}=1:2:1$ for 15 min. It is established that at chosen conditions of etching, the largest ability to pore formation is displayed by crystals of indium phosphide.

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