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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 відносне 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 A3B5

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 A3B5

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

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RESEARCH INTO REGULARITIES OF PORE FORMATION ON THE SURFACE OF SEMICONDUCTORS

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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 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 investigated 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.

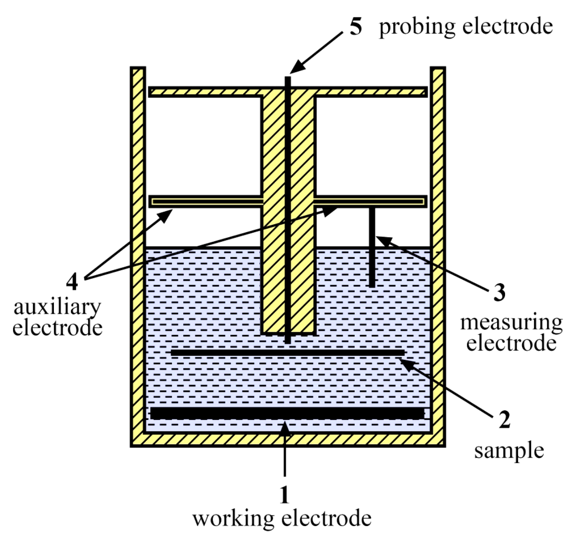

Fig. 1. Schematic of setup for electrochemical etching of semiconductors

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 of 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.

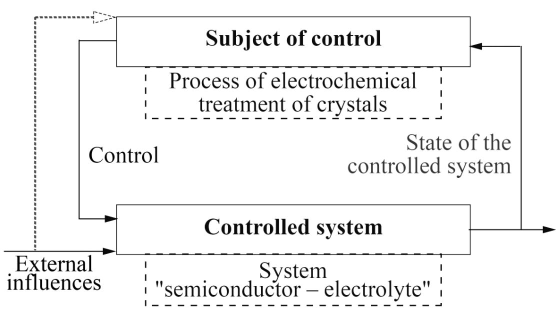

Fig. 2. General scheme of control over the process of electrochemical dissolution of crystal

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 of light 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.

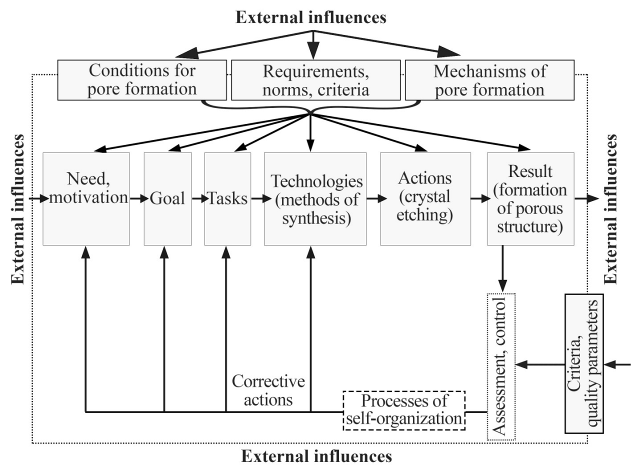

Fig. 3. Structural components of the detailed scheme of control over technological process of electrochemical dissolution of crystal

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.

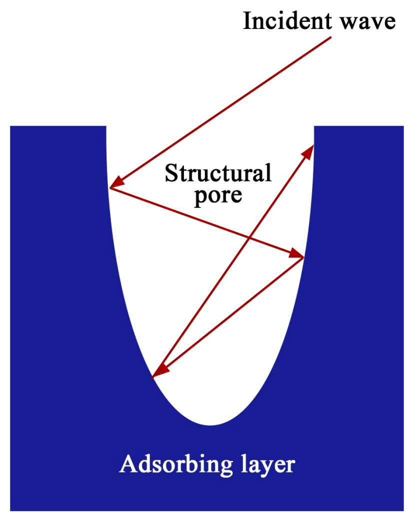

Fig. 4. Diagram of light absorption by a pore

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 – Un.

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". Un 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/cm^2. 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 $HF:C_2H_5OH:H_2O=1:2:1$; etching time is 15 min.

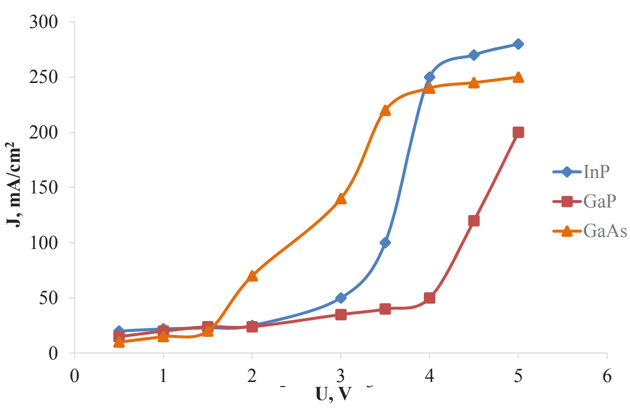

Fig. 5. Dependence of current density on the value of applied voltage during electrochemical treatment of crystals of indium phosphide, gallium phosphide, and gallium arsenide (electrolyte $HF:C_2H_5OH:H_2O=1:2:1$; etching time is 15 min)

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	Un, 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;
– Gooey layer (region of spatial charge of the electrolyte) (Fig. 6).

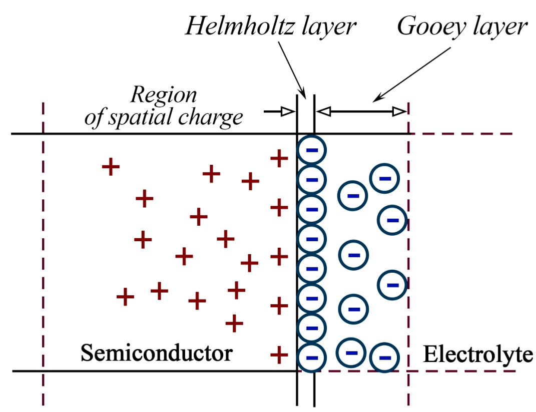

Fig. 6. Diagram of the structure of double electric layer at the boundary section semiconductor/electrolyte

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

$$\varphi_{1,2}=\varphi_1+\varphi_o+\psi, \quad (1)$$

where φ_1, φ_o, 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]:

$$\phi_1/\phi_o=L_1\varepsilon_1/(d_o\varepsilon_1), \quad (2)$$

$$\phi_1/\psi=L_1\varepsilon_2/(L_2\varepsilon_1), \quad (3)$$

where L_1 is the thickness of the region of spatial charge; L_2 is the thickness of the Gooey layer; d_o 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 $HF:C_2H_5OH:H_2O=1:2:1$), 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 Gooey 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.

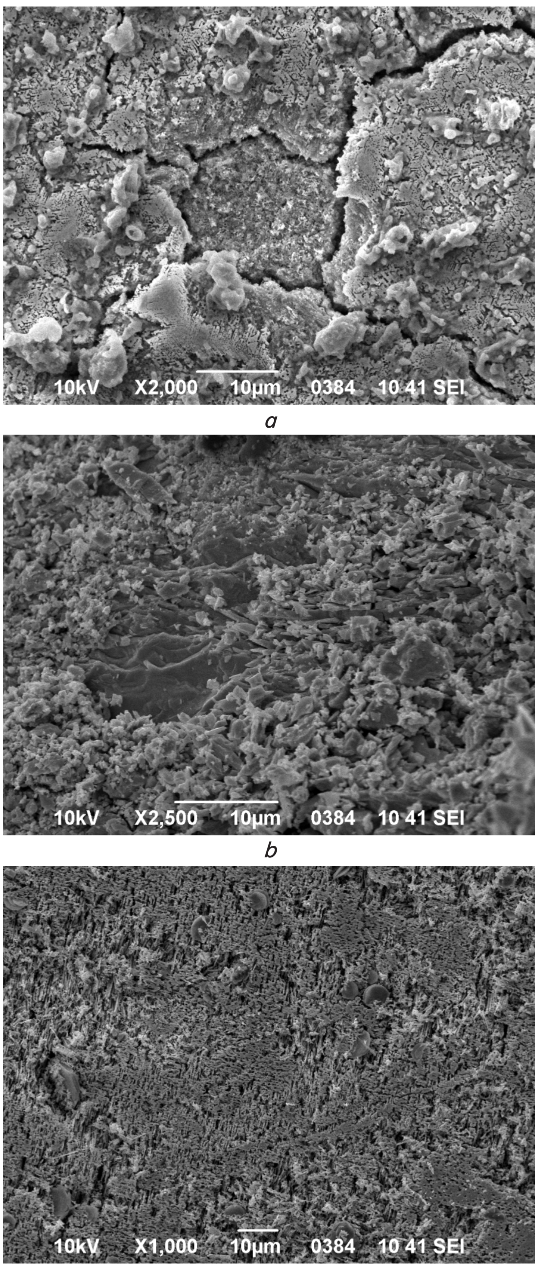

Fig. 7. Morphology of porous surfaces of semiconductors of group A3V5, obtained by the method of electrochemical etching in the solution of hydrofluoric acid for 15 min: *a* — InP; *b* — GaP; *c* — GaAs

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 $HF:C_2H_5OH:H_2O$=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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ABSTRACT AND REFERENCES

APPLIED PHYSICS

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REFINED CALCULATION OF INDUCTION MOTOR EQUIVALENT CIRCUIT NONLINEAR PARAMETERS BY AN ENERGY METHOD (p. 4-10)

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The topicality of the research aim is caused by the analysis of the processes of energy conversion taking into account the induction motor particular nonlinearities that reveal the physical properties and phenomena in structural materials under the action of electrical and electromagnetic impacts. Taking into consideration the nonlinearities of the induction motor equivalent circuit influences the accuracy of determination of the electric machine operating characteristics. Most conventional methods for parameter identification do not enable assessment of the induction motor nonlinear characteristics and properties.

It is proposed to use resistive impedance and inductance dependences on the rotor current to take into account the rotor nonlinear parameters. To form identification equations, the instantaneous power components for the rotor nonlinear resistive impedance and nonlinear inductance have been obtained. The solution of the identification equations resulted in determination of the equivalent circuit electromagnetic parameters taking into account the rotor nonlinear parameters and the amplitudes of the harmonics of the current cosine and sine components of the rotor and magnetization circuit. The results of identification of the induction motor equivalent circuit parameters taking into account the rotor nonlinear parameters have been obtained with sufficient accuracy. This is confirmed by a low error of determination of the electromagnetic parameters. The adequacy of the identified parameters is determined by comparison of the stator current experimental and calculated curves.

Keywords: induction motor, energy method, equivalent circuit, nonlinear electromagnetic parameters.

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METHOD FOR THE EROSION RATE MEASUREMENTS OF STATIONARY PLASMA THRUSTER INSULATORS (p. 11-17)

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Despite the fact that for many years the stationary plasma thruster (SPT) has been used in space, this technology has not yet been fully studied. One of the research directions to improve SPT characteristics is the increase of thruster lifetime. This can be achieved with the research of the discharge chamber (DCh) walls erosion rate as a function of different thruster operational modes.

The analysis of the SPT DCh wear rate diagnostics methods was carried out. It is shown that the considered methods cannot provide measurements of the insulators erosion rate separately during the experiment.

The method of optical emission spectroscopy with plasma scanning through collimator (OESSC) was developed. The method allows registration of radiation separately for each of SPT DCh walls directly during the experiment. According to this, the experimental time of different SPT operational regimes research was reduced by 98 %.

Experimental equipment of the OESSC method was developed, produced and tested. It consists of the high-resolution spectral block with the range of 240...820 nm and the optical detector positioning system.

A set of experiments were provided with the OESSC method. As a result, the inner and outer insulators erosion rates are researched as functions of thruster operational regimes. It is shown that for the outer and inner coils currents of 5 and 6 A, respectively, there is a uniform wear of DCh walls. For such SPT operational regime, the difference in the insulators specific relative erosion rates is 2 %.

The results of the OESSC method measurements allowed increasing the anode block lifetime significantly.

Keywords: stationary plasma thruster, optical emission spectroscopy, discharge chamber, relative erosion rate.

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RESEARCH INTO RESOURCE-SAVING MOLYBDENUM-CONTAINING ALLOYING ADDITIVE, OBTAINED BY THE METALLIZATION OF OXIDE CONCENTRATE (p. 18-23)

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We established that the phase composition of oxide molybdenum concentrate is re presented mainly by MoO_3, as well as MoO_2, WO_3, Mo_2C and associated ore impurities of Al_2O_3, CaO, SiO_2 and MgO. We found non-uniform microstructure in the form of plates, granules of round shape, and thread-like formations. It was determined that phase composition of metallized molybdenum concentrate, obtained by carbon-thermal technique, is mostly composed of metal Mo with the presence of MoC and Mo_2C. Unrestored component is represented by the oxy-carbide compound (Mo, O, C) and the lower molybdenum oxide MoO_2. We noted fragmented presence of Mo_8O_{23}. Spongy microstructure revealed areas where the molybdenum oxide restoration products dominate. The presence of residual oxygen confirms the existence, along with metal Mo, of unrestored oxide or oxy-carbide compounds. The residual oxygen could also be contained in the oxide compounds of Si, Al, Ca, Mg, K, Na as associated ore impurities. This is confirmed by discovery of the specified elements in the examined areas. The detected phases and compounds do not display significant susceptibility to sublimation. High restorative ability, due to the excess of carbon in the form of carbides, provides the post-restoration of oxide component in the liquid metal in the process of alloying, as well as protection against secondary oxidation.

Keywords: molybdenum concentrate, carbon-thermal restoration, metallization, sublimation, phase analysis, microstructure, resource saving.

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INVESTIGATION OF PROPERTIES OF ELECTROCONDUCTING NANOZONES IN MATERIALS OF VARIOUS NATURE BY THE ELECTRON PARAMAGNETIC RESONANCE METHOD (p. 24-30)

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This work studied properties of conductive nanozones in dependence on the material nature by the method of electron paramagnetic resonance. Delocalization of charge carriers and strong exchange interactions between paramagnetic centers take place in annealed biominerals, like in polyaniline in a form of emeraldine salt. Since biominerals, unlike polyaniline, can be subjected to a high-temperature anneal, this extends potentials of the EPR method in solving issues associated with the properties of conductive zones in nanosized polyaniline. It was shown that the EPR signals in the materials under consideration are conditioned by the electrical charge carriers and variations of electrical properties result in variation of the EPR signal characteristics. Consequently, information on the mechanisms of the EPR signal induction in one group of specimens can be used

for interpretation of the signal nature and elucidation of properties of local conductive zones in other materials.

It was ascertained that the characteristics of electron paramagnetic resonance of conductive nanozones in biominerals and the textile materials containing nanoparticles of polyaniline are similar. This feature is due to the similarity of paramagnetic charge carriers localized in nano-sized conductive zones of biominerals and organic polymers. Interconnected electron paramagnetic resonance studies of conductive zones in various materials can promote a more successful application of electron paramagnetic resonance in developing nanotechnologies for creation of conductive textile materials containing nanoparticles, especially nanoparticles of polyaniline with a high level of oxidation.

Keywords: nanoparticles, polyaniline, biomineral, enamel, bones, textile material, electron paramagnetic resonance, conductive nanozones.

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VERIFICATION RESULTS OF THE INNOVATIVE TECHNIQUE FOR IONIC NITRIDING OF STEELS WITH LOW TEMPERING TEMPERATURES (p. 31-36)

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The innovation technique of complex treatment for steels with the low temperature of tempering is proposed and tested in the course of present study. It includes nitriding in the vacuum gas discharge before hardening and tempering. In this case, during nitriding, the heating temperature influences little the process of high-temperature treatment. In this case, the process of diffusion of nitrogen atoms is accelerated considerably (since nitrogen atoms penetrate untempered steel more easily), which leads to an increase to 2000 μm in the depth of penetration of nitrogen atoms and in the thickness of the formed region with changed structure and hardness. It was established that, according to the properties, the region of exposure is divided into a surface layer (with a thickness of about 200 μm) with lowered hardness and the deeper operating layer with enhanced hardness. Layer with the greatest hardness is at depth of 400–800 μm. In this case, enhanced hardness, in comn parison with the base, is maintained at depth that exceeds 2000 μm. The surface layer with low hardness makes it possible to implement the allowance for finishing, in order to obtain the required accuracy of dimensions and surface finish. Hardness of the surface of articles after this sequence of operations for the steels with low temperature of tempering is at the level of 8–10 GPa. The phase composition of the nitrided layer with high hardness, detected by the X-ray diffraction method, is the lowest nitride Fe4N and the solution of nitrogen in α-Fe.

Keywords: ionic nitriding, complex treatment, diffusion, depth of impact, diffraction spectra.

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RESEARCH INTO REGULARITIES OF PORE FORMATION ON THE SURFACE OF SEMICONDUCTORS (p. 37-44)

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A general procedure is devised to control the process of formation of porous layers on semiconductor surfaces by the method of electrochemical etching. When controlling the process of pore formation on the surface of crystal, it is necessary to consider: conditions of pore formation, requirements that are put forward to quality of the obtained nanostructures, and mechanisms that underlie the process of pore formation. It is shown that the built scheme could be used for different cases of the synthesis of nanostructured semiconductors. We investigated the processes that underlie pore formation and define morphological properties of nanostructures. A thermodynamic analysis of processes at the boundary of contact «semiconductor – electrolyte» was performed. We examined a relative drop in potential in the Helmholtz layer, which is an important characteristic of the process of pore formation on the surface of crystal. Main morphological criteria are selected of quality of porous nanostructures for their application in solar batteries. These include diameter and depth of the pore, a degree of porosity of the surface of a nanostructured crystal. Taking into account these criteria, we received porous spaces on the surface of semiconductors A3V5 that could be used for solar cells. We determined the value of boundary voltage of the early pore formation for semiconductors of group A3V5 during etching in the electrolyte HF:C2H5OH:H2O=1:2:1 for 15 min. It was established that at chosen conditions of etching. the largest capacity to pore formation is displayed by crystals of indium phosphide. The results obtained demonstrate that at the same conditions of etching semiconductors possess different ability to form pores.

Keywords: quality of nanostructures, electrochemical etching, porous semiconductors, Helmholtz layer, morphology, semiconductors.

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RESEARCH INTO THE PROCESS OF PREPARATION OF UKRAINIAN COAL BY THE OIL AGGREGATION METHOD (p. 45-53)

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We conducted a study into concentration of Donetsk coal with varying degrees of coalification – anthracite and grade G – by the method of oil aggregation. We determined the character of impact of the following factors on the results of oil aggregation of coal: the ash content of original coal, pulp agitation duration, pulp density, consumption and type of reagent-binder.

The research demonstrated a possibility of effective preparation of finely- and thinly dispersed Donetsk coal the size of 0–0.1; 0–1 (2) mm and ash content from 10–15 % to 65–70 % by the method of oil aggregation.

In this case, it was established that with an increase in the ash content of original coal, the process of oil aggregation displays a capacity of self-leveling. By reducing the speed of aggregation, it is possible to attain practically stable technological results over the entire examined range of ash content of original coal – from 10–15 % to 65–70 %.

Obtained results substantiate the possibility of employing the process of oil aggregation for the re-preparation of waste, in particular, waste of flotation and gravitation separation of coal preparation.

Keywords: oil aggregation, coal preparation, ash content of coal, aggregation, self-leveling of oil aggregation.

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RESEARCH INTO CONSTRUCTIVE AND TECHNOLOGICAL FEATURES OF EPITAXIAL GALLIUM-ARSENIDE STRUCTURES FORMATION ON SILICON SUBSTRATES (p. 54-61)

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The technology of formation of LSI structures on GaAs epitaxial layers, formed on Si-substrates of large diameter, is developed, which makes it possible at least by an order of magnitude to reduce the production cost of crystals due to epitaxial growth of GaAs layers and the use of technological equipment of silicon technology. This technology also enables the useof heterostructures to increase the speed of the LSI.

An analysis of complex structures of different architecture of IC/LSI on GaAs epitaxial layers, formed on Si-substrates, is carried out. The influence of the scattering processes of charge carriers on the potential fluctuations on the magnitude and profile of the mobility of electrons along the thickness of the epitaxial structure is investigated. When using epitaxial technology in structures, there are no isoconcentric impurities of oxygen and carbon, which are the factors of scattering of charge carriers, which makes it possible to achieve high values of mobility of charge carriers.

It is shown that the use of epitaxial layers of gallium arsenide eliminates the effects of isoconcentration impurities of oxygen and carbon in gallium arsenide layers that increases their purity.

A test element was implemented that allows non-destructive measurement of the mobility of charge carriers in the technological cycle of the formation of LSI structures. This allows us to realise the electrophysical diagnosis of the reliability of the LSI at the stage of crystal manufacturing.

Keywords: complementary structures, semiconductors, epitaxy, integrated circuits, technological features.

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