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About synthesis mechanism of periodic oxide nanocrystallites on surface of single-crystal

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We have obtained unique periodic oxide nanocrystallites on the surface of indium phosphide. The morphological characteristics of the structures obtained and their component composition are investigated in the article. The main attention is focused on explaining the mechanism of the periodic structures, which are packed by the ‘parquet floor’ type. The mechanism based on sliding the sources of rectangular-shaped dislocation loops has been proposed. The system of the main and secondary (ingoing) dislocations that cause the formation of the steps has been considered. The mathematical interpretation of the described model has also been proposed.

Keywords: indium phosphide, nanocrystallites, periodic structures, nanowires.

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Introduction

The need for new “smart” materials is caused by the increasing demand for high-tech electronics [1, 2]. Composite materials have been widely used in photonic devices [3, 4]. The heterostructures are widely used as materials for lasers [5, 6]. Planar nanostructures are of the utmost interest for thin-film technologies, in particular, for the creation of light-emitting diodes [7, 8]. In recent times, the attention of researchers is focused on the materials with the surface architecture [9, 10]. Today, nanoneedles [11], nanowhiskers [12], flower-shaped structures [13], porous surfaces [14, 15], etc., are successfully synthesized. Furthermore, periodic structures are gaining in considerable popularity [16, 17]. The advantage of such structures over other types of nanostructured nanomaterials is caused by the possibility to control wavelengths, radiation angles, etc. [18, 19].

The following tasks are raised to scientists due to understanding of these trends: (1) selection of effective, inexpensive synthesis methods, which allow to create periodic nanostructures with the controlled properties [20, 21]; (2) selection of the components of the substrate/nanostructure system with well-formed

parameters [22].

Today, the synthesis methods of oxide semiconductors and their integration into substrates are being actively investigated [23 – 25]. So, the investigations are concerned with Ga₂O₃ [26, 27], In₂O₃ [28], ZnO [29], CdO [30], TiO₂ [31], etc. Such semiconductors show excellent ability for nanostructuring, as well as being chemically and thermally stable [32, 33]. Furthermore, the surface of the oxide films on the semiconductor show the passivating properties and allow extended service life of the devices [34, 35]. As a rule, the only problem when such heterostructures are formed is the mismatch of the crystal lattices, which causes a large number of stress states at the boundary of two heterostructure components [36]. On the other hand, such situation can be effectively used when structures with non-standard morphology are formed [37, 38]. In this case, the defects of the output semiconductor (substrate) are the source of the nanostructure on the surface [39]. The effective use of this phenomenon is quite a difficult task and requires a further detailed study.

In this study, we report the synthesis of the periodic structures on the surface of indium phosphide, which are packed in the “parquet floor” type. Such structures are promising for study, since periodicity can be effectively

used to create photocatalytic heterostructures and waveguides. We give a qualitative and quantitative interpretation of the mechanism of formation of the parquet oxide structure on the surface of a highly doped indium phosphide.

I. Experiment

The periodic parquet structures were formed by the combined chemical etching methods. A Teflon standard electrochemical cell was used. The cell was filled with the electrolyte. After that, the etching processes were started, with subsequent deposition of the reaction products on the sample surface. The first stage was carried out in the electrochemical anodization at a constant current density. Simple electrochemical etching without the action of a current was used at the second stage. The electrolyte solution was not changed during both stages. Table 1 shows the conditions for the chemical treatment of the samples.

Before carrying out the experiment, the samples were washed in the acetic solution and then degreased with the ethyl alcohol. Immediately after that, the experiment was started. After completion of the experiment, the samples were dried and stored in the open air.

The surface morphology was investigated using a Jeol microscope (Japan), equipped with an NCA Energy 350 spectrometer manufactured by Oxford Instruments Analytical (Great Britain). The topographic images of the synthesized nanostructure surfaces were obtained by raster electron microscopy with the use of secondary electron signals (SEI). Data on the surface inhomogeneity and composition were obtained in the back-scattered electron (BEC) mode. The energy of the electron beam was chosen in the range of (10-15) keV. The elemental composition of the samples was determined using an energy-dispersive spectrometer at an acceleration voltage of (15-20) kV and a beam current of (1-3) nA. The quantitative analysis of the chemical composition of the sample surface was performed by comparing the characteristic lines of the sample with those of standards.

II. Results

Fig. 1 shows the SEM image of InP surface after the electrochemical treatment. It can be seen that massive crystal nanowires, which are organized by “parquet floor” type, were formed on the surface. Table 2 shows the geometric sizes of the formed structures.

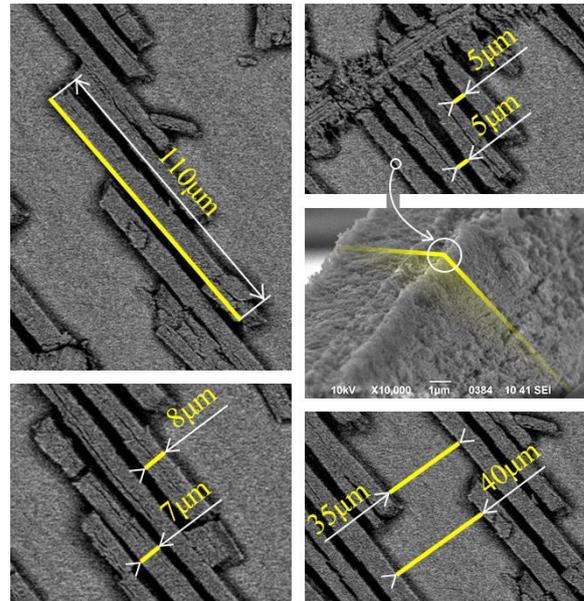


Fig. 1. SEM image of formed periodic structure on the InP surface.

Table 2.

Geometrical sizes of nanowires	
Characteristics	Size, μm
Thickness of nanowire	8-10
Height of nanowire	7-10
Length of nanowire	110-130
Distance between adjacent wires	3
Distance between “parquet layers”	20-50

Thus, it can be seen that the nanowires have the shape of a regular prism. It is interesting to note that the wires have the porous structure with a pore size of 80-120 nm.

Fig. 2 shows the results of the EDX analysis of the formed structure surface at the point on the crystallite surface. We can see that the crystallites were formed by indium oxide with a small content of phosphorus. This certainly points to the formation mechanism of the periodic nanocrystallites on the surface of the indium phosphide. The active etching of the indium and phosphorus atoms, which leads to electrolyte saturation, is observed during the first etching stage. It is followed by an alternative process, namely, the deposition of the reaction products on the surface.

Table 1.

Conditions of experiment				
Stages	Electrolyte	Current density, j , mA/cm^2	Time, min	Additional conditions
1 st stage	$\text{HF}:\text{H}_2\text{O}:\text{C}_2\text{H}_5\text{OH}=1:1:2$	150	10	Mixing of electrolyte; platinum plate was used as cathode
2 nd stage	$\text{HF}:\text{H}_2\text{O}:\text{C}_2\text{H}_5\text{OH}=1:1:2$	0	10	Room temperature, darkness

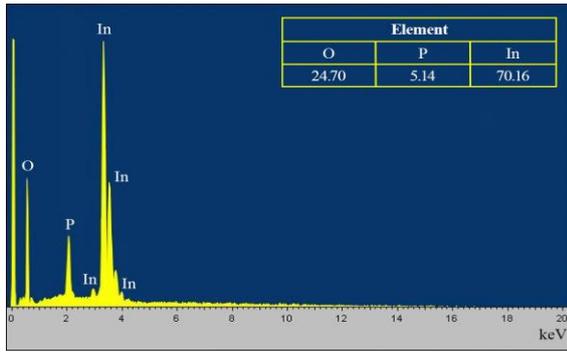


Fig. 2. The results of EDX analysis at the point on the crystallite surface.

III. Discussion

3.1 Mechanism of formation of the periodic structures on the surface of indium phosphide by “parquet floor” type

Single-crystal semiconductors, doped to the high concentrations of minor charge carriers, are characterized by a large number of the dislocations. For the experiment, we used the InP plates with the (111) surface orientation (Fig. 3a). The sources of rectangular-shaped dislocation loops, the sliding system of which can be represented as a parallelogram surface with faces along the (101) and (110) planes, will be observed in the volume of such sample (Fig. 3b).

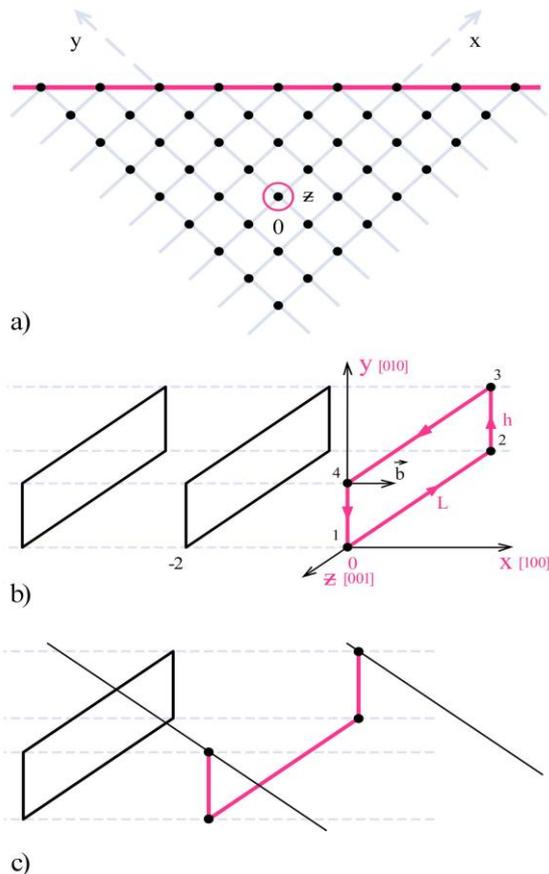


Fig. 3. Fixation model of dislocation loops: a) location of dislocation sliding planes; b) dislocation loops in the volume of n-InP (111); c) fixation of dislocation on sample surface, which will cause the steps.

The dislocation loops tend to spread and move due to the forces of mutual repulsion. This movement occurs in the direction of the sample surface and stops at the moment of emergence of the dislocations (Fig. 3 c). The exit of the first (main dislocation loop) to the surface causes the step. Three other loops (ingoing) in the dislocation system are fixed and their movement stops. Thus, we can observe the accumulations of dislocations.

According to Fig. 3b an important observation can also be made. The dislocation loop does not move along the [010] and [001] directions during movement toward the sample surface (Fig. 3b axes OY and OZ, respectively). In this case, the superposition of the Peach-Koehler force component is zero in all directions except for the [100] direction (OX axis). This will violate the symmetry of the primary and incoming loops, allowing one to displace the ingoing loop along the [010] direction. During further sliding of the ingoing dislocation, it will fall into the obstacle-free sliding system, caused by the main loop of the dislocation accumulations. Then, when exposed to the next dislocation loop, it will advance in the sample volume until it reaches the surface. The next dislocation loops will move according to the same mechanism.

Thus, the sources of the dislocation loops will initiate the steps on the sample surface, which are the periodic areas of the inhomogeneous deformation (Fig. 4a). This, in turn, will allow one to form the sample surface relief with a periodic architecture (Fig. 4b).

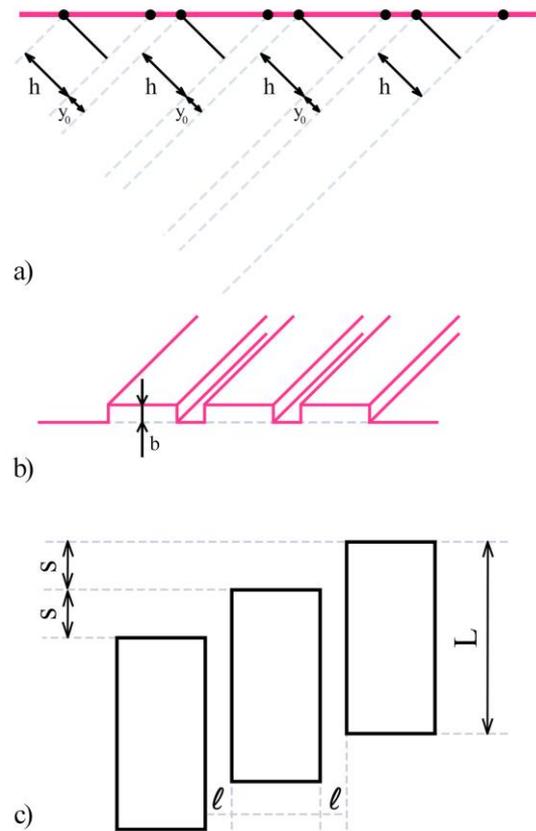


Fig. 4. The system of dislocation loops on the surface of InP (111) (a); schematic diagram of surface relief with parquet periodic crystallites (b) and relative displacement of steps caused by displacement of main and ingoing dislocation loops (c).

In addition to sliding dislocations, the actual crystals also have other type of defects in the volume. The symmetry of the mutual location of the dislocation loops (nonzero superposition of the Pitch-Keller force component) is violated due to this (Fig. 4c). Displacement will occur along the sliding direction [001]. Thus, the arrangement of nanocrystalline wires in the form of parquet steps will be observed on the InP (111) surface.

3.2 Mathematical interpretation of formation of parquet periodic nanostructures

From these considerations, we can present the described mechanism with a mathematical model. Thus, the Pitch-Keller force component acts on the unit length of the dislocation line:

$$f_i = e_{ikl}\tau_k\sigma_{lm}b_m \quad (1)$$

where e_{ikl} is the Levi-Civita symbol; τ_k is the component of the unit vector; σ_{lm} is the components of the mechanical tension tensor; b_m is the components of the Burgers vector.

The superposition of the Pitch-Keller force component, acting on the dislocation loop along the [010] direction, is described by the expression:

$$F_y = -b^2B \frac{h(3d^2+h^2)}{(d^2+h^2)^2}L, \quad (2)$$

$$B = \frac{\mu}{2\pi(1-\sigma)}$$

where b is the Burgers vector; μ is the shear modulus; σ is the Poisson's ratio; h is the size of the dislocation loop along the direction [010]; d is the distance between the dislocation loops; L is the size of the dislocation loop [001].

The Pitch-Keller force component in section (1-2) (Fig. 3b) is described by the expression:

$$F_y^{(1-2)} = -b^2B \frac{(h+y_0)[3d^2+(h+y_0)^2]}{[d^2+(h+y_0)^2]^2}L, \quad (3)$$

where y_0 is the distance module of the dislocation loop from the section (3-4) to the plane ZOx.

Accordingly, for section (3-4), the Pitch-Keller force component (Fig. 3b) is described by the expression:

$$F_y^{(3-4)} = b^2B \frac{y_0(3d^2+y_0^2)}{(d^2+y_0^2)}L. \quad (4)$$

On the basis thereof, the fixation condition of the dislocation loop corresponds to the expression:

$$F_y^{(1-2)} + F_y^{(3-4)} = 0. \quad (5)$$

An important observation is that the length of one parquet step is equal to the length of sections (1-2) and (3-4) of the dislocation loops.

The Pitch-Keller force, acting on the dislocation loop, parallel to the direction [010]:

$$F_y = \pm b^2B \frac{y(3d^2+y^2)}{(d^2+y^2)^2}L, \quad (6)$$

The extremes of the function $F(y)$ have the following values:

$$y_1 = \sqrt{(2\sqrt{3}-3)}d \approx 0,681d,$$

$$y_2 = -\sqrt{(2\sqrt{3}-3)}d \approx -0,681d,$$

The condition of stable equilibrium with respect to the displacement of the dislocation loop along the [010] direction:

$$y_0 = 0,681d - \frac{h}{2}. \quad (7)$$

Taking into account the fact that the angle between the sample surface and the sliding planes of the dislocations is equal to 45° , the step distance is calculated as follows:

$$l = \sqrt{2}y_0 = \sqrt{2}(0,681d - 0,5h) \quad (8)$$

We can also estimate the value of the longitudinal displacement S of the dislocation loop:

$$S = 2(h + y_0) = 2(0,5h) + 0,681d \quad (9)$$

The values of d and h are approximately equal to each other, which gives us the value of the step distance:

$$l \approx 0,256h; \quad S \approx 2,362h. \quad (10)$$

We can see that the conducted simulation gives us the full conformity between the calculated and experimental values.

Conclusions

The mechanism of the periodic nanostructures on the surface of highly doped single-crystal indium phosphide, which are oxide formations and packed on the surface by "parquet floor" type, were described in our paper. The morphological characteristics of the formed nanowires were investigated. In particular, it has been shown that the thickness of the nanowires has the average value of $10 \mu\text{m}$, the length is in the range of $(100 - 130) \mu\text{m}$. EDX analysis showed that the nanowires consist mainly of indium and oxygen atoms. The phase composition of the formed nanocomposites requires further detailed study.

The dislocation mechanism of nanowire formation has been proposed. According to this mechanism, the dislocations move in the direction of the sample surface and stop at the moment of the emergence of the dislocations. This leads to the "run-up" of the next dislocations, resulting in the appearance of the "steps" on the surface. These steps are the sources of the formation of the nanostructures located on the surface with a defined period.

The mathematical interpretation of the proposed

mechanism has been given, and the longitudinal displacement of the dislocation loop and the distance between the steps have been theoretically estimated. The simulation conducted gives us the full conformity between the calculated and experimental values.

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Про механізм синтезу періодичних оксидних нанокристалітів на поверхні монокристалічного InP

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Нами було отримано унікальні періодичні оксидні нанокристаліти на поверхні фосфіду індію. У статті досліджуються морфологічні характеристики одержаних структур та компонентний склад. Основна увага в статті зосереджена на поясненні механізму формування періодичних структур, які упаковані по типу «паркет». Запропоновано механізм, який ґрунтується на ковзанні джерел дислокаційних петель прямокутної форми. Розглянуто систему головної та вторинних (набігаючих) дислокацій, які спричиняють появу сходинок. Також запропоновано математичну інтерпретацію описаної моделі.

Ключові слова: фосфід індію, періодичні структури, дислокації, електрохімічне травлення, поруватий оксид.