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Chemical evaluation of the quality of nanostructures synthesized on the surface of indium phosphide

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ABSTRACT

Purpose: The article proposes a methodology for determining the chemical quality criterion of porous layers synthesized on the surface of semiconductors, based on taking into account the chemical parameters of the surface that can affect the properties of nanostructures.

Design/methodology/approach: The chemical quality criterion was evaluated in terms of stoichiometry, stability of structures over time, uniformity of distribution over the surface, and the presence of an oxide phase. As an example, a calculation is demonstrated for the por-InP/InP structure synthesized on a mono-InP surface. The results of calculating the chemical quality criterion were evaluated using the Harrington scale to rank samples by quality level.

Findings: A chemical criterion for the quality of porous layers synthesized on the surface of semiconductors has been developed. This criterion contains a set of indicators sufficient for a comprehensive assessment of the surface condition and is universal in nature. The studies carried out make it possible to reasonably approach the determination of the modes of electrochemical processing of semiconductors and open up new perspectives in the construction of a model of self-organization of a porous structure.

Research limitations/implications: The chemical quality criterion does not allow evaluating the obtained nanostructures in terms of geometric parameters. Therefore, in the future, there is a need to develop a morphological quality criterion and determine a methodology for assessing a generalized quality criterion for nanostructures synthesized on the surface of semiconductors, which may include economic, environmental, technological indicators, and the like.

Practical implications: Study results are expedient from a practical point of view, since they make it possible to reasonably approach the determination of the modes of electrochemical processing of semiconductors, synthesize nanostructures with predetermined properties, and create standard samples of nanomaterial composition.

Originality/value: Methodology for assessing the quality of porous semiconductors by a chemical criterion has been applied for the first time in engineering science. The article will be useful to engineers, who are engaged in the synthesis of nanostructures, researchers and scientists, as well as specialists in nanometrology.

Keywords: Semiconductors, Nanostructures, Chemical criterion, Indium phosphide, Quality indicators

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1. Introduction

Evaluating and ensuring the quality of nanomaterials is crucial for the modern materials science [1,2] holding key positions in the development of new engineering and functional materials of the new generation [3,4]. Reliable operation of such materials requires sufficient permanence and stability of properties over time [5,6].

High-rate developments in the domain of nanostructured materials primarily include synthesis technologies [7,8], the study of morphological parameters [9,10] and application niches [11,12], etc. Many studies focus on the surface and bulk flaws of nanostructured nanomaterials [13,14].

Today, we know more than 40 types of nanostructures [15], the most common of them being the nanostructures synthesized on the surface of semiconductors [16,17]. This is due to their widespread use in sensors [18], sources of alternative energy [19], photonics devices [20], etc. The prime advantages of such materials are the simplicity of their synthesis [21] and the stability of surface properties over time [22]. Various types of nanostructures are formed on the surfaces of such semiconductors as *GaAs* [23], *InP* [24], *GaN* [25], *GaP* [26], *ZnO* [27], *ZnSe* [28], *SiC* [29] with the most common among them being porous layers [30], thin films [31], quantum dots [32], whiskers, etc. [33].

In order to evaluate the quality of a nanomaterial in each specific case, it is necessary to solve the following tasks:

- to select criteria reflecting the functional purpose of the nanomaterial;
- to select quality indicators ensuring the suitability of the nanomaterial;
- to evaluate the quality level by a generalized indicator;
- to identify the interrelation between individual and generalized quality indicators in order to develop recommendations for quality assurance.

The morphological (geometric) indicators of nanostructures are the most profoundly studied today. Traditionally, the density of nanoobjects [34], the homogeneity of their distribution over the surface [35], their shape [36], size [37], etc. are evaluated.

Limited research has been done on establishing the chemical quality indicators of nanostructured semiconductors. However, this issue is extremely important. The shelf life depends on the chemical resistance of the nanostructure surface [38]. Surface active layers determine physical and electrical properties [39]. The chemical composition of nanostructures is of decisive importance when studying the recombination properties of a material [40].

The article suggests a method for determining the chemical quality criterion of porous layers synthesized on the surface of semiconductors. The calculation for por-InP/InP structure has been demonstrated as an example.

2. Approach for research

The chemical quality criterion is evaluated in terms of stoichiometry, structural stability over time, homogeneity of distribution over the surface, and the presence of the oxide phase (Fig. 1).

The stoichiometry of nanostructures synthesized on the surface of single-component semiconductors is taken as the optimal value, while binary semiconductors necessarily require determining the range of mass fractions of one of the elements. Stability over time is checked by photoluminescence spectroscopy, namely by determining the aging of the spectra over time. The PL method requires additional research and complicates data processing; therefore, it is advisable to use an indirect approach by estimating the time of surface passivation [41]. The more optimal the passivation time, the more stable the structure. It is expedient to check the homogeneity of the element distribution over the surface by INCA [42] while determining the quantification by Fisher's criterion [43]. The presence of oxides is confirmed by EDAX technique [44].

A significant advantage of the suggested approach is that in order to determine the quality level of nanostructures, only a scanning electron microscope (SEM) must be used; EDAX and INCA are supplementary to the SEM functionality.

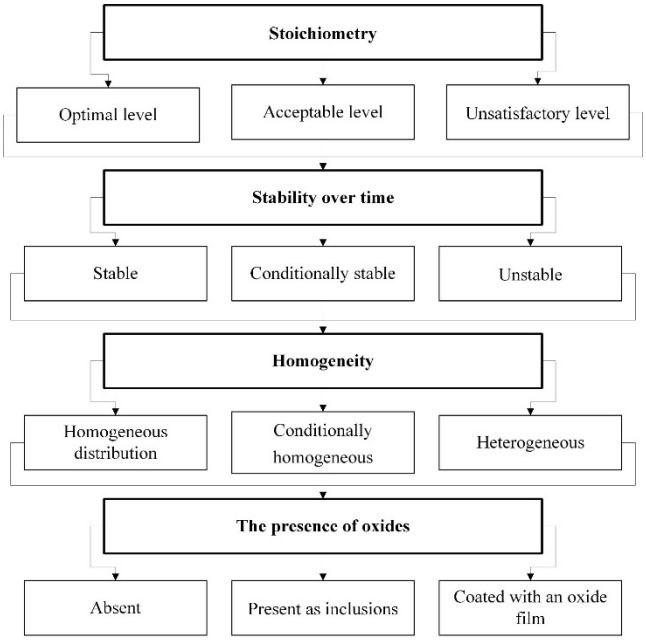

Fig. 1. Partial indicators of the chemical criterion for the quality of nanostructures

The convolution of the chemical quality criterion indicators will be carried out according to the linear law:

$$K_x = b_1 k_{x1} + b_2 k_{x2} + b_3 k_{x3} + b_4 k_{x4}, \quad (1)$$

where b_1, b_2, b_3, b_4 – weight coefficients;
k_{x1}, k_{x2}, k_{x3}, k_{x4} – partial indicators of the chemical quality criterion.

The indicator reflecting the stoichiometry of the surface porous layer for single-component semiconductors (Si, Ge) will always have the value $k_{x1} = 1$. As for binary semiconductors (*InP, GaAs, GaP, ZnSe, SiC, CdTe*), it will be determined by the content of one of the elements and will take the following values:

$$k_{x1} = \begin{cases} 1, C \subset \left[C_{\min}; C_{\max} \right]; \\ 0.5, C \subseteq \left[C_{\min} - 10\% C_{\min}; C_{\min} \right) \cup \left(C_{\max}; C_{\max} + 10\% C_{\max} \right]; \\ 0, C \subseteq \left[0\%; C_{\min} - 10\% C_{\min} \right) \cup \left(C_{\max} + 10\% C_{\max}; 100\% \right] \end{cases} \quad (2)$$

where C – mass fraction of the element, %;
C_{min} – the minimum allowable value of the mass fraction of the element under study, %;
C_{max} – the maximum allowable value of the mass fraction of the element under study, %.

The indicator reflecting the stability of the surface properties over time (chemical and electrical inertness) will be estimated by the time (t) of the chalcogenide passivation. This indicator can take the following values:

$$k_{x2} = \begin{cases} 1, t \subset \left[t_{\min}; t_{\max} \right]; \\ 0,5, t \subseteq \left[t_{\min} - 10\% t_{\min}; t_{\min} \right) \cup \left(t_{\max}; t_{\max} + 10\% t_{\max} \right]; \\ 0, t \subseteq \left[0; t_{\min} - 10\% t_{\min} \right) \cup \left(t \succ t_{\max} + 10\% t_{\max} \right) \end{cases} \quad (3)$$

where t – passivation time, min;
t_{min} – the minimum allowable value of the passivation time for the formation of a uniform passivating layer, min;
t_{max} – the maximum allowable value of the passivation time for the formation of a uniform passivating layer, min.

The indicator reflecting the homogeneity of the element distribution on the surface for one-component semiconductors (*Si, Ge*) will always have the value $k_{x3} = 1$. As for binary semiconductors (*InP, GaAs, GaP, ZnSe, SiC, CdTe*), it will be determined by EDAX. It is advisable to study the homogeneity of the element distribution over a porous surface at 10 points, at least. Calculations for one of the elements are sufficient to determine the homogeneity of the element distribution over the surface. The variation coefficient reflects the relative scatter of the totality values; it indicates its average scatter in proportion to its average quantity value. The variation coefficient will be determined by the formula:

$$\nu = \frac{\sigma}{\overline{C}}, \quad (4)$$

where $\overline{C}$ – the arithmetic mean value of the concentration of an element over the surface;

σ – mean-square deviation equal to the root mean square:

$$\sigma = \sqrt{D}, \quad (5)$$

$$D = \frac{\Sigma (C_i - \overline{C})^2}{n}, \quad (6)$$

Subsequently, the variation coefficient can be calculated by the formula:

$$\nu = \frac{\sqrt{\frac{\Sigma \left(C_i - \overline{C} \right)^2}{n}}}{\overline{C}}. \quad (7)$$

The greater the value of the variation coefficient, the relatively greater the scatter and the lower the evenness of the studied values (Tab. 1).

Table 1.
The value of the variation coefficient and the homogeneity of the variation series

1.	σ	Variation	Homogeneity of the series
2.	$\nu \prec 30\%$	Weak	Homogeneous
3.	$30 \le \nu \le 70\%$	Moderate	Insufficiently Homogeneous
4.	$\nu \succ 70\%$	Strong	Heterogeneous

Thus, k_{x3} indicator can take the following values:

$$k_{x3} = \begin{cases} 1, \nu \subset \left(0;\ 30\right)\%; \\ 0.5, \nu \subset \left[30;\ 70\right]\%; \\ 0, \nu \subset \left(70;\ 100\right)\% \end{cases}. \quad (8)$$

The indicator indicative of the presence of oxides on the surface can take the following values:
$k_{x4} = 1$, if there are no oxides on the porous surface;
$k_{x4} = 0.5$, if there are single inclusions of oxides as nanocrystallites;
$k_{x4} = 0$, if the porous surface is coated with an oxide film.

Table 2.
Harrington's Desirability Scale [45]

No	"d" Scale	Quality Indicators of "d" Desirability Indicator Scale
1.	1.00	Displays the extreme level of excellent quality with no need for any improvement.
2.	1.00 – 0.80	Acceptable at the level of "excellent". Reflects good quality.
3.	0.80 – 0.63	Acceptable at the level of "good". Reflects the level above the best level corresponding to the value d = 0.63.
4.	0.63 – 0.40	Acceptable at the level of "satisfactory". The quality is acceptable to the maximum allowable level but needs improvement.
5.	0.40 – 0.30	Ultimate level. In the presence of standard requirements to the indicators, some of the products will exceed the relevant limits (in case such indicators exactly match the specified minimum or maximum, the "d" value should be 0.36788=1/e).
6.	0.30 – 0.00	Unacceptable level.
7.	0.00	Completely unacceptable level.

Having calculated the morphological and chemical quality criteria, we subsequently determine the value of the generalized quality criterion. Next, we use Harrington's desirability scale (Tab. 2) in order to determine the quality level of the nanostructure.

If any of the chemical quality indicators cannot or should not be determined, it can be omitted.

3. Experiment

As an example, we will consider the porous structures of por-InP formed by electrochemical etching of single-crystal indium phosphide in a hydrogen solution of hydrochloric acid. Before the experiment, the samples have been cleaned in order to remove mechanical and chemical contaminants. The plates have been subsequently immersed in an electrochemical cell with platinum cathode and etching occurring at a constant current density of 150 mA/cm^2. The experimental conditions are given in Table. 3.

Table 3.
Conditions for the synthesis of porous layers on the surface of indium phosphide

No sample	Time, min	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

After the experiment, the samples have been annealed in ammonia solution in order to stabilize their properties. We have used a JEOL-6490 scanning electron microscope to study the morphology of the obtained structures, with chemical composition of the surface layers identified using EDAX and INCA techniques. The stoichiometry of the samples has been determined based on the concentration of phosphorus. In porous layers of indium phosphide, the phosphorus sublattice is etched away faster than indium; therefore, the stoichiometry is shifted towards the excess of indium atoms [46,47]. Porous samples should be stable over time with the elements evenly distributed over the surface, and with no oxide films. Reference quality indicators of porous layers on the surface of indium phosphide as well as weighting coefficients are presented in Table. 4.

Table 4.
Reference indicators of the quality of nanostructures formed on the surface of indium phosphide, and weighting coefficients (example)

No	Indicator	Reference Value	Weighting Coefficients
1.	Phosphorus concentration	(35-45) %	b_1=0.25
2.	Stability	stable over time	b_2=0.25
3.	Homogeneity	homogeneous distribution	b_3=0.3
4.	Oxides	none	b_4=0.2

4. Experimental results and discussion

The results of the scanning electron microscopy have demonstrated that all the study samples have a porous layer on the surface after electrochemical treatment in a hydrochloric acid solution.

There are no continuous oxide films on the surface of the samples; some samples have single oxide inclusions, not affecting the recombination significantly. We have determined the average content of elements on the surface by INCA Energy technique in order to study the stoichiometry for each sample (Tab. 5). We have also applied EDAX technique to determine the concentration of elements at 10 points for each of the samples and thus, to study the homogeneity of the element distribution on the surface. The results indicating the content of phosphorus at each test point of the samples are presented in Table 6. The calculation data required to study the homogeneity of phosphorus distribution over the surface as well as the indicator values reflecting such homogeneity of phosphorus distribution over por-InP surface are presented in Table 7.

Table 5.
Spectral analysis of elements on the surface of por-InP samples by INCA Energy and the value of the chemical quality criterion

No sample	Element concentration, y %			k_{x1}
	In	P	O	
1.	61.93	38.06	0.01	1
2.	63.60	36.10	0.30	1
3.	61.99	37.00	1.01	1
4.	65.91	33.09	1.00	0.5
5.	60.02	39.67	0.31	1
6.	56.11	41.83	2.06	1
7.	61.26	37.00	1.74	1
8.	60.86	34.07	5.07	0.5
9.	66.73	33.21	0.06	0.5

Calculations of the homogeneity of the element distribution over the surface allows us to see that all samples, except for samples No. 8 and No. 9, demonstrate absolutely homogeneous distribution of elements over the surface. The inhomogeneity of samples No. 8 and No. 9 can be explained by several factors such as the concentration inhomogeneity of the element distribution over the surface of the original crystal caused by growth defects; the presence of significant surface flows causing a faster etching of the sublattice of one of the elements; fluctuations in the distribution of elements and the like. In order to calculate the chemical quality criterion, we will determine the value of the product of partial criteria and the standardized coefficients, and calculate the chemical quality criterion directly (Tab. 8).

Table 6.
Concentration of phosphorus on the surface of por-InP samples with spectra collected at 10 points of each of the samples by EDAX

No sample	Element concentration, y %									
	1	2	3	4	5	6	7	8	9	10
1.	0.43	0.28	0.37	0.41	0.30	0.54	0.39	0.28	0.45	0.38
2.	0.36	0.30	0.30	0.28	0.40	0.6	0.32	0.30	0.43	0.45
3.	0.29	0.29	0.40	0.30	0.48	0.43	0.30	0.40	0.36	0.43
4.	0.18	0.23	0.48	0.51	0.28	0.36	0.40	0.35	0.29	0.31
5.	0.37	0.54	0.28	0.45	0.30	0.34	0.38	0.37	0.45	0.22
6.	0.46	0.43	0.45	0.43	0.40	0.6	0.28	0.29	0.41	0.34
7.	0.50	0.31	0.43	0.37	0.34	0.31	0.26	0.45	0.34	0.38
8.	0.20	0.19	0.36	0.38	0.30	0.51	0.35	0.25	0.38	0.54
9.	0.24	0.34	0.29	0.35	0.22	0.41	0.17	0.36	0.29	0.62

Table 7.
Calculation data and indicator values reflecting the homogeneity of phosphorus distribution

No sample	$\overline{C}$	D	σ	ν ,%	k_{x3}
1.	0.38	0.00604	0.0777	20.29	1
2.	0.37	0.0089	0.0944	25.23	1
3.	0.37	0.00438	0.0662	17.78	1
4.	0.34	0.00973	0.0986	29.1	1
5.	0.37	0.00782	0.08884	23.9	1
6.	0.41	0.00773	0.0879	21.5	1
7.	0.37	0.00481	0.0693	18.79	1
8.	0.35	0.0124	0.111	32.19	0.5
9.	0.33	0.0141	0.119	36.08	0.5

Table 8.
Calculation data and the value of the chemical quality criterion

No sample	b_1k_{x1}	b_2k_{x2}	b_3k_{x3}	b_4k_{x4}	K_x	Quality level on the Harrington scale
1.	0.25	0.25	0.25	0.20	0.95	excellent
2.	0.25	0.25	0.25	0.20	0.95	excellent
3.	0.25	0.25	0.25	0.20	0.95	excellent
4.	0.125	0.25	0.25	0.20	0.825	excellent
5.	0.25	0.25	0.25	0.20	0.95	excellent
6.	0.25	0.25	0.25	0.20	0.95	excellent
7.	0.25	0.25	0.25	0.20	0.95	excellent
8.	0.125	0.25	0.125	0.1	0.6	satisfactory
9.	0.125	0.25	0.125	0.20	0.7	good

When analyzing the values of the chemical quality criterion, we can see that all samples demonstrate acceptable and high levels of quality with no significant deviations.

The results obtained help us determine the conditions favorable for synthesizing nanostructures with indicator values close to the reference ones, that is, high-quality nanostructures; it also helps us determine the conditions deteriorating the said process. Samples No. 8 and No. 9 have been treated in a highly concentrated electrolyte solution ($10H_2O$+5HCl) for a long time (15 and 20 min, respectively). They demonstrate the lowest quality level. The calculation of the chemical quality criterion has proved such conditions to be unacceptable and rather rigid.

5. Conclusions

1. The article suggests a method for determining the chemical quality criterion of nanostructures in terms of stoichiometry, structural stability over time, homogeneity of distribution over the surface and the presence of the oxide phase.
2. Using the example of porous por-InP synthesized on a mono-InP surface, we have calculated the value of the chemical criterion for the samples obtained under different treatment conditions. The results evaluated according to Harrington's scale show that the porous layers treated in a solution of hydrochloric acid ($10H_2O$+1HCl) for (10-20) min demonstrate the highest quality level.
3. The presented studies are practically expedient since they allow to reasonably determine the conditions for electrochemical treatment of semiconductors. Theoretically, the studies open up new vistas in the construction of a model of a porous structure self-organization on the surface of semiconductors.
4. The chemical quality criterion allows to evaluate only the surface condition of nanostructured layers. It does not allow evaluating the obtained nanostructures in terms of geometric parameters. Therefore, there is a need to develop a morphological quality criterion and determine an effective method for evaluating a generalized quality criterion for nanostructures synthesized on the surface of semiconductors in the future.

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