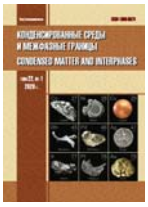

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Influence of Nanoscale Layers of the $Mn_3(P_{0.1}V_{0.9}O_4)_2$ Chemostimulator-Modifier on the Process of Thermal Oxidation of GaAs, its Composition, and Morphology of the Resulting Films

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Abstract
Chemostimulated thermal oxidation is one of the approaches to the formation of functional nanoscale films on an $A^{III}B^{V}$ surface. In order to obtain the desired result, it is necessary to reasonably choose an object that can act as a chemostimulator of the process or a modifier of the structure and properties of films formed as a result of oxidation. The use of complex compounds capable of combining both of these functions seems to be effective. The purpose of the study was an investigation into the effect of nanoscale layers of the $Mn_3(P_{0.1}V_{0.9}O_4)_2$ chemostimulator-modifier on the process of thermal oxidation of GaAs, its composition, and morphology of the formed films.
The object of study was gallium arsenide (100) with nanosized layers of manganese vanadate-phosphate $Mn_3(P_{0.1}V_{0.9}O_4)_2$ deposited on its surface. In order to increase the speed of the process and ensure the high chemical homogeneity of the product, it was proposed to use microwave activation of the synthesis of the chemostimulator-modifier $Mn_3(P_{0.1}V_{0.9}O_4)_2$ and its further deposition onto the surface of the semiconductor by the spin-coating method. The formed $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructures were thermally oxidized in the temperature range 490–550 °C for 60 min in an oxygen stream. The thickness of the growing films (by laser and spectral ellipsometry), their composition (X-ray phase analysis, Auger electron spectroscopy), and surface morphology (atomic force microscopy) were controlled.
Studies of the kinetics of thermal oxidation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/ GaAs heterostructures showed that the determining process is the solid-phase reaction, limited by diffusion in the solid phase, and the transit character of the chemostimulator without the catalytic effect occurs. It was revealed that manganese vanadate-phosphate promoted an increase in the growth of the formed film by an average of 70–220% compared to the standard oxidation of GaAs, leads to the intensification of secondary interactions of the oxides of the substrate components with the products of thermolysis of $Mn_3(P_{0.1}V_{0.9}O_4)_2$ and the absence of segregation of arsenic in the film in a non-oxidized state.
Thermal oxidation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructures results in the formation of nanoscale (50-200 nm) films with a fairly pronounced relief. Further study of the electrophysical characteristics of the films is necessary, since composition data suggest they possess a dielectric nature. This can be used in practice for the formation of films on the surface of $A^{III}B^{V}$ with functional purposes and with widely varying characteristics.

Keywords: gallium arsenide, manganese vanadate-phosphate, nanoscale films, chemostimulated oxidation, microwave synthesis.

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1. Introduction

Heterostructures on gallium arsenide are widely used in technologies for the production of microwave integrated circuits, various optoelectronic devices, and field-effect transistors [1–6]. The result of the appearance of gallium arsenide microelectronics was the creation of efficient and powerful injection lasers and LEDs based on GaAs/GaAlAs heterostructures in the wavelength range of 600–900 nm [7–9]. Moreover, the variety of objects formed on the GaAs surface and possessing a wide range of properties is extremely large: quantum dots, one-dimensional nanostructures, and thin films. In the latter case, the application of a wide variety of oxides, sulphides, nitrides, and complex compounds is possible, and now there are a number of physical and chemical methods for the creation of such $A^{III}B^{V}$ based thin-film heterostructures [10–13]. However, the problem of synthesizing functional nanoscale films by relatively simple and technologically advanced methods has not yet been solved.

The formation of functional nanosized films on the GaAs surface by thermal oxidation requires the use of reasonably selected synthesis chemostimulators and film modifiers [14, 15]. Chemostimulators change the semiconductor oxidation mechanism and prevent segregation of arsenic in a free state at the inner boundary of the film. Modifiers in the process of thermal oxidation are embedded in the film, enabling its rapid growth, and a targeted change in its composition and nanostructure.

The use of complex compounds in the process of thermal oxidation of $A^{III}B^{V}$ seems to be effective. Their cationic component includes a chemostimulator. An anionic component can act as a modifier, by being included in the composition of the film (phosphates, sulphates, etc.) as a group, and can also contain a second, additional, chemostimulator (for example, vanadates). In this case, there is an additional opportunity to fine-tune the regulation of the synthesis processes using the combined effects of the chemostimulator-modifier. In this study, manganese vanadate-phosphate $Mn_3(P_{0.1}V_{0.9}O_4)$ was chosen as a chemostimulator-modifier. Manganese oxides are effective chemostimulators of the oxidation processes of $A^{III}B^{V}$ [16]. Vanadate groups VO_4^{3-}, isostructural to arsenate anions AsO_4^{3-} [17], are ready fragments of films formed during the oxidation process. In addition, oxygen compounds of vanadium, are able to exhibit the function of a chemostimulator even in the anionic component [18]. The doping of manganese vanadate with phosphorus ensures the inclusion of phosphate groups PO_4^{3-} exhibiting dielectric properties in the films [19].

The purpose of the study was the investigation of the effect of nanoscale layers of the chemostimulator-modifier $Mn_3(P_{0.1}V_{0.9}O_4)_2$ on the process of thermal oxidation of GaAs and the composition and morphology of the formed films.

2. Experimental

The synthesis reactions of manganese vanadate-phosphate $Mn_3(P_{0.1}V_{0.9}O_4)_2$ from a solution of precursors was carried out under the influence of microwave radiation (operating frequency 2450 MHz, P_{max} of source – 800 W) [20]. Vanadium (V) oxide V_2O_5 (analytical grade, Russian Federation Purity Standard TU 6-09-4093-88) was dissolved in an excess of a 20 % NaOH solution (analytical grade, Russian Federation Purity Standard GOST 432877), which led to the formation of sodium metavanadate $NaVO_3$. $Na_2HPO_4 \cdot 12H_2O$ (analytical grade, Russian Federation Purity Standard GOST 4172-76) and $MnCl_2 \cdot 4H_2O$ (International Purity Standard analytical reagent) solutions were added to a $NaVO_3$ solution for the synthesis of $Mn_3(P_{0.1}V_{0.9}O_4)_2$. Exposure to microwave irradiation of 600 W was performed for 10 min. The resulting suspension was cooled to room temperature and the $Mn_3(P_{0.1}V_{0.9}O_4)_2$ precipitate was separated from it using a vacuum filter. Then the precipitate was washed, dried, and annealed at a temperature of 400 °C for 2 h in a muffle furnace (SNOL 8.2/1100).

The phase composition was determined by X-ray phase analysis (XRDs) using an ARL X'TRA diffractometer in continuous mode. The angular range of the study was in the range from 10 to 80 ° (Cu$K_{\alpha 1}$ c λ = 1.540562 Å) at 25 °C. The size of the coherent scattering regions (CSR) according to X-ray phase analysis (XRD) for the synthesized $Mn_3(P_{0.1}V_{0.9}O_4)_2$ powder was calculated according to the Scherrer equation [21]:

$$D_{hkl} = \frac{kx\lambda}{\beta_{hkl}\cos\theta},$$

where D_{hkl} – average particle size, Å, k – correction factor (for cubic and orthorhombic structure $k = 0.9$), λ – X-ray tube wavelength, θ – the position of the peak maximum, deg., β_{hkl} – intrinsic physical broadening of the diffraction maximum, rad.

Monocrystal (100) gallium arsenide wafers of AGTSCh-1 grade were used as semiconductor substrates. GaAs was doped with zinc, the concentration of the main charge carriers was $11.5 \cdot 10^{18}$–$2.5 \cdot 10^{18}$ cm^{-3}. For the creation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructures with nanosized vanadate phosphate layers, the spin coating method was used [22]. Distilled water was added to the $Mn_3(P_{0.1}V_{0.9}O_4)_2$ powder. Dispersion was then carried out in an ultrasonic bath (VU-09-"Ya-FP"-0) for 15 min. A small amount of gelatine was added for the improvement of the adhesion to the substrate during spin-coating application. The final solution was stirred at 80 °C for 15 min with a magnetic stirrer (Magnetic Stirrer MSH-300).

Before the formation of thin-film heterostructures, the GaAs semiconductor substrates were treated with concentrated HF (49 %) for 10 min [23].

The thermal oxidation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructures was carried out in a flowing quartz reactor of a horizontal resistive heating furnace (MTP-2M-50-500) with temperature control with an error of 1 °C (ARIES TRM-201). The process was carried out in flowing oxygen (medical (99.5 %) GOST 5583-78, National product classification code: 21 1411 0200) with a volumetric flow rate of 30 l /h (linear gas flow rate in the reactor was 10 cm/min). Oxidation of the samples was carried out in the temperature range 490–550 °C for 60 minutes.

The thickness of the deposited layers of vanadate phosphate and films grown by thermal oxidation was measured using a LEF-754 laser ellipsometer (LE, accuracy ± 1 nm) and an Ellipse-1891 spectral ellipsometer (SE) [24]. According to the LE and SE data, the layer thickness of $Mn_3(P_{0.1}V_{0.9}O_4)_2$ on the semiconductor surface was 25±1 nm.

The elemental composition of oxide films on GaAs and the distribution of components based on their thickness were determined by the Auger electron spectroscopy (AES) method using an ESO-3 spectrometer with a DESA-100 analyser, determination accuracy ± 10 %. In this study, the layer-by-layer etching of films by argon ions was used for obtaining information on the distribution of elements along the film depth.

The imaging of the samples was performed by atomic force microscopy (AFM) using a Solver P47 Pro (NT-MDT) scanning probe microscope with the HA_NC Etalon cantilever.

3. Results and discussion

Microwave activation of the synthesis of a chemostimulator modifier $Mn_3(P_{0.1}V_{0.9}O_4)_2$ for a significant increase in the speed of the process and provision of high chemical homogeneity of the product was proposed in studies [25–27].

According to the study [28], decomposition of crystalline hydrates of 3*d*-elements in the microwave field was carried out in several stages to the oxide phase. Initially, crystalline hydrate solutions absorb microwave radiation due to the water of crystallization. At temperatures of 130–180 °C, hydrolysis of salts starts, with the formation of oxo- and hydroxo- compounds as intermediate products. Fine oxide particles that are formed after decomposition of salt compositions are uniformly distributed over the reaction volume and are able to actively interact with each other. A significant contribution is also made by the specific "non-thermal" effect of microwave radiation associated with the generation of ion currents at intercrystalline boundaries, the intensity of which increases significantly in highly dispersed systems.

XRD results of synthesized $Mn_3(P_{0.1}V_{0.9}O_4)_2$ powder in addition to the presence of the target phase showed the presence of impurities in the form of $Mn_3(PO_4)_2$ (d_{hkl} = 2.8745 Å; 1.8610 Å) and V_2O_5 (d_{hkl} = 4.3611 Å; 4.0797 Å; 3.3979 Å; 2.7566 Å). Despite the excess of sodium hydroxide during the synthesis of metavanadate, it was not possible to fully use V_2O_5, which according to the literature [29] can be observed in similar reactions. However, the presence of vanadium (V) oxide in the synthesized nanocrystalline powder is not a drawback, since previously [18] an effective chemostimulating effect of V_2O_5 by the catalytic mechanism was revealed in the processes of thermal oxidation of $A^{III}B^{V}$ semiconductors. The average size of the CSR was 30 nm.

The kinetic characteristics of the oxidation processes of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructures processed using the equation $d = (k\tau)^n$, are presented in Table 1.

Table 1. Kinetic parameters of the equation $d = k^n t^n$ for the process of thermal oxidation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructures

Chemostimulator-modifier	Oxidation temperature range, °C	$n_{wed} \pm \Delta n$, $nm^{1/n}min^{-1}$	EAE, kJ/mol	Maximum relative increase of film thickness,%
$Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs	490–550	0.39±0.01	156	220
GaAs (standard)	450–550	0.56±0.01	110	–

During the thermal oxidation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructures the value of n_{av} was less than 0.5, indicating that in this temperature range the determining process is the solid-phase reaction, limited by diffusion in the solid phase [16]. The EAE value of the studied process was somewhat higher (156 kJ/mol) compared with the intrinsic thermal oxidation of GaAs (110 kJ/mol). It is typical for a solid-solid reaction without a catalytic effect and indicates the transit nature of the action of the chemostimulator in the considered process [14].

The relative increase in film thickness g during chemically stimulated thermal oxidation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs (Table 2) in comparison with the intrinsic oxidation of the semiconductor was calculated by the formula:

$$g = \frac{\Delta d_{Mn_3(P_{0.1}V_{0.9}O_4)_2/GaAs}}{\Delta d_{GaAs}} \cdot 100\ \%,$$

where $\Delta d_{Mn3(P0.1V0.9O4)2/GaAs}$ – change in the thickness of the film formed in the process of thermal oxidation of the studied heterostructures with a deposited chemostimulator layer minus the thickness of the latter, and Δd_{GaAs} – change in the oxide film thickness during intrinsic oxidation of gallium arsenide.

When the chemostimulator-modifier $Mn_3(P_{0.1}V_{0.9}O_4)_2$ was used over the entire temperature–time range, an increase in the growth of the formed film by an average of 70–220 % was revealed compared with the oxidation of GaAs. This, apparently, was due to both the incorporation of ready isostructural phosphate and vanadate groups into growing films, and the effective chemostimulating effect of the cationic component of vanadate phosphate and V_2O_5 detected in the initial powder.

Films formed by the oxidation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructures contained $MnAsO_4$, $Mn_3(VO_4)_2$, $Mn_3(PO_4)_2$, GaAs, $GaAsO_4$ (Table 3). With an increase in the oxidation temperature, the intensities of the reflections of gallium arsenate and manganese arsenate were observed, confirming the intensification of the secondary interactions of the oxides of the substrate components with the products of thermolysis of the chemostimulator.

The analysis of the distribution profiles of elements along the depth of the film formed by the oxidation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructure in the regime of 510 °C, 60 min, demonstrated (Fig. 1) that the film was enriched with oxygen (up to 50-60 %) over the entire thickness. This indicates the absence of segregation of arsenic in the film in an unoxidized state (which is an attribute of the process of intrinsic thermal oxidation of gallium arsenide), and hence the effective chemostimulating effect of manganese vanadate-phosphate. The presence of gallium and arsenic on the film surface confirms their significant diffusion into the chemostimulator layer. Phosphorus in the film was practically not fixed, which was probably associated with its small amount due to evaporation in the form of oxides and the detection limit of elements

Table 2. Relative increase in oxide film thickness during thermal oxidation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructures in comparison with the GaAs standard

Образец	Relative increase in thickness as a function of oxidation time, %						
	T, °C/*t*, min	10	20	30	40	50	60
$Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs	530	68	83	84	82	83	90
	550	69	90	115	190	196	220

Table 3. Composition of films formed by the thermal oxidation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructures (XRD data [30])

Heterostructure, thermal oxidation mode	d_{hkl}, Å	Angle 2θ, degrees	Phase
$Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs, 500 °C, 60 min	1.7829	51.195	$MnAsO_4$
	1.3898	67.320	$Mn_3(VO_4)_2$
	3.5777; 2.7806	24.867; 32.166	$Mn_3(PO_4)_2$
	3.2541	27.386;	GaAs
	1.6299; 2.0674	56.406; 43.752	$GaAsO_4$
$Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs, 530 °C, 60 min	1.7909	24.823	$MnAsO_4$
	3.2542	27.385	GaAs
	3.5840; 2.774	24.823; 32.204	$Mn_3(PO_4)_2$
	2.0679	43.739	$GaAsO_4$

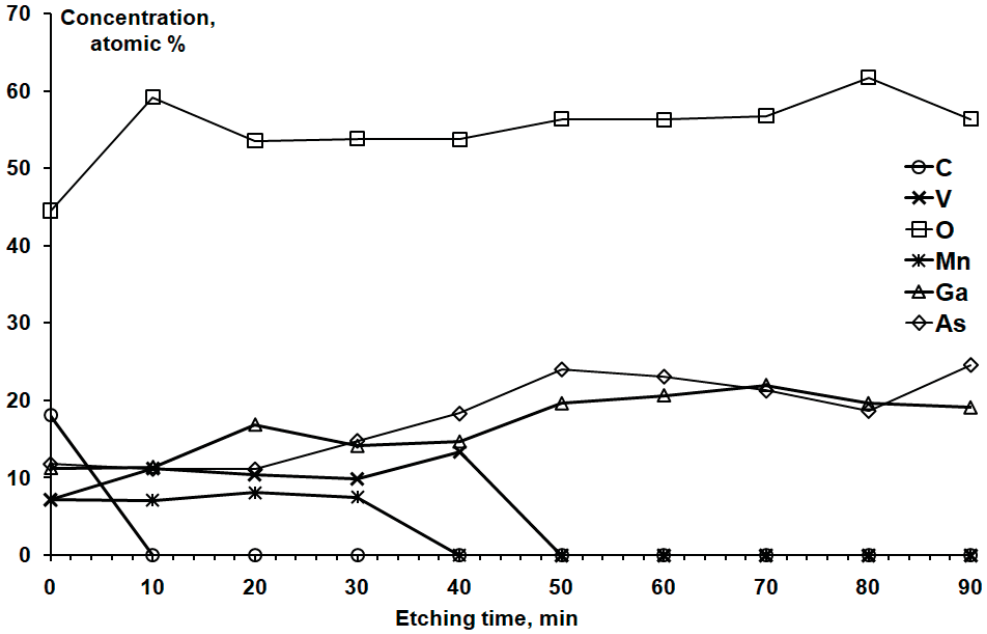


Fig. 1. Auger profiles of the distribution of elements in the film formed by the oxidation of an $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructure at 510 °C, 60 min

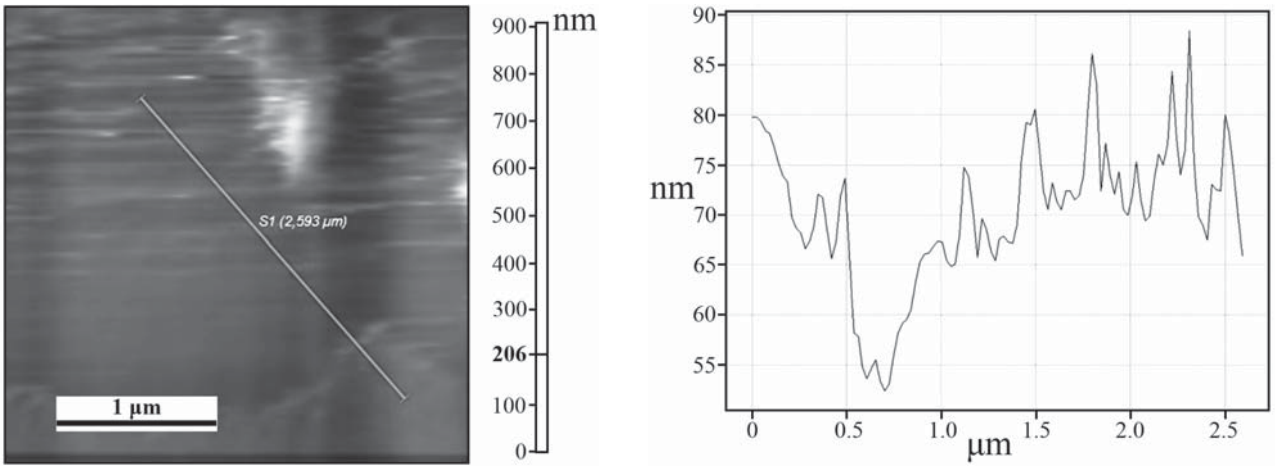
by the Auger electron spectroscopy method. With further propagation into the film, oxidized gallium and arsenic exist in the form of gallium arsenate, which correlates with XRD data.

According to AFM data for a non-oxidized $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructure, the average difference in the relief height was about 15 nm (Fig. 2). For $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructure, oxidized at 490 °C for 60 min, the difference in the relief height decreased to 8–10 nm. The average grain size was within 200 nm (Fig. 3a, b) With an increase in the oxidation temperature to 550 °C, the films had more expressed relief (Fig. 3c, d) The height difference increased to 20–25 nm, the average grain size increased to 300 nm.

Thus, nanoscale (thickness range of 50–200 nm) films with a fairly expressed relief were formed during the thermal oxidation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructures. As the oxidation temperature increased, the grain structure of the films became more expressed.

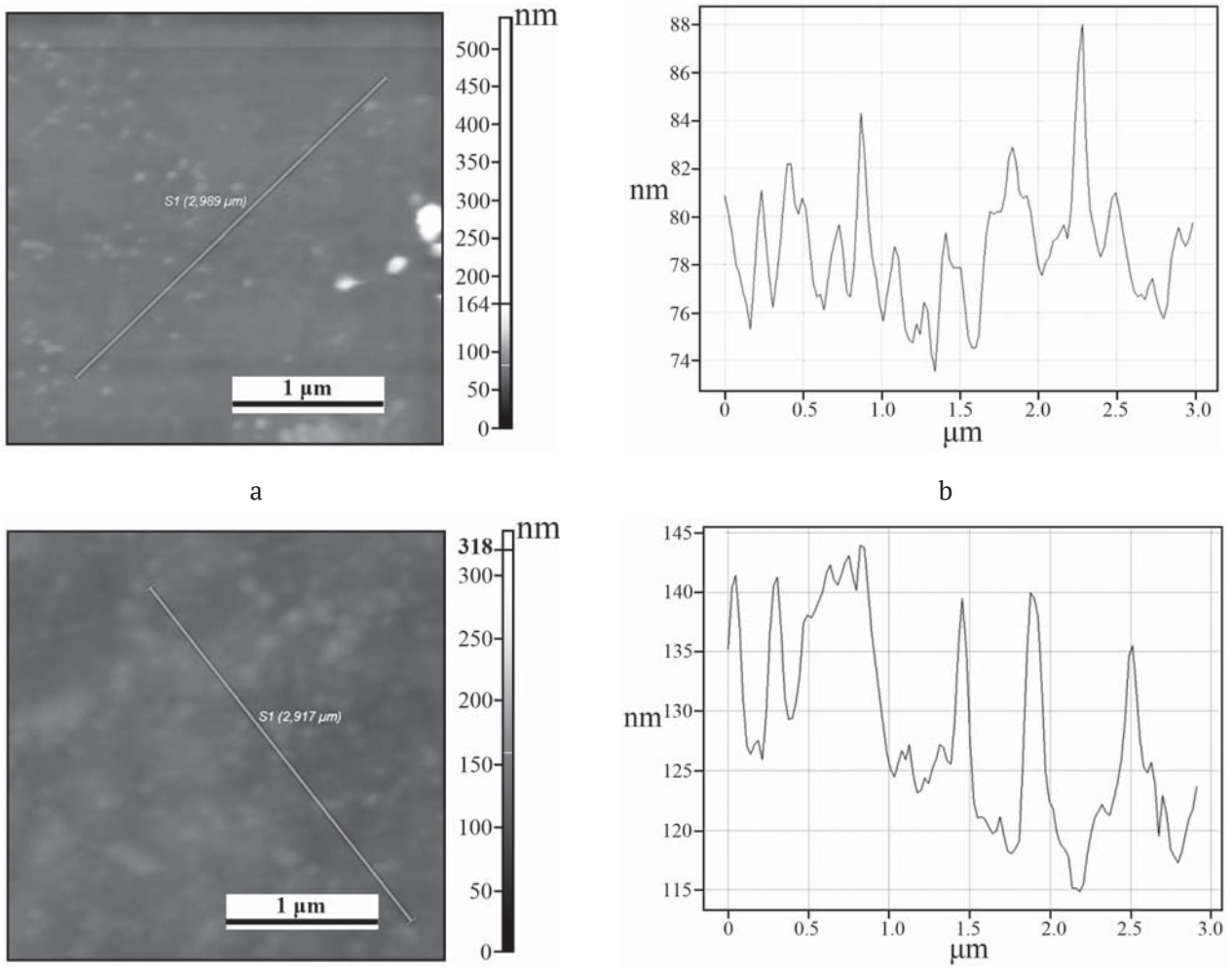
4. Conclusions

The chemically stimulated oxidation of gallium arsenide with a nanoscale layer of



a b

Fig. 2. AFM image of the surface of a non-oxidized $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructure: a) topography; b) profile. The size of the scanning area 3×3 μm²



a b

c d

Fig. 3. AFM images of the films surface (a, c) and profile (b, d) formed by the oxidation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructures at 490 °C, 60 min. (a, b) and 550 °C, 60 min. (c, d). The size of the scanning area is 3×3 μm²

manganese vanadate-phosphate on the surface proceeds via a transit mechanism. This was evidenced by the EAE value of the process (of the order of 156 kJ/mol), which was somewhat higher in comparison with the standard oxidation of GaAs (~ 110 kJ/mol). According to the XRD results, V_2O_5, a chemostimulator with a pronounced catalytic mechanism of action, initially present in the starting vanadate-phosphate was not detected in the films, which confirms the absence of a catalyst regeneration cycle $V_2O_5 \leftrightarrow VO_2$. Films formed by the oxidation of $Mn_3(P_{0.1}V_{0.9}O_4)_2$/GaAs heterostructures were in the thickness range of 50-200 nm. They mainly contained manganese and gallium arsenates and had expressed relief with a grain size in the range of 200-300 nm.

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Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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