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**MICRO AND NANO TECHNOLOGIES
SPACE TECHNOLOGIES AND PLANETARY SCIENCE**

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**MICRO AND NANO TECHNOLOGIES,
SPACE TECHNOLOGIES AND PLANETARY SCIENCE**

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ELECTROCHEMICAL ETCHING OF P-TYPE GALLIUM PHOSPHIDE**Dr. Gauhar Mussabek**^{1,2}**Dana Yermukhamed**¹**Saniya Sarsembek**¹**Kazakhbay Almasuly**¹**Dr. Vladimir Sivakov**³¹al-Farabi Kazakh National University, **Kazakhstan**²National Research Nuclear University "MEPhI", **Russia**³Leibniz Institute of Photonic Technology, **Germany****ABSTRACT**

Gallium phosphide is a semiconductor material of great interest for new LED technologies. Porous $A^{III}B^V$ are of great interest because of their interesting optical properties different of those for bulk material. The main goal of this research work is to obtain nanostructured porous gallium phosphide (por-GaP) layers by electrochemical etching of the monocrystalline (100) p-type GaP substrate surface. The structure and surface morphology of obtained samples were studied by scanning electron microscopy (SEM), atomic force microscopy (AFM) and Raman spectroscopy. The chemical composition of nanostructured GaP surfaces was studied by Energy Dispersive X-Ray (EDX) spectroscopy. It was found that shape and size of structures is strongly depending on electrochemical etching conditions. At constant applied voltage and varied current we observe that the pore size is increasing from 10 microns to 50 microns, and at fixed current, but varying the voltage the pore diameters can be achieved in the range between 100 to 200 nanometers.

Keywords: gallium phosphide, porous structure, electrochemical etching, SEM, AFM.

INTRODUCTION

Due to their unique physical and chemical properties, semiconductor nanomaterials include a wide range of applications [1-5]. Among modern electronic device technologies LED is one of the fast growing in the world [6]. Gallium phosphide and related nanomaterials are of great interest as a research object for modern LED technologies [7]. Porous gallium phosphide (por-GaP) is a very promising material for various photonic applications [8]. The first reports on por-GaP obtaining and characterization are referred since 1990s [9]. One of the most effective ways of obtaining porous structure from the $A^{III}B^V$ materials is electrochemical etching [10]. The advantages of the method are good process controllability and relative cheapness. By changing the parameters of electrochemical etching, such as current density, voltage and duration of etching, one can obtain porous structures of different morphology [11]. However, in most of reports there are description of n-type por-GaP, and the less is for p-type ones [12, 13]. Present paper is devoted for investigation of formation features of p-type por-GaP layers and its structure.

EXPERIMENTAL

Porous GaP films were obtained by electrochemical etching of the single crystalline (100) oriented, p-type GaP substrate surface using electrolyte containing the mixture of ethanol (C_2H_5OH) and 40% hydrofluoric acid (HF), taken in a volume ratio of 1:1. Electrochemical etching was carried out in a fluoroplastic etching cell. In our experiments applied voltage was varied from 30 to 65 V, current - from 1 to 10 mA and etching time - between 10-40 minutes. *Figure 1* shows the schematic representation of experimental electrochemical etching process.

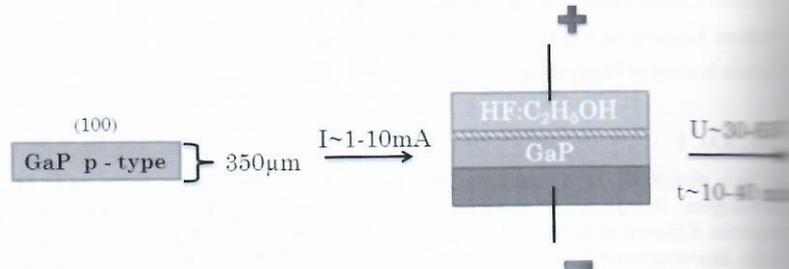


Figure 1. Schematically representation of electrochemical etching process of the surface of GaP substrate.

The structure and morphology of obtained samples were studied by SEM using an ULTRA 55 FE-SEM (Carl Zeiss) microscope, AFM measurements were provided on Integra Spectra (NT-MDT). Raman spectra of samples were measured on Sander Spectrum (NT-MDT), excitation wavelength 473 nm, acquisition time was 30 s, diameter of laser spot was 2 μ m. The chemical composition of obtained surfaces was studied by Energy Dispersive X-Ray (EDX) spectroscopy. Energy-dispersive X-ray spectra of samples were measured using the Bruker Quantax Flatquadr. Raman spectroscopy measurements were carried out in air at room temperature.

RESULTS AND DISCUSSION

The process of porosity correlates directly with voltage, current density and etching time. One of the aims of our experimental study was to find up the optimal technological conditions of electrochemical etching for porous structures formation. The top view SEM images of por-GaP samples, obtained under different modes of electrochemical etching are shown in *Figure 2a and 2b*. One can see that the structural properties of por-GaP layers are directly depend on the parameters of electrochemical etching. It is experimentally shown that the formation of a uniform porosity on the surface is observed when the etching voltage is greater than 50 V. In this case, the surface structure of the samples is uniformly flat, and pore size are almost the same. The sample shown in *Fig. 2a* pores sizes varied from 10 to 100 nm, thus structure could be considered as a nanoporous material. In addition, structure shown in *Fig. 2b* has non-

uniform structure with pore sizes varied from 10 to 100 nm. The phenomenon can obviously be explained by the influence of electromagnetic field on the distribution of material in one (see *Fig. 2b*).

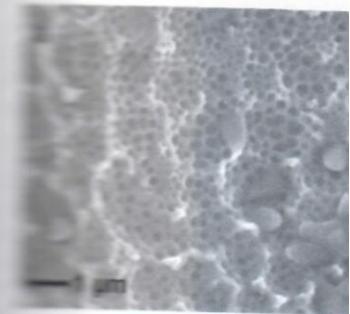


Figure 2. A top view SEM images of porous GaP surface obtained under: (a) $U = 65$ V, $I = 10$ mA, $t = 30$ min.

The surface morphology was also studied by AFM. The AFM images of the porous GaP surface are shown in *Figure 3a and 3b*. The surface of the sample is easily polished. The AFM images were obtained using the Bruker MultiMode head with scanning currents densities up to 5 mA/cm² for up to 30 minutes.

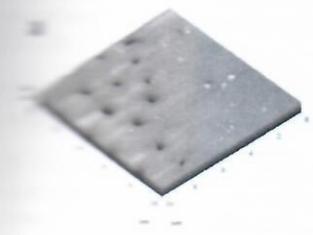
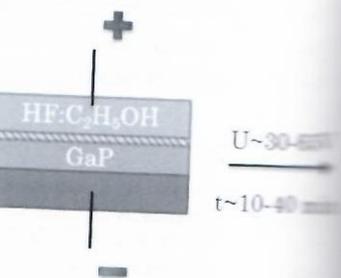


Figure 3. AFM images of porous GaP surface obtained under: (a) $U = 30$ V, $I = 5$ mA, $t = 25$ min.

chemical etching of the single crystalline GaP substrate in an electrolyte containing the mixture of hydrofluoric acid (HF) and ethanol (EtOH), taken in a volume ratio of 1:1. The etching was performed in a fluoroplastic etching cell. In order to study the influence of the voltage up to 65 V, current - from 1 to 10 mA and etching time up to 60 min. Figure 2 shows the schematic representation of the electrochemical etching process of the GaP substrate.



Electrochemical etching process of the GaP substrate.

The surface morphology of the obtained samples was studied by SEM using an S-3400N scanning electron microscope. AFM measurements were provided on the obtained samples. The AFM images were measured on Solver 3.0 software. The acquisition time was 30 s. The composition of obtained surfaces was studied by X-ray photoelectron spectroscopy. Energy-dispersive X-ray analysis was performed on a Bruker Quantax Flatquad. Measurements were taken at room temperature.

The influence of the voltage, current density and etching time on the surface morphology was studied. The aim of the study was to find up the optimal parameters for porous structures formation. The obtained porous structures are shown in Figure 2b. One can see that the structure of the porous layers is dependent on the parameters of electrochemical etching. The formation of a uniform porosity on the surface of GaP is reached at a voltage higher than 50 V. In this case, the pore sizes are almost the same. The pore size is about 100 nm, thus structure could be used for the applications shown in Fig. 2b has main

features. The porous structure with pore sizes varied from 200 nm to 40 μm. It is clear that the surface morphology of samples obtained under low voltage and current intensity is non-uniform. This phenomenon can obviously be explained due to the different rate of dissolution under the influence of electromagnetic fields of different powers, which leads to anisotropic dissolution of material in one (see Fig. 2a) or several directions (see Fig. 2b).

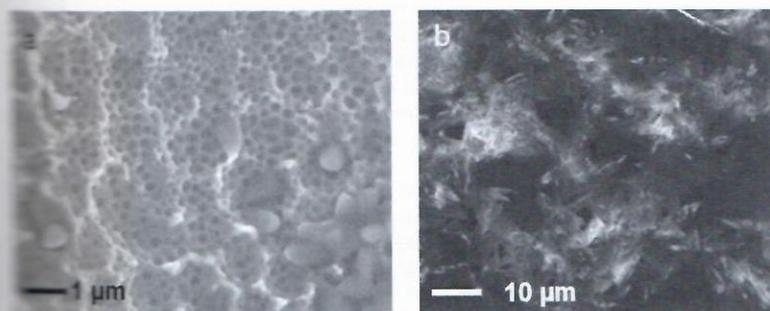


Figure 2. A top view SEM images of por-GaP surface obtained by electrochemical etching under: (a) $U = 65 \text{ V}$, $I = 10 \text{ mA}$, $t = 15 \text{ min}$ and (b) $U = 30 \text{ V}$, $I = 7 \text{ mA}$, $t = 17 \text{ min}$.

The surface morphology was also studied by atomic force microscopy (AFM) which allowed us to observe the pores size distribution on the surface of por-GaP layers. The top view AFM images of the por-GaP obtained under different electrochemical etching parameters are shown in Figure 3a and 3b. It is notable that the surface of porous layers becomes flattened after electrochemical treatment. When the etching time is longer, the surface of the sample is easily polished. The lightly polishing effect can be reached at low modifying currents densities up to 5 mA/cm^2 , voltages up to 40 V and the etching time up to 30 minutes.

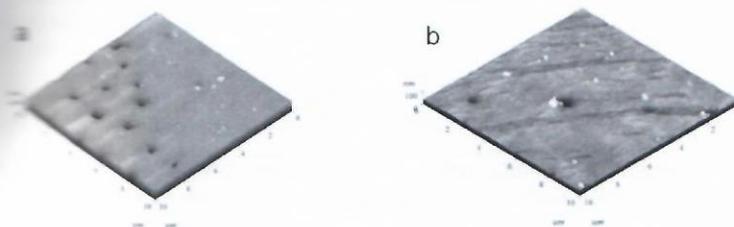


Figure 3. AFM images of por-GaP surface obtained by electrochemical etching under: (a) $U = 30 \text{ V}$, $I = 5 \text{ mA}$, $t = 25 \text{ min}$ and (b) $U = 40 \text{ V}$, $I = 1 \text{ mA}$, $t = 60 \text{ min}$.

The method of energy-dispersive X-ray spectroscopy was used in order to identify the elemental composition of samples. As shown in *Figure 4*, the basic element of the por-GaP is mainly gallium. The reason of such appearance is in the faster dissolution rate of phosphorus atoms rather the gallium ones. Since the initial substrate of crystalline p-type GaP is doped by Zn, there is also a signal of Zn appeared in EDS spectra. One can see that formation of pores during electrochemical etching process is mainly due to dissolution of phosphorus in electrolyte. Broken bonds of Ga are passivized by oxygen. Thus por-GaP walls separating the pores are formed by Ga₂O₃. Therefore, most of the porous skeletons are composed of Ga and GaO. According to data the sample also contains C, F, Zn, and Al, except of Ga, P and O elements in small amounts.

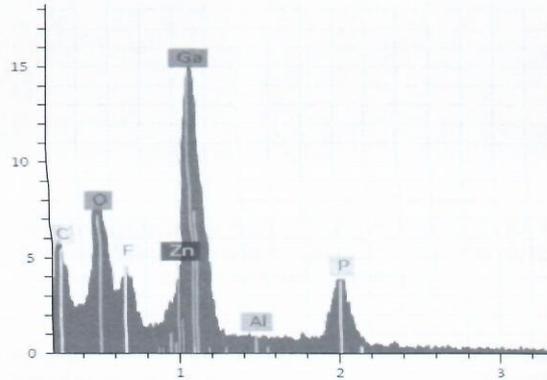


Figure 4. Energy-dispersive X-ray spectrum of por-GaP sample obtained by electrochemical etching under $U=30$ V; $I=5$ mA; $t=40$ min.

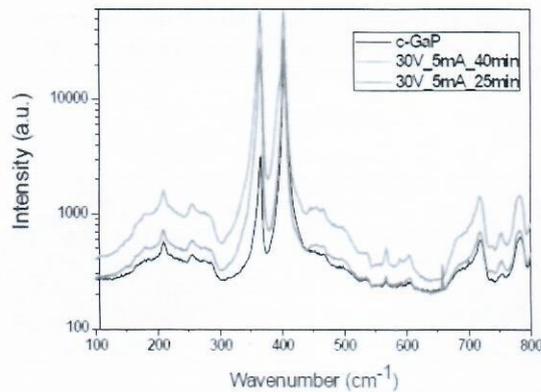


Figure 5. Raman spectra of por-GaP samples obtained by electrochemical etching under different conditions.

One of the linear optical methods for nanostructures is Raman spectroscopy. The acquisition time was 30 s, diameter was 100 μ m, exciting laser power 30 W, crystalline GaP and por-GaP samples were etched for different times. It should be noted that both peaks (360 cm^{-1} and 404 cm^{-1}) appeared in por-GaP. The 360 cm^{-1} signal has a high intensity, which increases with etching time. The 404 cm^{-1} signal appears at the beginning of phase transition.

CONCLUSIONS

The electrochemical etching method was used to study the effect of technological conditions on the properties of the obtained porous gallium phosphide. The uniformity of the porous structure was investigated. The uniformity of the porous structure was higher when the anodizing voltage was higher. In this case, the surface morphology appears. Energy-dispersive X-ray spectroscopy shows that formation of pores during electrochemical etching is due to the dissolution of phosphorus in electrolyte. The porous structure of the long etched (40 minutes) sample is more uniform. The concentration of LO- and TO-oscillation in the porous GaP is preserved. Obtained porous GaP has different physical and chemical properties of p-type GaP. It has different applications in photonics and optoelectronics.

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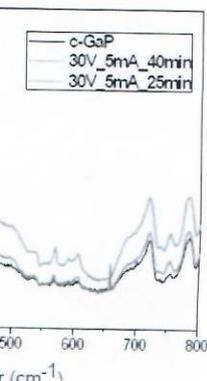
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scopy was used in order to identify... shown in Figure 4, the basic element... such appearance is in the faster... um ones. Since the initial substrate... is also a signal of Zn appeared in... during electrochemical etching process... in electrolyte. Broken bonds of Ga... separating the pores are formed by Ga... composed of Ga and GaO. According... l, except of Ga, P and O elements in anal...



Spectrum of por-GaP sample obtained by... J=30 V; I=5 mA; t=40 min.



obtained by electrochemical etching under... conditions.

One of the linear optical methods for estimating the surface morphology and structure of nanostructures is Raman spectroscopy. In our experiments excitation wavelength was 633nm, acquisition time was 30 s, diameter of laser spot was 2µm, enhancement factor was 100x, exciting laser power 30W. Figure 5 represents Raman spectra of the crystalline GaP and por-GaP samples obtained by electrochemical etching at different etching times. It should be note that both LO and TO oscillations modes of GaP (at 366 cm⁻¹ and 404cm⁻¹) appeared in por-GaP layers spectra. One can see that at long etching time signal has a high intensity, which shows that the structure has changed. Por-GaP sample etched for 40 minutes also has extended shoulders in Raman spectra, which shows the beginning of phase transition from monocrystalline to the amorphous phase.

CONCLUSIONS

Electrochemical etching method was used to obtain porous gallium phosphide, and effective technological conditions were formed. The dependence of the structural properties of the obtained porous gallium phosphide on the electrochemical etching parameters was investigated. The uniform porosity structure formation was observed when the anodizing voltage was higher than 50 V. In such a case, the surface structure of the samples becomes evenly flat. In such modes, the effect of light polishing on surface morphology appears. Energy-dispersive X-ray spectrum of por-GaP samples shows that formation of pores during electrochemical etching process is mainly due to dissolution of phosphorus in electrolyte. Raman spectroscopy showed that the structure of the long etched (40 minutes) samples was close to the amorphous phase. The high concentration of LO- and TO-oscillations shows that the crystalline direction of the first monocrystal GaP is preserved. Obtained results can be useful in further investigations of physical and chemical properties of p-type porous gallium phosphide nanostructures for different applications in photonics and opto-electronics.

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ELECTROCHEMICAL METHOD FOR NANOSTRUCTURATION AND FUNCTIONALIZATION OF SURFACES AND BIOMATERIALS

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ABSTRACT

Electrochemical method for the preparation of functionalized surfaces through active biomolecules. There are two applied electrochemical methods: (i) electrochemical oxidation or anodization of metal surfaces to form nanostructured oxide films and (ii) electrochemical deposition process of nanocomposite layers. This article presents a summary of results obtained by electrochemical techniques in obtaining advanced functionalized surfaces. The characterization in terms of surface roughness and layers thicknesses, corrosion properties as nanohardness or wear resistance, and electrochemical deposition with other electrochemical methods can lead to a large class of nanocomposite coatings (films) on different substrates for a future based on nanotechnology. The properties of materials face of aggressive environments in specific environments give applications by increasing the efficiency and durability. **Keywords:** electrochemical methods, nanocomposite coatings, electrochemical oxidation, electrodeposition.

INTRODUCTION

In recent years, the development of nanotechnology has led to an explosion of new functional coatings and materials in the coming years. Functional and intelligent coatings have attracted enormous technological interest [1-12]. Out of the various surface modification and protection, the advantages of electrochemical methods applied to improve their properties. Electrochemical methods are within the author's field of expertise [1-12].

The electrochemical methods are applied in various fields with specific applications, but both with a view to the resistance to degradation of surfaces in specific