

# Evolution of Pore Growth in GaAs in Transitory Anodization Regime from One Applied Voltage to Another

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**Abstract**—The paper reports the results of investigation of the pore growth during anodic etching of (111)-oriented wafers of Si-doped *n*-GaAs in an environmentally friendly NaCl based electrolyte, with switching the applied voltage from a high voltage to lower one and vice-versa. Switching of the applied voltage in the process of anodization was found to cause the formation of layered porous structures with different degrees of porosity. Crystallographically oriented pores shaped as triangular prisms were produced in a stationary regime of anodization, while a more complex morphology of pores was observed at the interface between the two layers with different degrees of porosity, including pores composed of three circular ones. Based on the results of the morphology study using scanning electron microscopy, a possible mechanism of the formation of such kind of pores in the dynamic transitory regime of anodizing is discussed.

**Keywords:** successive anodization, porous GaAs, crystallographically oriented pores, triangular shape, round shape

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## INTRODUCTION

Porous templates with tailored architecture on the submicrometer scale are nowadays widely used in nanofabrication and production of various composite materials [1–4]. Among porous materials, semiconductor compounds provide a wide space for tailored nanofabrication, due to their diversity of compositions, bandgaps, and mechanisms of the pore growth [5]. Over the last two decades, it has been demonstrated that electrochemistry is one of the most accessible and cost-effective approaches for the fabrication of porous semiconductor materials with various morphologies and properties, which open large potential for applications [6–9].

It is worth to mention that semiconductor compounds have some important advantages in comparison with silicon. For example, (i) gallium arsenide has six times higher electron mobility than silicon, which allows a faster operation [7]; (ii) a wider band gap, which allows operation of power devices at higher temperatures and gives a lower thermal noise to low power devices at room temperature [10, 11]; (iii) the direct band gap provides conditions for the occurrence of more pronounced optoelectronic properties in GaAs than in silicon with its indirect band gap; (iv) GaAs can be alloyed to ternary or quaternary compo-

sitions adjusting in this way the width of the band gap, thus allowing light emission at chosen wavelengths.

The morphologies and properties of the produced porous semiconductor materials are determined by the mechanisms of the pore growth during electrochemical etching of the bulk semiconductor wafers [5]. Depending on the mechanism of growth, pores with different characteristics are formed in terms of their shape, velocity of growth, etc. On the other hand, the pore growing mechanism depends on the characteristics of the initial bulk semiconductor material and the specific anodizing conditions [12]. Usually pores grow perpendicular to the semiconductor surface. To make the porous nanotemplates perspective for nanofabrication processes, the authors developed methods for the preparation of templates with pores oriented parallel to the top surface of the substrate [13]. Such kind of porous structures are of pronounced interest for the fabrication of two-dimensional and three-dimensional photonic crystals including metallo-dielectric ones, since this geometry allows a wide implementation of structures due to a large surface of samples as compared to the geometry with pores propagating perpendicularly to the surface. Moreover, this approach provides conditions for reaching spectacular porous architectures by applying specially designed masks on the sample surface subjected to anodization [14].

It was found that three types of pores can be generated in semiconductor compounds: current line oriented (CLO or curro pores), crystallographically oriented (CO or crysto pores), and fractal pores [5, 6]. Pores classified as fractal due to their fractal-like way of growth have been observed in Si, III–V and II–VI semiconductor compounds. While a fractal is normally defined as an object that can be divided into parts, and each of these parts will be similar to the original object, each point of a fractal pore can be a source for one or more similar pores growing in totally different directions.

Only curro pores have been observed up to now in II–VI semiconductor compounds such as ZnSe and CdSe [15–18], while both types of CO and CLO pores have been observed in III–V semiconductor compounds [5, 6, 19], except for GaAs, in which no curro pores have been observed so far. At the same time, in single crystalline GaAs, an intersection of crysto pores was observed in premiere [20] which allowed to propose and fabricate three-dimensional photonic structures with pores propagating along  $\langle 111 \rangle$  crystallographic directions (the so-called “*Moldavite*” structures [21]). In sphalerite crystalline phase inherent to GaAs, the pores grow along  $\langle 111 \rangle_B$  crystallographic directions, whatever the initial surface orientation is, the angle between pores being approximately of  $109^\circ$  [5]. Apart from that, a new type of pores, namely tetrahedron-like pores, have been brought to light during anodic etching of (100) oriented *n*-GaAs [12].

The growth of curro pores follows the current flow. As a result, they are always perpendicular to the equipotential surfaces in the sample. Therefore, they grow mostly perpendicularly to the plate surface exposed to the electrolyte, the current flow being provided by an electrical contact on the opposite surface of the sample. It was suggested that the growth of curro pores is determined by a high ionicity of II–VI compounds [22, 23]. It was established that in compounds with a lower ionicity, such as III–V ones, CO pores grow at current densities lower than a certain threshold value, whereas CLO pores grow at current densities higher than the threshold value [6]. The threshold value of the current density was found to be determined by a combination of several factors, such as the free carrier density in semiconductor crystal, electrolyte concentration, and temperature.

The purpose of this work was to reveal the features of the pore growth in GaAs in a dynamic regime of anodization occurring at transition from one applied voltage to another one, in order to understand if the curro pore growth is also possible or only crysto pores are formed, similarly to the regime of stationary anodization. A NaCl based electrolyte was chosen, taking into account its environmentally friendly nature.

## DESCRIPTION OF MATERIALS AND TECHNOLOGICAL PROCEDURES

In this study, crystalline 500- $\mu\text{m}$  thick (111)-oriented substrates of Si-doped *n*-GaAs with a free electron concentration of  $2 \times 10^{18} \text{ cm}^{-3}$  were used. Before anodizing, the wafers were cleaned by sonication in acetone for 15 min, washing in distilled water, and dried. The native oxide was removed from the surface by dipping the sample in an HCl/H<sub>2</sub>O solution with a ratio 1 : 3 for 2 min. The sample with the electrical contact prepared with a silver paste was pressed against an O-ring in a Teflon cell as shown in Fig. 1, and the surface of 0.2 cm<sup>2</sup> area was exposed to a 1.75 M NaCl electrolyte. The anodizing was performed in a potentiostatic regime at room temperature ( $T = 23^\circ\text{C}$ ) in three electrode configurations, with a Pt mesh with the surface area of 6 cm<sup>2</sup> acting as counterelectrode, a saturated Ag/AgCl reference electrode, and the sample as working electrode. The potentiostat was fully controlled via a PC unit. The steering of the electrolyte was provided by a Teflon agitator connected to the motor. The rotation speed of 100 rpm was controlled by the applied potential from an external power supply to the motor. The morphology and chemical composition analyses of the anodized GaAs wafers were investigated using Zeiss Sigma and TESCAN Vega TS 5130 MM scanning electron microscopes (SEM).

## RESULTS AND DISCUSSIONS

Figures 2a, 2b compare the pore growth in GaAs wafers with different crystallographic orientations. As mentioned above, pores grow always along the  $\langle 111 \rangle_B$  directions in GaAs. This fact is demonstrated by the experiments which show that pore etching on  $\{111\}_A$  and  $\{111\}_B$  surfaces produce different results [24]. The morphology of the porous structure produced by anodizing the (100) surface consists of two sets of  $\langle 111 \rangle$  crystallographically oriented pores intersecting each other [5]. The direction of the pore growth forms an angle of  $54.5^\circ$ , with the normal to the (100) surface of the anodized wafer (Fig. 2a). However, this refers only to the primary pores. Li et al. observed secondary branched pores growing in the  $\langle 001 \rangle$  direction, i.e. perpendicularly to the (100) plate surface [25]. On the other hand, the primary pores grow perpendicularly to the (111)<sub>B</sub> oriented GaAs plate (Fig. 2b). Note that the pores tend to have a triangular cross-section, the pore walls and tips exhibiting a pronounced crystallographic anisotropy with  $\{112\}$  plane as pore walls [5] (see the inset in Fig. 2a). With growing the triangular cross section at long anodization duration and higher applied voltage, the pore walls are etched and high aspect ratio nanowires with triangular cross section were produced [24]. Triangular pillar arrays were also produced by anisotropic chemical etching of triangular pore arrays formed initially by anodic etching in an HCl electrolyte of (111) GaAs [26].

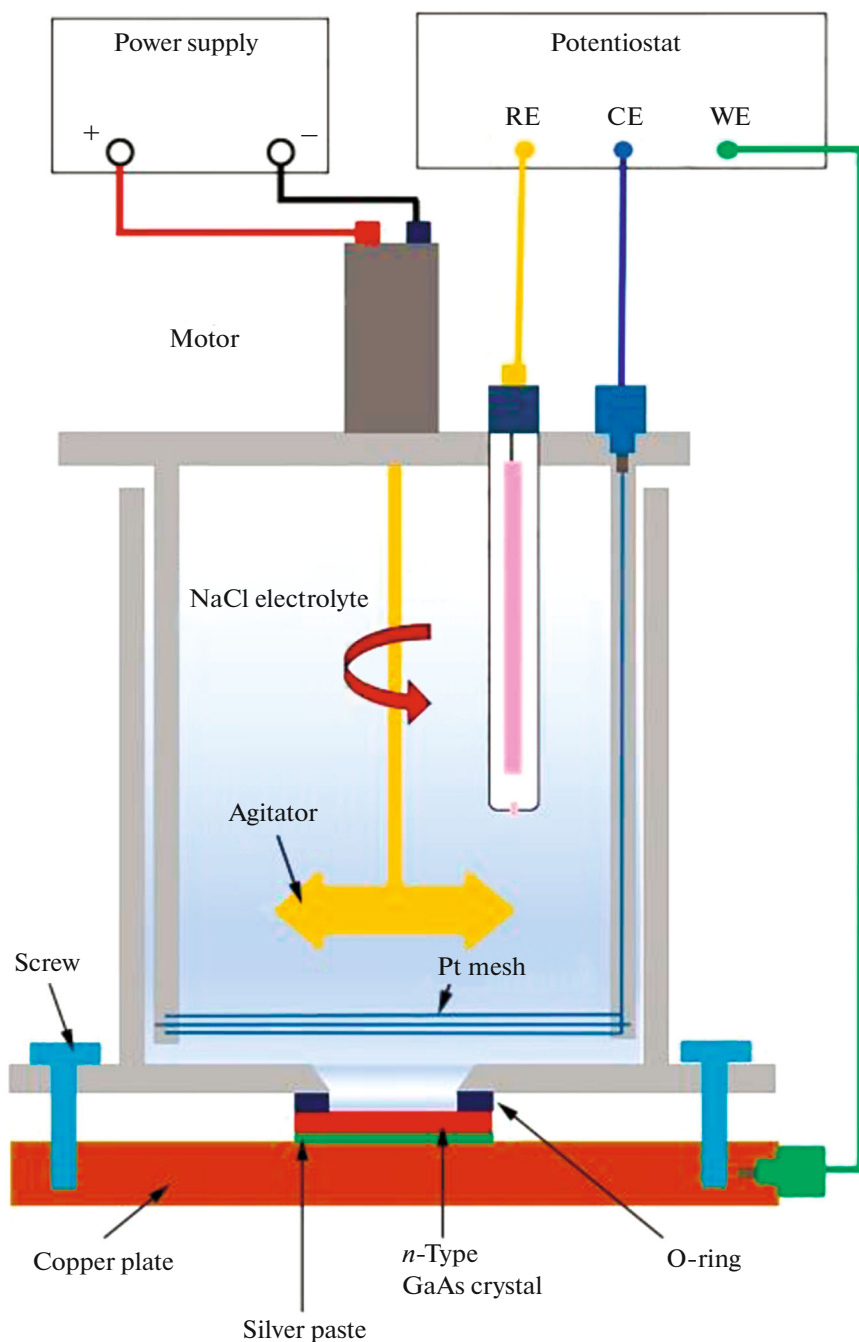
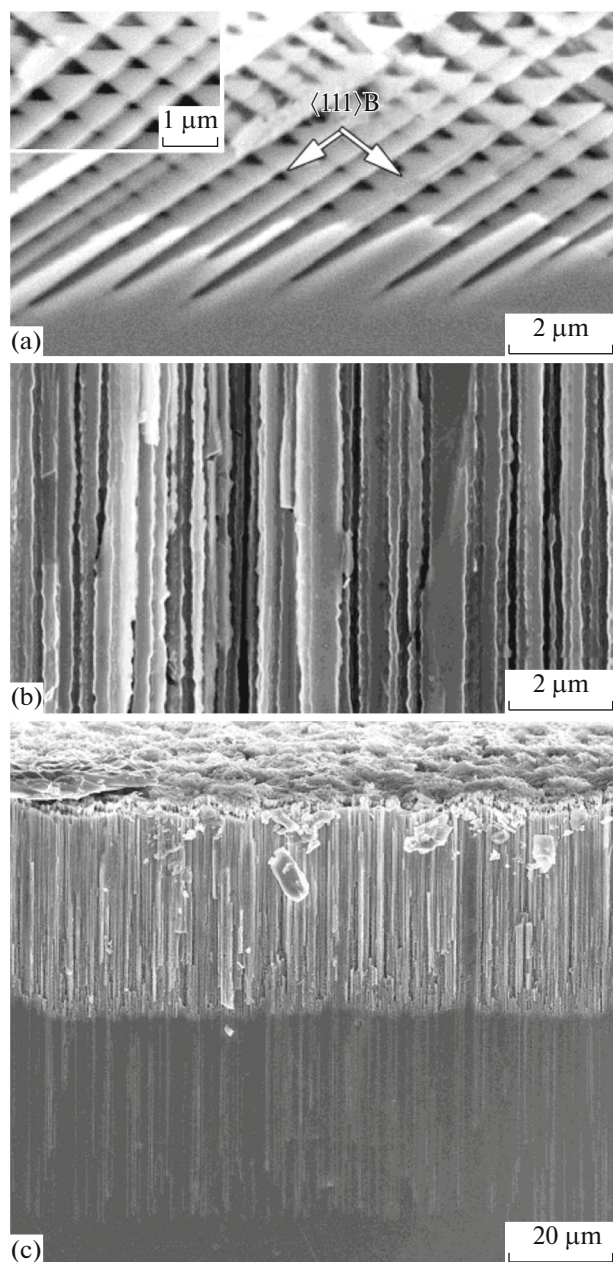


Fig. 1. Schematic presentation of the electrochemical etching setup.

Further investigations discussed in this paper are focused on pore growing in a dynamic regime of a transition voltage from 3 to 2 V at the interface of layers with different degrees of porosity. One can see from Fig. 3a that the pores formed at the upper surface of the GaAs plate have a triangular shape indicating that they are crystal pores. However, the shape of pores is more complex at the interface between the two layers with different degrees of porosity produced

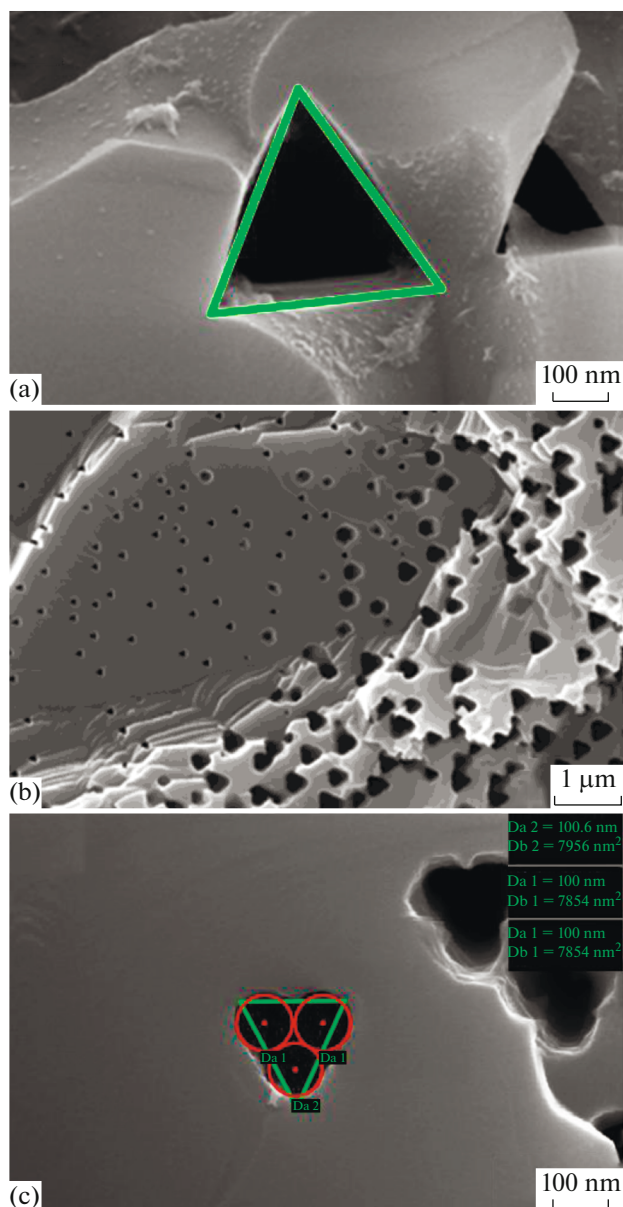
as a result of changing the applied voltage from 3 to 2 V (Fig. 3b).

It is well known that the diameter of the produced pores is directly influenced by the value of the applied voltage during anodizing. Pores with different sizes are observed in Fig. 3b. Note that the pores belong to different steps of cleavage. At the same time, one can find pores with different sizes even in the same cleavage plane. This observation can be explained by a slightly



**Fig. 2.** (a) SEM image in cross-section of a porous GaAs layer produced on *n*-GaAs (100) wafer anodized in a 5% H<sub>2</sub>SO<sub>4</sub> electrolyte in the galvanostatic regime at constant current  $j = 20 \text{ mA/cm}^2$  during 60 min. For the uniform nucleation of pores, the surface was preliminarily subjected to pulsed anodizing with a pulse amplitude  $j = 300 \text{ mA/cm}^2$  and a pulse width  $t = 0.8 \text{ s}$ . (Reproduced with permission from Ref. 5); (b) SEM image of a porous layer produced by anodizing the (111)B GaAs wafer in 1.75 M NaCl electrolyte in potentiostatic regime with applied voltage of 3 V. (Reproduced with permission from Ref. 24); (c) a two-layer porous structure produced by changing the applied voltage from 3 to 2 V during anodization in 1.75 M NaCl electrolyte. (Reproduced with permission from Ref. 24).

different growth speed of pores, which results in evidencing pores at different levels of transition in a cleavage plane. Apart from that, a detailed analysis of



**Fig. 3.** (a) SEM image of pores at the surface of the GaAs plat obtained at the applied voltage of 3 V; (b) morphology of pores at a cleavage between the porous layers with different degrees of porosity; (c) analysis of the shape of pores at the interface.

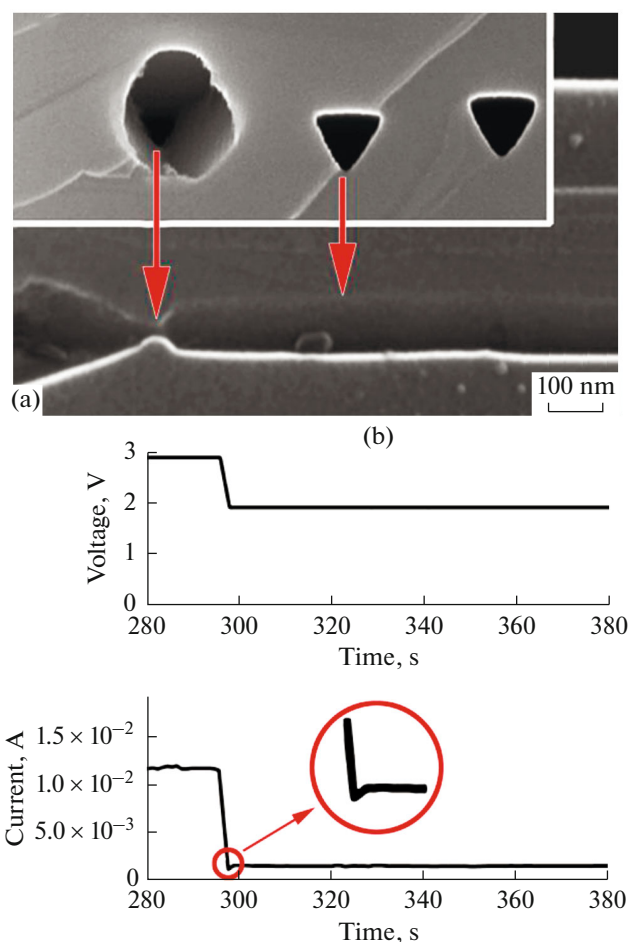
those pores (Fig. 3c) indicates that their triangular shape is formed of three circular pores with a diameter around 100 nm. Usually, curro pores have a circular form. However, as mentioned in the Introduction section, the analysis of literature data revealed no curro pore formation in GaAs. In this context, a question arises about the nature of pores with the shape illustrated in Fig. 3c formed in the transitory regime from a high to a low voltage.

The evolution of the pores in the depth of the transition region was investigated to throw light on this issue. As one can see from a comparison of images in Figs. 3a and 4a, that the transition of a triangular pore with a big size of 400 nm to a triangular one with a small size of 100 nm occurs not instantly with switching the anodizing voltage from 3 to 2 V, but smoothly through the intermediary formation of three circular pores. However, those circular pores are not curro pores. As shown in Fig. 4b, the change of the voltage from 3 to 2 V results in a more significant decrease of the current from 12 to 2 mA. Apart from that, even a deeper decrease of the current occurs before the stabilization of the current at the level of 2 mA, as shown in the inset of Fig. 4b. This peculiarity leads to the formation of a bottleneck-like feature in the cross-section of the pore marked by the left-hand arrow in Fig. 4b.

The transformation of triangular pores produced initially by anodic etching in an HCl electrolyte of (111) GaAs to larger circular pores oriented perpendicularly to the (111)B surface was previously observed upon the second stage of anodization in citric acid performed at a high applied voltage (100 V) [27]. However, those pores are not curro pores because they do not grow in a direction perpendicularly to the surface. Actually, the second stage of anodization just enlarges primarily formed triangular pores, and the circular form is obtained due to the isotropic etching of primary pore walls. One can suppose that a similar isotropic etching occurs in the dynamic regime of anodizing determined by the derivative of the current ( $dI/dt$ ), which results in the formation of three circular pores instead of one triangular pore, as shown in Fig. 4b.

It is worth to mention that circular shaped pores were also reported at the surface of (100) GaAs wafers anodized in a mixture of sulfuric acid with dimethylformamide solutions [28]. Again, those pores cannot be classified as curro pores, in spite of their circular shape. First of all, they were circular pits on the GaAs plate surface rather than authentic pores since they propagated in the wafer at depths much shorter than one micron. Apart from that, the morphology of the pores was not investigated in the cross section to demonstrate that their propagation followed the current flow, not being produced by just isotropic etching initiated at specific sites of pore nucleation.

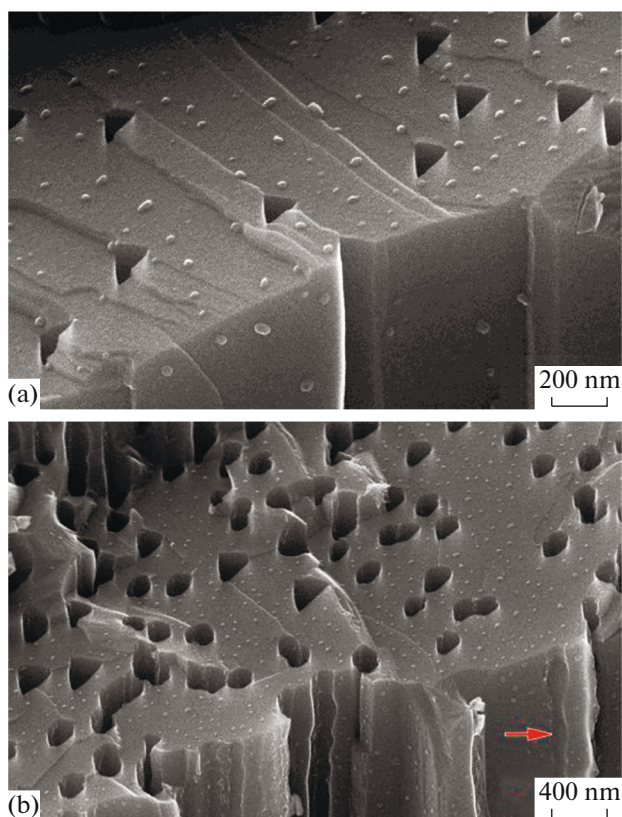
Apart from functions of isotropic etching inherent to the dynamic regime of anodizing, the etching process resemble also, to some extent, the production of a chain of tetrahedron-like pores in GaAs through a “tetrahedron sliding process” taking place in the dynamic regime of voltage oscillations [5, 12]. Those similarities are even more evident when the applied voltage during anodizing is switched from a low value to a higher one. Figure 5 compares the morphology of pores produced in the stationary regime at a low volt-



**Fig. 4.** (a) SEM image in cross-section illustrating the transition of a pore with big size to one with small size upon switching the anodizing voltage from 3 to 2 V. Inset is the frontal view; (b) the diagram of voltage and current in the transition process.

age (2 V) with the morphology of pores obtained in a dynamic regime of switching from a low applied voltage (2 V) to a high one (3 V).

Again, the shape of pores changes from small triangular pores (around 100 nm) growing deep inside the GaAs wafer to larger pores with a more complex shape, including circular pores and pores composed of three circular pores, produced in the transitory regime. Therefore, the transitory regime does matter, i.e. the existence of the derivative of the current is important, but not the sign of the derivative. Whatever is the sign of the derivative of the current, negative or positive, the pore growth mechanism is the same. The only difference is the emergence of current oscillations when the current is switched from a low value to a higher one, as illustrated in Fig. 6. Such oscillations lead to modulations of the diameters of pores, as shown by an arrow in Fig. 5b, and those oscillations

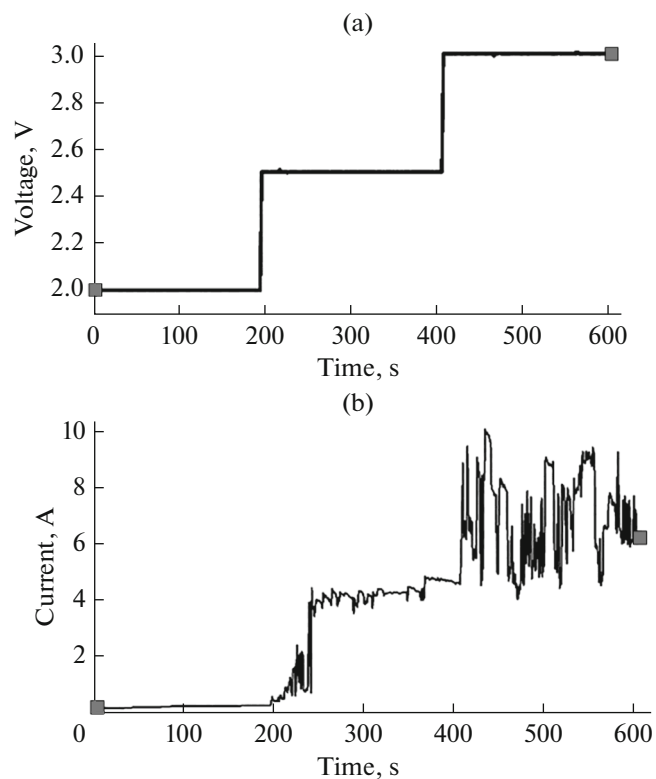


**Fig. 5.** (a) SEM image of pores produced in a GaAs wafer anodized at a stationary applied voltage of 2 V; (b) SEM image of pores obtained in a transitory regime of anodizing with switching the applied voltage from 2 to 3 V.

resemble the mechanism of the tetrahedron-like pore growth, as mentioned above.

## CONCLUSIONS

The results of this study demonstrate the difference between the mechanisms of the pore growth during electrochemical etching of GaAs wafers in the stationary and dynamic regimes of anodizing. Crystallographically oriented pores with the shape of triangular prisms with  $\{112\}$  plane as pore walls were found to grow along the  $\langle 111 \rangle_B$  direction in a stationary regime of anodizing. At the same time, complex pores composed of three circular pores were observed to be produced in the dynamic transitory regime of anodizing during the switching of the applied voltage from a high value to a lower one or vice-versa. The analysis of such pores suggests that they are not current line oriented, in spite of their circular form, in concordance with previous literature data. Such shape of pores can be explained by isotropic etching of a primary triangular pore wall formed during stationary anodizing, which occurs in the subsequent dynamic regime of anodizing determined by the derivative of the current. As a result of comparison of the features of pore formation in the



**Fig. 6.** The diagrams of: (a) voltage; and (b) current, during a consecutive switching of voltage from 2 V to 2.5 V and to 3 V.

dynamic regime of anodizing under two different signs of the derivative of the current, it was established that the trends of pore evolution are similar. However, switching of the applied voltage from a low value to a higher one was found to induce current oscillations, in contrast to the opposite switching. Some similarities were brought to light between the pore growth in such dynamic regime of etching and tetrahedron-like pore growth previously observed in GaAs.

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