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Oxide crystals on the surface of porous indium phosphide

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ABSTRACT

Purpose: If this paper is to is to establish the patterns of oxide formation on the surface of indium phosphide during electrochemical etching of mono-InP.

Design/methodology/approach: A porous surface was formed with the anode electrolytic etching. Morphology of the surface was studied with the help of scanning electron microscope JSM-6490. The analysis of chemical composition of porous surface of samples was also performed.

Findings: It was shown that during the electrochemical etching of indium phosphide, oxide films and crystallites form on the surface. It has been established that crystalline oxides are formed mainly on the surface of n-type indium phosphide. Continuous oxide films are predominantly formed on the surface of p-lnP.

Research limitations/implications: The research was carried out for indium phosphide samples synthesized in the solution of hydrofluoric acid, though, carrying out of similar experiments for crystalline oxides on the surface of porous indium phosphide obtained in other conditions, is necessary.

Practical implications: The study of oxide crystals on the surface of porous indium phosphide has great practical importance since it is the reproducibility of experimental results that is the main problem of modern materials science, the more nanoengineering. Oxides can significantly affect the properties of materials. On the one hand, oxides significantly affect the recombination properties of materials, this can impair the operation of semiconductor devices. On the other hand, oxide films can serve as a passivating coating for the surface of a porous semiconductor. Such an oxide property will be useful for the practical application of nanostructured indium phosphide. Therefore, questions of the conditions for the formation of semiconductor intrinsic oxides, their structure, and chemical composition, and also the effect of oxides on the physical and technical characteristics of materials are important.

Originality/value: The patterns of oxide formation on the surface of indium phosphide during electrochemical etching are investigated in this work. It is shown for the first time that the structure of an oxide depends on the orientation of the surface of the semiconductor. It was shown that continuous oxide films are formed on the surface of p-lnP, and oxide crystalline clusters are formed on the surface of n-lnP.

Keywords: Indium phosphide, Oxide clusters, Dislocations, Electrochemical etching

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MATERIALS

1. Introduction

Technologies based on the nanostructurization of semiconductor surface have come to replace semiconductor technology [1,2]. One of the most perspective trends in the development of nanotechnologies is to obtain porous semiconductors [3,4]. The characteristic property of porous semiconductors is a great total area of its internal surface [5,6]. A wide field of application of porous silicon confirms one of the tendencies of the up-to-date solid-state electronics: to solve many practical tasks there is no need to use ideal, defect-free super-pure monocrystals [7,8]. Here, some materials with guided structure can be of help, the example of which are porous silicon and other semiconducting materials [9,10].

As a rule, in crystals of A3B5 type, electrochemical etching provokes the emergence of etch holes – pores, the surface density of which can reach 80% [11,12]. However, electrochemical process can be accompanied by a number of alternative chemical and physical reactions, the result of which can be a polishing of surface [13], texturing [14], formation of compact indissoluble films [15,16], emergence of oxides [17,18].

Technological compatibility of proper oxides with the semiconducting material stimulates the interest of researchers to seek the ways of increasing the quality of oxide dielectric layers and their interfaces with the use of varied diagnostic means [19,20].

It is shown in the work [21] that the films of oxide can serve as passivating coating that prevents the destruction of indium phosphide surface. Authors of the work [22] inform that indium oxide In_2O_3 is the semiconductor of the high-capacity band. Therefore, it can find application as detectors of toxic gases. In addition, it is reported in papers [23,24] that due to its transparency and high electric conduction, it can be applied for solar elements and diode devices.

However, the quality criteria for porous materials and materials with oxide films on the surface that will allow their use on an industrial scale have not yet been determined [25,26].

Therefore, the issues of ensuring the quality of porous semiconductors, the presence of oxides on the surface, and studying the conditions for the formation of intrinsic oxide during etching remain unresolved [27].

The aim of this work is to establish the patterns of the formation of oxides on the surface of indium phosphide during electrochemical etching of single-crystal InP n- and p-type of conductivity.

2. Description of the approach, work methodology, materials for research, assumptions, experiments etc.

Single-crystal samples of InP of n- and p-type with the orientation of surface (111), sulfur alloyed to the concentration of carriers 2.3 x1018 cm⁻³, were used for the experiment. The samples of indium phosphide were grown according to Czochralski method in the laboratory of company "Molecular Technology GmbH". The plates were cut out perpendicular to the growing axis and polished on both sides.

Before the experiment, the samples were cleaned in ethyl alcohol. A porous surface was formed with the anode electrolytic etching. As the base of electrolyte, we have chosen a hydrofluoric acid (HF). The solution of hydrofluoric acid (48%), ethyl alcohol (C₂H₅OH) and water was used in various concentrations of oxidizing reagent. A regime of fixed current density was chosen that was controlled within the range of 110 mA/cm² that was sufficient to produce and spread of pores in the crystals with the violation of crystallographic structure, particularly at the expense of dislocations. The experiment was carried out in two stages. First, the samples were etched for 10 min. Then they were kept for 2 hours in the air. After this, the samples were subjected again to electrochemical etching for 10 min. Thus, a total time of etching constitutes 15 minutes. Etching in two stages was carried out in order to oxidize the surface of the crystal in order to provoke a more intensive growth of oxide.

A plate of platinum served as cathode. Operating surface of samples is 5 cm². The experiments were carried out at the room temperature. Morphology of the surface was studied with the help of scanning electron microscope JSM-6490. The analysis of chemical composition of porous surface of samples was also performed.

3. Description of achieved results of own researches

After the first stage of etching, a porous layer was formed on the surface of indium phosphide of n-type (Fig. 1). The size of pores is within a wide range – from units of nanometers to tens of microns. Such spread of diameters testifies that porous surface was formed according to two competing mechanisms. The first mechanism is associated with the property of the crystal to etch in the defective and disordered areas of the surface (massive pores) [28]. The second mechanism – accidental spontaneous pore-formation that provokes the emergence of pores of nanometric size [29].

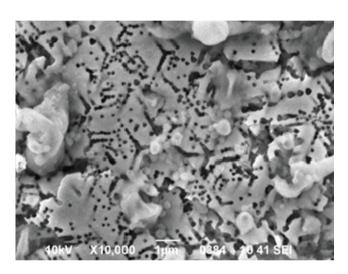


Fig. 1. SEM-images of porous surface of indium phosphide of n-type after etching in the solution of electrolyte for 10 min

After carrying out repeated etching, massive oxide crystallites with the size from 100 to 300 μ m (Fig. 2) were formed on the porous surface of indium phosphide. Crystallites have a rather complex stellate shape.

Figure 2 shows that crystallites have grown as non-uniform layers. The thickness of the lower layers makes up units of micrometers, the upper layers – approximately 15-20 $\mu m.$ Thus, it can be confirmed that these structures were formed during etching as the result of chemical reaction between semiconductor and electrolyte.

In Figure 3, EDAX spectra of the investigated structure are given. The results of chemical analysis of the surface of porous indium phosphide are given in Table 1. Spectral data were collected in 6 points of the sample surface (according to Fig. 2).

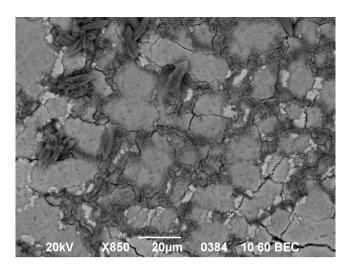


Fig. 2. SEM-images of oxide crystallites on the surface of porous indium phosphide of n-type

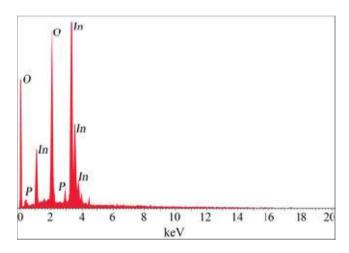


Fig. 3. EDAX results of the sample identifying each of the atomic components of In, O and P

Table 1.

The percentage composition of chemical elements of the surface of crystallites obtained with the help of EDAX method

Point of the	Element			- Total
surface	О	P	In	- Total
a	25.72	1.02	73.26	100.00
b	28.78	2.75	68.47	100.00
С	31.21	0.05	68.74	100.00
d	31.98	0.79	67.23	100.00
e	24.57	1.98	73.45	100.00
f	28.01	3.09	68.90	100.00
Average value	28.38	1.61	70.01	100.00

Table 1 demonstrates that crystallites are composed of atoms of oxygen, indium and phosphorus. Phosphorus is present in the least concentrations (1%). This means that crystallites represent oxides of indium.

Predominant places of growth of crystalline oxide on the surface of indium phosphide are atomic defects [30], surface-steps [31] and dislocations [28,32]. Micromorphology of the surface of studied samples testifies that porous layer was formed not in the whole surface of the crystal, but as radial bands. These bands - the result of distribution of dope additives during crystal growth [33]. As it is known, indium phosphide is grown according to Czochralski method [34]. In the process, a certain feature of components distribution is observed. To the extent of moving away from the center of the surface growth, the concentration of charge carriers increases and their mobility decreases. Elastic remote-acting mechanical stresses prevent the formation of geometrical and concentration uniformity. The sources of these mechanical stresses are segregated bands, dominating mechanism of their formation is dislocations (and their multiplication) that are originated in the internal defects of growth [35]. Oxide crystallites are formed in the places of pore accumulation, i.e., along the line of segregation.

In the course of the oxide layer formation, semiconductor and reagent are separated from one another with this layer. The continuation of the oxide growth is the result of reagent migration through this surface layer. If the layer of oxide formed on the surface is porous, the oxidation continues until the time when the whole semiconductor is oxidized. Figure 4 shows a porous oxide layer on the surface of n-type of conductivity indium phosphide.

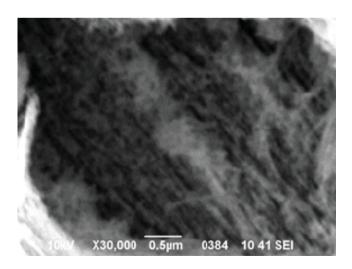


Fig. 4. SEM-images of oxide crystallites on the surface of porous indium phosphide of n-type

If the layer of oxide is compact, densely adheres to the surface and does not contain pores, it protects a semi-conductor from further oxidation (Fig. 5). This is due to the fact that oxide continuous films are a passivating coating for the surface of indium phosphide. Owing to this, the film formed does not permit the front of etching to advance in the depth of crystal – a porous layer is not formed on the surface of semiconductor [36].

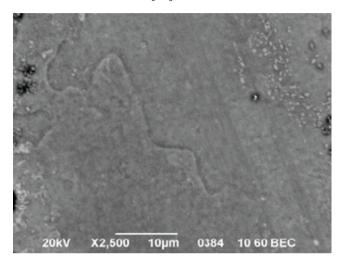


Fig. 5. Dense layer of oxide on the surface of InP

The experiment has shown that compact films of oxide are formed on the surface of indium phosphide of p-type conductivity. Such films densely adhere to the surface of the crystal. Their removal from the surface is possible only on condition that one more stage of etching is carried out in the darkness. Porous oxide layers and oxide crystallites are bound to the surface of the crystal not so firmly.

The analysis of the transverse split of obtained structures testifies that compact films are very thin on the crystals – their thickness is less than 2 μ . However, porous layers of oxide have a thickness up to 10 μ . The structure of oxide layer depends on the structure of semiconductor and conditions of etching. The orientation of oxide crystallites is determined by experimental conditions, such as the temperature and presence of oxygen. When the oxidation passes in several stages, the composition and properties of the resulting oxide film are also determined by experimental conditions.

The oxidation of semiconductors is not limited by the effect of atmospheric air or increased temperatures. The oxidation passes also during electrochemical etching. As a chemical analysis has shown, the oxide, formed in the result, should not be without fail stoichiometric or homogenous, in which connection both porous and compact films are possible.

The formation of surface oxide films can change the whole process of dissolution [37-39]. Densely adjoining and compact surface films slow down the process of dissolution. However, a chemical effect can be localized on the areas more accessible for the electrolyte due to development cracks and openings on them. Puffy and not tightly adjacent films that formed in the oxidizing etching agents, not always have any noticeable effect on the process of etching [40]. Passivation can be prevented by the presence of component in the solution, usually called as complex-forming that turns oxide into a soluble compound [41].

Oxidation-reduction etching agents for semiconductors, as a rule, admit a transfer of two or a less number of charges and give rise to a chemical spot on the surface of the semiconductor (Fig. 6).

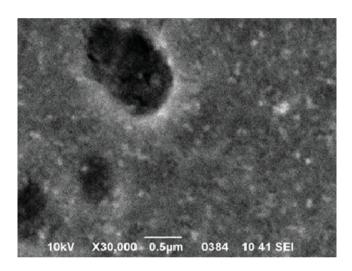


Fig. 6. Oxide spot on the surface of indium phosphide

This spot arises as the result of incomplete oxidation of the semiconductor at the expense of rapid depletion of the oxidizing reagent near the interface semiconductor/etching agent. Formation of the spot occurs particularly at low concentrations of oxidizing reagent.

It should be noted that the investigation of conditions of forming proper oxides on the surface of indium phosphide is the important technological task. At present, mechanisms are not finally determined, by which the formation of oxide films occur. However, already today we can state that indium oxides have wide perspectives of industrial application as the passivating layer, antireflection coating, etc. On the other hand, oxides can significantly affect the characteristics of devices made on the basis of nanostructured semiconductors. This effect is primarily due

to the appearance of additional bands in the photoluminescence spectra of the studied semiconductors. In this work [42] we have studied the quality of porous indium phosphide layers, but the effect of oxides was not taken into account. In the study, we show that the surface of the crystal is capable of oxidation, and this can fundamentally change the morphology and physicochemical parameters of porous indium phosphide layers. Therefore, this problem requires carrying out further researches. Particularly interesting is the investigation of thermo-physical and electron properties of the proper oxides of indium phosphide. In addition, it is necessary to determine under what conditions the oxides will be useful, and under which they will require removal from the surface of the crystal.

4. Conclusions

The phenomenon of oxidation of the surface of indium phosphide during its electrochemical etching, in particular, the formation of its own oxide, is studied. Based on the research, the following important observations can be made:

- The surface of indium phosphide during electrochemical etching and after etching exhibits the ability to oxidize. Own oxides are formed on the surface, namely indium oxides.
- 2. The structure of the oxide depends on the etching conditions and the parameters of the initial crystal. The oxides can be in the form of continuous films, crystallites, and the like.
- 3. Crystalline oxide is formed on the surface of n-type indium phosphide. Crystalline oxides can reach a height of 15-20 microns. Such oxide crystallites do not interfere with a further etching of the crystal and make it possible to obtain a porous layer on the surface of indium phosphide.
- 4. Predominantly continuous oxide films are formed on the surface of p-InP. The film thickness is about 2 microns. Such continuous films prevent further etching of the crystal. Namely, they act as a passivating coating to the surface of the crystal.

Additional information

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