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ZnO nanowires for photoelectric converter applications

Abstract. This work is focused on the creation and study of photosensitive structures based on zinc oxide nanofibers, which are promising for solar energy. Zinc oxide nanowires were obtained on the porous zinc selenide surface. The porous substrate was obtained by electrochemical etching of a low-resistance n-ZnSe plate (110) with a polished surface. Nanowires were deposited by radical-beam epitaxy. The annealing temperature was varied from 400° C to 500° C. The oxygen radical flux was $1.5 \cdot 10^{17}$ $cm^{-2}s^{-1}$. The process duration was 50 minutes. According to the scanning electron microscopy results, the nanowires length reaches 10 μm, the nanowires diameter is ~1 μm. The predominant X-ray diffraction reflex at 2θ=34.44° indicates the polycrystalline nature of the manufactured ZnO coatings with a wurtzite-type hexagonal lattice. The study of nanowires ZnO luminescence at room temperature contains an ultraviolet peak around 385 nm. This peak is related to the zinc oxide edge luminescence. Based on the fabricated structure, the design of the photoconverter was developed. The upper contact of the fabricated photoelectric converter was created by vacuum thermal sputtering of aluminum through a mask. The deposition was carried out at a substrate temperature of 200° C. Ohmic contacts were made using conductive silver paste. The reverse ohmic contact was formed by applying Al paste to the entire reverse side of the surface. The upper layer of the structure is an array of ZnO nanowires. The active base layer is ZnSe. The light volt-ampere characteristics of the obtained structure were measured in the AM 1.5 illumination mode. No-load voltage U_{xx}, short-circuit current I_{sc} and the fill factor of the current-voltage characteristic FF of the solar element were measured. The efficiency of the manufactured photoconverter was 13.7 %.

Key words: photosensitive structures, electrochemical etching, radical-beam epitaxy, nanowires, solar energy, photoconverter.

Introduction

Conversion of solar energy into electrical energy is a promising area of research. Various designs of converters of solar energy, mechanical energy and combined converters of several types of environmental energy into electrical energy based on semiconductor materials are known [1-2]. An important solar converters characteristic is the efficiency. It is known that efficiency value of 44.5% was achieved in laboratory conditions [3]. However, the silicon photocells' efficiency is only 15%. That is why one of the main problems of microelectronics is to increase the conversion efficiency of solar energy into electrical energy, to identify ways to increase the efficiency, to reduce the electricity cost.

One of the main approaches in solving this problem is the use of new materials, in particular nanomaterials. The study of photoconverter structures is becoming an actual direction in the development of efficient photovoltaic converters (PV) of the optical range.

Zinc oxide is a promising material for the PV manufacture, and its distinctive feature is good electronic conductivity and high chemical resistance. The use of zinc oxide in the form of cut nanorods can significantly increase the collection of charge carriers and the short-circuit current of devices [4]. 1D-structures are particularly attractive for these purposes due to the combination of a perfect crystal structure with a developed surface.

Therefore, the development of controlled growth methods for high-quality single-crystalline ZnO nanorods 0.2-0.5 μm in diameter and research aimed at the creation of miniature, bright, and cost-effective short-wave light sources [5] are most relevant.

Literature review and problem statement

Metal oxides (TiO_2, ZnO, Al_2O_3, Fe_2O_3, etc.) are successfully used in solar energy. Among the above-mentioned oxides, the study of zinc oxide is of particular interest due to the promising use of solar cells as photoelectrodes. The prospect of using ZnO is associated with its stability, good electronic conductivity, high chemical resistance, wide band gap (3.1-3.3 eV), etc.

Typically, various techniques are used in the manufacture of ZnO films, including spray pyrolysis [6], molecular beam evaporation [7], chemical vapor

deposition (CVD) [8], organometallic deposition (MOCVD) [9], and so on.

In [10, 11] an original method of obtaining oxide coatings was proposed. The authors of these works show that annealing in the flow of atomic oxygen promotes the formation of ZnO surface layers with changes in the photoluminescence spectra and electrical properties. As a result of annealing in the flow of atomic oxygen of ZnO single crystals implanted with phosphorus or nitrogen, under certain technological modes there are thin films of ZnO, the intensity of ultraviolet luminescence and the type of conductivity which can be controlled by temperature treatments.

In one of our works [12], the use of a porous surface of indium phosphide was proposed as a substrate for oxide coatings. As a result of annealing, the inner surface of the walls of each pore was covered with oxygen along its entire length from the base (column of indium phosphide) to the surface of the sample, forming oxygen-containing nanotubes that repeat the shape of the pores.

ZnO is widely used in various forms of inorganic heterojunction solar cells (quantum dot solar cells, thin film solar cells, and exciton solar cells).

In [13] one of the first descriptions of the use of ZnO in a thin-film solar cell Cu (InGa)Se_2:CDS is encountered. The authors of this work used ZnO as a buffer layer between the active layer and the cathode. Gradually, due to its outstanding chemical, physical and optical properties, ZnO began to be used as an electron transport layer for beneficial electron transport and blocking holes in organic solar cells [14-16] and flat solar cells based on perovskite [17-19] and also as an antireflection coating in solar cells with a heterojunction [20-23].

However, as studies show [24-26], nanosized zinc oxide, namely 1D-structures, dramatically change in properties compared to their bulk analogs [27]. Compared to films, ZnO NWs have a number of advantages. This is a high mechanical stability and adsorption capacity [28], radioactive stiffness, penetration in the visible range of electromagnetism and a wide straight fenced zone [29], before that stink is characterized by a rapid collection of carriers generated across the entire device [30], large surface area [31] and low reflectance due to scattering and light capture [32].

These properties make ZnO promising in many fields, such as gas sensors [33-34], acoustic wave sensor [35], piezoelectric nano-generator [36] and supercapacitors [37]. Well aligned 1D ZnO structures have also been widely studied as photoelectrodes for converting solar energy [38-39].

Various methods are used to synthesize 1D oxide structures: chemical vapor deposition (CVD), high-frequency vacuum spraying, deposition from aqueous solutions, sol-gel method, electrochemistry, inkjet printing, atomic layer deposition and sputtering techniques, etc. However, the existing methods are not selective enough. The products of the methods are extremely heterogeneous and contain, along with nanotubes, many other oxide nanoscale morphologies (ribbons, threads, nails, etc.). Therefore, this issue still remains unresolved.

In addition, studies of heterostructures based on ZnO nanorods, which are used as photovoltaic converters, show that the properties of the manufactured PVCs strongly depend on the substrates for growing ZnO.

The authors of [40] obtained ZnO NWs on a Zn substrate by hydrothermal treatment of Zn foil in an aqueous ammonia/alcohol solution. This was the first attempt to obtain ZnO nanofibers by the hydrothermal method in the absence of catalysts and at a relatively low temperature. In 2011, ZnO nanowires have been synthesized on an n-type Si substrate using a hydrothermal method where surfactant acted as a modifying and protecting agent sponsored by the work [41]. The authors of [42] have ascertained the morphological and optical power of heterostructures based on ZnO NWs on Si, sapphire, and GaN substrates. Well oriented ZnO NWs were grown on Si (100) substrates with a ZnO buffer layer, which was grown by deposition of atomic layers. However, the best photocurrents of 4.75 and 3.24 mA/cm^2 at 1.5 V (compared to Ag/AgCl) were shown by ZnO NWs grown on GaN and Si substrates, respectively.

Attempts have also been made to grow high-quality and less expensive structures based on ZnO nanorods on glass, soda-lime glass (SLG), indium tin oxide, and polyethylene terephthalate (PET), CdS, aluminum substrates, and others. However, the final results did not show higher efficacy than previously reported for the best structures of this type.

However, when joining dissimilar semiconductors, a problem arises associated primarily with a significant difference in the lattice constants of the semiconductor materials used. This problem leads to the appearance of dislocations in the structure. The concentration of such penetrating dislocations can be so high that the photovoltaic properties of the material will be significantly impaired. Recently, a widespread way to reduce the concentration of

penetrating dislocations is to use the porous surface of the substrate [43-45] as intermediate buffer layers. Such layers relieve lattice stress, preventing the propagation of dislocations. As the works [22, 46-47] show, heterostructures with an intermediate porous layer can have rather high values of the efficiency of photoelectric converters. Therefore, the actual task is to develop designs of high efficiency transducers on oxide NWs, research and improvement of technologies for synthesizing nanostructured materials, etc.

The aim and objectives of the study

The aim of the work is to develop a controlled synthesis of ZnO nanowires by radical-beam epitaxy method with unique morphological, electrical and optical properties, as well as to consider the design of a photoelectric converter based on the fabricated structure.

To accomplish the aim, the following tasks have been set:

- to analyze the physical and technological bases of obtaining ZnO nanotubes;
- to obtain ZnO/porous-ZnSe/ZnSe heterostructures by radical-beam epitaxy method. Investigate the morphological and electrophysical properties of the obtained structure;
- consider the design of heterojunctive photoelectric converters;
- to propose a model of a solar cell based on the ZnO/porous-ZnSe/ZnSe heterostructure and measure the light characteristics.

Materials and methods of research

In this work, the method of radical-beam epitaxy [48], which consists in the annealing of porous semiconductor substrates in a stream of atomic oxygen, is proposed for the production of oxide nanowires. A porous ZnSe surface was chosen as the substrate. The spongy open structure and large specific surface area make the porous substrate a convenient material to place in the ZnO pores and thus provide a good basis for nucleation, which is necessary for ZnO growth along the preferred orientation.

The experiment was conducted in two stages (Fig. 1).

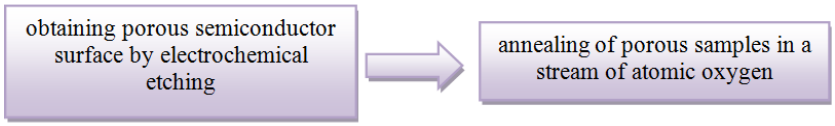


Figure 1 – Stages of the experiment

At the first stage, porous ZnSe was producing by anodic electrochemical etching of single-crystalline zinc selenide plates [49]. The ZnSe plate size was 10^{-4} m^2.

The experiment was performed at room temperature in several stages (Fig. 2).

Samples of low-resistance ZnSe n-type single crystals oriented in the plane (110) with a polished surface were used for experiments. The voltage source was a precision power supply based on the KIS-3R33S module. The current kinetics of the digestion process were recorded by the ADC and displayed on the computer screen in real time.

As the electrolyte used mixtures of hydrofluoric, hydrochloric and nitric acids, namely: $HF:HNO_3:HCl=2:3:3$ and $HF:HNO_3:HCl=2:3:2$. The duration of the etching process was from 10 to 30 minutes. The current densities ranged from 30 to 270 mA/cm^2. A precision power supply based on the KIS-3R33S module served as a voltage source. The current kinetics of the etching process was recorded by ADC and displayed on the computer screen in real time. The highest quality porous structure was obtained using the electrolyte $HF:HNO_3:HCl=2:1:3$ at a current density of 130 mA/cm^2.

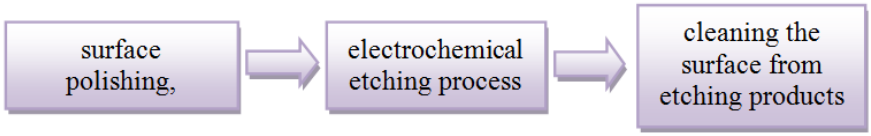
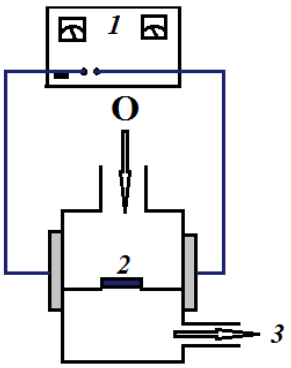


Figure 2 – Stages of electrochemical etching

At the second stage, the porous samples were annealed in an atomic oxygen flow [12].

The porous sample was loaded into the flow system on one side of which atomic oxygen was supplied and to the other side a fore vacuum pump was connected in order to discharge the residual gas (Fig. 3).

The process temperature was gradually increased in increments of 50°C. The initial annealing temperature was set at 400°C. The temperature was brought to 500°C for 50 minutes. The initial oxygen pressure in the growth chamber was 10^{-3} Pa, oxygen radical flow $J_O=1.5\cdot10^{17}$ $cm^{-2}s^{-1}$.



Figrку 3 – Schematic representation of the installation for annealing: 1 – generator of ultra high frequency, 2 – sample, 3 – gas evacuation system

The resulting structures were characterized by scanning electron microscopy and X-ray diffraction. The morphology was investigated using a JSM-6490 scanning electron microscope with a resolution of x60000. The chemical composition was determined using a JAMP-10S microprobe by JEOL and X-ray spectral microanalysis. The phase analysis of the resulting heterostructures was determined using a DRON-3M X-ray unit (CuKα radiation, λ=1.5405 Å) with a graphite monochromator in the range 2θ=15-65°. To study the photoluminescence, the ILGI-503 nitrogen laser with a wavelength of 337.1 nm and a pulse length of 10 ns was used. The resulting spectra were analyzed using an MDR-12 monochromator.

Experimental Results and their Discussion

Study of the composition and properties of the structure

After each step, the surface of the samples was examined using scanning microscopy.

Electron microscopic studies of the samples after electrochemical etching show that this material is a system of vertically oriented columns formed in the plate volume.

The study of the porous surface chemical composition (Table 1) indicates insignificant non-stoichiometricity. In addition to zinc and selenium, a small amount of oxygen is present on the surface; no other reaction products were found either on the surface or in the pores.

Table 1 – Elemental composition of the porous surface

Element	Percentage, %
O	0.89
Zn	51.83
Se	47.28

After the second stage (annealing), the porous surface undergoes significant changes. The substrate surface changes its color from yellow-green to white, which indicates the formation of a new chemical compound. The new compound is formed as a result of the displacement of selenium atoms by oxygen atoms.

SEM microphotography (Fig. 4) shows that the surface consists of column-shaped nanocrystals. At the same time, the NWs formed on the surface are arranged perpendicular to the substrate surface. The length of the nanowires reaches ten microns, with a filament diameter of ~1 μm.

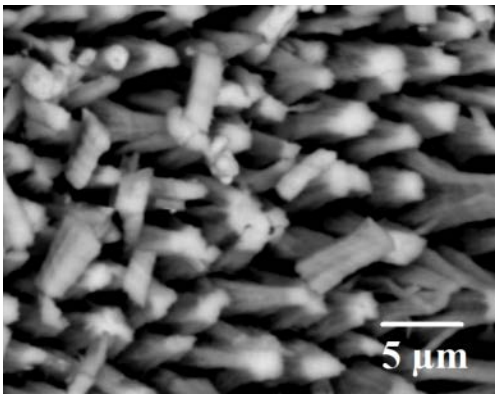


Figure 4 – SEM images nanotubes ZnO obtained on the surface porous-ZnSe

By studying the chemical composition of the surface of the formed heterostructure (Table 2), it was found that a coating consisting of oxygen and zinc was formed on the porous ZnSe surface. The studies allow us to state that the nanocolumns formed by electrochemical etching on the ZnSe surface are converted into ZnO NWs (Se atoms are stored in insignificant amounts).

Table 2 – Elemental composition of formed nanotubes

Element	Percentage, %
O	50.97
Zn	48.63
Se	0.40

According to electron microscopy, the parameters of the ZnSe and ZnO layers were established and listed in Table 3.

Table 3 – Parameters of the ZnSe and ZnO layers

Parameter	Value	
	ZnSe	ZnO
The height of the column h, μm	9.0	10.0
The diameter of the column d, μm	0.9	1.0
The distance between the columns, μm	0.8	1.2

X-ray studies of the ZnO NWs crystal structure showed that they are polycrystalline in nature with a wurtzite-type hexagonal lattice, as evidenced by the preferential reflex [002] at 2θ=34.44°. The angular positions of the peaks agree well with the tabulated ZnO data for nominally pure zinc oxide.

In addition to the [002] peak, peaks at 36.38°, 56.70°, 69.07°, and 72.42° are observed, which correspond to the crystallographic orientations [101], [110], [201] and [004].

The results of luminescence studies of ZnO nanowires at room temperature indicate a peak in the ultraviolet (UV) radiation area (385 nm). Except for the UV peak, other luminescence bands in the visible region of the spectrum do not appear. According to [50], the observed UV peak can be attributed to the edge luminescence of zinc oxide (free excitons luminescence).

The mechanism of formation of oxide nanotubes

When growing ZnO nanowires, the substitution process is diffusion in nature. In the initial stages of annealing in the heterophase system, two mechanisms of defect formation compete: quasi-epitaxial and atom substitution.

When O atoms are deposited on the ZnSe surface, one of the important problems is the replacement of Se atoms by O atoms, which leads to the formation of a thin enveloping ZnO layer on the ZnSe surface. With a further step-by-step increase in temperature, the ZnO coating will grow due to the diffusion of oxygen into the volume.

The process will continue until the oxygen atoms completely displace the selenium atoms. The depth of the diffusion layer depends on the annealing conditions. At high annealing temperatures, intense desorption of selenium with ZnSe is observed. When annealing with oxygen not only breaks the bond of zinc with selenium, but also the release of selenium outside the material, which forms a compound of selenium and oxygen with the formula SeO_2. The oxide, which is present in the air in sufficient quantities, is isovalent with respect to ZnSe.

A schematic representation of the formation of ZnO nanowires on the porous surface of ZnSe is presented in Fig. 5.

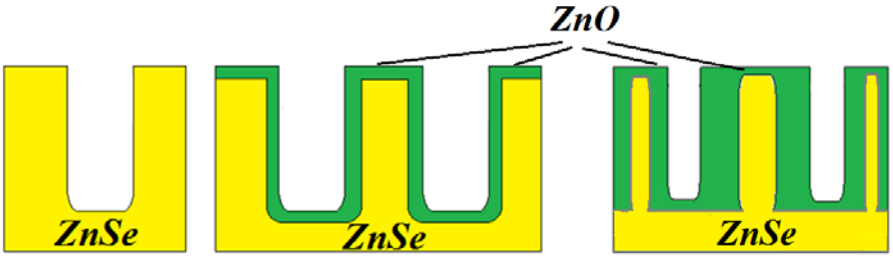


Figure 5 – Schematic representation of the formation of ZnO nanotubes on the porous surface of ZnSe

Photoconverter based on ZnO/porous-ZnSe/ZnSe heterostructure

On the basis of the structure obtained, it was proposed to manufacture a solar cell. The design of the manufactured photoelectric converter is shown in Fig. 6. The upper contact was created by vacuum thermal sputtering of aluminum through a mask. The deposition was carried out at a substrate temperature of 200° C. Ohmic contacts were made using conductive silver paste. The reverse ohmic contact was formed by applying Al paste to the entire reverse side of the surface. The ZnO nanowires on the top layer of the structure allow light to pass through this layer. Inside the active ZnSe base layer, electron-hole

pairs are generated by incident light with a suitable wavelength. These photogenerated holes and electrons are separated by diffusion and drift processes, which leads to the appearance of a photocurrent.

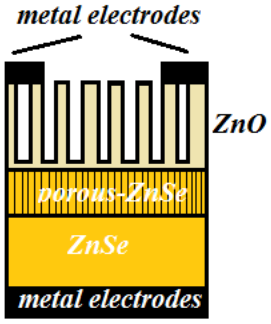


Figure 6 – Scheme of a solar battery based on a fabricated structure

Measurements of light volt-ampere characterristics of the obtained structure were carried out in the lighting mode AM 1.5. No-load voltage U_{xx}, short-circuit current I_{sc} and fill factor of the photovoltaic converter FF volt-ampere characteristic were measured.

The results of the conducted investigations are given in Table 4.

Table 4 – The results of experimental tests of manufactured solar cells

Parameter	Value
No-load voltage U_{xx}, mV	640
Short-circuit current I_{sc}, A	3.4
Fill factor of volt-ampere characteristic FF	0.63
Efficiency factor, %	13.7

The efficiency value is somewhat lower than the previously obtained values for photovoltaic converter based on ZnO nanostructures [51-52]. Nevertheless, the sample demonstrates sensitivity to illumination in the visible range. We should also note the non-zero current in the absence of illumination, which is associated with the background IR irradiation of the sample.

Conclusion

The work demonstrates the successful synthesis of homogeneous coatings from zinc oxide nanorods consisting of two stages: obtaining porous semiconductor surface by electrochemical etching and formation of zinc oxide nanowires by radical-beam epitaxy.

The porous surface of ZnSe was obtained by the method of electrochemical etching of single-crystal plates. It was found that the size of the formed columns is 0.9 and 9 μm in diameter and height, respectively.

On the basis of the process of atom substitution due to thermal annealing in the atomic oxygen stream, ZnO oxide nanowires on the porous surface of ZnSe were obtained. It is established that the substitution process is diffusion in nature. The length of the obtained nanowires reaches ~10 μm. The average thread diameter is 1 μm.

The study of the surface properties of the formed structure testifies to the polycrystalline nature of the coating with a hexagonal lattice of the wurtzite type. The results of luminescence studies of ZnO nanowires at room temperature indicate a peak in the ultraviolet radiation area.

It is shown that for the creation of photovoltaic converters based on zinc oxide nanowires it is possible to use the made structure ZnO/porous-ZnSe/ZnSe, which is an urgent task for the creation of environmentally friendly low-cost solar modules. The solar cell design is proposed, and the parameters of the made structure are calculated. It is established that the efficiency of the manufactured photoconverter was 13.7 %

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