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ENGINEERING OF SEMICONDUCTOR COMPOUNDS VIA ELECTROCHEMICAL TECHNOLOGIES FOR NANO-MICROELECTRONIC APPLICATIONS

Eduard Monaico, ORCID: 0000-0003-3293-8645

National Center for Materials Study and Testing, Technical University of Moldova, 168 Stefan cel Mare Blvd., Chisinau, MD-2004, Republic of Moldova
*Corresponding author: Eduard Monaico, *eduard.monaico@cnstm.utm.md*

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Abstract. The paper is focused on electrochemical approaches for nanostructuring of semiconductor compounds with further applications in nano - microelectronic devices. A cost-effective technology for nanowires and nanotubes obtaining by pulsed electrochemical deposition is presented. Functionalization of elaborated nanostructures with gold or platinum via electroplating improves the properties of the nanostructures. An optimization of the varicap design to increase the capacitance is proposed and discussed as well as the optimization of pulsed electrochemical deposition of several hundred micrometer long Pt nanotubes is performed. Herein, the elaboration of contacts to GaAs nanowires via different approaches for photoelectrical investigations is reported.

Keywords: *electrochemistry, nanostructures, nanowires, photodetector, contacts, varicap device.*

Rezumat. Lucrarea se concentrează pe abordări electrochimice pentru nanostructurarea compușilor semiconductori cu aplicații ulterioare în dispozitive nano - microelectronice. Este prezentată o tehnologie cost-efectivă pentru obținerea de nanofire și nanotuburi prin depunere electrochimică în impulsuri. Funcționalizarea nanostructurilor elaborate cu aur sau platină prin galvanizare îmbunătățește proprietățile nanostructurilor. Este propusă și discutată o metodologie de optimizare a designului varicap pentru a crește capacitatea și optimizarea depunerii electrochimice pulsate a Pt pe nanotuburi de câteva sute de micrometri. Este raportată elaborarea contactelor cu nanofire de GaAs prin diferite abordări pentru investigații fotoelectrice.

Cuvinte cheie: *electrochimie, nanostructuri, nanofire, fotodetector, contacte, dispozitiv varicap.*

Introduction

Nowadays, a lot of applications e.g. in electronics, photonics, sensors, communications, etc implies the synthesis of functional low-dimensional materials and devices. Nanotechnology is a developing field in which researchers elaborate technological approaches to manipulate with materials at the atomic and molecular scale to obtain nanomaterials with improved or even completely new properties. It is worth to mention that the properties of material at dimensions of a few nanometers often differ significantly from the characteristics of the same bulk material. It can be explained by the enhancement of properties of low-dimensional materials and devices caused by high surface aria to volume ratio [1, 2].

Usually, semiconductor nanowires are fabricated by means of well-known techniques like electrochemical deposition in porous templates, chemical transport, chemical vapor deposition using Au catalytic seed, etc. [3 - 5] representing bottom-up technologies. During the last two decades it was demonstrated that electrochemical etching of massive semiconductor crystals can be used as a cost-effective approach for nanostructuring [6 - 8]. Moreover, optimization of electrochemical parameters leads to fabrication of a huge amount of semiconductor nanowires aligned perpendicular to the crystal surface. The preparation of nanowires by anodization has some advantages: short time of etching; anodization is performed at room temperature; no costly equipment is required; small amount of electrolyte; etc. Moreover, the possibility of cost-effective fabrication of InP nanowires via "fast electrochemical etching" of InP semiconductor compound was demonstrated [9]. Using this approach, the authors fabricated semiconductor nanowires with a length of 2 μm during 3 seconds of electrochemical etching, reaching an etching rate about 40 μm/min.

In comparison to Si, nanostructures based on III-V or II-VI semiconductor compounds offer larger possibilities for engineered nanofabrication due to the chemical composition, larger band-gap which give the possibilities to use these nanostructures even in visible spectral range, diversity of produced pores, and novel characteristics resulting in broad application possibilities. Furthermore, semiconductor nanotemplates, have a lot of advantages compared with dielectric nanotemplates based on porous anodic aluminum oxide or etched ion-track membranes. First of all, the properties of semiconductor nanotemplates can be adjusted by application of external electric field, illumination, etc, lead to more opportunities of application in nanofabrication. The second major advantage consist in the issue that the walls of the porous semiconductor skeleton, which have a higher electrical conductivity compared to the walls of dielectric nanotemplates, provide favorable conditions for metal nucleation and uniform deposition on the pores walls [6, 10, 11]. In other words, using a combination of two approaches and namely: anodization and electroplating of metal give the possibilities of cost-effective fabrication of metallic nanowires or nanotubes with well-defined diameters and shapes. The elaborated metal nanostructures possessing unique properties can find applications in nanoelectronics and optoelectronics as flexible electrodes, interconnectors with well-designed direction of propagation, electrodes, magnetic devices and sensors [6, 11].

An important issue to be solved for device fabrication and characterization is related to the contacting of elaborated nanostructures. In this work we will focus on the realization of the electrical contacts via different approaches to the single GaAs nanowire for photoelectrical investigations. At the same time, an optimized design of varicap device with the aim to increase the capacitance is proposed and technological realization is demonstrated.

The technological process and materials

Pt nanotubes deposition in porous GaP

The n-GaP with a free electron concentration of $1.2\mathrm{x}10^{17}$ cm^{-3} with crystallographic orientation (100) and substrate thickness of 500 μm were subjected to electrochemical anodization in 5% H_2SO_4 aqueous electrolyte for porous matrix obtaining. To obtain the desired deepness of the porous layer, the duration of anodization was changed from 30 min up to 2 h. Platinum electrodeposition was performed at 40°C temperature of electrolyte during 6 hours in fabricated porous GaP templates characterized by several important parameters as follows: (i) thickness of porous layer about 70 μm; (i) pore diameter 200 nm; and (iii) pore wall thickness about 150-200 nm. The configuration of cell with two electrodes was used. The porous GaP sample served as working electrode and as counter electrode a mesh from Pt wire with the total surface 6 cm^2 was used. To assure the uniform deposition inside the porous matrix, the pulsed electrochemical deposition was used at applied negative cathodic potential of 20 V with the pulse length of 200 μs and duration between the pulses as long as 1 s. During this delay, a refreshment of the electrolyte inside of the pores is realized.

GaAs nanowires obtaining. For GaAs nanowires obtaining, the n-type GaAs semiconductor crystals with crystallographic orientation (111) and free carriers concentration of $2x10^{18}$ cm^{-3} were used. More detailed description of GaAs nanowires obtaining and optimization of the electrochemical parameters, sample surface, etc can be found in early published paper [7]. Briefly, prior the anodization, to remove the native oxide, the samples were etched for 2 min in $HCl:H_2O$ solution with ratio (1:3). The electrical contact to the back side of the sample was realized with the silver paste. The anodization was carried out on the (111)B surface of GaAs through a 0.2 cm^2 surface area in 1M HNO_3 electrolyte at applied anodic potential of 3 V. For anodization, the configuration of cell with three electrodes was used. A piece of GaAs sample, with a surface of 1 cm^2 served as working electrode while a mesh from Pt wire with the total surface 6 cm^2 was used as counter electrode. The third one is a saturated Ag/AgCl electrode. A Scanning Electron Microscope (SEM) TESCAN Vega TS 5130 MM in configuration with an INCA Energy EDX system from Oxford Instruments operated at 20 kV was used for morphology and chemical composition analysis of obtained GaAs nanowires.

Contacts manufacturing to GaAs nanowires

Focused Ion Beam (FIB) contacting: The FIB instrument used in this work was a FEI Helios Nanolab 600. At the beginning, the sample with GaAs nanowires was placed in a small amount of deionized water and sonicated for 5 seconds in order to detach the nanowires from the substrate. 1 ml of obtained solution with GaAs nanowires was dropped on a Si substrate and let dry until the water was completely evaporated. For investigation of electrical and sensing properties of a single nanowire, a special chip was fabricated on a Quartz substrate with the distance between the contacts varying from 5 to 20 μm. A single nanowire was transferred from Si substrate to the prefabricated chip with contacts inside the FIB chamber using an Omniprobe micromanipulator. The contacts to the nanowires were performed with Pt using $(CH_3)_3Pt(CpCH_3)$ as the precursor gas under the Ion Beam.

Laser Beam Lithography contacting: The contacts to the produced GaAs nanowires via electrochemical etching in one step were performed by means of Laser Beam Lithography μPG 101 from Heidelberg Instruments. To detach the nanowires from the GaAs substrate, the samples were subjected to sonication for 15 s in an ultrasound bath containing ethanol. In the second step, a suspension containing dispersed GaAs nanowires in ethanol were deposited on the glass substrate with the dimensions of 1 cm^2. The glass substrate with the GaAs nanowires was covered with the photorezist (LOR 3B and ma-P 1205) and a designed contact pads were transferred by illumination using Laser writer μPG 101 from Heidelberg. Prior to the metal deposition by magnetron sputtering for contacts consisting from thin layers of Cr and Au with thickness of 50 nm and 250 nm respectively, the contacts pads were developed in photorezist remover. A final step was lift-off with Microposit remover 1165 at 50°C. To mention that on the same glass substrate it is possible to fabricate several contacted nanowires.

Photoelectrical study

The photoconductivity of the fabricated photodetectors based on single GaAs nanowire was exited with a Xenon lamp DKSS-150. Due to the band-gap of GaAs, the infrared region is expected to give perspective results. However, also UV spectral region has been investigated. Optical filters were used to select the desired spectral range (ultraviolet, near infrared (IR) and visible). The optical power in IR region after optical filter was 100 mW. The current in the dark and under illumination was recorded at 300 K by means of Keithley 2400 Source Measure Unit connected to the computer via IEEE 488 interface.

Results and discussions

As it was mentioned in Introduction section, porous nanotemplate-based nanomaterials are promising for a lot of applications, especially in micro-nanoelectronics. A good example is a fabricated device consisting from deposited metal in porous semiconductor matrix resulting in high contact area at the metal/semiconductor interface [12]. In comparison with the case of 2D contact of metal and semiconductor, the approach with use of porous template, representing 3D structure leads to an enormous capacitance which has been used for realization of the variable capacitance device. The device was obtained via pulsed electroplating of Pt on the whole GaP pores walls resulting in formation of Pt nanotubes [13]. The formation of Schottky contact is clearly seen from SEM images of investigated metallo-semiconductor networks. As a rule, the metal is not charging during the scanning because have a higher conductivity than semiconductor matrix. In our case, the deposited nanotubes look bright, indicating the charge accumulation in metal due to the high Schottky contact. The height of the formed Schottky contact depends from the used metal. For GaP, some metal like Zn and Mg give an ohmic contact, while Pt possesses the highest value of difference between work function of the metal and electron affinity of the used semiconductor (ϕ_m - χ_s) which is 1.68 eV.

The schematic representation of the fabricated variable capacitance device is presented in Figure 1a, as well as the SEM image of the deposited Pt nanotubes in porous GaP template presented in the inset of Figure 1a. The GaP porous layer (2 from Figure 1a) was created in a bulk GaP substrate (1) with a 500-µm thickness at depth of 70 µm. The In metal, giving ohmic contact (1), was deposited on the back side of the GaP substrate. The Schottky contact was formed on the top and internal surfaces of the GaP pores via successive pulsed electrochemical deposition of Pt. Successive pulsed electrochemical deposition means that after formation of Pt nanotubes on internal surface of pores, the duration (length) of pulses was increased to force the deposition of more quantity of metal ions, also the duration between pulses was reduced with the aim to not allow the penetration of metal ions deeply inside of porous layer. In this pulsed electrochemical deposition regime, we provide the deposition at interface of porous layer and electrolyte, thereby, growing up, each Pt nanotube are interconnected with the deposited thin film on the surface.

The measured I-V characteristics demonstrated the existence of the Schottky contact reaching a rectifying ratio of 10^5 at applied potential of 12 V (see Figure 1b). The relationship between device capacitance and voltage is presented in Figure 1c.

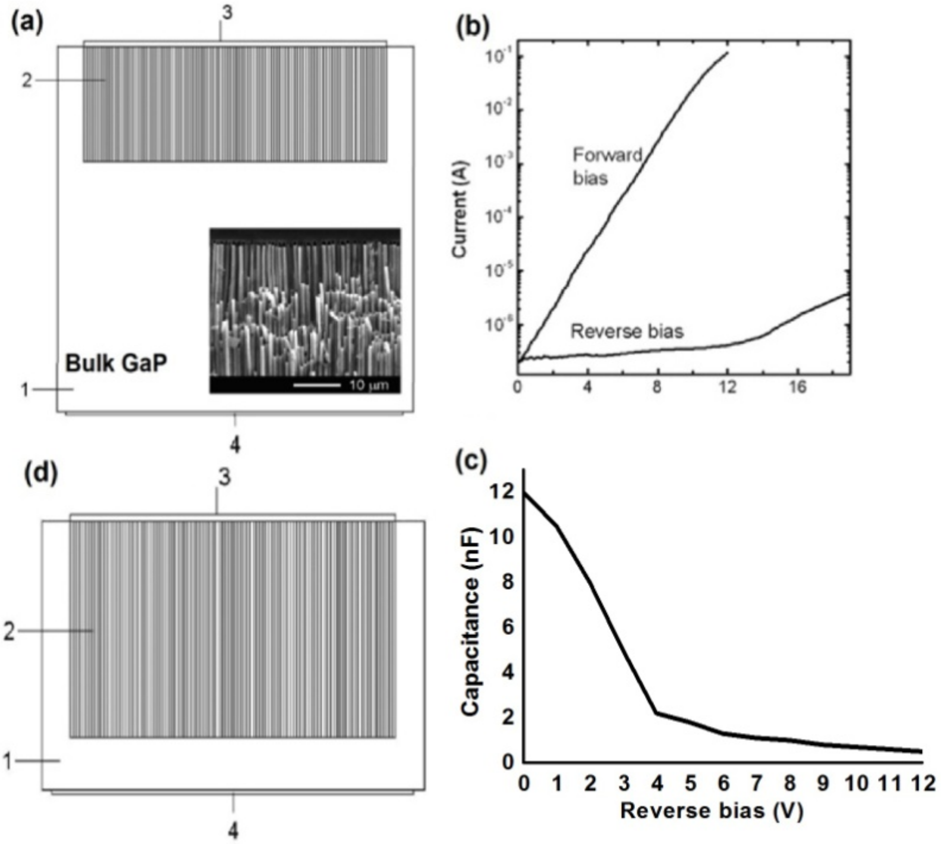

Figure 1. (a) The variable capacitance device schematics with deepness of 70 µm are as follows: 1 - GaP substrate; 2 - Pt nanotube-filled porous GaP template; 3 - Pt Schottky contact interconnecting deposited Pt nanotubes in pores and thin Pt film on the top surface; 4 - Ohmic contact. (b) Characteristics of current and voltage. (c) Capacitance-voltage characteristics of the varicap device elaborated in (a). (d) Proposed design with improved capacitance-voltage characteristics by 5 times due to use of 300 µm thick porous layer. The inset in (a) represent SEM image of Pt/GaP nanocomposite used for device fabrication.

It is worth to mention that the capacitance of varicap device, usually named barrier capacitance, is measured at reverse polarization. It can be seen, that in the range of 0.5 to 4 V of applied voltage, the capacitance value drops sharply from 12 to 2 nF, indicating that the device reaches a capacitance density variation of about $6x10^{-3}$ pF/V for 1 μm^2 of surface. This obtained value is much higher in comparison with other 2D variable capacitance device consisting from flat p-n junction or deposited two-dimensional thin film on the semiconductor surface.

In this paper, based on our calculations and preliminary results, there have been established that taking into account that the deposited Pt nanotubes were on depth of 70 µm, the capacitance value can be increased using porous layers with deepness of several hundred of micrometers (see Figure 1d). For this purpose, an optimization of pulsed electrochemical deposition of Pt is required. The optimization of parameters of pulsed electrochemical deposition showed that uniform deposition of Pt nanotubes inside pores along 300 µm thickness occurs at pulse duration of 100 µs and duration between the pulses of 3 sec. The total time of electrodeposition is 15 hours. Beside this, a mechanical steering of the electrolyte is performed.

Via electrochemical etching not only porous structures can be obtained but also due to optimization of electrochemical parameters during the anodization leads to the formation of arrays of nanowires [7 - 9, 14]. Recently, the fabrication of photodetector based on single GaAs nanowire was reported [7]. As was discussed in the introduction section, nanowires obtaining via anodization is a cost-effective technology, while the contacts fabrication to single nanowire is a challenge and require more investigations. In continuation, the formation of contacts via three different methods is investigated. The first one is fabrication using the FIB. The SEM image of contacted GaAs nanowire is presented in Figure 2a. In this case, the measured I-V characteristic demonstrated formation of Schottky contacts requiring an applied voltage with amplitude about 8 V for fabricated device. It is worth to mention that the fabrication of contacts with FIB it is very expensive while require a lot of work-hours in clean room and expensive equipment. The second approach is based on contacts fabrication via Laser Beam Lithography. Used equipment in this case is not so expensive. The optical image of contacted GaAs nanowire is presented in Figure 2b. The possibilities to choose the deposited material in magnetron sputtering (e.g. Cr/Au) give the advantage to obtain ohmic contact resulting in linear dependence of photodetectivity.

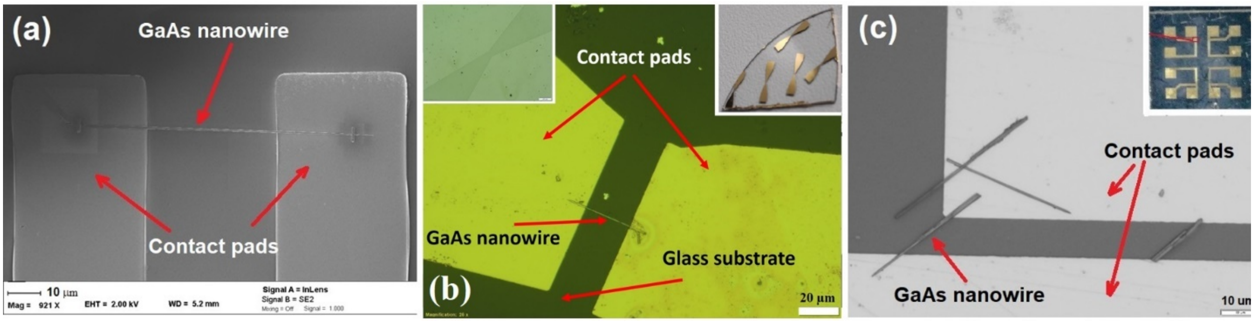

Figure 2. (a) SEM image of contacted GaAs nanowire with FIB technique. Photos at optical microscope of contacted GaAs nanowires via Laser Beam Lithography in (b) and prefabricated Si chip with contacts in (c). The inset in (b) represents design of contacts (left) and photo of 4 fabricated photodetectors (right) while inset in (c) shows the Si chip (1×1 cm) with contacted nanowire.

The third approach, representing a low-cost fabrication, could serve the use of Si/SiO_2 chips with prefabricated contacts (see inset in Figure 2c). The technological route implies the following steps: nanowires fabrication via anodization, sonication to assure the release of nanowires from the substrate to ethanol; a drop of ethanol suspension with nanowires is deposited on the surface of the chip with prefabricated contacts; evaporation of the ethanol; the chip is subjected to investigation, until the condition that at least one nanowire is caught between contacts is met. Otherwise, the procedure is repeated until electrical contact with a nanowire is created. Despite the accessibility and the low cost of this method, it should be mentioned that the photodetector manufactured by this approach demonstrated a degradation over the time (after 3 months). We assume that the cause is the formation of unstable contact, the nanowire is just placed on the contact and any mechanical action, electrical stress, and temperature gradient can affect the integrity of the contact between the nanowire and the contact pad. So, this method could be recommended just for rapid investigation of electrical properties of fabricated nanowires with further deposition of contacts by Laser Beam Lithography or FIB.

Schematic of photodetector realization on the basis of single GaAs nanowire is presented in Figure 3a. The measured photoresponse demonstrated the increase of the current in the case of illumination about 4 times (see Figure 3c).

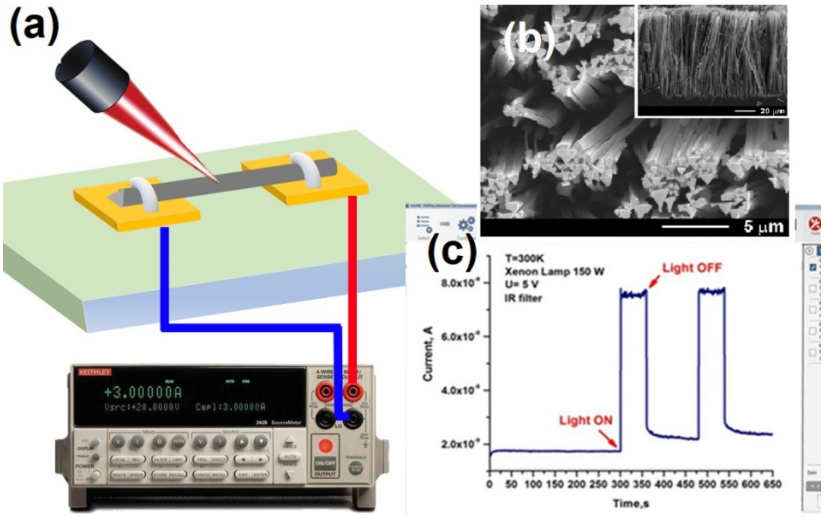

Figure 3. (a) Schematic representation of realization of IR photodetector based on single GaAs nanowire. (b) SEM image from top view and inset cross-section view of obtained GaAs nanowires via electrochemical etching. (c) Photoresponse as a function of time measured at infrared radiation with the excitation density of 800 mW/cm^2 of elaborated photodetectors on the basis of single GaAs nanowire.

The responsivity of the photodetector R is calculated according to the formula [15]:

$$R = \frac{I_{photo} - I_{dark}}{P_{ill}} \tag{1}$$

where I_{photo} and I_{dark} represent the measured current of the elaborated photodetector at illumination and in the dark respectively, while P_{ill} is the illumination power. As was mentioned above, the value of the band-gap of GaAs define the use of IR radiation.

The calculation of the right illumination power is a very important issue. In our case, beam power (P_{beam}) was 100 mW and beam diameter (d) 9 mm. Taking into account that the active area of the photodetector was (S_{phd}) 20 × 0.4 μm^2 (length and diameter of the nanowire respectively).

The excitation density per square centimeter is calculated in equation 2:

$$P_{exc} = \frac{P_{beam}}{S_{beam}} = \frac{100\times10^{-3}}{0.1} = 100 \text{ mW/cm}^2 \tag{2}$$

To calculate how much light drops on photodetector, the obtained excitation density needs to be multiplied with the area of the photodetector giving a power of illumination P_{ill}=800 mW/cm^2. Accordingly, to the results presented in Figure 3c, the calculated responsivity of the elaborated photodetector based on single GaAs nanowire is equals to 100 mA/W at the applied potential of 5 V.

Another important parameter for the detection capability of the photoelectric device is the detectivity which can be calculated from equation 3 [15].

$$D^* = \frac{R\sqrt{A}}{\sqrt{2eI_{dark}}} \quad (3)$$

where A - represent the active area of the nanowire exposed to the illumination, e is the elementary charge. Introducing the calculated responsivity in the equation 3 we get the value ≈$1.2\text{x}10^9$ cm $\text{Hz}^{1/2}$/W.

Thus, the elaborated photodetector based on single GaAs nanowire is characterized by good sensitivity and dynamic characteristics in the IR region of the spectrum.

As was mentioned above, the band-gap of GaAs define the use of this material in IR region of the spectrum. Notwithstanding this, the elaborated photodetectors were studied also in the UV region of the spectrum with *cut*-off *filters*. In this case, a small enhancement in the photocurrent was registered at polarization voltage of 10 V for UV region of the spectrum, as can be seen in Figure 4.

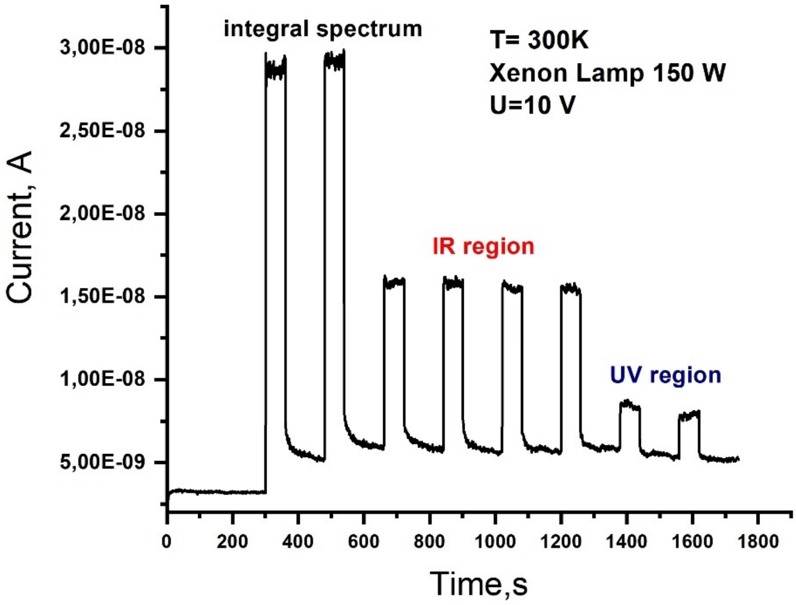

Figure 4. Photoresponse as a function of time measured at integral spectral range, infrared and ultraviolet radiation of elaborated photodetectors on the basis of single GaAs nanowire biased at applied voltage 10 V. The integral spectrum means that no optical filters were used.

Conclusions

Nowadays, anodization of semiconductor compounds represents a cost-effective top-down approach in nanofabrication. Cost-effective technology means that it not requires sophisticated equipment, high temperatures and long durations of nanostructuring in comparison with bottom-up approach. To extend the area of applications it is proposed to combine these two approaches for micro-nanodevice manufacturing. This route was used to fabricate varicap device based on Pt/porous GaP with increased capacitance. At the beginning, the porous templates were fabricated by anodization and then, Pt nanotubes were electroplated via pulsed electrochemical deposition. An optimized design of varicap device with the aim to increase the capacitance was proposed and technological conditions of pulsed electrochemical deposition for realization of metal/semiconductor with deepness of 300 μm were elaborated.

The influence and formation of contacts to the single GaAs nanowire via three different methods was investigated. It was demonstrated that, contacting with means of laser beam lithography represent an efficient and cost-effective approach for photodetector elaboration. The elaborated photodetector based on single GaAs nanowire is characterized by good sensitivity and dynamic characteristics in the IR region of the spectrum. Moreover, the developed photodetector shoved photosensivity in the UV region of the spectrum at applied potential of 10 V.

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