Thin films of semiconductors for magneto-operated diodes and memristors

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Abstract

The thin films of VO_2, TiO_2, InSb:Mn, and the heterostructures on their basis have been produced by the PLD method. The memristive effect has been revealed in the heterostructures of Au/VO_2/VO_{2-x}/Au, and Au/TiO_2/TiO_{2-x}/Au. The value of x was varied during the structure growth by changing the oxygen pressure. The dependence of the *I-V* curves of the diode heterostructure *p*-(InSb:Mn)/*n*-InSb on the magnetic field orientation both in the plane of the structure and perpendicular to it has been revealed. The diode current in the field 0,15T perpendicular to the diode plane decreased almost by a factor of 9, that is indicative of the effect of giant magnetoresistance of the *p*-(InSb:Mn)/*n*-InSb diode.

Keywords: PLD method; thin films of VO_2; TiO_2; InSb: Mn; memristive effect in Au/VO_2/VO_{2-x}/Au and Au/TiO_2/TiO_{2-x}/Au heterostructures; giant magnetoresistance of *p*-(InSb:Mn)/*n*-InSb diode

1. Introduction

The method of pulsed laser deposition (PLD) allows producing thin films of various materials on single-crystal, amorphous and flexible organic substrates [1]. The droplet-free mode of film deposition provides smooth homogeneous high-quality films even at the ambient temperature of the substrate thanks to elimination of the drops which are emitted from the target onto the substrate in the deposition process [2-4]. The films of the diluted magnetic semiconductors with high Curie temperature and high extent of spin polarization of carriers [5] were obtained by this method. The investigation of thin films of ferromagnetic semiconductors (AIII-BV): Mn is of great interest in connection with a possibility of their use in the magnetoelectronic and spintronic devices [6]. A bulk sample of the InSb-MnSb composite eutectic represents the narrow band semiconductor of *p*-type that has the lattice parameters approaching those of InSb and is characterized by high mobility of carriers. The Curie temperature in In1−xMnxSb alloys reaches 600 K. Low solubility of manganese in InSb is responsible for formation of nanoclusters of MnSb in volume alloys with concentrations higher than 3,5% [7]. Though solubility of manganese in InSb is small, the PLD method makes possible obtaining homogeneous thin films of InSb:Mn with concentration of manganese to 20% from the targets of InSb-MnSb alloys. The magnetoresistance in thin films of InSb:Mn can have influence on the characteristics of the magneto-operated *p*-(InSb+MnSb)/*n*-InSb diodes in which the effect of giant magnetoresistance is observed [8,9].

New prospects in creation of computer systems are opened by the use of analog architecture of artificial neural networks [10]. The basis of the proposed neuromorphic systems is made by the memristors – the bipolar devices in which electrical resistance changes in proportion to the charge flowing through them. The electric characteristics of the memristor are defined by the background of its functioning, which is similar to the properties of the synapse of biological neural systems. Until recent time memristor systems were only used as mathematical abstractions in simulation of signal processing operations, behavior of non-linear semiconductor systems, electrochemical processes, and in modeling the work of neurons of human brain. However in practice the effect of memristivity (change and "storage" of material resistance in the course of current flowing through it) was not shown, as for microscopic structures a variation in electrical resistance was negligible. With the advent of a possibility of formation of nanothickness structures the existence of memristive effect in the metal-dielectric-metal nanostructures in the film of titanium dioxide TiO_2 ~5 nm thick was experimentally demonstrated for the first time [11].

The first thin-film memristor was manufactured on the basis of a TiO_2 film of nanometer thickness in 2008 [11]. In this device a layer of TiO_{2-x} and a layer of stoichiometric TiO_2 were arranged between two platinum electrodes. Since then the memristor on TiO_2 became a reference specimen used by many authors to research the mechanisms of resistive switching in memristors. Various mechanisms of memristive effect in the TiO_2 based memristors were suggested. The explanation of the mechanism of resistive switching by formation and destruction of fine conductive fibers (filaments) in the volume of a dielectric is given in [13]. The influence of energy barrier changing on the memristive effect is considered in [14]. Since work [11] was published, many authors have considered the memristor resistance as the sum of resistances of the alloyed and unalloyed areas in which the border between these two regions can move. Consideration was given to the models in which modification of the specific resistance of the metal/dielectric interface is conditioned by participation of the defects or traps of the carriers [15-17] or by formation of the localized chains of atoms of the metals which become the bridges between the electrode materials under the influence of the electric field [18]. Work [19] deals with the memristor device having a single homogeneous TiO_{2-x} a layer arranged between the metal electrodes which displays good memristive characteristics. The authors suggest the memristor model that allows for the interaction of the conductive fibers with the interfaces at the border with metal. Coexistence of several various mechanisms of memristor switching is quite possible, and various mechanisms can be dominating both in different materials, and under various modes.

Our work studies the memristive properties of the structures which incorporate a homogeneous $Au/TiO_{2-x}/Au$ a layer with various contents of oxygen vacancies, or a TiO_{2-x} layer with the smoothly varying or abrupt change of the x value.

The multilayer structures of $Au/VO_2/VO_{2-x}/Au$ have been obtained and investigated by the droplet-free PLD method on the substrates of c-sapphire with the use of mask technologies.

Using the data of *I-V* curves we have investigated for the first time the effect of magnetoresistance in the thin films of InSb:Mn produced on the substrates of c-sapphire from the InSb+MnSb composite alloy targets with the contents of MnSb to 20% by the PLD method in the droplet-free mode. The temperature dependence of magnetoresistance of the films in the range of temperatures from 10K to 300K has been studied. The diode structures *p*-(InSb+MnSb)/*n*-InSb have been formed on the single-crystal substrates of n-InSb from the InSb+MnSb targets by the PLD method. The current-voltage characteristics of the diode structure of *p*-(96%InSb+4%MnSb)/*n*-InSb have been explored at ambient temperature in the absence of the field and at various orientations of the magnetic field of 0,15 T both in the plane of structure, and perpendicular to it.

2. The experimental part

2.1. Experimental procedure

The InSb: Mn films were deposited by the PLD method in a droplet-free mode on the c-sapphire substrates from the targets of InSb + MnSb composite alloy with MnSb content of 4%, 5%, 10% and 20%. The composition homogeneity and the morphology of the films were investigated by the SEM method with an attachment for energy-dispersive X-ray spectroscopy, by X-ray phase analysis (XPS), and by the AFM method with a magnetic cantilever. The thickness of the films was determined with the aid of the MII-4 interferometer. The conductivity type, the concentration and mobility of the current carriers were measured by the Hall method. The films exhibited p-type conductivity. The multilayer structures of Au / VO_2 / VO_{2-x} / Au were obtained by the droplet-free PLD method on c-sapphire substrates with the use of mask technologies. The active region of the memristor, consisting of two layers (a layer of depleted vanadium oxide VO_{2-x} and a layer of vanadium dioxide VO_2), was located between two metal electrodes. The value of *x* was varied in the process of structures growth by changing the oxygen pressure in the vacuum chamber, which provided the required conductivity in the depleted injection layer of VO_{2-x}. Ablation of the metal targets of vanadium and gold was carried out by an excimer KrF laser radiation at the wavelength of 248 nm with energy density on the target of at least 3 J/cm^2. The oxygen pressure in the vacuum chamber during the growth of the films varied from 0.1 mTorr to 40 mTorr. The thicknesses of the layers of VO_{2-x} and VO_2 ranged from 10 to 100 nm. Deposition of all the layers was carried out at room temperature in the droplet-free regime, which provided the smooth, homogeneous films of high quality by eliminating the droplets emitted from the target onto the substrate during deposition. The memristive structures of $Au/TiO_{2-x}/Au$ were obtained, in which a homogeneous TiO_{2-x} layer with a different content of oxygen vacancies or a TiO_{2-x} layer with a smooth or abrupt change in the value of *x* was located between the electrodes of gold.

2.2. Magnetoresistance of thin InSb:Mn films

The thin InSb:Mn films were grown on sapphire (0001) substrates by ablation with an excimer KrF laser using the droplet-free pulsed laser deposition (PLD) method. The InSb+MnSb composite alloy target contained up to 20% of MnSb. We used Hall method to measure the conductivity type, concentration and mobility of the charge carriers in the films. It has been found that the concentration and mobility of the charge carriers and the resistivity of the films depend on the substrate temperature during deposition and on the energy density at the target surface. The homogeneity of composition and the morphology of the film surfaces were investigated using the SEM method with an energy-dispersive X-ray spectroscopy device, by X-ray phase analysis and by the AFM method with a magnetic cantilever. The investigation of the films with SEM showed the homogeneous distribution of the In, Sb and Mn elements in the surface layer of the film. According to the results of energy-dispersive X-ray spectroscopy, the content of these elements corresponded to the composition of the initial target. The results of the AFM analysis using a magnetic cantilever point to the presence of ferromagnetic clusters of the size of 4 nm. In this paper, we have for the first time investigated the magnetoresistive effect in the thin InSb:Mn films using the *I-V* curve method. The magnetoresistance of the *p*-(InSbMn) films was measured under the constant magnetic field of two values, 0.15 T and 1 T. The field of 0.15 *T* was applied collinearly to the current flowing in the film, and the field of 0.15 T or the field of 1 T was applied perpendicular to the film plane and to the current flowing in the film. The indium ohmic contacts were formed on the film surface before measuring of the I-V curve using the Keithley 2612 device. The *I-V* curve was measured in the range from -10 V to +10 V in steps of 1 mV. Switching of the voltage polarity allowed reversing the polarity of the electric field. The magnetoresistance MR (U) was determined on the basis of *I-V* curve in the temperature range from 10 K to 300 K using the formula $(R(H) - R(0)) / R(0) *$ 100%. It was observed that the magnetoresistance of the sample depended on the magnetic field orientation with respect to the current direction at room temperature. Figure 1 illustrates the dependence of the magnetoresistance of the *p*-InSb:Mn thin films on the voltage for collinear electric and magnetic (0.15 T) fields located in the plane of the film.

The measurements were made of the dependence of the film magnetoresistance on the voltage for the magnetic field perpendicular to the film surface for two magnetic field values B = 0.15 T and B = 1 T. The electric field is localized within the film plane (Fig.2).

The observed different behavior of the magnetoresistance of the films depending on mutual orientation of the collinear magnetic and electric fields can be caused by different mean free paths of the charge carriers for the parallel and antiparallel directions of the fields E and H in the thin InSb:Mn films. For the magnetic field normal to the film a similar dependence on the magnitude of the field is disclosed [8].

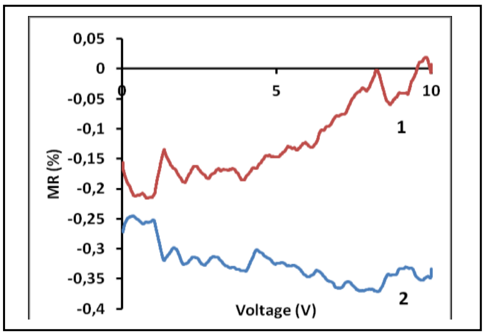

Fig. 1. The magnetoresistance of the *p*-InSb:Mn thin films vs. the voltage for collinear electric and magnetic fields located in the plane of the film (1 - $E\uparrow\downarrow B$, 2 - $E\uparrow\uparrow B$), B=0.15T.

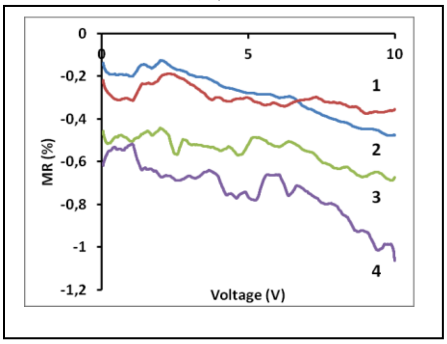

Fig. 2. The magnetoresistance of the film vs. the voltage for the magnetic field perpendicular to the film (1 – B = 0,15 T ↑, 2 – B = 0,15 T ↓, 3 – B = 1 T ↑, 4 – B = 1 T ↓).

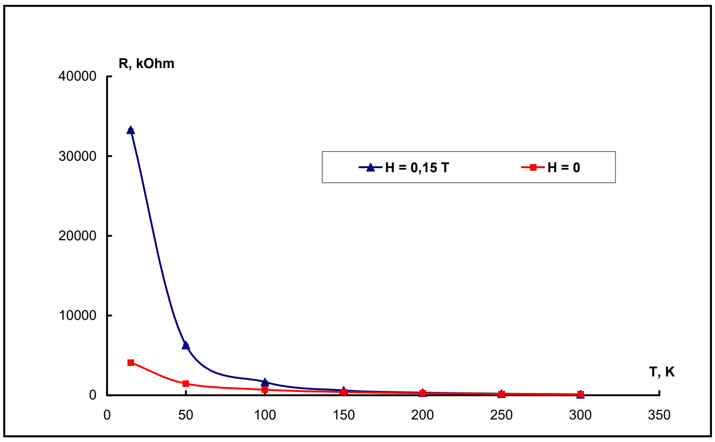

Fig. 3. The dependence of the film InSb:Mn resistance on the temperature in the absence (squares) and in the presence of a magnetic field of 0.15 T (triangles). The voltage applied to the sample is 10V.

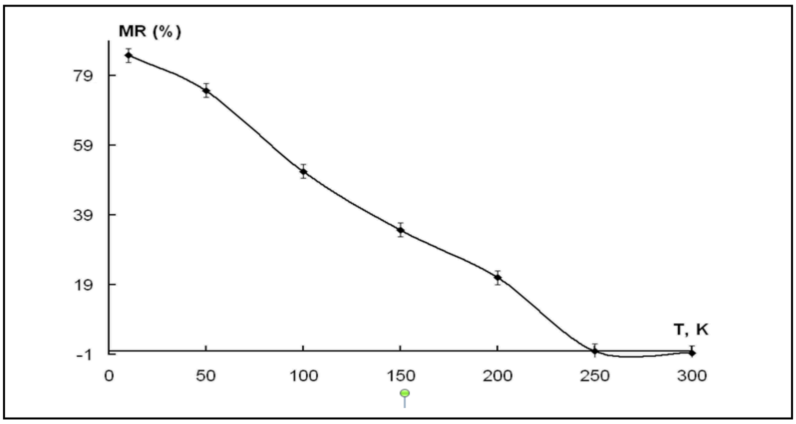

Fig. 4. The dependence of the thin InSb:Mn film magnetoresistance on the temperature at an applied voltage of 10V and under magnetic field of 0.15 T in the plane of the film.

The temperature dependence of the magnetoresistance of *p*-InSb:Mn films with the MnSb content of 20% was studied in the temperature range from 10 K to 300 K. The investigations were carried out by measuring and analyzing their *I*-*V* curves in the temperature range from 10 K to 300 K. The measurement wires were connected to the indium ohmic contacts which were formed on the surface of each film. The films were placed in a cryostat. The measurements of the *I*-*V* curves were performed in the field with permanent magnet and without it. The dependences of the magnetoresistance for a fixed temperature and magnetic field were calculated on the basis of the *I-V* curve. The results of measurements *R* and calculations *MR* are shown in Fig. 3, 4.

Figure 4 shows that the crossover from positive to negative giant magnetoresistance (*GMR*) is observed at the temperature of 250 K during cooling. As the sample cooled down, the value of *GMR* increased and reached 600% at 15 K in the magnetic field of 0.15 T directed perpendicular to the plane of the film.

Thus, it has been shown that the magnetoresistance of the InSb:Mn thin films is registered even at room temperature under small magnetic fields, which opens up considerable prospects for using this material to create various spintronic devices.

The diode structures *p*-(InSb + MnSb) / *n*-InSb were grown on the single-crystalline *n*-type InSb substrates from the InSb + MnSb targets using the PLD method. The *I-V* curves of the *p*-(96% InSb + 4% MnSb) / *n*-InSb structure have been investigated at room temperature under the magnetic field of 0.15 T both in the plane of the structure and perpendicular to it. Figure 5 shows the *I-V* curves of such a diode structure at room temperature in the absence of the magnetic field and at different orientations of the magnetic field of 0.15 *T* both in the plane of the structure and perpendicular to it.

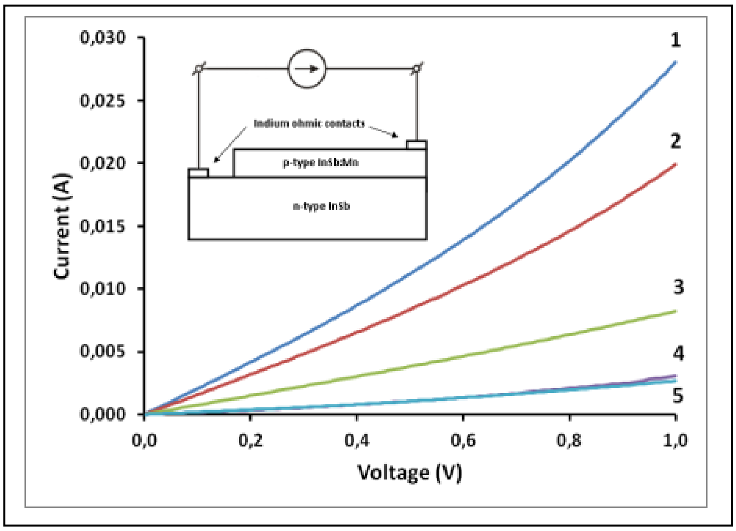

Fig. 5. The *I-V* curves of the heterostructure *p*-(InSb:Mn)/*n*-InSb with *B* = 0,15T (1 – *B* = 0; 2 - *B* ↑↓ *E*; 3 - *B* ↑↑ *E*) in the plane of the film and *B*= 0,15T perpendicularly to the plane of the film 4 – *B* (↑); 5- *B* (↓); the inset presents the scheme of the heterostructure.

In the structures *p*-(InSb:Mn)/*n*-InSb represented at the inset of Fig. 5 both the electrical contacts are located in the heterostructure plane. As seen from Fig. 5, when the direction of the field *H* is changed in the film plane, the *I-V* curves differ, which may be due to the difference in the magnetoresistance of the *p*-film (96% InSb + 4% MnSb) for different mutual orientations of the electric and magnetic fields. This can be seen from curves 2 and 3 in Fig. 5. The current decreased almost 9 times from 0.35 *A* to 0.04 *A* at the voltage of 1 *V* under the magnetic field of 0.15 *T* perpendicular to the plane of the diode structure. The results obtained show that in all cases of magnetic field application the phenomenon of giant magnetic resistance (*GMR*) is observed. The observed crossover of *GMR* is associated with spin-selective carrier scattering due to the *p-d* exchange interaction [8]. This interaction leads to splitting of the valence band of the diode *p*-region into two spin-up and spin-down bands. In the presence of a magnetic field, the difference in the conductivity of the two bands results in a relative change of the diode current.

Thus, we have studied the spin-dependent properties of the *p*-InMnSb/*n*-InSb heterojunction where *p*-InMnSb is a magnetic semiconductor thin film grown by pulsed laser deposition. The measured *I-V* curves of the diode structure revealed that positive magnetoresistance is observed under direct bias and applying of a magnetic field at 298 K.

2.3. Memristor Based on Vanadium Dioxide

The multilayer structures of Au/VO_2/VO_{2-x}/Au were obtained by the droplet-free PLD method on c-sapphire substrates with the use of mask technologies. The active region of the memristor, consisting of two layers (a layer of oxygen depleted vanadium oxide VO_{2-x} and a layer of vanadium dioxide VO_2), was located between two metal electrodes (Figure 6). The value of x was varied during the growth of the structures by changing the oxygen pressure in the vacuum chamber, which provided the required conductivity in the depleted injection layer of VO_{2-x}. Ablation of the metal targets of vanadium and gold was carried out by an excimer KrF laser radiation at the wavelength of 248 nm with energy density on the target of at least 3 J/cm^2. The oxygen pressure in the vacuum chamber during the growth of the films varied from 0.1 mTorr to 40 mTorr. The thicknesses of the layers VO_{2-x} and VO_2 ranged from 10 to 100 nm. Deposition of all the layers was carried out at room temperature in the droplet-free regime, which ensured the production of smooth, homogeneous films of high quality by eliminating droplets emitted from the target onto the substrate during deposition.

To study the memristive properties at room temperature, the Au/VO_2/VO_{2-x}/Au structure, in which the thickness of the VO_2/VO_{2-x} layers was 10/30 nm, was placed on a copper plate for the purpose to thermally stabilize the structure when current and voltage were supplied. It is established that under the action of a sequence of short unipolar voltage pulses applied to this structure, its resistance decreases after each pulse, demonstrating a memristive effect (Fig. 7).

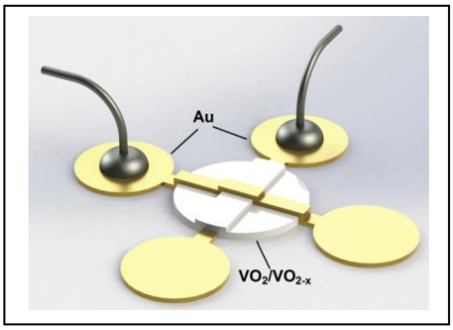

Fig. 6. Schematic representation of the multilayer structure of $Au/VO_2/VO_{2-x}/Au$ synthesized on c-sapphire.

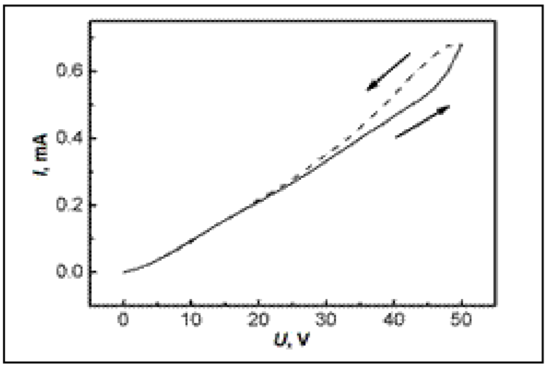

Fig. 7. A change in the resistance and current of the multilayer structure $Au/VO_2/VO_{2-x}/Au$ under the action of short impulses of the applied voltage.

A typical current-voltage characteristic of such a structure is shown in Fig. 8.

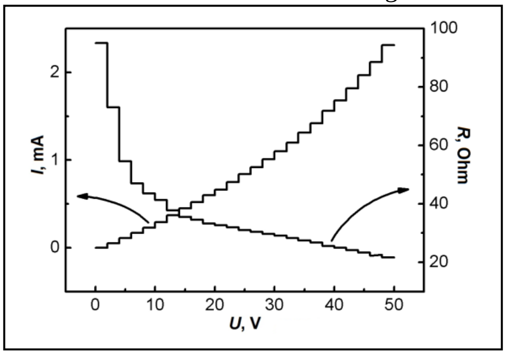

Fig. 8. *I-V* characteristic of the multilayer structure $Au/VO_2/VO_{2-x}/Au$; unipolar current pulses are applied. Solid line – an increase in the pulse amplitude, dotted line – a decrease in the pulse amplitude. The arrows indicate the direction in which the voltage applied to the structure changes.

As can be seen from Fig. 8, the *I-V* characteristic has the form of a hysteresis loop. This kind of dependence can be explained within the framework of the memristor model proposed in [11] and based on the redistribution of the thickness of the active layers VO_{2-x} and VO_2 of the memristor. According to this model, when electric field bias is applied to the control electrode, the diffusion of charged oxygen vacancies begins, which leads to a change in the effective thicknesses of each of the layers. Oxygen vacancies act in VO_{2-x} as an n-type dopant, so the VO_{2-x} layer has a significantly lower resistance than the defect-free VO_2. At a certain voltage and magnitude of the charge that has passed through the structure, the VO_2 layer will completely turn into VO_y (y <2), and the resistance of the entire structure will be determined by the smallest possible resistance (solid line, up arrow in Figure 8). With a subsequent decrease in voltage, the curve of the current-voltage characteristic is different (dotted line, down arrow in Figure 8), which depends on the changes that have occurred in the active region of the memristor. Thus, for the first time we have demonstrated the possibility of using vanadium dioxide as a working material for creating memristors in the vertical (Fig. 6) geometry of $Au/VO_2/VO_{2-x}/Au$.

2.4. Memristor based on titanium dioxide

The memristive structures of $Au/TiO_{2-x}/Au$ with various contents of oxygen vacancies have been created by the droplet-free PLD method on *c*-sapphire substrates using mask technologies, and their properties have been studied. A continuous gold film was first grown on the single-crystal substrate at high vacuum and served as the bottom electrode. Then the thin TiO_x films were coated from a high purity titanium target on a part of the Au electrode. The films with different oxygen contents were deposited by changing the oxygen pressure in the chamber. The gold contact pads serving as the upper electrodes were applied on a TiO_{2-x} film surface by the grid mask. Thus, a set of a large number of identical memristive transitions located on one substrate was obtained. An indium contact platform was deposited on the mutual bottom gold electrode; one of two measuring probes was placed on it. The second probe was located on the top electrode of the memristor under study. The probes were the thin tungsten needles coated by gold. The *I-V* characteristics were measured with these probes by the Keithley 2612 device using a two-contact scheme. To measure the *I-V* characteristics a voltage pulse was applied on a sample and the current was measured. Then the voltage value was set to 0, and with a certain delay the following pulse was applied. The amplitudes of the pulses changed under the preset law with a certain step. The pulse duration and filling factor could be adjusted. Further the *I-V* curves were plotted by the resulting pairs of voltage and current values.

On smooth variation of the concentration of oxygen vacancies (oxygen pressure in the chamber changes from $1*10^{-3}$ Torr to $2*10^{-3}$ Torr during an oxide layer growth) in the TiO_{2-x} film and on delivery of a pulse train of positive polarity with triangular envelope, the structure exhibits a memristive effect with R_{on}=8 Ohm and R_{off}=18 Ohm, however when an identical pulse train shows negative polarity, the structure demonstrates an ohmic character of dependence with R=8 Ohm (Fig.9).

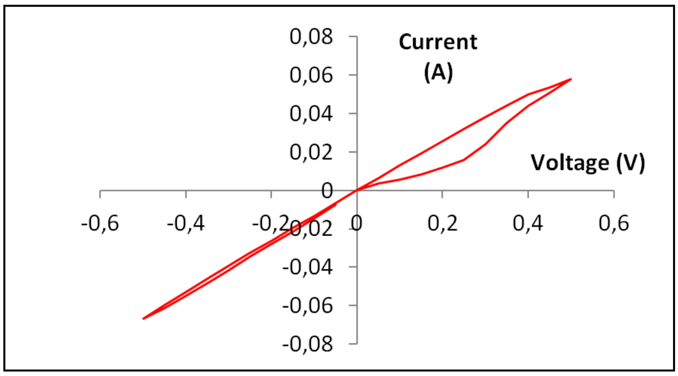

Fig. 9. The I-V curve of the memristive structure of Au/TiO2-x/Au with smooth change of x under bipolar switching.

After the fourth cycle of bipolar switchings the *I-V* curve attained an ohmic character. The sharp change of concentration of oxygen vacancies (the pressure in the chamber is $2*10^{-3}$ Torr at the first stage of TiO_{2-x} film growth and $3*10^{-3}$ Torr at the second stage) results in increasing of the specific resistance of the memristor in the switched-off condition up to 2000 Ohm·cm. Fig. 10 represents the *I-V* curve of the memristor at the first positive bipolar switching.

In this case, R_{on}=18 Ohm and R_{off} =42 Ohm on the positive branch of the *I-V* curve and R_{on}=18 Ohm and R_{off}=21 Ohm on its negative branch. After 100 cycles of bipolar switching the *I-V* hysteresis was significantly narrowed, and the *I-V* curve took a symmetric form. The *I-V* curve is presented by Fig. 11.

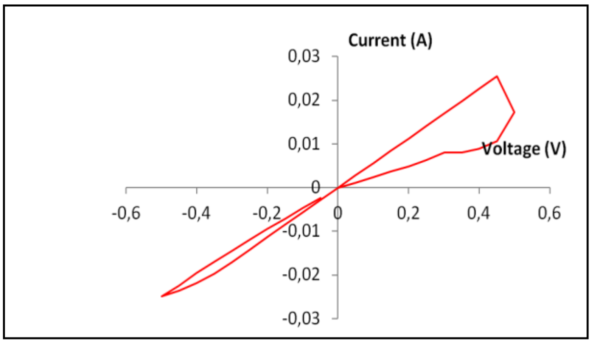

Fig. 10. The *I-V* curve of the memristor with a sharp boundary of oxygen vacancy concentration in a TiO_{2-x} film (the oxygen pressure in the chamber is $2*10^{-3}$ Torr and $3*10^{-3}$ Torr) at the first bipolar switching.

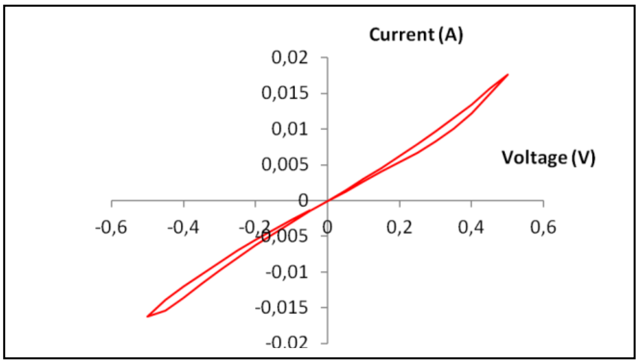

Fig. 11. The *I-V* curve of the memristor after 100 cycles of bipolar switching.

The resistances of the memristor were R_{on}=34 Ohm in the "*on*" state and R_{off}=38 Ohm in the "*off*" state, respectively. At the same time, the exponential dependence of the current on the voltage is tracked in both states, which is testimony to the tunnel transition of electrons through the interface barriers [20]. Thus, the dependence of the memristive properties of Au/TiO_{2-x}/Au structures on oxygen pressure in the growth chamber during pulsed laser deposition is demonstrated.

3. Conclusion

The measurements were made of the dependence of the film magnetoresistance on the voltage for the magnetic field within the film plane and perpendicular to the film surface. The magnetoresistance of the thin InSb: Mn films is recorded even at room temperature in small magnetic fields, which opens up considerable prospects for using this material to create various spintronics devices. We have studied the spin-dependent properties of the *p*-InMnSb/*n*-InSb heterojunction obtained by pulsed laser deposition. The *I-V* curves of the diode structure *p*-InMnSb/*n*-InSb, where *p*-InMnSb is a magnetic semiconductor, have been obtained. These curves showed that at forward bias and application of a magnetic field positive magnetoresistance is observed at room temperature.

It has been established that under the action of a sequence of short unipolar voltage pulses applied to the Au/VO_2 /VO_{2-x} /Au structure, its resistance decreases after each pulse, demonstrating a memristive effect.

The memristive structures Au/TiO_{2-x} /Au, synthesized at the oxygen pressure from $2\cdot10^{-3}$Torr to $3\cdot10^{-3}$Torr in the chamber, exhibit a hysteresis of the *I-V* curves in both the positive and negative branches of the applied voltage, which indicates the ability of these structures to change their electrical resistance when the current flows.

Acknowledgements

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