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Photosensitive $CuFeO_2$/*n*-InSe Heterojunctions

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Photosensitive anisotypic $CuFeO_2$/*n*-InSe heterojunctions were fabricated by the method of low-temperature spray pyrolysis. An aqueous solution of copper dichloride $CuCl_2 \cdot 2H_2O$ and iron trichloride $FeCl_3 \cdot 6H_2O$ was sprayed onto the InSe substrate heated to 623 K. As a result, *p*-type $CuFeO_2$ films with a thickness of ~ 0.3 μm and a band gap of 2.6 eV were obtained. Contacts were formed using silver-based conductive paste. The *I-V* characteristics were studied at temperatures from 295 to 336 K. It was shown that the temperature dependence of the height of the potential barrier is linear. Based on the analysis of the temperature dependences of forward and reverse *I-V* characteristics, the dynamics of change of energy parameters was established and the role of energy states at the boundary of the heterojunction in the formation of the contact potential difference was clarified. The approximation of *I-V* characteristics was carried out within the framework of the model, which takes into account the influence of series and shunt resistances. The values of the diode coefficient, series and shunt resistances of the heterojunction were found. The mechanisms of formation of direct and reverse currents through the $CuFeO_2$/*n*-InSe energy barrier were determined. The spectral dependence of the quantum efficiency of heterojunctions in the range of photon energies from 1.2 to 3.2 eV was studied. The effect of light absorption in heterostructure materials on its general photosensitivity was analyzed. The obtained results confirmed the promise of $CuFeO_2$/*n*-InSe heterojunctions for photoelectronics.

Keywords: Indium Selenide, $CuFeO_2$, Electrical Conductivity, Photosensitivity.

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1. INTRODUCTION

Indium monoselenide (InSe) has a band gap E_g = 1.26 eV which is in the range of optimal values for photoelectric conversion of the solar spectrum in terrestrial conditions. The layered structure of InSe crystals with a weak Van der Waals bond between the layers provides them with an advantage over other semiconductors in the manufacture of substrates for heterostructures by avoiding ingot cutting operations, mechanical and chemical surface treatment. In addition, the resistance of InSe to radiation expands the scope of its use. The use of indium selenide as a base material allows to create various electronic components such as photodetectors [1, 2], solar cells [3, 4], Van der Waals heterojunctions [5, 6] and other.

The presence of a weak Van der Waals bond between the layers and a strong ion-covalent bond in the layers in InSe determines the features of the physical properties of crystals. In particular, the existing structural defects significantly affect the electrical properties [7]. Stacking faults, dislocation grids placed in the (0001) plane, create additional energy barriers E_δ for the movement of charge carriers along the *c* axis, which causes large values of electrical conductivity anisotropy. Due to the existence of vacancies and dislocations, localized states appear near the Fermi level [8].

Transparent conductive oxides are materials with high electrical conductivity and low optical absorption of visible light. Thin films of transparent conductive oxides are widely used in various devices such as flat panel displays, touch panels and solar panels. It is known that heterojunctions based on such oxides and InSe have good rectifying properties and photosensitivity [4, 9, 10]. Delafossites are triple oxides of copper and iron with the basic formula ABO_2, where A represents monovalent cations, such as Cu or Ag, and B represents trivalent metals from Al to La. $CuFeO_2$ delafossite is a *p*-type semiconductor, the band gap of which can vary from 0.91 to 3.35 eV [11]. $CuFeO_2$ has a relatively high electrical conductivity compared to other delafossites, only $CuCrO_2$ is higher. $CuFeO_2$ can exhibit both the properties of multipheroism and spintronics. Investigation of magnetic and magnetoelectric properties of $CuFeO_2$ is intensively studied [12, 13].

This paper presents the results of the study of the electrical properties and photosensitivity of $CuFeO_2$/*n*-InSe heterojunction fabricated by spray pyrolysis of pyrite thin films on *n*-InSe substrates.

2. EXPERIMENTAL

Monocrystalline *n*-InSe grown by the Bridgman method was used to make heterojunctions. Plane-parallel plates 5 × 5 × 1.2 mm³, which had perfect mirror surfaces, were chipped from the InSe crystal ingot along the cleavage plane. Chipping was performed in air.

Thin films of $CuFeO_2$ *p*-type conductivity with a thickness of ~ 0.3 μm were made by spray pyrolysis of 0.1 M aqueous solutions of salts of copper dichloride $CuCl_2 \cdot 2H_2O$ and iron chloride $FeCl_3 \cdot 6H_2O$. When dissolving metal salts, double-distilled water was used. Separately prepared solutions of copper and iron salts before application were mixed in a ratio of 1:1 by volume using a magnetic stirrer for 30 min at room tem-

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perature. The pyrolysis temperature when obtaining samples of $CuFeO_2$ thin films on substrates of sodium-calcium glass, sitall and indium selenide was $T_S = 623$ K. Compressed air was used as the carrier gas. Glass and sieve substrates were degreased in ammonia-peroxide solution H_2O_2:$(NH_2)OH$:H_2O before treatment with $CuFeO_2$ films, treated with a 5 % solution of potassium dichromate $K_2Cr_2O_7$ in sulfuric acid and washed in double-distilled water. Samples of $CuFeO_2$ films made on 18×18 mm^2 glass were used to study the optical transmission spectrum. Optical band gap of manufactured $CuFeO_2$ films was $E_g = 2.6$ eV. The process of spray pyrolysis allowed simultaneous deposition of films on different substrates. Specially made masks were used to obtain the film steps used in determining the thickness. The thickness of $CuFeO_2$ films was measured with a MII-4 Linnik microinterferometer. The surface electrical resistance of $CuFeO_2$ films on dielectric sieve substrates immediately after the manufacturing process was monitored by the four-probe method and was $\rho_s = 10^4$ Ω/square.

Contacts to the InSe base material and to the $CuFeO_2$ film were formed using a silver-based conductive paste. The *I-V* characteristics of $CuFeO_2$/*n*-InSe heterojunctions were studied on a Solartron 1255 measuring complex in the temperature range from 295 to 336 K. The photosensitivity spectra of heterojunctions were measured at room temperature on a monochromator MDR-3 with a resolution of 2.6 nm/mm. For the spectra, normalization was performed with respect to the photon flux.

3. RESULTS AND DISCUSSION

Fig. 1 shows the forward *I-V* characteristics of $CuFeO_2$/*n*-InSe heterojunctions measured at different temperatures. They are well described by the formula within the model, which takes into account the influence of series (R_s) and shunt resistances (R_{sh}) [14]:

$$I = I_s\left[\exp\left(\frac{e\left(V - IR_s\right)}{nkT}\right) - 1\right] + \frac{V - IR_s}{R_{sh}}, \qquad (1)$$

where I_s is the reverse saturation current, which can be found by extrapolation to zero voltage, n is the diode coefficient.

Curves in Fig. 1 represent the results of the *I-V* characteristics approximation by formula (1). The fitting parameters are given in Table. 1.

The values of the potential barrier height φ_0 of the heterostructure at different temperatures are determined by extrapolating the linear sections of the *I-V* characteristics to the intersection with the voltage axis. It is established that the temperature dependence of $\varphi_0(T)$ for $CuFeO_2$/*n*-InSe heterojunctions is well described by the equation (Fig. 2):

$$\varphi_0\left(T\right) = \varphi_0\left(0\right) - \beta_\varphi T, \qquad (2)$$

where $\beta_\varphi = 5.5 \cdot 10^{-3}$ eV·K^{-1} is the temperature coefficient of the potential barrier height, and $\varphi_0(0) = 2.56$ eV is the value of the potential barrier height of the studied heterostructure at absolute zero temperature.

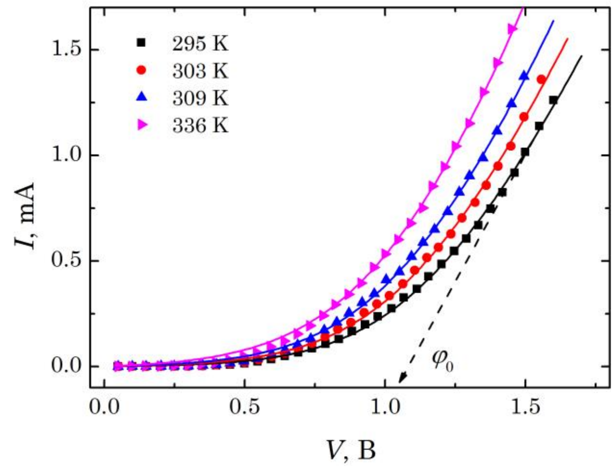

Fig. 1 – Forward *I-V* characteristics of the $CuFeO_2$/*n*-InSe heterojunction at different temperatures (points are experimental data, curves are approximation by formula (1))

Table 1 – Fitting parameters

T, K	n	I_s, μA	R_s, Om	R_{sh}, Om
295	8	2.5	272	10^6
303		3.7	253	
309		5.4	232	
336		11	190	

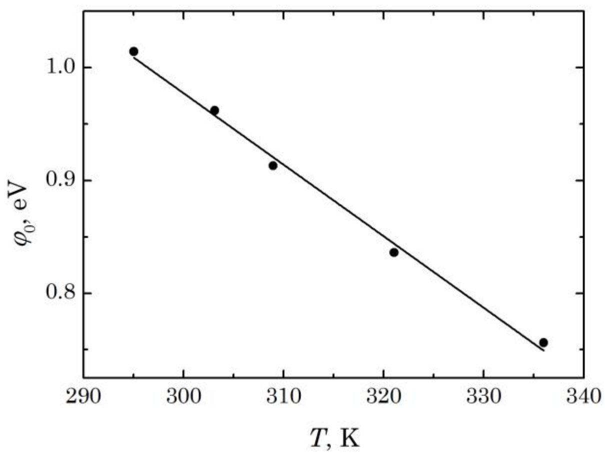

Fig. 2 – Temperature dependence of the potential barrier height of the $CuFeO_2$/*n*-InSe heterojunction

The large value of β_φ compared to the temperature coefficient of the band gap for InSe ($\beta_{Eg} = 2.3 \cdot 10^{-4}$ eV/K) may be due to the high concentration of surface defects N_{ss} at the $CuFeO_2$/*n*-InSe interface, which is a consequence of a significant difference between the constant lattice and materials heterojunction.

In the first approximation $N_{ss} \propto x^{-2}$ [14], where the distance between the dislocations of the mismatch x is determined from the following expression:

$$x = a_{\mathrm{InSe}} a_{\mathrm{CuFeO_2}} / \left(a_{\mathrm{InSe}} - a_{\mathrm{CuFeO_2}}\right). \qquad (3)$$

For $a_{\mathrm{InSe}} = 4.24$ Å and $a_{\mathrm{CuFeO2}} = 5.43$ Å, we obtain $x = 19.3$ Å and $N_{ss} = 2.67 \cdot 10^{13}$ cm^{-2}. At this density, surface states can play the role of effective centers of capture or recombination, and significantly affect the electrical properties of heterostructures.

Fig. 3 shows the dependence of the differential resistance R_{dif} on the voltage V, on the basis of which it is possible to determine the value of the series resistance of the heterostructure R_s. It is seen that in the voltage region greater than the potential barrier height, curves $R_{dif}(V)$ reach saturation. This indicates that the voltage in the barrier region of the diode ceases to change, i.e., the barrier is almost open, and the current through the heterojunction is limited by its series resistance R_s, which is determined by extrapolating the saturation region to the intersection with the differential resistance axis.

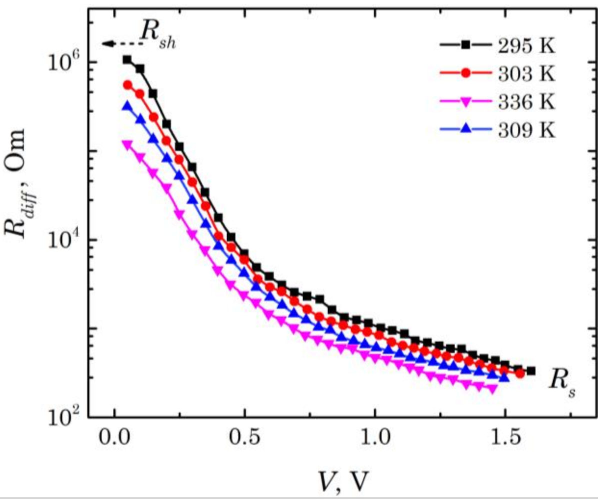

Fig. 3 – Dependence of differential resistance of the $CuFeO_2$/n-InSe heterojunction on voltage at different temperatures

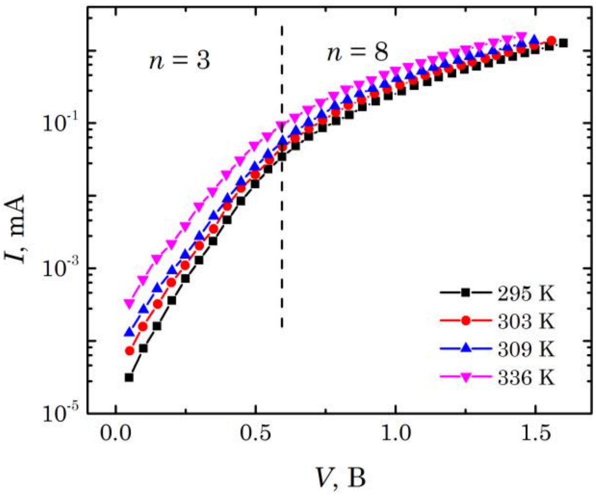

Fig. 4 – Forward I-V characteristics of the $CuFeO_2$/n-InSe heterojunction at different temperatures

The series resistance of the heterojunction is determined by the resistance of the substrate (InSe). At room temperature $\rho_{\|c}$(InSe) ~ 10^3 Om·cm. Then for a sample of size $5 \times 5 \times 1.2$ mm^3 we can estimate $R_S = \rho \cdot S/l = 480$ Om·cm, which correlates with the data of Fig. 3.

The forward I-V characteristics of the heterojunction in semi-logarithmic coordinates at different temperatures are shown in Fig. 4. As can be seen from the figure, in the area of direct bias $V > 3kT/e$ rectilinear sections are observed.

Analysis of the forward I-V characteristics of the $CuFeO_2$/n-InSe heterojunction constructed on a semi-logarithmic scale showed that dependence $\ln(I) = f(V)$ consists of two rectilinear sections, which indicates the exponential dependence of current on voltage. The determined values of the diode coefficient ($\Delta\ln(I)/\Delta V = e/nkT$) are $n = 3$ ($V < 0.6$ V) and $n = 8$ ($V > 0.6$ V).

The large value of the diode coefficient and the weak slope of the dependences $\ln(I) = f(V)$ at different temperatures is evidence of the tunneling nature of the current transfer mechanism. At small displacements, the space charge region is too thin for direct tunneling, which is described by Newman's formula. Therefore, given the above-assessed high concentration of N_{ss} mismatch dislocations, multistage tunneling-recombination processes involving surface states at the $CuFeO_2$/n-InSe interface can be considered the only mechanism of current transfer. The forward bias current is defined by the following expression [14]:

$$I = B\exp\left(-\alpha\left(\varphi_0\left(T\right) - eV\right)\right), \qquad (4)$$

where B is a value that is weakly dependent on temperature, and φ_0 is the potential barrier height.

Rewrite expression (4) in another form:

$$I = B\exp\left(-\alpha\varphi_0\left(T\right)\right)\exp\left(\alpha eV\right) = I_0\exp\left(\alpha eV\right), \qquad (5)$$

where $I_0 = B \cdot \exp(-\alpha\varphi_0(T))$ is the cut-off current, which does not depend on the applied voltage.

From expression (5), it follows that the slope $\Delta\ln(I)/\Delta V$ of the initial sections of the forward I-V characteristics determines the coefficient α, which takes the value of ≈ 10 eV^{-1}.

Substituting this formula into an expression for the cut-off current, we obtain the following expression:

$$\begin{aligned} I_0 &= B\exp\left(-\alpha\left(\varphi_0\left(0\right) - \beta_\varphi T\right)\right) = \\ &= B\exp\left(-\alpha\varphi_0\left(0\right)\right)\exp\left(\alpha\beta_\varphi T\right) = I_C\exp\left(\alpha\beta_\varphi T\right), \end{aligned} \qquad (6)$$

where I_C is a constant.

The coefficient α can be determined from the slope of the rectilinear dependence $\ln(I_0) = f(T)$: $\alpha = \beta_\varphi^{-1}$ $(\Delta\ln(I_c)/T) = 10.5$ eV^{-1}. The close values of the coefficient α, determined from different dependences (formulas (5) and (6)), confirm the reliability of the analysis of the initial I-V characteristics of $CuFeO_2$/n-InSe heterojunction characteristics within the multistage tunnel recombination current transfer mechanism with the participation of surface states at the n-InSe/$InFe_2$ interface.

In the voltage range $V > 0.5$ V, the dependence of $I(V)$ is well described by Newman's formula for tunnel current [14]:

$$I = I_t^0\exp\left(\beta T\right)\exp\left(\gamma V\right) = I_t\exp\left(\gamma V\right), \qquad (7)$$

where $I_t = I_t^0 \cdot \exp(\beta T)$ is cut-off current, γ and β are constants.

From the last expression, it is seen that the slope of $\Delta\ln(I)/\Delta V$ of the forward I-V characteristics determines the coefficient γ (formula (7)), which takes the value of 5.8 V^{-1}.

At a reverse bias in the case of a sharp transition, the expression for the tunnel current has the form [14]:

$$I_{rev}^{t} \approx a_0 \exp\left(\frac{b_0}{\sqrt{\varphi_0(T)-eV}}\right), \qquad (8)$$

where α_0 and b_0 are voltage-independent parameters.

The fact that the reverse *I-V* characteristics in Fig. 5 are straight lines in $\ln(I^t_{rev}) = f(\varphi_0 - eV)^{-1/2}$ coordinates, according to equation (8), confirms the dominance of the tunneling mechanism of current transfer in the region of inverse bias $|V| > 3kT/e$.

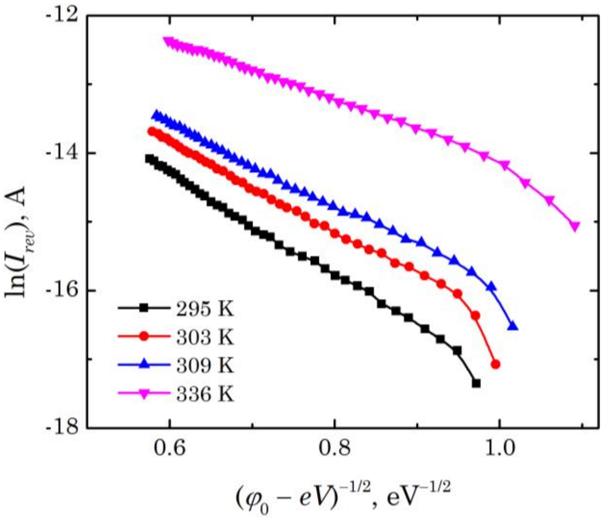

Fig. 5 – Reverse *I-V* characteristics of the $CuFeO_2$/*n*-InSe heterojunction at different temperatures

Reducing the slope of rectilinear sections in Fig. 5 with increasing temperature is associated with a decrease in the parameter b_0, which is determined from the following expression:

$$b_0 = CW_1(T)\varphi_0(T)^{3/2}, \qquad (9)$$

where C is a constant, W_1 is the width of the space charge region at $\varphi_0 - eV = 1$ eV.

For an asymmetric sharp heterojunction, the width of the space charge region is determined from the following expression [14]:

$$W = \sqrt{\frac{2\varepsilon_0\varepsilon_p(\varphi_0 - eV)}{e(N_A - N_D)}}, \qquad (10)$$

where ε_0 is the absolute dielectric constant of vacuum, ε_p is the relative dielectric constant.

The parameter α_0 is determined by the probability of filling the energy level from which the tunneling occurs during the reverse displacement. From the energy slope of the temperature dependence $\ln(\alpha_0) = f(10^3/T)$ it is possible to determine the depth of its occurrence. But the obtained experimental data indicate the temperature independence of the parameter α_0. This situation is typical of a metal-semiconductor contact when tunneling from metal levels. The temperature independence of the parameter α_0 may indicate participation in the processes of tunneling of surface states with a uniform energy distribution.

The spectral dependence of the quantum efficiency of the $CuFeO_2$/*n*-InSe heterostructure irradiated from the side of the $CuFeO_2$ film is in the range of photon energies from 1.2 to 3.2 eV with a maximum at 2.3 eV (Fig. 6). The long-wavelength edge of photosensitivity at $h\nu \approx 1.2$ eV is due to the edge of fundamental absorption in *n*-InSe. $CuFeO_2$ thin films are polycrystalline, as a result of which the intrinsic absorption edge is blurred due to partial absorption at grain boundaries compared to monocrystalline materials. At energies $h\nu < E_g = 2.4$ eV, a part of the radiation is absorbed at grain boundaries. In this case, light that is able to be absorbed in *n*-InSe does not penetrate into the base region due to the absorption in $CuFeO_2$. The spectral characteristic shows a decrease in photosensitivity. At a quantum energy $h\nu > 2.4$ eV, free minor charge carriers are generated in the $CuFeO_2$ film, which diffuse to the heterojunction and form a current. Photosensitivity increases. The full width at half height $\delta_{1/2}$ of the relative quantum efficiency spectrum is equal to 1.78.

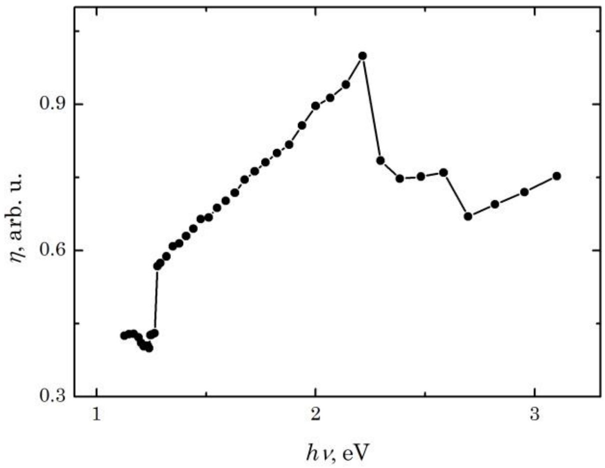

Fig. 6 – Spectral dependence of the relative quantum efficiency of the $CuFeO_2$/*n*-InSe heterojunction

It is known that when InSe is heated in air to temperatures of 573÷773 K, In_2Se_3 and $In_2(SeO_4)_3$ phases are formed, and In_2O_3 at higher temperatures [15, 16]. These phases can cause changes in the photosensitivity spectra. In [17], it was shown that the band gap of α-In_2Se_3 films increases with decreasing thickness and can reach 2.8 eV for 3.1 nm. Since, in the manufacture of a heterojunction, the substrate is heated to 623 K, we can assume that a thin film of In_2Se_3 is formed on the surface of InSe, which determines the maximum on the curve.

4. CONCLUSIONS

It is shown that $CuFeO_2$/*n*-InSe heterojunctions made by low-temperature spray pyrolysis of $CuFeO_2$ thin films onto *n*-InSe crystalline substrates have rectifying properties and good photosensitivity. The character of the temperature dependences of the *I-V* characteristics does not change in the temperature range from 295 to 336 K. The deviation of the *I-V* characteristics from the ideal diode model due to the influence of series and shunt resistances is discussed. It is established that a tunneling-recombination mechanism of current transfer is dominant in $CuFeO_2$/n-InSe heterojunctions. In a logarithmic scale, the direct I-V characteristics have two straight sections with different values of the diode coefficient: $n = 3$ for $V < 0.6$ V and $n = 8$ for $V > 0.6$ V. The high values of n are evidence of the tunneling nature of the current transfer.

$CuFeO_2$/n-InSe heterojunctions are photosensitive in the range of photon energies from 1.2 to 3.2 eV with a maximum at 2.3 eV. The long-wavelength edge of photosensitivity at $h\nu \approx 1.2$ eV is due to the edge of fundamental absorption in n-InSe. The photosensitivity is determined not only by light absorption in the InSe base region, but also by absorption in $CuFeO_2$ and in In_2Se_3 thin film, which is obtained as a result of heating the substrate during sputtering. This extends the range of photosensitivity of the heterojunctions towards higher energies.

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Фоточутливі гетеропереходи $CuFeO_2$/n-InSe

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Методом низькотемпературного спрей-піролізу виготовлено фоточутливі анізотипні гетеропереходів $CuFeO_2$/n-InSe. На нагріту до 623 К підкладку InSe розпилювався водний розчин солей двохлористої міді $CuCl_2 \cdot 2H_2O$ і трихлористого заліза $FeCl_3 \cdot 6H_2O$. В результаті отримувались плівки $CuFeO_2$ p-типу із товщиною ~ 0,3 мкм та шириною забороненої зони 2,6 еВ. Контакти формувалися з використанням струмопровідної пасти на основі срібла. Проведено дослідження ВАХ при температурах від 295 до 336 К. Показано, що температурна залежність висоти потенціального бар'єру є лінійною. На основі аналізу температурних залежностей прямих та обернених ВАХ встановлено динаміку зміни енергетичних параметрів та з'ясовано роль енергетичних станів на межі гетеропереходу у формуванні контактної різниці потенціалів. Проведено апроксимацію ВАХ в рамках моделі, що враховує вплив послідовного та шунтуючого опорів. Знайдено значення діодного коефіцієнту, послідовного та шунтуючого опорів гетеропереходу. Визначено механізми формування прямого та зворотного струмів через енергетичний бар'єр $CuFeO_2$/n-InSe. Досліджено спектральну залежність квантової ефективності опроміненої з боку $CuFeO_2$ гетероструктури в діапазоні енергій фотонів від 1,2 до 3,2 еВ. Проаналізовано вплив поглинання світла в матеріалах гетероструктури на її загальну фоточутливість. Отримані результати підтверджують перспективність гетероструктур $CuFeO_2$/n-InSe для фотоелектроніки.

Ключові слова: Селенід індію, $CuFeO_2$, Електропровідність, Фоточутливість.