EFFECT OF THE DIFFUSION OF COPPER ATOMS IN POLYCRYSTALLINE CdTe FILMS DOPED WITH Pb ATOMS[†]

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The process of diffusion of labeled copper ^{61}Cu atoms in p-CdTe<Pb> coarse-block films with a columnar grain structure has been studied. The CdTe<Pb> film is a p-type semiconductor, where an increase in the Pb concentration in the composition of the CdTe films increases the resistivity ρ of the structure. When the Pb concentration in CdTe changes from 10^{18} to $5\cdot10^{19}$ cm^{-3}, the hole concentration decreases by more than 3 orders of magnitude at a constant operating level depth of $E_V + (0.4 \pm 0.02)$ eV. This may indicate that the concentration of acceptor defects, which are formed in the films due to self-compensation upon doping with a Pb_{Cd} donor, exceeds the number of the latter. Electrical measurements by the Hall method were carried out at a direct current and a temperature of 300 K. As a result, an increase in the temperature of films on a Mo-p-CdTe<Pb> substrate during annealing affects the electrical parameter of charge carrier mobility μ, it decreases significantly. X-ray diffraction analysis showed that on the diffraction patterns of samples of p-CdTe<Pb> films, all available reflections correspond to the CdTe phase and up to x = 0.08 do not contain reflections of impurity phases and have a cubic modification. Based on the results of the calculation, it was established that the low values of the diffusion coefficient of Cu atoms are due to the formation of associates of the A type $Cu_i^+ Pb_{Cd}^-$, which are directly dependent on the concentration of Pb_{Cd} atoms. Diffusion length L_n and lifetime τ_n of minority current carriers in large-block p-type cadmium telluride films, which can also be controlled by introducing lead atoms into cadmium telluride.

Keywords: *Diffusion; Associate; Lifetime; Film; Acceptor center; Radioactive isotope; Distribution; Mobility, Resistivity; Diffusion coefficient; Enthalpy*
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INTRODUCTION

In recent years, significant progress has been made in the technology of manufacturing thin-film solar cells based on A^2B^2 semiconductor compounds [1–4]. Unfortunately, $Cu_2S − CdS(ZnCdS)$ and $Cu_2S − CdTe$ thin-film solar cells tend to change their parameters during operation. The degradation of such solar cells is mainly associated with a change in the heterojunction interface and shunting of the base thin CdS(ZnCdS) and CdTe layers due to the high diffusion rate of copper atoms.

In [5], a hypothetical model was proposed - associations of doped Cu atoms with ionized acceptors of non-vacancy origin, leading to a sharp decrease in the diffusion rate in cadmium telluride films.

However, the microparameters of these local acceptor centers are not specified, their nature is not disclosed, it is only indicated that at the annealing temperature the Fermi level should be above the level of acceptor centers A′ of non-vacancy origin. This condition allows acceptor centers A to be in a charged state and form an associate (complex) with interstitial ions of copper atoms of the $Cu_i^+ A′$ type.

The development of a controlled reproducible technology for producing thin-film solar cells and their modules using layers of honey sulfide and telluride is mainly associated with the search for and determination of impurities that could localize copper atoms in the space of crystal lattices of cadmium sulfide and telluride by forming stable complexes.

It was shown in work [6] that impurities of Sn, Ge and Pb atoms in cadmium telluride dissolve in high concentration (up to $10^{19}÷10^{20}$ cm^{-3}) and form levels, respectively, $E_C – (0,6÷0,9)$ eV; $E_V + 0,6$ eV; $E_V + 0,4$ eV.

Since Ge, Sn, and Pb impurities are located at the sites of the cadmium sublattice in cadmium telluride crystals, they can, in principle, play the role of ionized acceptor centers leading to the formation of associates with doped copper atoms. Among these impurities, Pb atoms have a certain advantage. Pb is highly soluble in CdTe, secondly, Pb can be doped at relatively low temperatures (773÷973 K), thirdly, Pb in CdTe gives a "shallower" level than impurities of Sn and Ge atoms.

The aim of the authors is to study the diffusion of labeled copper atoms in p-CdTe large-block films with different contents of Pb atoms in the temperature range 573÷723K.

MATERIALS AND METHODS

The distribution profiles of the concentration of labeled ^{64}Cu atoms in polycrystalline p-CdTe films in the temperature range 573÷723 K are shown in Fig. 1.
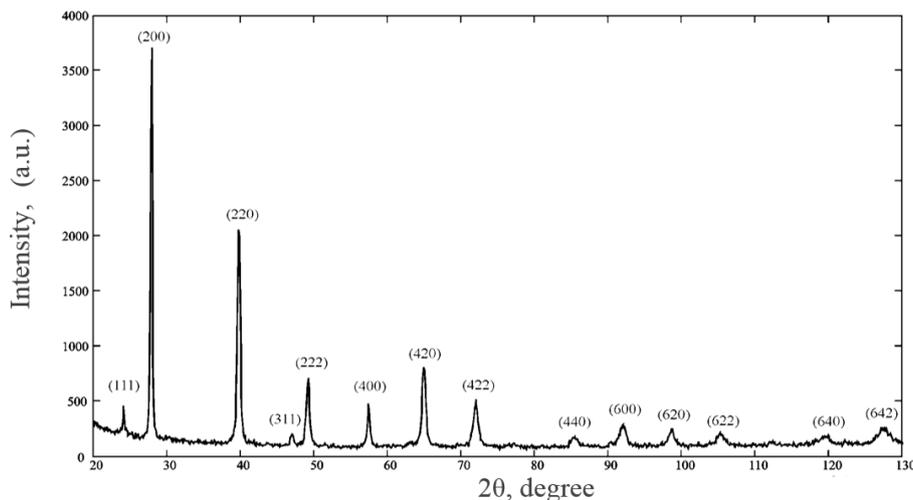
Figure 1. X-ray diffraction pattern of p-CdTe<Pb> films

The study showed that the concentration distributions of copper atoms over the thickness of the p-CdTe film in this temperature range are described by the function erfc (Fig. 2).

The initial p-CdTe films were synthesized by the gas transport method in a quasi-closed volume in a flow of purified hydrogen [7]. Pb atoms were introduced into a growing p-CdTe film during synthesis from a molybdenum substrate, which had a lead layer with a thickness of d = 2 mm deposited by magnetron ion sputtering on the surface of molybdenum [8]. Pb atoms were introduced into the growing p-CdTe film in the course of synthesis from a molybdenum substrate. The content of Pb impurities was controlled by the neutron activation method [9]. The thickness of the grown p-CdTe<Pb> films is $100 \div 120$ μm, the working area $S \geq 1$ cm^2. In such films, grains (crystallites) are oriented in the direction of growth, and their dimensions are not less than the film thickness. The diffusion volume was $\equiv 9$ cm^3. The radioactive isotope ^{64}Cu was used as a diffusant. The sample weight in the ampoule was 2 mg, which corresponded to a diffusant vapor pressure of $0,15 \div 0,2$ atm. Diffusion annealing was carried out in electric furnaces for 4 h, the temperature was maintained with an accuracy of ± 3 K.

The composition of the p-CdTe<Pb> film samples obtained was monitored using a Jeol JSM-6380LV scanning electron microscope equipped with an IN-CAx-sight X-ray spectral microanalysis of the elemental composition. X-ray analysis of p-CdTe<Pb> films was carried out on a DR0N-4-07 diffractometer with a step of 0.01° and exposure at a point of 15 s (Fig. 1). The results of the indexing of the fingerprint comparison of the obtained results with a set of reference X-ray patterns (ASTM) made it possible to determine the composition of the p-CdTe<Pb> films. film composition, as predominantly homogeneous, cubic modification.

In samples, as a result of lead doping in all types of samples, the mobility μ decreases deeper into the thickness and the value of resistivity ρ increases. Table 1 shows the parameters of mobility μ and resistivity ρ of doped CdTe layers during layer-by-layer removal of film thickness d at room temperature.

Table 1. Electrophysical parameters of p-CdTe<Pb> films at a temperature of 300K

№	Film thickness, d, μm	Conductivity type	Hall mobility, μ, cm^2/(V·s)	Resistivity, ρ, Ohm·cm
1.	20	p	78	2.88- 10^4
2.	30	p	72	1.64- 10^4
3.	40	p	66	7.76- 10^4
5.	50	p	62	3.13 • 10^5
6.	60	p	56	3.66 • 10^5
7.	70	p	47	6.38- 10^5
8.	80	p	41	1.86- 10^6

The distribution profile of the concentration of labeled ^{64}Cu atoms in polycrystalline p-CdTe films with a columnar grain structure was recorded using the activation analysis method [9], the CdTe layers doped with ^{64}Cu isotopes were removed by chemical etching with Br:C$_2$H$_2$OH (1:5) bromomethyl. The activity of the removed layers was measured on a gamma spectrometer for 1 h.

RESULTS AND DISCUSSION

The distribution profiles of the concentration of labeled ^{64}Cu atoms in p-CdTe polycrystalline films in the temperature range $573 \div 723$ K are shown in Fig. 2.

The study showed that the concentration distributions of copper atoms over the depth of the p-CdTe film in this temperature range are described by the erfc function (Fig. 3).
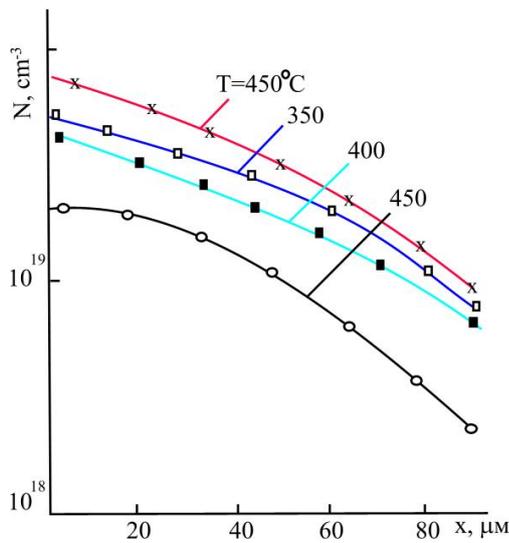
Figure 2. The distribution profiles of the concentration of labeled ^{64}Cu atoms in p-CdTe polycrystalline films
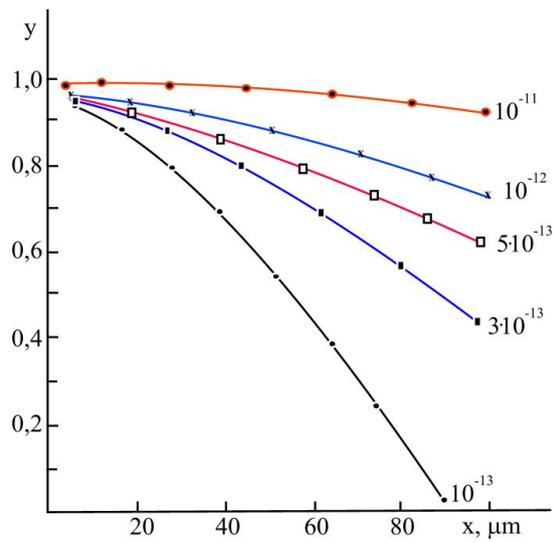

Figure 3. Calculated curves of the concentration distribution of impurity atoms over the thickness of the films at the constructed time t=4 h depending on the diffusion coefficient

In this case, the concentration and diffusion coefficient of copper atoms on temperature are described by an exponential dependence (Fig. 4) and the following analytical expressions

$$N_{Cu}(T) = 7.2 \cdot 10^{20} \exp\left(-\frac{0.32}{kT}\right) cm^{-3}, \tag{1}$$

$$D_{Cu}(T) = 6.7 \cdot 10^{-9} \exp\left(-\frac{0.49}{kT}\right) cm^2 \cdot cm^{-1}, \tag{2}$$

An analysis of the obtained results shows that the pre-exponential tallow factor D_0 and the diffusion activation energy Q correspond to the formula obtained in [10]:

$$Q_0 = A\exp\left(-\frac{Q}{kT_{melt.}}\right), \tag{3}$$

where $T_{melt.}$ is the melting point of the material;

$$A = Pa^2\nu \exp\left(-\frac{\Delta G}{kT}\right), \tag{4}$$

P - dimensionless coefficient depending on the geometry of the unit cell of the crystal and the diffusion mechanism; a is the period of the crystal lattice; ν - Debye frequency; ΔG - Gibbs free energy of activation at a temperature equal to $T_{melt.}$.
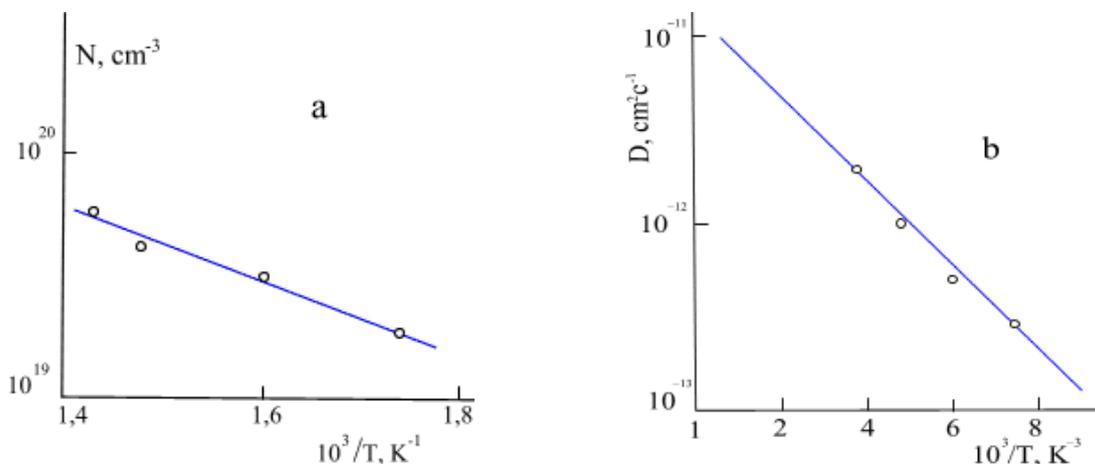

Figure 4. Temperature dependences of the concentration of doped impurity atoms (a), and the diffusion coefficient (b)

The numerical value of the coefficient A is determined by the impurity diffusion mechanism, A = 9.6·10^{-11} cm^2·s^{-1} during the diffusion of copper atoms in large-block p-CdTe films. Approximately for semiconductor materials, according to [11],

$$Q = 2.1 \cdot 10^{-4}[10 + \lg{(D_0, \text{cm}^2/c)}] \cdot T_{\text{melt}}. \tag{5}$$

Substituting the experimental value of the pre-exponential factor $D_0 = 6.7 \cdot 10^{-9}$ cm^2·s^{-1} and T_{melt}(CdTe) = 1315 K into (5), we find Q = 0.5 eV. The value of Q calculated in this way satisfactorily agrees with the value determined directly from the experiment, which confirms the correctness of expression (3) in the calculation of the diffusion parameters of labeled copper atoms in large-block p-CdTe<Pb> films.

The low values of the effective diffusion coefficient of copper atoms $D(T) = 2{,}7 \cdot 10^{-13} \div 1.8 \cdot 10^{-12}$ cm^2·s^{-1} in the temperature range 573÷723 K show that Cu diffuses in p-CdTe polycrystalline films by a complex mechanism. The most probable is diffusion with association [12]. This is also supported by the small value of $A = 9.6 \cdot 10^{-11}$ cm^2·s^{-1} in expression (3). It shows that the associate between the ions of Cu and Pb atoms is stable even at the melting temperature of cadmium telluride.

Assuming that the Pb acceptors are located in the cation sublattice, and the interstitial Cu atoms occupy tetrahedral interstices in equilibrium, we obtain the shortest distance between ions r = 2,8 Å. Assuming that only the Coulomb interaction contributes to the enthalpy of the associate, we estimate ΔH by the formula [13]: $\Delta H = e^2/4\pi\xi\xi0 = 0.50$ eV, where ξ = 10.3 is the low-frequency (static) permittivity of cadmium telluride.

The associate enthalpy value $Cu_i^+ Pb_{Cd}^-$ ΔH = 0.50 eV coincides with the diffusion activation energy Q = 0.49 eV of Cu atoms in p-CdTe<Pb> coarse films. This result shows that all diffusing Cu atoms are completely bound in complexes with ionized Pb_{Cd}^- acceptor centers since $N(Pb_{Cd}^-) \geq Cu_i^+$. Therefore, the activation energy Q of diffusion of copper atoms is almost entirely determined by the binding energy of the $Cu_i^+ Pb_{Cd}^-$. associate. In this case, the rate of diffusion of interstitial copper ions depends on the probability of decay of the associate, and the diffusion process itself consists of the stages of decay and formation of $Cu_i^+ Pb_{Cd}^-$. The electronic structure of the ionized Pb_{Cd}^-. acceptor center probably plays an important role in the formation of such an associate. This, probably, explains the sharp difference between the diffusion coefficient of copper atoms $D(T=160°C) \cong 10^{-8}$ cm^2·s^{-1} and the value of the pre-exponential factor $D_0 = 1.4$ cm^2·s^{-1} obtained in [14,15], from our data.

The difference in the values of D(T) and D_0 is most likely due to the thermodynamic state of the system, the associate. Probably, in [16] an associate $Cu_i^+ Á$, is formed, in which the atomic energy of atoms is high, therefore, in the process of diffusion, when atoms pass into activated states, the change in entropy is large. Since D_0 is related to the entropy ΔS by the following relationship [17].

$$D_0 = avd^2 \exp\left(\frac{\Delta S}{R}\right), \tag{6}$$

then a noticeable change in ΔS leads to large values of D_0.

In the case of the formation of the $Cu_i^+ Pb_{Cd}^-$ associate, the system is apparently in a more stable position, closer to the equilibrium state. In such a material, diffusible Cu atoms rapidly dissolve; are mixed, and without much effort form associates of the $Cu_i^+ Pb_{Cd}^-$ type, which is facilitated by the Coulomb interaction between the Cu_i^+ and Pb_{Cd}^- ions. In this process, the change in the vibrational entropy can be neglected; therefore, the value of D_0 is small, which is observed in the experiment (see (6)).

Let us now consider the influence of the Pb content in p-CdTe on the diffusion parameters of Cu atoms. Pb concentration in films is within $N_{Pb}^0 = 10^{18} \div 10^{20}$ cm^{-3}. Studies have established the dependence of the diffusion coefficient of Cu atoms in p-CdTe on the concentration N_{Cd}^0, which is clearly manifested at $N_{Pb}^0 \leq 10^{19}$ cm^{-3}. For example, when the Pb concentration changes by two orders of magnitude, i.e., from 10^{20} to 10^{18} cm^{-3}, the diffusion coefficient increases from $2 \cdot 10^{-13}$ to $1{,}8 \cdot 10^{-9}$ cm^2·s^{-1}, almost by four orders of magnitude, at 573 K. Moreover, the increase in DT is mainly provided by increasing D_0, since the activation energy of diffusion of copper atoms in this case changes insignificantly, only by 0,07÷0,08 eV, and becomes Q = 0,55÷0,56 eV.

The presented experimental results confirm the correctness of the proposed model of diffusion of Cu atoms, i.e., diffusion mechanism with the association $Cu_i^+ Pb_{Cd}^-$. Indeed, at concentrations $N_{Pb}^0 \leq 5 \cdot 10^{18}$ cm^{-3} in p-CdTe films, extra Cu atoms appear that are not bound in complexes, which begin to diffuse through other channels, among which dissociative diffusion is most likely, since free vacancies of cadmium atoms V_{Cd} are formed.

Recombination parameters were determined on p-CdTe<Pb> samples with different Pb content: diffusion length L_p, minority carrier lifetime τ_p, and surface recombination rate S.

To measure L_P in CdTe, a heterostructure with an upper optical window from a wide-gap semiconductor CdS was formed by vacuum deposition in a quasi-closed volume with an area of 1 cm^2 according to the technology described in [18]. The front contact, from the side of which illumination is provided, is made of indium deposited in a vacuum of ~10^{-5} Torr in the form of a comb. In this case, the width of the contact strip was 0.8 mm, and the distance between the strips was ~2 mm. The back contact was a molybdenum substrate. The deposited indium contact on CdS was connected to the heterojunction in the blocking direction.

L_p was measured by the diffusion method described in [19], as well as by the method [20], including the measurement of the photocurrent depending on the absorption depth α^{-1} at a constant intensity F in the region of the intrinsic absorption spectrum. Both methods give almost identical results.

At high concentrations of Pb (N = $5\cdot10^{19}\div10^{20}$ cm^{-3}) in p-CdTe<Pb>, the electron diffusion length reaches its maximum value, $\cong$60 μm. With a decrease in N_{Pb}, the value of L_p also decreases at $N_{Pb} = 10^{18}$ cm^{-3}, $L_p \cong$14 μm.

From the short-wavelength region of the photocurrent spectrum I_{ph}(hv) (hv$\geq$2 eV), the surface recombination rate S was determined for holes at the p-n junction boundary adjacent to the CdS wide-gap filter. The results show that S depends on the Pb content in CdTe, and it increases with an increase in the Pb content, and S$\cong10^5$ cm·s^{-1} is maximum at $N_{Pb}= 5\cdot10^{19}$ cm^{-3}. Further, as N_{Pb} increases to 10^{20} cm^{-3}, the surface recombination rate remains constant. Note that S for holes has the lowest value of $\cong 7\cdot10^3$ cm·s^{-1} in the absence of impurities of Pb atoms in the samples under study.

The lifetime τ_p of minority current carriers was also determined on p-CdTe<Pb> samples by the phase difference method. At the same time, a certain correlation between τ_p and N_{Pb} was revealed. It has been established that τn reaches its maximum value $\cong 2\cdot10^{-6}$ s at $N_{Pb}\cong10^{20}$ cm^{-3}, and at $N_{Pb}\cong10^{18}$ cm^{-3} $\tau_p=10^{-7}$ s.

The mobility of minority carriers μ_n was calculated using the experimental values of L_p and τ_p using the known formula $L_p = (kT/q\mu_n\tau_n)^{1/2}$. Found $\mu_p = 690$ cm^2/V·s at $L_p = 60$ μm, $\tau_p = 2\cdot10^{-6}$ s and $N_{Pb} = 5\cdot10^{19}\div10^{20}$ cm^{-3}. μ_n remains practically constant in p-CdTe<Pb> samples with different contents of Pb impurities, so $\mu_n \cong 680$ cm^2/V s at $L_n =14$ μm, $\tau_n =10^{-7}$ s and $N_{Pb}\cong5\cdot10^{18}$ cm^{-3}.

Thus, the presence of Pb atoms in large-block p-CdTe films leads to the formation of recombination centers with sharply different electron and hole capture cross sections, the ratio of which is directly dependent on the N_{Pb} concentration. These results do not contradict the data obtained in [21].

The diffusion and recombination parameters of control samples of large-block p-CdTe films with $\rho \cong 10^3$–10^4 Ω·cm, in which there are no Pb atoms, have also been studied. The control p-CdTe films and the p-CdTe<Pb> films were synthesized in identical technological modes, the only difference being that the control p-CdTe films were grown on mica substrates, while the p-CdTe<Pb> films were grown on substrates with an impurity of Pb atoms, of which, during the synthesis, Pb atoms were doped into growing films.

As for the recombination parameters, they are low in p-CdTe control samples and correspond to the literature data, for example, $L_p = 0{,}5\div0{,}6$ mkm and $\tau_p = 10^{-8} \div 5\cdot10^{-9}$ s coincides with the results [20].

CONCLUSIONS

Thus, by introducing impurities of Pb atoms, it is possible, firstly, to control the diffusion rate of copper Cu atoms in p-CdTe films, this opens up wide opportunities for using thin-film solar cells with Cu_2Te-CdTe and Cu_2S-CdS (ZnCdS) structures in terrestrial conditions. Secondly, the possibility of controlling the charge states of recombination centers in large-block p-CdTe films by introducing atomic impurities. Pb makes it possible to obtain semiconductor base materials with desired properties, i.e., given values of the microparameters Ln and τn, which is extremely important in materials science, for the microelectronic and semiconductor industries, especially for the creation of thin-film solar cells.

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ВПЛИВ ДИФУЗІЇ АТОМІВ МІДІ В ПОЛІКРИСТАЛІЧНИХ ПЛІВКАХ CdTe, ЛЕГОВАНИХ АТОМАМИ Pb

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Досліджено процес дифузії мічених атомів міді ^{61}Cu в крупноблокових плівках p-CdTe<Pb> зі стовпчастою зернистою структурою. Плівка CdTe<Pb> є напівпровідником p-типу, де збільшення концентрації Pb у складі плівок CdTe збільшує питомий опір ρ структури. При зміні концентрації Pb в CdTe від 10^{18} до $5 \cdot 10^{19}$ см$^{-3}$ концентрація дірок зменшується більш ніж на 3 порядки при постійній глибині робочого рівня $E_V + (0,4 \pm 0,02)$ еВ. Це може свідчити про те, що концентрація акцепторних дефектів, які утворюються в плівках внаслідок самокомпенсації при легуванні донором Pb_{Cd}, перевищує кількість останнього. Електричні вимірювання методом Холла проводили при постійному струмі та температурі 300 К. У результаті підвищення температури плівок на підкладці Mo-p-CdTe<Pb> під час відпалу впливає на електричний параметр заряду. мобільність носія μ, вона значно зменшується. Рентгеноструктурний аналіз показав, що на дифрактограмах зразків плівок p-CdTe<Pb> усі наявні рефлекси відповідають фазі CdTe і до x = 0,08 не містять рефлексів домішкових фаз і мають кубічну модифікацію. За результатами розрахунку встановлено, що низькі значення коефіцієнта дифузії атомів Cu зумовлені утворенням асоціатів типу A $Cu_i^+ Pb_{Cd}^-$, які знаходяться в прямій залежності від концентрації Pb_{Cd}. Дифузійна довжина Ln і час життя τ_n неосновних носіїв струму у великоблочних плівках телуриду кадмію p-типу, якими також можна керувати шляхом введення атомів свинцю в телурид кадмію.

Ключові слова: *дифузія; асоційований; час життя; плівка; акцепторний центр; радіоактивний ізотоп; розподіл; рухливість, питомий опір; коефіцієнт дифузії; ентальпія*