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Spectral Distribution, Optical and Thermal Quenching of Longitudinal Photoconductivity in CdSe/SnO$_2$ Films

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Abstract

A method has been developed for producing a photosensitive film made of CdSe, including thermal vacuum deposition of CdSe on a substrate with a transparent layer of SnO$_2$ and thermal treatment of the freshly prepared film in a quasi-closed chamber in air in the presence of CdCl$_2$ or CuCl$_2$ vapors, ensuring uniform diffusion of the sensitizing substances. From the analysis of the longitudinal photoconductivity spectrum it is shown that freshly prepared cadmium selenide films have local levels with the activation energy: $E_V + 0.63$; $E_V + 0.8$; $E_V + 0.925$; $E_C - 0.55$ and $E_C - 0.41$ eV. After heat treatment in air in the presence of CuCl$_2$ new photosensitive centers are formed: complexes of the chlorine atom with a cadmium vacancy with the activation energy of ($E_V + 0.42 \pm 0.05$) eV, cadmium vacancies $-E_V + (0.6 \pm 0.05)$, as well as a fast recombination center $E_V - (0.95 \pm 0.05)$ eV.

Keywords

Thin Films, Photodetectors, Longitudinal Photoconductivity, Transparent Contact, Cadmium Selenide, Tin Dioxide, Cathode Sputtering, Thermal Vacuum Deposition

1. Introduction

Among the wide-bandgap II-VI semiconductor compounds, cadmium selenide (CdSe) is one of the most sensitive in the visible and near-infrared regions of the spectrum. Recent advancements in the technology of producing polycrystalline CdSe films functioning in the longitudinal photoconductivity mode have led to the development of various optoelectronic devices for microelectronics, acoustoelectronics, automation, and nonlinear optics [1]-[7]. In these devices, the film substructure is formed in the direction perpendicular to the substrate, as is the current in longitudinal photosensitive structures. One of the most commonly used methods for producing such CdSe films is vacuum deposition at temperatures ranging from 200˚C to 400˚C. It is well known [6]-[9] that freshly prepared films typically exhibit low photosensitivity. An effective method for tuning the photoelectric properties of cadmium selenide films is thermal treatment (annealing)—in vacuum, in air, or in the presence of vapors containing various chlorides, among others. During the annealing process, not only do the electrophysical properties change, but also the phase composition, crystal lattice parameters, preferred crystallite orientation, block sizes, and internal microstrains. However, the influence of structural imperfections introduced by annealing on the photoelectric properties in the longitudinal photoconductivity mode has not been fully elucidated. The available literature data on the substructure, composition, and photoelectric properties of CdSe films remain insufficient. Therefore, the aim of this work is to study the influence of annealing processes in different environments on the photosensitivity of freshly prepared CdSe films operating in the longitudinal photoconductivity mode with stable electrophysical characteristics. It is shown that the electrical, photoelectric, and spectral properties of cadmium selenide films are significantly affected by localized levels within the forbidden bandgap. The crystal lattice defects responsible for these levels act as trapping centers for majority and minority carriers, as well as recombination centers. Thus, a more detailed study of the activation energies of localized levels in CdSe, depending on the preparation and annealing conditions in various environments, allows for a deeper understanding of the physical processes occurring in the fabricated films. By analyzing the spectral distribution curves, optical and thermal quenching of photoconductivity, as well as thermally stimulated current (TSC) measurements, the activation energies of sensitizing centers and trapping levels for electrons and holes were determined for films with longitudinal photoconductivity fabricated under various technological conditions.

2. Experimental Results and Discussion

A method has been developed for producing photosensitive CdSe films, which includes thermal vacuum deposition of CdSe with a thickness of 3 - 5 μm onto a glass substrate pre-coated with a transparent SnO_2 layer, followed by thermal treatment of the freshly prepared film in a quasi-closed chamber in air in the presence of $CdCl_2$ or $CuCl_2$ vapors. This ensures uniform diffusion of sensitizing agents [10] [11]. By varying the deposition time (5 - 50 minutes) and the amount of antimony introduced as an impurity, SnO_2 films were obtained with thicknesses ranging from 0.1 to 1 μm, resistances of 150 - 200 Ohms, and transmittance between 70% - 85%, which meets the requirements for transparent contacts used in longitudinal photoconductive devices.

Studies of the spectral distribution of photoconductivity (σ_{ph}) showed that the initial films, prepared at different substrate temperatures, have similar localized level spectra. The spectral dependence curves for films obtained at a substrate temperature of 350˚C and measured at 300 K are shown in **Figure 1**. Based on comparison of experimental results (curve 1) with theoretical curves (curve 2) calculated using the Lukovsky formula [12], and from graphical analysis, it was found that freshly prepared films contain localized levels with activation energies: $E_V + 0.63$ eV, $E_V + 0.8$ eV, $E_V + 0.925$ eV, $E_C - 0.55$ eV, and $E_C - 0.41$ eV. In all freshly prepared films deposited at different substrate temperatures, small steps are observed in the spectral curves near photon energies of ~0.6 eV and ~1.2 eV. Graphical analysis showed that these steps are due to localized levels at $(E_V + 0.63)$ eV and $(E_C - 0.41)$ eV.

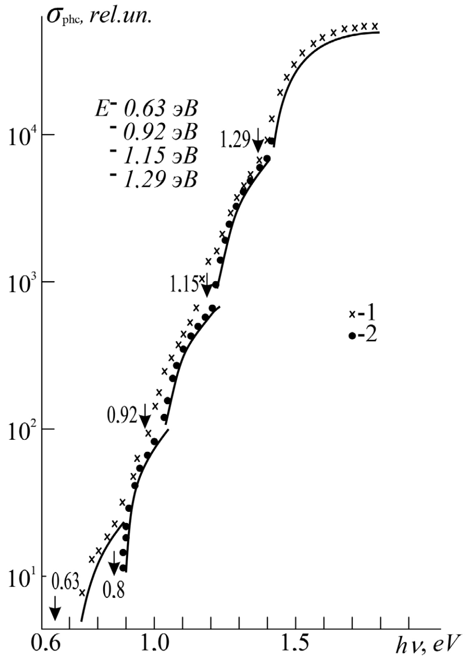

Figure 1. Spectral dependence σ_{phc} of CdSe films obtained at $T_s = 350$˚C. Curves 1—experiment, solid-theory, 2—difference between experiment and theory.

To increase the photosensitivity, the freshly prepared films were subjected to thermal treatment in various environments. Even after short-term annealing in air in the presence of $CdCl_2$ vapors, an additional localized level near the bottom of the conduction band ($E_C - 0.24$ eV) becomes evident in the spectral photoconductivity distribution (**Figure 2**), and a level at $(E_V + 0.43)$ eV near the top of the valence band is observed in the optical quenching curves (**Figure 3**).

Points labeled "2" in **Figure 1** and **Figure 2** were obtained by subtracting the theoretical curves from experimental data. Arrows on the figures indicate the optical ionization energies used in the theoretical model (solid lines). With increasing annealing time, the spectrum of localized levels changes insignificantly (see **Figures 4-6**). In the long-wavelength region of the spectrum ($h\nu < 1$ eV), the behavior of the photoconductivity spectra is governed by localized levels located in both the upper and lower halves of the forbidden bandgap. Increased concentration of such levels results in higher photosensitivity in this spectral region. Levels responsible for this behavior were confirmed by infrared quenching bands. Based on the spectral curves, electron trapping levels were determined as $E_C - (0.19 \pm 0.03)$ eV and $E_C - (0.42 \pm 0.02)$ eV (see **Figure 4** and **Figure 5**).

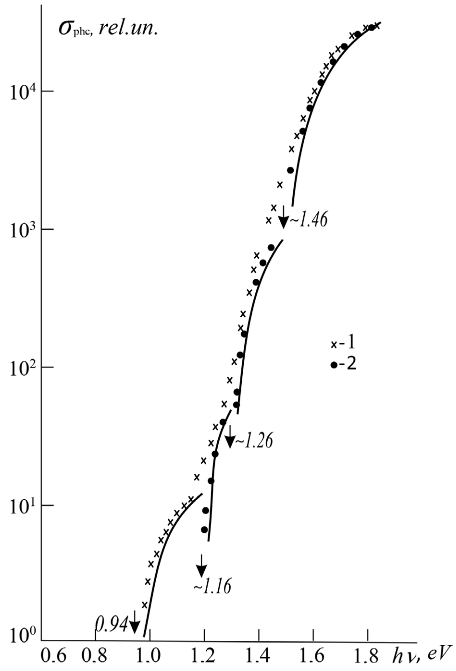

Figure 2. Spectral dependence σ_{phc} of CdSe films heat-treated in air in the presence of $CdCl_2$ vapor for 4 min.

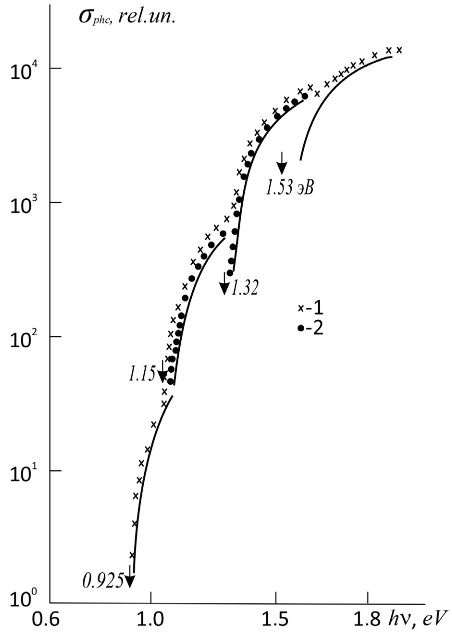

Figure 3. Optical quenching curves of photoconductivity for CdSe films, annealed in air in the presence of $CdCl_2$ ($t = 4$ min).

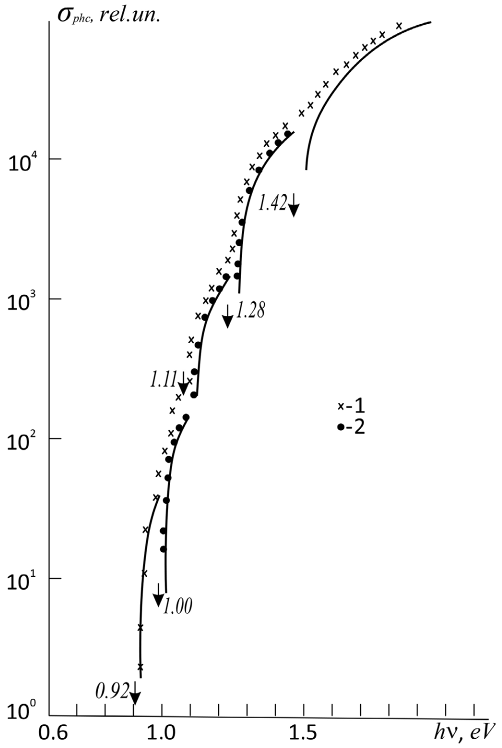

Figure 4. Spectral dependence σ_{phc} of CdSe films heat-treated in air in the presence of $CdCl_2$ vapor for 6 min.

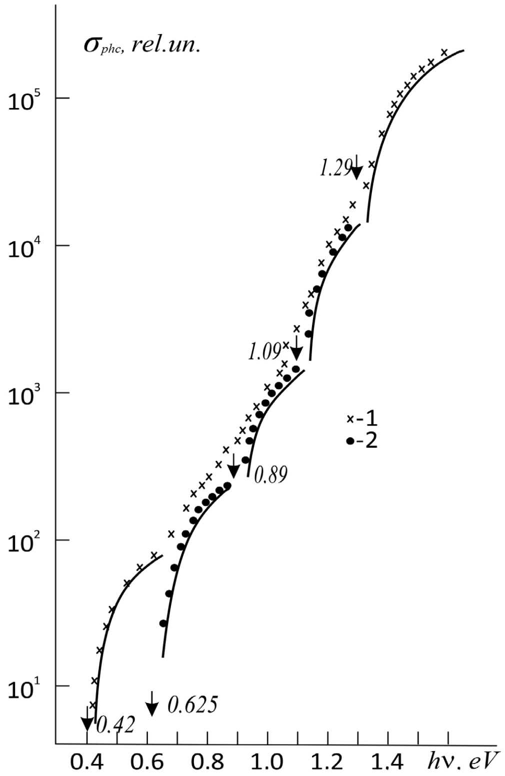

Figure 5. Spectral dependence σ_{phc} of CdSe films heat-treated in air in the presence of $CdCl_2$ vapor for 8 min.

As is known, the possible types of simple intrinsic defects in CdSe include interstitial cadmium and selenium atoms (Cd_i, Se_i) and their vacancies (V_{Cd}, V_{Se}). At temperatures close to room temperature, significant concentrations of defect complexes and impurity-defect complexes may form. Prior studies [13] [14] have shown that donor levels can be singly or doubly charged, and the same applies to acceptor levels [15]. Thus, even basic intrinsic defects in CdSe can give rise to eight distinct types of localized levels.

In the freshly prepared films studied here, the localized levels are evidently associated with intrinsic defects of the CdSe crystal, since highly pure cadmium selenide was used for deposition. For example, the donor level ($E_C - 0.41$) eV is attributed to complexes of doubly charged selenium vacancies with interstitial cadmium or selenium atoms, while the level ($E_C - 0.55$) eV is due to doubly charged selenium vacancies, consistent with the literature [15]. The acceptor levels ($E_V + 0.95$) and ($E_V + 0.63$) eV are known to be associated with cadmium vacancies [16].

Thermal treatment in various environments alters the concentration of these defects, and thus the concentration of the associated localized levels. It also leads to the formation of new levels associated with dopant impurities. An impurity atom may occupy a lattice site, an interstitial site, or reside in the intercrystallite regions. Its location is mainly determined by the size of its ionic radius. If the radius is smaller than that of the film components, it will likely occupy a lattice site of the component whose ionic radius is not smaller than the impurity's. Whether the impurity acts as a donor or acceptor depends on the number of valence electrons relative to the substituted atom. If it has more, it behaves as a donor; if fewer, as an acceptor.

In the early stages of thermal treatment in air with $CdCl_2$ vapors, dissociated chlorine atoms diffuse into the volume of the crystallites and substitute selenium or fill selenium vacancies. This leads to the formation of a shallow donor level. Some chlorine atoms also react with cadmium atoms and cadmium vacancies, resulting in the formation of a compensating acceptor level ($E_V + 0.42$ eV), associated with cadmium vacancy-chlorine $\left(V_{Cd}^{--}D_{Se}^{+}\right)$ complexes (**Figure 6**). The appearance of photosensitive centers with this activation energy ($E_V + 0.42$ eV) contributes to an increase in the photosensitivity (**Figure 4**). Increasing the annealing time to 6 - 8 minutes leads to a higher concentration of cadmium vacancies and cadmium-chlorine complexes, *i.e.*, a higher concentration of sensitizing centers ($E_V + 0.42$ eV and $E_V + 0.6 \pm 0.05$ eV), and consequently further enhances the photosensitivity.

In the case of annealing in air with $CuCl_2$ vapors, dissociated copper and chlorine atoms diffuse into the crystallites and intercrystallite regions of the film. Since the covalent radius of copper is smaller than that of cadmium, the copper atom substitutes cadmium in the crystal lattice. Chlorine atoms facilitate this process by reacting with cadmium and increasing the concentration of cadmium vacancies, making it easier for copper atoms to occupy these vacancies. This leads to the formation of a compensating acceptor level ($E_V + 0.66$ eV) (**Figure 7**), and an increase in its concentration enhances the photosensitivity.

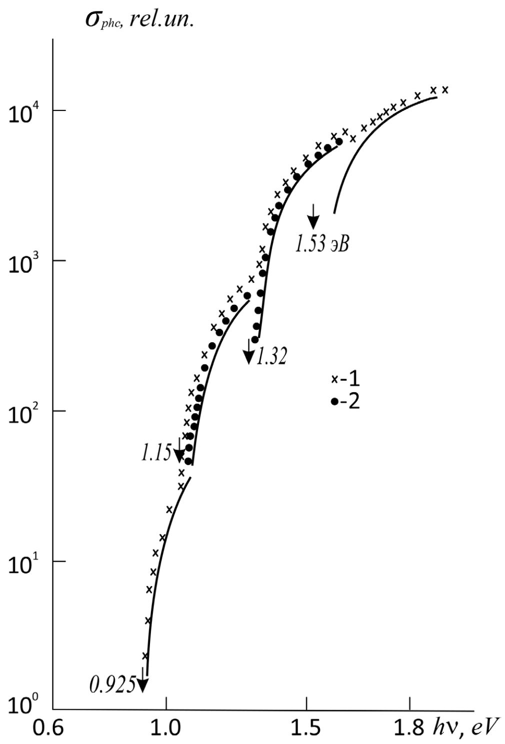

Figure 6. Spectral dependence σ_{phc} of CdSe films heat-treated in air in the presence of $CdCl_2$ vapor for 12 min.

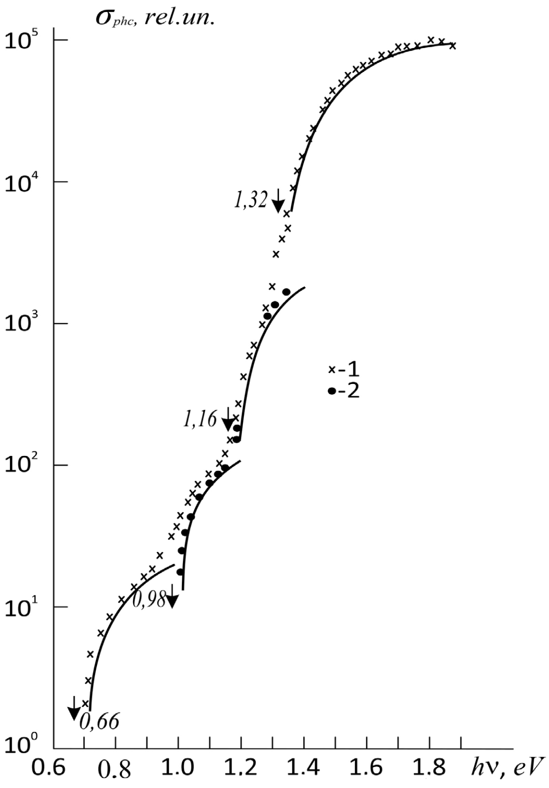

Figure 7. Spectral dependence σ_{phc} of CdSe films heat-treated in air in the presence of $CdCl_2$ vapor for 4 min (T_s = 350˚C).

Thus, during photosensitization by thermal treatment in air with $CuCl_2$, chlorine atoms play the dominant role by forming and increasing the concentration of photosensitive centers. When a chlorine atom forms a complex with a cadmium vacancy, a level with an activation energy of (E_V + 0.42) eV appears. When a cadmium vacancy forms independently, a level at E_V + (0.6 ± 0.06) eV is formed. In addition, a fast recombination center at E_V – (0.95 ± 0.05) eV may arise [17]-[19]. The formation of selenium vacancies due to chlorine incorporation and its reaction with cadmium leads to the appearance of a trapping level at E_C – (0.19 ± 0.03) eV. In the case of $CuCl_2$ treatment, both copper and chlorine atoms contribute to the formation of compensating acceptor levels and an increase in donor center concentration.

The activation energy of sensitizing centers was determined from photoconductivity thermal quenching (TQ) curves. It was found that in CdSe films fabricated under various conditions, pronounced TQ effects occur only after a certain threshold value of photoconductivity is achieved. TQ curves for CdSe films annealed in air in the presence of $CdCl_2$ at different illumination levels are shown in **Figure 8**. As seen in the figure, as the temperature increases up to about 250 K, the conductivity does not change significantly. However, upon further heating, the photoconductivity sharply decreases, indicating quenching. With increasing illumination intensity, the temperature at which quenching begins also increases.

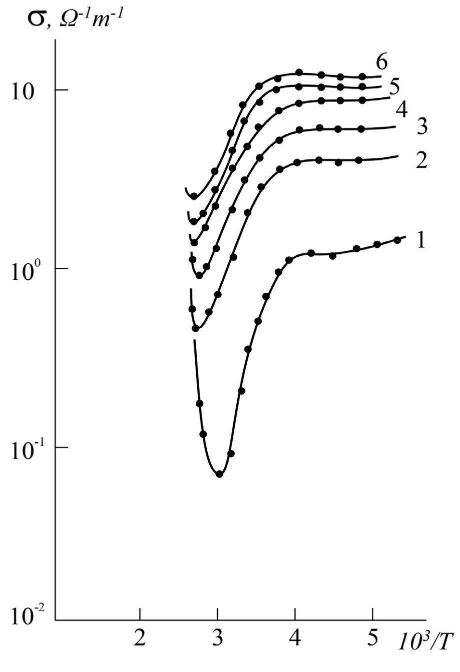

Figure 8. TQ curves for a CdSe film obtained by thermal treatment in air in the presence of $CdCl_2$ at different illumination intensities *L*, *lx*: 1-10; 2-50; 3-100; 3-200; 4-300; 5-400; 6-500.

This behavior can be explained as follows. Under constant light intensity at low temperatures, the probability of hole capture at *r*-centers exceeds their thermal excitation probability into the valence band. As a result, *r*-centers become preferentially occupied by holes, and *s*-centers by electrons, since the capture cross sections satisfy the condition S_{pr}, S_{ps}, $S_{ns} \gg S_{nr}$ (where S_{nr} and S_{pr} are the capture cross sections for electrons and holes by *r*-centers, respectively, and S_{ns} and S_{ps} for *s*-centers). Under these conditions, recombination of electrons occurs mainly via *r*-centers. As the temperature increases under constant illumination, the demarcation level for holes shifts upward and intersects the *s*-center level, making them effective trapping centers for holes. This causes holes to relocalize to *s*-centers. Since $S_{nr} \ll S_{ns}$, the recombination rate of free carriers increases, which reduces the lifetime of electrons in the conduction band—this results in thermal quenching of photoconductivity.

The thermal ionization energy E_r^T was determined using Bube's method [16], where the transition from high to low sensitivity with increasing temperature under constant light intensity is described by the equation:

$$\ln n = \ln\left(\frac{N_V S_{pr}}{S_{nr}}\right) - \frac{E_r^T}{kT}, \tag{1}$$

where N_V—is the effective density of states in the valence band, $\frac{S_{pr}}{S_{nr}}$ —is the ratio of hole to electron capture cross sections at the recombination center, *k*—is Boltzmann's constant, *T*-is absolute temperature, and *n*-is the concentration of free carriers.

To determine the sensitivity parameters using the method [16], sections of the curves were used where the TQ process just begins or ends. The activation energy of the r-center was determined from the slope of curves $\ln\sigma_p = f\left(\frac{1}{T}\right)$ constructed at the points where the TQ begins or ends, taken at different illumination intensities.

Based on the experimental results, it was established that the value of E_r^T for films produced by heat treatment in air in the presence of $CdCl_2$ was equal to 0.48 eV. From the point of intersection of the curve $\ln\sigma_p\left(\frac{10^3}{T}\right)$ with the angular coefficient $-\frac{E_r}{k}$, constructed according to the points where the TQ begins, with the ordinate axis $\frac{10^3}{T} = 0$, the values of $\ln N_V S_{pr} - \ln S_{nr}$ were found. At $N_V = 10^{19}\ \text{cm}^{-3}$, the ratios of the capture cross-sections of electrons and holes were determined. They turned out to be equal to $\sim 10^5 \div 10^6$.

In order to obtain information about the parameters of electron traps, TSC in films were studied. Measurements were carried out in a vacuum of 10^{-3} mm Hg with a linear increase in temperature at a rate of $\beta = 0.1$ deg/s.

It is known that the TSC is caused by the thermal release of free carriers captured in traps into the conduction zone. The depth of the traps was determined by the method [16]

$$\Delta E_t = kT_m \ln \frac{e\mu N_c}{\sigma_m} \tag{2}$$

and the method [13] [14], which is based on the analysis of the initial sections of the TSC increase, where the current increases exponentially with temperature as follows:

$$I \sim \exp\left(-\frac{\Delta E}{kT}\right) \tag{3}$$

and the conductivity:

$$\sigma = g\mu_n n_{t_0} \tau_n S_{nt} \vartheta_t N_c \exp\left(-\frac{\Delta E}{kT}\right). \tag{4}$$

Then for the electron concentration we can write

$$n = \gamma Q_c \exp\left(-\frac{\Delta E}{kT}\right) \tag{5}$$

where, $\gamma = S_{nt}/S_{nr}$, g—is the electron charge, Q—is the stat. factor of the conduction band, S_{nr}—is the cross section of electron capture by the r-center, S_{nt}—is the cross section of electron capture by the t-center of attachment.

With further increase in temperature, the decreasing section of the TSC is described by the expression

$$n' = \frac{\beta E_{rt}}{\vartheta_n S_{nr} kT^2}, \tag{6}$$

where β—is the heating rate, ϑ_n—is the thermal velocity of the charge carriers. The cross section of electron capture by traps was determined by the formula

$$S_{nt} = \frac{I_o \beta}{Q_{TSC} \vartheta N_c}, \tag{7}$$

where I_o—is the value of current extrapolated to the exponential initial section of the TSC at $(1/T) = 0$, Q_{tsc}—is the area under the maximum of the TSC.

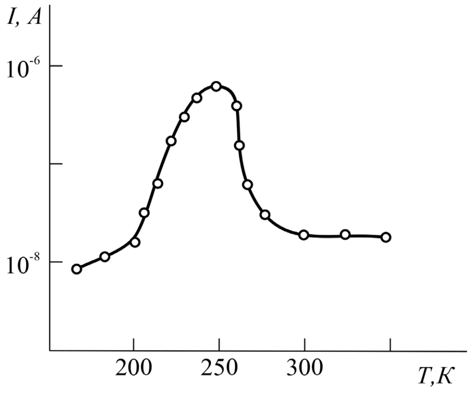

Figure 9. TSC curves for a CdSe film obtained by thermal treatment in air in the presence of $CdCl_2$ vapor.

The concentration of traps N_t was determined using the expression for the area under the TST curve [16]

$$Q_{TSC} = \beta^{-1}\int_{T_1}^{T_2} \sigma \mathrm{d}T = g\mu_n \varepsilon_n N_t . \tag{8}$$

Figure 9 shows the TSC curves for films annealed in air in the presence of $CdCl_2$. It was found that the ionization energy of the traps for the films under study lies in the range of (0.21 ÷ 0.41) eV. Their concentration is $10^{13} \div 10^{14}$ cm^3, and the electron capture cross section by traps S_{nt} is $10^{-18} \div 10^{-19}$ cm^2. As we can see, the depths of the sensing centers and traps, determined from optical and thermal measurements, coincide.

3. Conclusions

1) A method has been developed for fabricating polycrystalline CdSe films with longitudinal photoconductivity. The method includes thermal vacuum deposition of CdSe onto a glass substrate pre-coated with a transparent SnO_2 layer, followed by thermal treatment of the freshly prepared film in a quasi-closed chamber in air in the presence of $CdCl_2$ or $CuCl_2$ vapors. This process ensures uniform diffusion of photosensitizing impurities into the film structure.

2) Based on the analysis of spectral distribution curves, optical and thermal quenching of photoconductivity, as well as thermally stimulated current (TSC) measurements, the activation energies of photosensitizing centers and trapping levels for electrons and holes were determined in $CdSe/SnO_2$ films fabricated under different technological conditions.

3) Freshly prepared films obtained at a substrate temperature of 350˚C exhibit localized energy levels with activation energies: $E_V + 0.63$ eV; $E_V + 0.8$ eV; $E_V +$ 0.925 eV; $E_C - 0.55$ eV, and $E_C - 0.41$ eV. During photosensitization by thermal treatment of CdSe films in air in the presence of $CuCl_2$, chlorine atoms play a dominant role in forming and increasing the concentration of photosensitive centers. When a chlorine atom forms a complex with a cadmium vacancy, a center with activation energy ($E_V + 0.42$ eV) appears. Formation of cadmium vacancies leads to a level at $E_V + (0.6 \pm 0.06)$ eV. Additionally, a fast recombination center form at $E_V - (0.95 \pm 0.05)$ eV.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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