








INVESTIGATION OF THE MICROSTRUCTURE OF PHOTSENSITIVE *CdSe* AND *CdSe:Cd,Cl* THIN FILMS

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The substructure of freshly prepared photosensitive *CdSe* and doped *CdSe:Cd,Cl* thin films were investigated with respect to the influence of substrate temperature T_s and heat-treatment time in air in the presence of $CdCl_2$ vapor. The results of electron diffraction and electron microscopy studies for films prepared under different technological conditions are also presented. It was established that the texture axis of the as-prepared *CdSe* films is perpendicular to the substrate plane. As T_s increases from 250 to 400°C, the texture-axis dispersion angle, the fraction of the hexagonal phase, the crystallite size, and the coherent X-ray scattering region size D_{CSR} increase. After annealing in air in the presence of $CdCl_2$ vapor at 300°C, films prepared at $T_s=250^\circ\text{C}$ exhibit reorientation of crystallites from the $(111)_c + (0002)_h$ plane, which is parallel to the substrate plane, to the $(10\bar{1}3)$ orientation through the $(311)_c + (11\bar{1}2)_h$ planes. This reorientation is accompanied by an increase in crystallite size and D_{CSR} , and by a decrease in the lattice parameter and the minimum dislocation density.

Keywords: Photosensitive film; *CdSe*; *CdSe:Cd,Cl*; Longitudinal photoconductivity; Polycrystal; Texture; Substrate temperature; Heat treatment; Sensitization kinetics; Coherent X-ray scattering size

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INTRODUCTION

Polycrystalline cadmium selenide (*CdSe*) thin films have attracted sustained research interest due to their wide applications in microelectronics and photonics, including photoconductors, light-emitting devices, visible and gamma-ray detectors, and memory elements [1–5]. The efficiency of *CdSe*-based photovoltaic devices is determined by key electrophysical and photoelectric parameters, such as charge carrier mobility and lifetime, defect types and concentrations, and photosensitivity. The structural, optical, and electrical properties of *CdSe* thin films have also been widely discussed in the literature for different preparation methods and application fields [6]. Numerous studies have shown that these parameters are highly sensitive to the microstructural state of the films, including grain size, porosity, crystallographic texture, and phase composition, which are significantly dependent on additional thermal treatment. In addition, the structural, optical, and electrical properties of *CdSe* thin films are influenced by film thickness [7]. In particular, process conditions greatly influence the crystal structure of grains and significantly affect charge transport processes. Depending on the fabrication conditions, polycrystalline *CdSe* thin films can crystallize in cubic (sphalerite) or hexagonal (wurtzite) forms, or exhibit the coexistence of mixed phases [3, 10-12]. The presence and evolution of these structural modifications play a decisive role in the formation of the crystallographic texture and substructure, thereby determining the microstructural development and functional characteristics of *CdSe*-based films.

Despite extensive investigations of the electrical and optical properties of *CdSe* thin films, quantitative studies of their substructure parameters performed using a combined set of complementary structural techniques remain relatively limited. In particular, a systematic correlation between substrate temperature, post-deposition thermal treatment conditions, and the evolution of substructure characteristics has not yet been sufficiently clarified. This limitation is mainly associated with the complexity of linking volume-sensitive X-ray diffraction data with surface-sensitive electron-microscopic observations, especially in textured and polyphase films.

In this context, the aim of the present work is to comprehensively investigate the substructure of photosensitive *CdSe* and *CdSe:Cd,Cl* thin films as a function of substrate temperature and annealing time in air in the presence of $CdCl_2$ vapor. The study is based on a combined application of X-ray diffraction, electron diffraction, and electron microscopy, enabling a correlated analysis of crystallographic texture, phase composition, coherent scattering region size, lattice parameters, and defect-related characteristics.

The novelty of this work lies in revealing the systematic correlation of substructure parameters with key physico-technological factors over a wide range of deposition and annealing conditions. Special attention is paid to the evolution of crystallographic texture, phase transformations between cubic and hexagonal modifications, and the mechanisms of crystallite reorientation during thermal treatment. The obtained results provide deeper insight into the structural perfection

of CdSe-based thin films and establish a reliable structural basis for optimizing their photoelectric and electrophysical properties.

TECHNOLOGY AND MEASUREMENT METHODS

Thin CdSe and CdSe: Cd, Cl films with an area of 5×15 mm² were produced by vacuum thermal evaporation on standard glass substrates with a predeposited transparent SnO₂ conductive layer. Deposition was performed at a residual gas pressure of approximately 10⁻³ Pa. To study the effect of substrate temperature on the structural properties of the films, the substrate temperature (*T_s*) during deposition was varied from 250°C to 400°C. The films were deposited for a time sufficient to achieve the desired thickness. The deposition time and substrate temperature were carefully controlled to achieve the desired crystal structure and phase composition.

Once the films were deposited, they underwent a post-deposition thermal treatment process in air, where CdCl₂ vapor was introduced. The annealing temperature was set to 300°C, and the annealing time was varied to evaluate the effects of thermal treatment on the microstructure and the phase composition of the films. The CdCl₂ vapor was used to promote the formation of the desired crystalline phases and improve the structural quality of the films. This treatment plays a crucial role in the enhancement of film crystallinity and the reduction of defects.

For structural characterization, X-ray diffraction (XRD) was utilized to analyze the crystallographic texture and phase composition of the films. The XRD measurements were conducted at room temperature, with diffraction angles (2θ) ranging from 20° to 80°. The XRD technique enabled the identification of the crystalline phases present in the films, as well as the determination of the crystalline quality. Electron diffraction (ED) was also used to further investigate the crystallographic texture and coherence of the films.

To examine the surface morphology and grain structure of the films, scanning electron microscopy (SEM) was employed. SEM images were taken to assess the surface features and to measure the average grain size. The grain orientation and distribution were analyzed based on SEM micrographs. The grain size was calculated using the Scherrer equation, which is based on the full width at half maximum (FWHM) of the XRD peaks. This allowed for the estimation of the average crystallite size in the films.

Additionally, the texture coefficients were calculated to evaluate the preferred orientation of the grains in the films. The texture coefficients provide insights into the crystallographic alignment and orientation of the grains relative to the substrate. The SEM and XRD data were used in combination to obtain a comprehensive understanding of the films' microstructural properties.

RESULTS OF THE EXPERIMENT AND THEIR DISCUSSIONS

X-ray studies carried out in the present work showed that the films obtained by vacuum evaporation of CdSe at a pressure of about 10⁻³ Pa, under perpendicular incidence of the vapor flux onto the glass substrate surface, with a condensation rate of 2500 Å/min and a thickness of 5–10 μm, are textured and polyphase within the substrate temperature range *T_s*=250–400°C. They consist of both cubic and hexagonal modifications. In this case, the crystallites are arranged predominantly so that the crystallographic planes (111)_c and (0002)_h are oriented parallel to the substrate surface (see Figure 1, the first strong line from the center of the X-ray pattern). From the presented X-ray photographs, it follows that with increasing *T_s*, the dispersion angle of the texture axis also increases (Table 1). A significant preferential orientation of crystallites with the plane (10 $\bar{1}$ 3)_h parallel to the substrate surface is also observed.

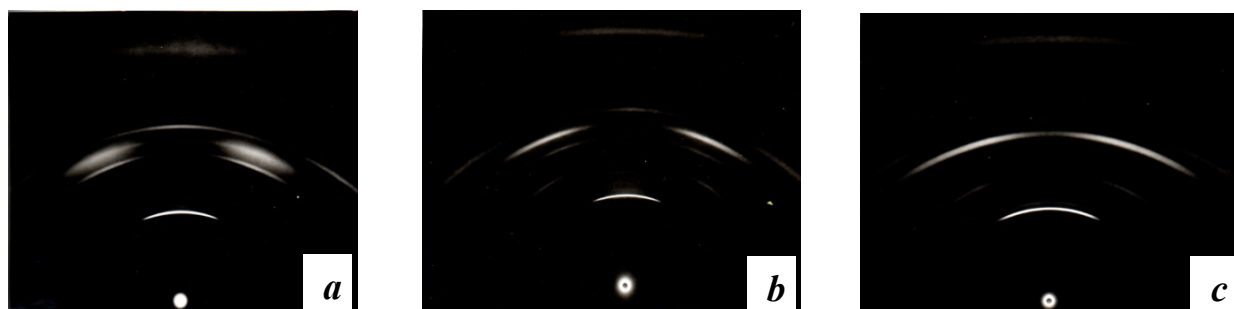


Figure 1. Photographic X-ray diffraction images of CdSe films under filtered FeK α radiation, condensed at substrate temperatures *T_s*: (a) – 250°C; (b) – 300°C; (c) – 400°C.

Table 1. Dependence of the texture axis dispersion angle (φ) for the (111)_c + (0002)_h line of CdSe films condensed at various substrate temperatures *T_s*

<i>T_s</i> , °C	250	300	350	400
φ , °	38	44	57	65

It should be noted that with increasing substrate temperature, both the width and the position of the X-ray lines change. Quantitative phase analysis from the film diffractograms is complicated due to the overlap of X-ray reflections

from the cubic and hexagonal modifications of cadmium selenide and the presence of texture. Therefore, phase analysis was performed on powder samples. For this purpose, a freshly prepared film was separated from the substrate, ground in an agate mortar, and the resulting powder was mixed with BM-1 diffusion pump oil, which does not produce X-ray diffraction lines. The prepared mixture was then applied to a glass substrate. By analyzing the integrated intensity of diffractometric reflections from the $(0002)_h + (111)_c$ planes, which have the same diffraction angle, the relative fraction of the hexagonal modification (H) in the phase composition was determined according to the following relation given in references [8,9]:

$$\frac{I(10\bar{1}1)}{I\{(111)+(0002)\}} = \frac{\alpha H}{\beta + (1-\beta)H}, \quad (1)$$

where $I(10\bar{1}1)_h$ and $I\{(111)_c + (0002)_h\}$ are the integrated intensities of the reflections from the corresponding planes, and α and β are coefficients equal to 1.3 and 4.0, respectively. It was established that the fraction of the hexagonal modification within the above substrate temperature range is between 35 and 44%.

Figure 2 shows the X-ray strip diagrams of the diffractograms of films condensed at $T_s=350$ and 400 °C. It is evident that the strongest line is $(111)_c + (0002)_h$. The other lines are much weaker or are not observed at all. Therefore, the analysis of the films' substructure parameters was carried out using the $(111)_c + (0002)_h$ line by the fourth-moment method, which requires only a single interference line.

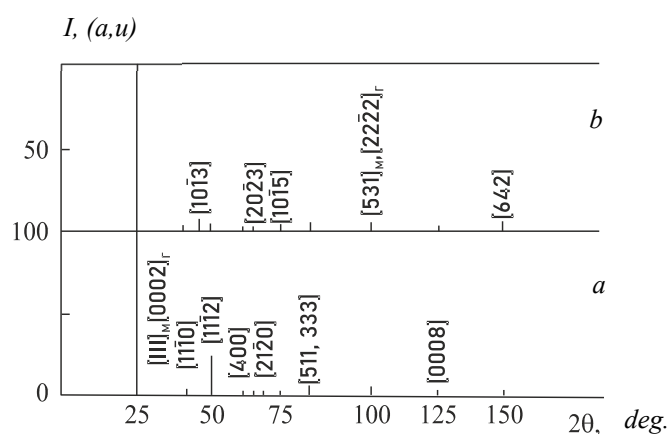


Figure 2. Line diagrams of X-ray diffraction patterns of freshly prepared CdSe films obtained at substrate temperatures T_s : (a) – 350 °C; (b) – 400 °C.

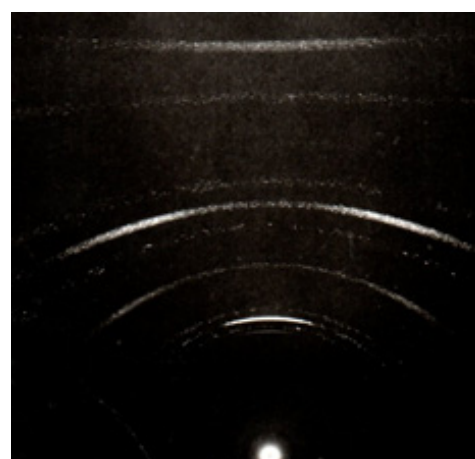


Figure 3. Photographic X-ray diffraction image of the reference sample obtained under filtered FeK_α radiation.

When determining the substructure parameters, it is necessary to exclude the contribution from instrumental factors in the profiles of the lines used. This contribution can be eliminated either theoretically or experimentally by using a standard sample. In this work, the second method was employed. The standard sample consisted of CdSe films prepared by thermal evaporation of the source material in a vacuum of 10^{-3} Pa onto glass substrates at 400 °C, followed by thermal treatment in a cadmium chloride gas environment at 470 °C for 20 minutes, and then cooled at a rate of 3 °C/min. Films prepared in this manner met the requirements of a standard both in terms of substructure parameters and the degree of texturing (Figure 3).

Based on the experimental data, it was established that, with increasing substrate temperature, the size of the coherent scattering region of X-rays increases [8]:

$$D_{csr} = \frac{\lambda(\theta_m^3 - 9\theta_m\mu f_2)}{3\pi^2 \cos \theta [\mu f_4 - 3(\mu f_2)^2]} \quad (2)$$

where λ is the wavelength of the X-ray radiation, θ_m - is the abscissa of the maximum of a line profile in the X-ray 2θ -diagram, and μf_2 and μf_4 are the second- and fourth-order central moments of the physical broadening function of the X-ray line. It was found that D_{csr} increases from 850 to 970 Å.

The microstrains of the crystal lattice were determined from the relation

$$\left(\overline{\varepsilon^2}\right)^{\frac{1}{2}} = \left[\left(\frac{\Delta d}{d}\right)^2\right]^{\frac{1}{2}}, \quad (3)$$

where d is the interplanar spacing. It was found that the microstrain changes from $2.5 \cdot 10^{-3}$ to $6.84 \cdot 10^{-3}$.

From the position of the most intense line on the diffractograms, the lattice parameters were determined. It was shown that, with an increase in the substrate temperature T_s from 250 to 400°C, the lattice parameters decrease from 0.6083 to 0.6082 nm.

Electron microscopic studies of the surface morphology of *CdSe* films showed that their visible structure depends on the substrate temperature T_s . Figure 4 presents electron micrographs (SEM) of the film surfaces obtained in the substrate temperature range of 250–400°C. The images show that crystallites with well-defined facets emerge on the film surface, and their size increases with increasing T_s . In electronograms obtained for the films' surface layer, the line spectra are richer than those on the X-ray diffractograms. These spectra are interpreted in terms of cubic and hexagonal modifications.

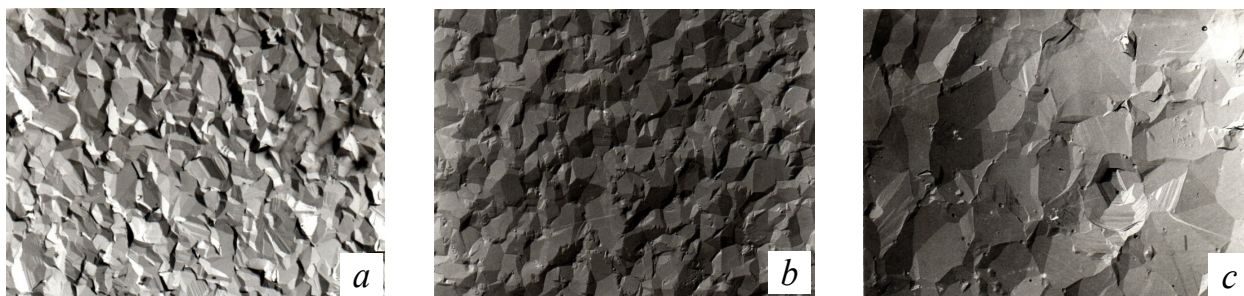


Figure 4. Electron micrographs of the surface of freshly prepared *CdSe* films, condensed at substrate temperatures T_s , °C: (a) – 250, (b) – 300, (c) – 400 ($\times 10000$)

To obtain quantitative data on the crystallite parameters observed on the film surfaces, measurements of their linear dimensions (d') were carried out based on the electron micrographs of films prepared at different substrate temperatures T_s (here, the linear dimension of a crystallite is defined as the diameter d of a circle whose area equals the average area of a crystallite on the film surface). For this purpose, a section of the electron micrograph was randomly selected to include a sufficient number of crystallites for a statistical size distribution. The value of d' was determined by dividing the total area occupied by the crystallites by the number of crystallites. It was found that with an increase in T_s from 250 to 400°C, the average d' increases from 500 to 1500 nm.

Fractographic studies of the film cross-sections (Figure 5) revealed that the upper, predominant part of the film has a columnar structure. This structure is disrupted near the substrate, with a thinner, finer-crystalline layer measuring 0.5-1 μm in thickness.

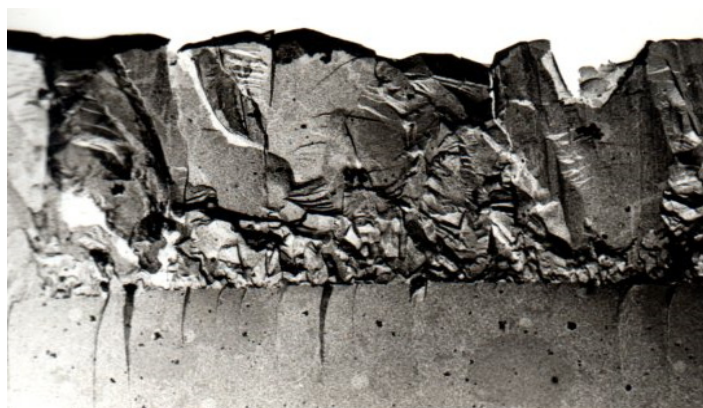


Figure 5. Fractographic image of the cross-section of a *CdSe* film prepared at $T_s=250^\circ\text{C}$ ($\times 7500$).

Freshly prepared polycrystalline cadmium selenide films are generally weakly photosensitive and are usually subjected to thermal treatment in the presence of oxygen or chloride-containing activators in order to improve their electrophysical and photoelectric properties [11,13,14]. Here, we consider the changes in the substructure parameters of *CdSe* films during TT in air in the presence of CdCl_2 . One of the important technological factors is the annealing temperature. Based on experimental data, it has been established that the most stable *CdSe: Cd: Cl* films with good electrophysical properties are obtained when annealed in a CdCl_2 atmosphere at 450–470 °C for 4–8 minutes [15,16].

To determine the effect of annealing on the films' substructure, a series of identical samples was prepared. Each film was subjected to thermal treatment for each time interval only once. The structure was studied after annealing and after the samples had cooled to room temperature.

Thermally treated *CdSe: Cd, Cl* films also exhibited texturing with a well-defined axial texture $(111)_k + (0002)_h$, the dispersion angle of which increases with increasing substrate temperature (T_s). Figure 6 shows a comparison of photoradiographs of films prepared at the same $T_s=250^\circ\text{C}$ and with a constant annealing temperature $T=470^\circ\text{C}$. In this case, the predominant orientation of the plane $(111)_k + (0002)_h$ parallel to the substrate surface gradually shifts to other

orientations as the annealing time increases. For example, in the (311) , $(11\bar{1}2)$ plane after annealing for $t_{ann}=2$ min, and then in another $(10\bar{1}3)$ plane after $t_{ann}=4$ min. It should be noted that in the latter case, there are also crystallites oriented randomly; further increasing the annealing time does not lead to significant changes in the predominant orientation. During the annealing of films prepared at higher substrate temperatures, the change in film texture occurs similarly to the case of $T_s=250$ °C. However, the reorientation of crystallites from one predominant orientation to another occurs over a shorter annealing time as T_s increases.

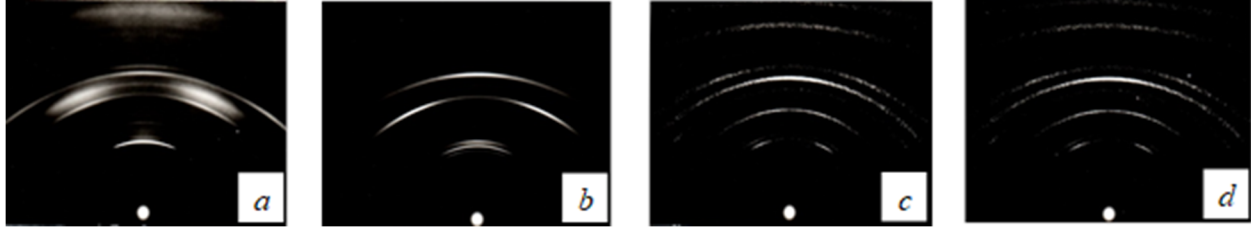


Figure 6. X-ray photographs of CdSe films before and after thermal annealing in air in the presence of CdCl₂: (a) – as-deposited; (b) – t=2 min; (c) – t=4 min; (d) – t=10 min. Substrate temperature $T_s=250$ °C.

From the photo-radiographs in Figure 6, one can also observe qualitative changes in the relative intensities of the X-ray lines, which were recorded with the X-ray beam incident on a stationary sample at an angle of 20°. It is evident that the most intense diffracted reflection lines occur in the range $\varphi=25$ –50°. Quantitative changes in the relative intensities of the X-ray lines, obtained from diffraction analysis, are shown in the bar charts for films deposited at a condensation temperature of 350 °C (Figure 7).

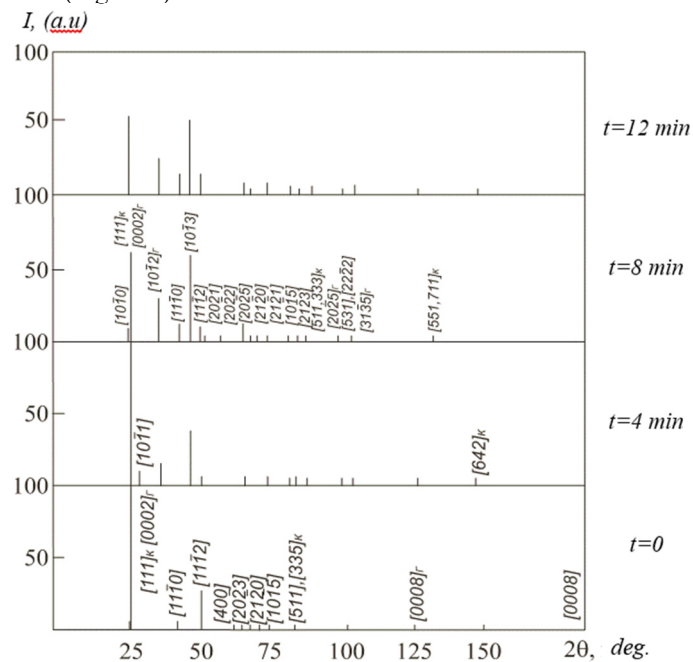


Figure 7. X-ray strip diagrams for CdSe films deposited at a condensation temperature of 350 °C before and after heat treatment in air in the presence of CdCl₂.

It was established that the fraction of the hexagonal modification H in the annealed films increases with the duration of the thermal treatment. In films prepared at $T_s=350$ °C and annealed for 6 min, the value of H reaches 72 %.

The experimental results for the determination of the coherent scattering region sizes D_{CSR} as a function of annealing time for CdSe: Cd, Cl films prepared at $T_s=350$ °C and 400 °C are shown in Figure 8a,b. It can be seen that at the initial stages of thermal treatment, D_{CSR} increases, followed by a plateau in the annealing time interval of 4–10 min, during which changes are minimal. Further increasing the annealing time ($t_{ann}>10$ min) leads to a rather sharp increase in D_{CSR} .

The same figures also show the dependence of the minimum dislocation density on the annealing time, determined based on the following formula given in reference [9]:

$$\rho_D = 3n/D_{CSR}^2, \quad (4)$$

where n is the number of dislocations per coherent scattering domain D_{CSR} (for calculations, $n=1$ was assumed).

In films obtained at $T_s=400$ °C, the dislocation density ρ_D in the plateau region ($t_{ann}=4$ -10 min) is almost an order of magnitude lower than in films obtained at $T_s=350$ °C, while the corresponding D_{CSR} values for these films are approximately 50 nm and 150 nm, respectively.

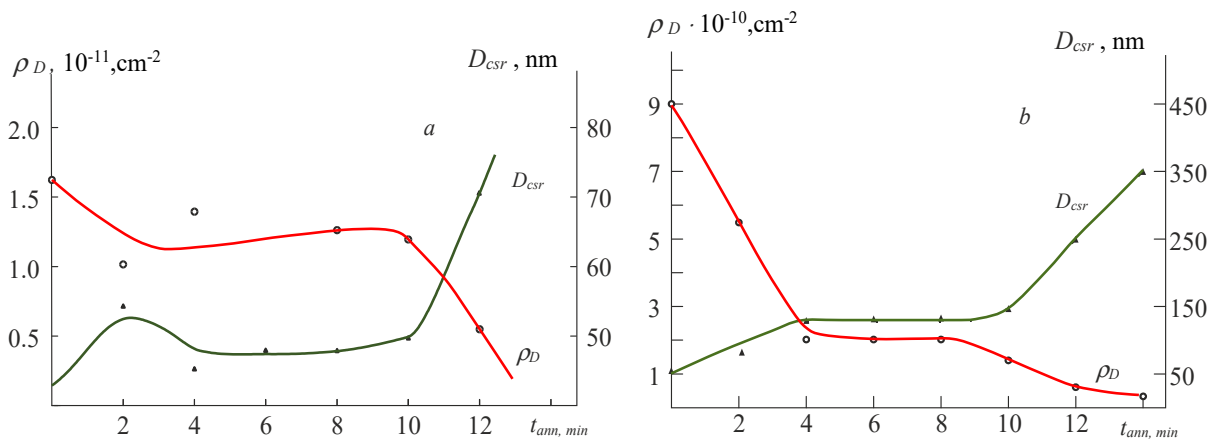


Figure 8. Dependence of D_{csr} and ρ_D on annealing time in air in the presence of CdCl_2 for CdSe films deposited at substrate temperatures $T_s=350^\circ\text{C}$ (a) and $T_s=400^\circ\text{C}$ (b).

One of the significant parameters characterizing the structure is the lattice parameter a . Since the lattice parameter can be determined with higher accuracy from diffraction lines at large reflection angles, it was determined using the (333) line. Figure 9 presents the results of the study of the lattice parameter a for films obtained at $T_s=350^\circ\text{C}$ as a function of annealing time. The figure shows that with increasing annealing time up to the optimal value ($t_{ann}=6$ min), the lattice parameter a decreases and approaches the lattice parameter of cadmium selenide in equilibrium, $a=6.035 \text{ \AA}$ [17]. In the annealing time range of 4–10 min, a changes only slightly, which can be explained by the transformation of the cubic modification into the hexagonal one.

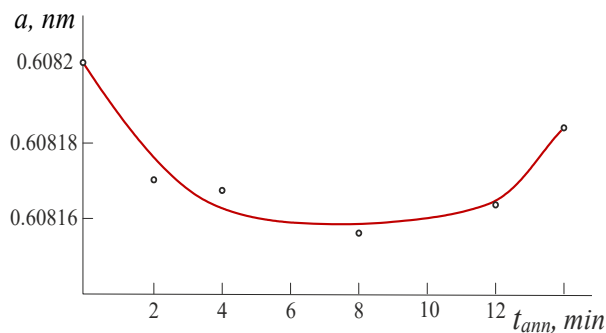


Figure 9. Dependence of the lattice parameter a of the CdSe: Cd, Cl film on annealing time, determined from the position of the X-ray (333) reflection.

The results of electron-microscopic studies also show that annealing the films leads to changes in the crystallite sizes. From the micrographs of the surfaces of sensitized films obtained at $T_s=250^\circ\text{C}$ and 350°C (Figure 10a,b), it is evident that after annealing, the grain sizes increase due to the merging of adjacent grains, the boundaries straighten, and the junction angles approach equilibrium values. The average grain size for films obtained at $T_s=250^\circ\text{C}$ is approximately 1900 nm, while for films obtained at $T_s=350^\circ\text{C}$, it is about 2100 nm. Individual grains for these films reach sizes of roughly 3–4 μm .

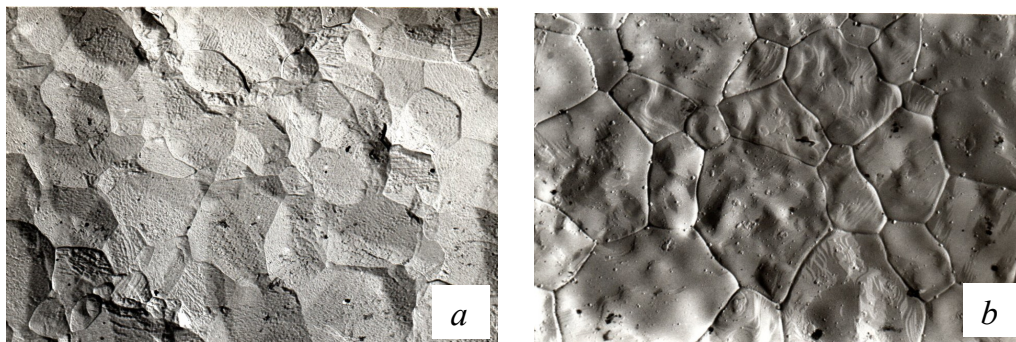


Figure 10. Electron micrographs of optimally sensitized CdSe: CdCl_2 films: (a) films obtained at $T_s=250^\circ\text{C}$; (b) films obtained at $T_s=350^\circ\text{C}$

The study of the cross-sectional structure of the films showed that the columnar structure, in some cases, extends through the entire thickness of the film (Figure 11a, b). However, it should be noted that although the cross-sectional

structure appears monolithic in the fracture micrographs, in reality the average size of the coherent scattering regions is much smaller, as determined from the study, $D_{CSR}=50\text{--}350$ nm (see, for example, Figure 8b). In the cross-sectional fracture images, the SnO_2 layer between the glass substrate and the CdSe-based film is indicated schematically.

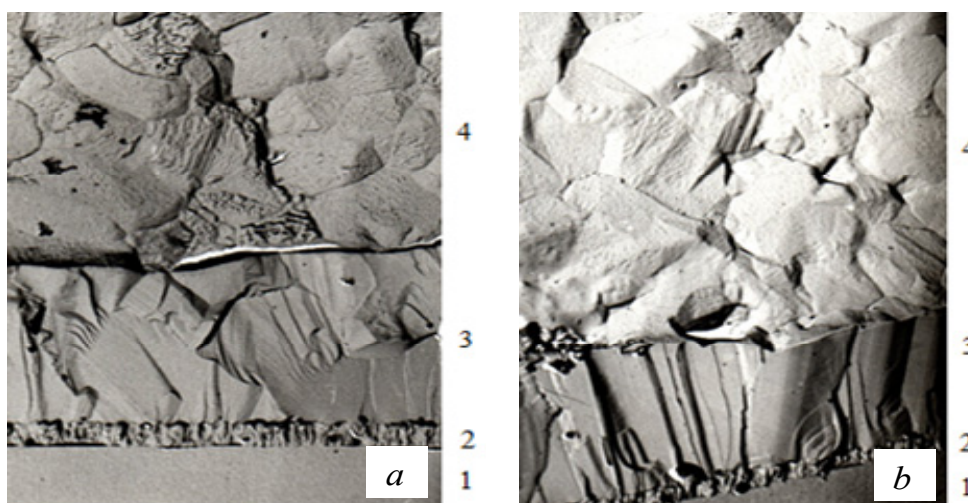


Figure 11. Fractographs of the cross-sectional fracture of optimally sensitized CdSe: Cd, Cl films: (a) films deposited at $T_s=250^\circ\text{C}$; (b) films deposited at $T_s=350^\circ\text{C}$. 1 – substrate; 2 – SnO_2 layer; 3 – cross-sectional fracture; 4 – surface.

CONCLUSIONS

Freshly prepared CdSe films are textured, with the texture axis coinciding with the crystallographic direction $(111)_c + (0002)_h$ and perpendicular to the substrate plane. As the substrate temperature T_s increases from 250 to 400°C , the texture axis dispersion angle, the fraction of the hexagonal modification, the crystallite size, and the coherent scattering regions of X-rays increase, while the lattice parameters and microstrains of the crystal lattice decrease.

Thermal treatment of freshly prepared CdSe films with a substrate temperature $T_s=250^\circ\text{C}$ in air in the presence of CdCl_2 vapor ($T_{ann}=470^\circ\text{C}$) for $t_{ann}=4$ min leads to an increase in the coherent scattering region D_{CSR} and, correspondingly, the size of the crystalline grains, as well as an increase in the fraction of the hexagonal modification. The texture axis dispersion angle increases significantly, and the predominant orientation becomes the plane $(10\bar{1}3)$. Such reorientation of crystallites necessarily occurs via $(311)_c + (11\bar{1}2)_h$ planes parallel to the substrate; however, with further increase in the annealing time, the original orientation $(111)_c + (0002)_h$ becomes predominant again. During the initial stage of annealing up to $t_{ann}=6$ min, the lattice parameter decreases, while at $t_{ann}>10$ min it increases. At the same time, the crystallite sizes and the coherent scattering region D_{CSR} increase, while the lattice parameter a and the minimum dislocation density ρ_D decrease.

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ДОСЛІДЖЕННЯ МІКРОСТРУКТУРИ ФОТОСЕНСИТИВНИХ ТОНКИХ ПЛІВОК CdSe ТА CdSe:Cd,Cl

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Субструктурні характеристики свіжоприготовлених фоточутливих тонких плівок CdSe та легованих CdSe:Cd,Cl було досліджено з огляду на вплив температури підкладки T_s і тривалості термообробки на повітрі в присутності парів CdCl₂. Також наведено результати електронно-графічних та електронно-мікроскопічних досліджень плівок, одержаних за різних технологічних умов. Встановлено, що вісь текстури свіжоприготованих плівок CdSe перпендикулярна до площини підкладки. Із підвищенням T_s від 250 до 400°C зростають кут розсіювання осі текстури, частка гексагональної фази, розмір кристалітів і розмір області когерентного рентгенівського розсіювання D_{CSR} . Після відпалу на повітрі в присутності парів CdCl₂ при температурі 300°C плівки, отримані за $T_s=250^\circ\text{C}$, виявляють переорієнтацію кристалітів від площини $(111)_c + (0002)_h$, паралельної площині підкладки, до орієнтації $(10\bar{1}3)$ через площини $(311)_c + (11\bar{1}2)_h$. Така переорієнтація супроводжується збільшенням розміру кристалітів і D_{CSR} , а також зменшенням параметра ґратки та мінімальної густини дислокацій.

Ключові слова: фоточутлива плівка; CdSe; CdSe:Cd,Cl; подовження фотопровідність; полікристал; текстура; температура підкладки; термічне оброблення; кінетика сенсibiлізації; розмір області когерентного рентгенівського розсіювання