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Structure and Optical Characteristics of Mercury-Modified $Se_{100-x}As_x$ Amorphous Films

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The structure and optical characteristics (transmission spectra and dispersion of the refraction index) of nominally pure and mercury-modified amorphous $Se_{100-x}As_x$ films (x = 0, 3, 5, 7, 9) were studied. It was ascertained that nominally pure films are X-ray amorphous. Exposure of the films in mercury vapor leads to the formation of HgSe crystalline inclusions in their near-surface layers. The width of the pseudo-forbidden gap E_g and the refraction indices n_2, n_2' and n_3 of both nominally pure (n_2) and those exposed to mercury vapor (n_2' and n_3) films were determined. Significant changes in the n_2' and n_3 values of amorphous films are caused by structural transformations that occur in them during modification.

Keywords: chalcogenide amorphous films, X-ray diffractometry, structure, transmission spectra, dispersion of the refraction index.

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Introduction

In [1-7], the results of studies of the structure of nominally pure and mercury-modified amorphous Se, $Se_{100-x}Te_x$ (x = 3, 5, 10 15) and $Se_{100-x}Sb_x$ (x = 3, 5, 7, 9) films by using the Raman spectroscopy were presented. It was established that the matrix of amorphous selenium films is built mainly with elements of Se_8 rings and Se_n spiral chains. The structure of $Se_{100-x}Te_x$ amorphous films is similar to amorphous Se, however, in the chain fragments of Se_n, part of selenium atoms are replaced with tellurium atoms [2]. The Raman spectra of $Se_{100-x}Sb_x$ films reveal features that indicate the presence of both elements of Se_8 ring and those of Se_n chains, as well as $SbSe_{3/2}$ structural groups in their matrix [6].

When amorphous films of the specified compositions are exposed to mercury vapor, crystalline inclusions of mercury selenide (HgSe) are formed in their near-surface layers [2, 3, 6, 8, 9]. It is the formation of HgSe inclusions in the amorphous films of $Se_{100-x}Te(Sb)_x$ systems, as well as $Se_{100-x}As_x$ films (x = 3, 5, 7, 9), according to the authors of works [10-14], that is caused by a sharp decrease (by 4 - 7 orders) of the electrical resistance of the films when they are exposed in mercury vapor. It should be noted that direct studies of the structure of the mercury-modified films of the Se-As system have not been carried out so far. The detected effect of a sharp decrease in the electrical resistance of the amorphous films of the Se-Te(Sb,As) systems indicates the possibility of creating the sensitive elements of sensors for the presence of mercury vapor based on these films.

Taking into account the results of research of the electrical parameters of amorphous films, it can be assumed that under the influence of mercury vapor, their other characteristics will also change, including and optical ones.

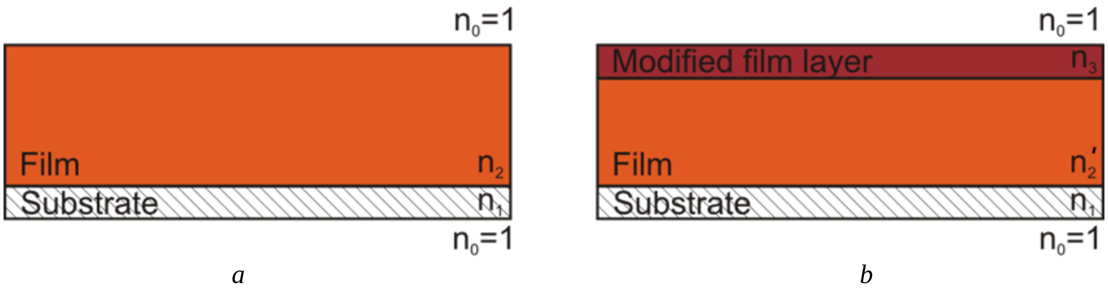

Fig. 1. Schemes of transmission of nominally pure (a) and mercury-modified (b) thin film.

This paper presents the results of research of the structure of mercury-modified amorphous films of the Se-As system with a low content of arsenic and their optical characteristics – transmission spectra and dispersion of refraction indices.

I. Methodology of the experiment

Amorphous films of the Se-As system with an arsenic content of 0, 3, 5, 7, and 9 at.% were prepared by using the vacuum evaporation of glasses of the appropriate compositions from quasi-closed effusion cells onto unheated glass substrates. The thickness of the films was ~1 μm. Modification of $Se_{100-x}As_x$ films was carried out in special hermetic containers by keeping them in mercury vapor for 24, 48, and 72 hours at the temperatures of 291 to 293 K. At these temperatures, the values of saturated Hg vapor pressure and its concentration are 0.136 - 0.162 Pa and 11.2 - 13.3 mg/m^3, respectively.

The study of X-ray diffractograms of amorphous and mercury-modified films of the Se-As system was carried out on a modernized X-ray setup "DRON-4" by using CuKα radiation (λ = 1.5418 Å) and a nickel filter.

The dispersion of the refraction index of the films of Se-As system was studied using a HORIBA Smart SE spectral ellipsometer. Optical constants were determined in the spectral range of 440 to1000 nm at the incidence angle 70°. The obtained experimental spectra were analyzed taking into account multilayer reflection in the samples (Fig. 1) with the help of the DeltaPsi2 software by using the Tauc–Lorentz model for nominally pure films (Fig. 1,a) and the Tauc–Lorentz + Drude model (Fig. 1,b) for mercury-modified films.

Studies of transmission spectra of films in the region of 450 to 1050 nm were carried out at room temperature by using a MDR-3 diffraction monochromator. The spectral resolution was no more than 10^{-3} eV.

For nominally pure films of the Se-As system, the absorption coefficient α was determined from the transmission spectra $T(\lambda)$ taking into account multilayer reflection in the samples (Fig. 1,a):

$$\alpha = \frac{1}{d}\ln\frac{(1-R_1)(1-R_2)(1-R_3)}{T}, \quad (1)$$

$$R_1 = \left(\frac{n_1-1}{n_1+1}\right)^2, \quad R_2 = \left(\frac{n_2-1}{n_2+1}\right)^2, \quad R_3 = \left(\frac{n_1-n_2}{n_1+n_2}\right)^2, \quad (2)$$

where d is the thickness of the sample, T is the transmission coefficient, R_1, R_2, R_3 are the reflection coefficients at the interface of the layers – substrate-air, film-air, substrate-film, respectively. n_1 – refraction index of the substrate, n_2 – refraction index of the film.

For mercury-modified films of the Se-As system, α was determined using the model shown in Fig. 1,b:

$$\alpha = \frac{1}{d}\ln\frac{(1-R_1)(1-R_2)(1-R_3)(1-R_4)}{T}, \quad (3)$$

$$R_1 = \left(\frac{n_1-1}{n_1+1}\right)^2, \quad R_2 = \left(\frac{n_1-n_2'}{n_1+n_2'}\right)^2, \quad R_3 = \left(\frac{n_2'-n_3}{n_2'+n_3}\right)^2,$$

$$R_4 = \left(\frac{n_3-1}{n_3+1}\right)^2, \quad (4)$$

where d is the sample thickness, T is the transmission coefficient, R_1, R_2, R_3, R_4 are the reflection coefficients at the interface of the layers – substrate-air, substrate-inner layer of the film, the inner layer of the film - the near-surface layer of the film, the near-surface layer of the film - air, respectively. n_1 is the refraction index of the substrate, n_2' is the refraction index of the inner layer of the film, n_3 is the refraction index of the near-surface layer of the film.

II. Experimental results

Studies have shown that all nominally pure films of the Se-As system are X-ray amorphous, that is, there are no pronounced reflections in their diffractograms. A typical (for all the research) X-ray diffractogram of mercury-modified amorphous film $Se_{95}As_5$ for 72 hours is shown in Fig. 2. It contains reflexes at 22.9, 25.49, 29.9, 42.0, 49.7 and 51.3 degrees. For films of other compositions, the positions of pronounced reflexes in their diffractograms differ by no more than 0.9 degrees and are located at:

$Se_{97}As_3$
22.5 , 25.40 , 30.1 , 41.8 , 50.2 , 51.5 degree
$Se_{93}As_7$
22.9 , 25.43 , 29.7 , 42.2 , 49.9 , 51.3 degree
$Se_{93}As_9$
23.2 , 25.49 , 29.8 , 41.9 , 50.1 , 51.4 degree

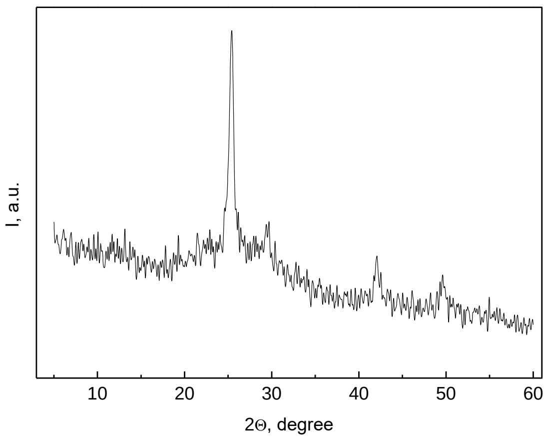

Fig. 2. X-ray diffractogram of the mercury-modified for 72 hours amorphous $Se_{95}As_5$ film.

Note that the intensity of reflexes increases as the time of exposure of amorphous films in mercury vapor increases, and decreases as the content of arsenic in their composition increases.

Comparison of the positions of reflexes in the obtained X-ray diffractograms with the positions of reflexes in the diffractograms of HgSe films obtained using the hydrochemical synthesis [15] and exposed in mercury vapor amorphous Se [3, 8, 9] and $Se_{100-x}Sb_x$ films (x = 3, 5, 7 and 9) [6] allows us to state that during the modification of amorphous $Se_{100-x}As_x$ films, the crystalline inclusions of mercury selenide are formed in their near-surface layers.

For example, the diffractogram of the HgSe film obtained using the hydrochemical synthesis revealed reflexes at 25.84, 29.73, 42.51 and 50.08 degrees, and for amorphous films Se and $Se_{97}Sb_3$ modified with mercury within 48 hours – at 25.37, 29.33, 41.99 and 49.65 degrees as well as 23.5, 25.27, 29.3, 41.9 and 49.6 degrees, respectively. Some difference in the position of reflexes in the diffractograms of modified films Se [3, 8, 9], $Sb_{100-x}Sb_x$ [6], $Se_{100-x}As_x$ is due, most likely, to a different degree of deviation of crystalline inclusions of mercury selenide from stoichiometry due to differences in the chemical composition of the films and conditions of their modification (temperature and exposure time in mercury vapor).

Fig. 3 shows the transmission spectra of nominally pure and mercury-modified $Se_{100-x}As_x$ films (x = 0, 7, 9) typical for all the studied samples. For films with x = 3 and 5, the spectra are similar. As the content of As in the composition of nominally pure films increases, the absorption edge shifts slightly to the long-wavelength part of the spectrum, indicating a decrease in the width of their pseudo-forbidden gap E_g. The absorption edge of films also shifts when they are modified with mercury. At the same time, due to the significant scattering and absorption of light in the near-surface layers, the transmittance of the films is significantly reduced (Fig. 3).

The E_g value of the films was determined from the Tauc relationship [16]:

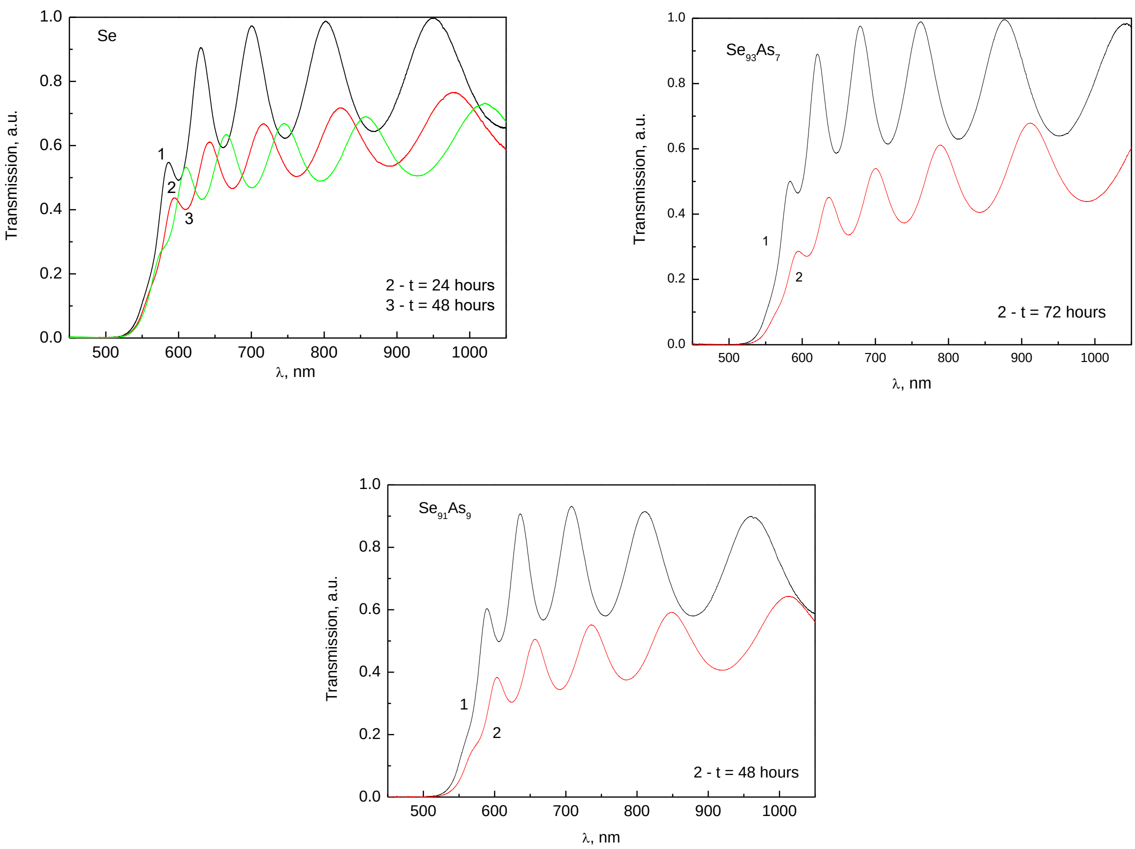

Fig. 3. Transmission spectra of nominally pure (1) and mercury-modified for different times t (2, 3) amorphous films of the Se-As system.

$$\alpha(h\nu) = \frac{B(h\nu - E_g)^2}{h\nu}, \quad (5)$$

where α(*h*ν) is the absorption coefficient, *h*ν is the photon energy, *B* is a constant that characterizes the slope of the Tauc absorption edge. The values of E_g of nominally pure films and films exposed in mercury vapor were determined by extrapolation of the dependences $[\alpha(h\nu) \cdot h\nu]^{1/2} \sim f(h\nu)$ to $\alpha(h\nu) = 0$ (Fig. 4) are given in Table 1. It can be seen that an increase in the content of arsenic in the composition of nominally pure films leads to a decrease in the width of the pseudo-forbidden gap. Note that a similar pattern was found for films of the Se-Sb system with a low antimony content. When the concentration of Sb increases from 3 to 10 at.%, E_g decreases from 1.95 to 1.93 eV [17].

Fig. 5 shows the dispersion of the refraction indices of nominally pure (curves 1) and exposed in mercury vapor for 24, 48, and 72 hours (curves 2 and 3) $Se_{100-x}As_x$ amorphous films with x = 0, 3, and 5. For other film compositions (x = 7 and 9) the spectral dependences of the refraction indices are similar. In the region of transparency (λ > 700 nm) for both nominally pure and mercury-modified films, the normal course of the spectral dependences of the refraction indices n_2, n_2', n_3 is observed.

The determined values of the refraction index n_2 at the wavelength λ = 1 μm of nominally pure amorphous films of the Se-As system are given in Table 1. It can be seen that with an increase in the content of arsenic in their composition, it increases slightly – from 2.53 for Se to 2.57 for $Se_{91}As_9$. Small changes in the refraction index n_2 were also recorded when the antimony content increased (up to 10 at.%) in the composition of films of the Se-Sb system [17].

Analysis of experimental results given in Table 1 shows that with an As content of 5-7 at.% in the composition of $Se_{100-x}As_x$ films, the slope of the dependences $E_g(x)$ and $n_2(x)$ changes. Such changes in the width of the pseudo-forbidden band of the refraction index of the As-Se system films are caused by structural changes that occur in them with increasing the arsenic content. As already noted, the structural network of amorphous Se films is built by fragments of Se_8 rings and Se_n chains. When arsenic is introduced into Se and its concentration increases, selenium chains are cross-linked [18], i.e., the matrix of $Se_{100-x}As_x$ amorphous films contains a certain amount of $AsSe_{3/2}$ structural groups.

An increase in the content of As in the composition of the films leads to branching of the structural network and a decrease in the length of the selenium chains, i.e., a topological transition occurs from a one-dimensional (1D) to a two-dimensional (2D) structure that is more stable as compared to the (1D) structure. According to the results of studies of photoinduced mass transport in films of the

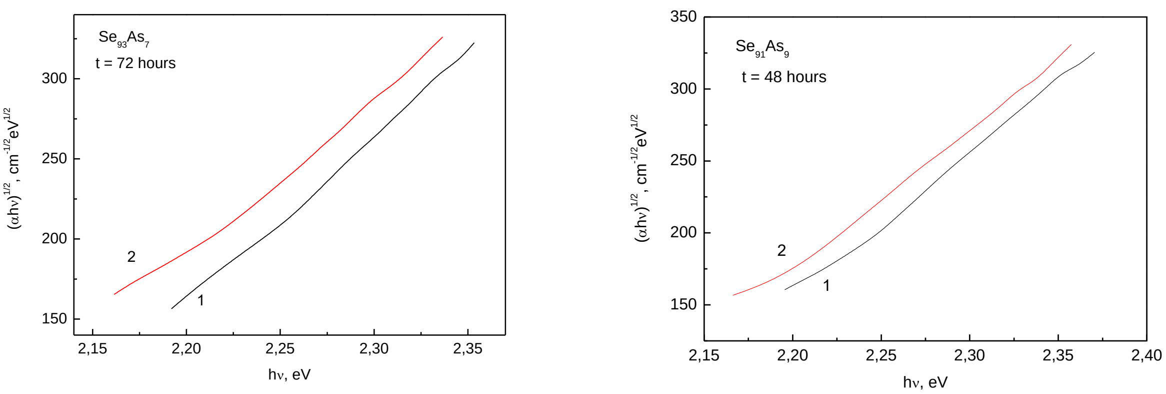

Fig. 4. Dependences of the absorption edge $[\alpha(h\nu) \cdot h\nu]^{1/2}$ on the energy of photons of nominally pure (1) and mercury-modified (2) during t = 72 and 48 h of $Se_{93}As_7$ (a) and $Se_{91}As_9$ (b) films.

Table 1.

The values of E_g, n_2, n_2' and n_3 of nominally pure and mercury-modified for 24, 48, and 72 hours amorphous films of the Se-As system

Composition	E_g, eV	n_2(λ=1μm)	n_2' (λ=1μm)	n_3 (λ=1μm)
Se	2,065	2.53	-	-
Se+Hg(24)	2,060	-	2.87	3.04
Se+Hg(48)	2,049	-	2.77	2.98
$Se_{97}As_3$	2,064	2.54	-	-
$Se_{97}As_3$+Hg (48)	2,003	-	2.72	3.12
$Se_{95}As_5$	2,063	2.55	-	-
$Se_{95}As_5$+Hg (72)	1,938	-	2.71	3.03
$Se_{93}As_7$	2,062	2.56	-	-
$Se_{93}As_7$+Hg (72)	2,024	-	2.57	3.09
$Se_{91}As_9$	2,056	2.57	-	-
$Se_{91}As_9$+Hg (48)	2,019	-	2.59	3.15

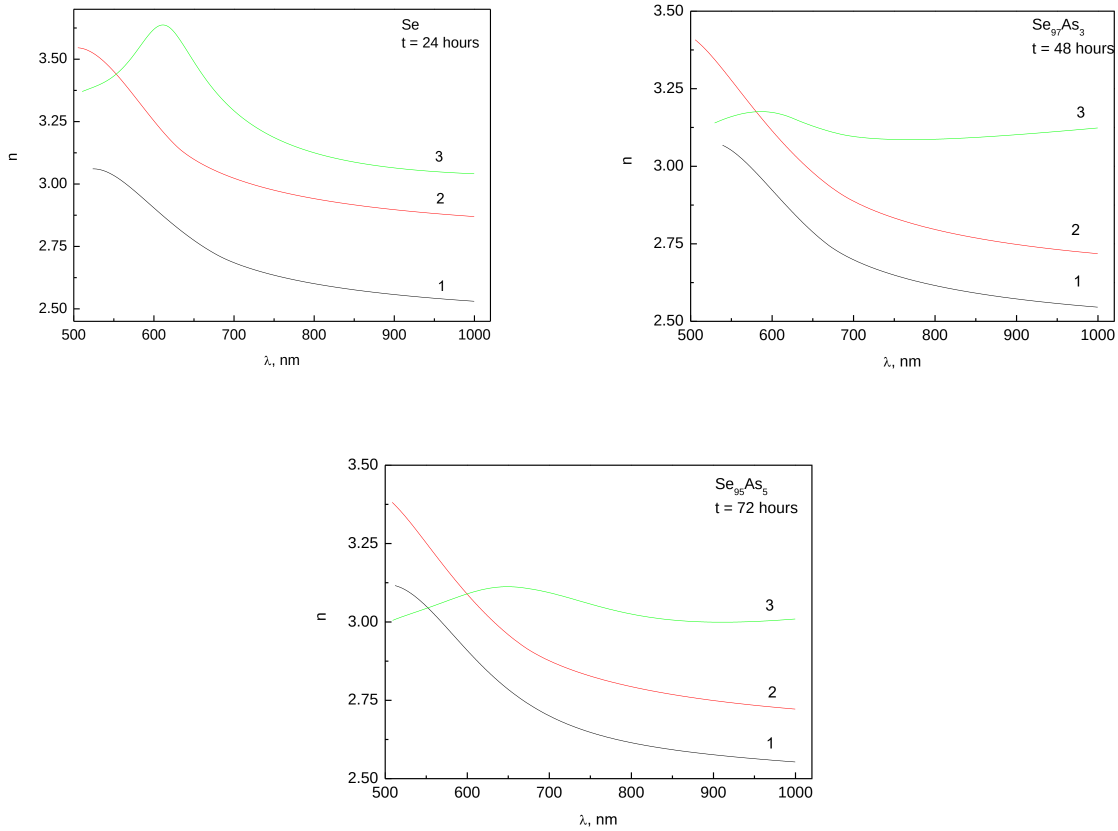

Fig. 5. Dispersion of refraction indices n_2, n_2' and n_3 of nominally pure (1) and mercury-modified during different time t (2, 3) amorphous films of the Se-As system.

Se-As system and physical parameters in glasses of the Se-Sb system, this transition occurs when the content of As and Sb in the samples is about 5 at.% [19, 20].

For $Se_{100-x}As_x$ films exposed in Hg vapors, slightly different values of pseudo-forbidden bands and refraction indices were found. As can be seen from Table 1, the mercury-modified amorphous Se-As films are characterized by smaller values of the width of the pseudo-forbidden band as compared to the nominally pure films. However, the difference in E_g values is small. Perhaps this is due to the fact that the absorption edge of mercury selenide, which was formed in the near-surface layers of the films after their exposure in mercury vapor, is close to the absorption edge of nominally pure films. Note that the fundamental absorption edge of the HgSe film obtained using the hydrochemical synthesis is 2.0 eV [15].

The situation is different for the refraction indices of modified films. According to calculations made taking into account multilayer reflection in the samples (Fig.1,b), the refraction index n_3 of the near-surface layers of the films is 2.98…3.15 (Table 1). We will remind that crystalline inclusions of HgSe were formed in this layer. The difference in the n_3 values is most likely caused by the sizes of the inclusions and the degree of their deviation from stoichiometry.

Much larger changes were found in the values of the refraction index n_2' of the inner layers of the mercury-modified films. The refraction indices of the inner layers of $Se_{100-x}As_x$ films with $x = 0, 3, 5$ are significantly higher than for nominally pure films. At the same time, a significant difference was found in the n_2' values of films with As content before and after 5 at.%.

When the content of arsenic in the composition of modified Se-As films increases to 5 at.%, the value of n_2' decreases from 2.87 for Se to 2.72 for $Se_{95}As_5$. For the $Se_{93}As_7$ and $Se_{91}As_9$ films, the n_2' values are 2.53 and 2.56, respectively. That is, the change in the refraction index n_2' of the inner layers of films with an As content of 7 and 9 at.% is much smaller than the change in n_2' for films with a lower content of arsenic. Note that the difference in the refraction index values of the nominally pure and inner layers of the modified films is much smaller than in films with an As content of 3 and 5 at.% (Table 1).

Such a difference in the values of the refraction indices n_2' of the films of the Se-As system with different arsenic content is caused, in our opinion, by the lower impact of mercury on the inner layers of the $Se_{93}As_7$ and $Se_{91}As_9$ films due to the greater stability of their structure as compared to the structure of the Se, $Se_{97}As_3$ and $Se_{95}As_5$ films.

Conclusions

X-ray diffractograms, transmission spectra, and dispersion of refraction indices of nominally pure and mercury-modified amorphous films of the Se-As system with arsenic content of 0, 3, 5, 7, and 9 at.% were studied. It is shown that exposure of amorphous films in mercury vapor leads to the formation of crystalline inclusions of mercury selenide in their near-surface layers. It was established that with increasing the As content in the composition of nominally pure amorphous films, the width of their pseudo-forbidden gap E_g decreases, and the refraction index n_2 increases.

For $Se_{100-x}As_x$ films with $x = 5, 7$, changes in the slopes of the dependences $E_g(x)$ and $n_2(x)$ were revealed, they may be associated with the presence in them of a topological transition from a one-dimensional to a two-dimensional (more stable) structure. The mercury-modified films are characterized by slightly lower values of E_g and higher values of the refraction indices of the inner (n_2') and near-surface (n_3) layers of the films. Due to the more stable structure of films with x = 7 and 9, the influence of mercury vapor on the value of n_2' is significantly smaller.

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Структура та оптичні характеристики модифікованих ртуттю аморфних плівок $Se_{100-x}As_x$

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Досліджені структура та оптичні характеристики (спектри пропускання та дисперсія показника заломлення) номінально чистих і модифікованих ртуттю аморфних плівок $Se_{100-x}As_x$ (x = 0, 3, 5, 7, 9). Встановлено, що номінально чисті плівки є рентгеноаморфними. Витримка плівок в парах ртуті призводить до формування в їх приповерхневих шарах кристалічних включень HgSe. Визначені величини ширини псевдозабороненої зони E_g та показників заломлення n_2, n_2' і n_3 як номінально чистих (n_2), так і витриманих в парах ртуті (n_2' і n_3) плівок. Суттєві зміни в значеннях n_2' і n_3 аморфних плівок викликані структурними перетвореннями, які відбуваються в них при модифікуванні.

Ключові слова: халькогенідні аморфні плівки, Х-променева дифрактометрія, структура, спектри пропускання, дисперсія показника заломлення.