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Structural Studies of Mercury-Modified Amorphous Films of the Selenium-Antimony System

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The structure of mercury-modified amorphous films of the Se-Sb system was studied using the methods of X-ray diffractometry and Raman spectroscopy. It was ascertained that the structure of the phase that formed in the amorphous matrix of the films after their exposure in mercury vapor corresponds to the structure of crystalline mercury selenide in cubic modification.

The structure of nominally pure and mercury-modified amorphous $Se_{100}Sb_x$ films (x = 3, 5, 7, 9) was investigated using X-ray diffractometry and Raman spectroscopy. It is shown that the matrix of nominally pure films is built mainly by elements of Se_8 rings and Se_n helical chains and contains a certain number of $SbSe_{3/2}$ structural groups. Exposure of the films in mercury vapor leads to formation of mercury selenide (HgSe) crystalline inclusions in the cubic modification in their near-surface layers, as well as to the activation of the process of formation and growth of Se crystalline inclusions of the trigonal modification in the film bulk.

Keywords: chalcogenide amorphous films, X-ray diffractometry, Raman spectroscopy, structure.

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Introduction

The results of studies of electrical characteristics of amorphous films Se, $Se_{100-x}Te_x$, $Se_{100-x}Sb_x$ and $Se_{100-x}As_x$ allowed the authors of the works [1-10] to conclude about the possibility of using these films as sensitive elements of sensors in the presence of mercury vapor in the environment. This conclusion is based on the effect of a sharp decrease (by 4-7 orders of magnitude) in the electrical resistance (R) of the films when they are modified with mercury.

Measurements of the change in R of chalcogenide amorphous films (CAF) as a function of exposure time (t) in mercury vapor were carried out using both planar structures "Ni(Cr) layer – CAF-Ni(Cr) layer" [1-7, 9] and the samples of "graphite probe-CAF-graphite probe" [1,8,10]. At the same time, in both cases, similar dependences of R on t were obtained. This indicates that the change in the electrical resistance of selenium-containing amorphous films modified with mercury is mainly determined by the change in the surface electrical conductivity of the films.

The performed analysis of the results obtained in [1-10] allows us to draw the following conclusions: 1 – amorphous Se films are the most sensitive to the action of mercury vapor; 2 – there is a latent period during which the resistance of the samples is practically unchanged. Its duration, depending on the mercury concentration, temperature and chemical composition of the films, ranges from several minutes to one hour; 3 – with increasing the temperature and concentration of Hg, the transition time from a high-resistance state to a low-resistance state decreases; 4 – when tellurium, antimony, and arsenic are introduced into amorphous selenium and their concentrations in the film composition are increased, the transition time increases, and the value of the change in resistance decreases.

Research of the structure and surface morphology of mercury-modified amorphous films Se and $Se_{100-x}Te_x$ ($3 \le x \le 15$) by using the X-ray diffractometry, Raman spectroscopy and scanning electron microscopy, the results of which are given in [11-15], showed that exposure of films of the above compositions in mercury vapor leads to formation of HgSe crystalline inclusions in their matrix, which have high electrical conductivity. It is the formation of such inclusions that causes a sharp decrease in the electrical resistance of amorphous Se and Se i $Se_{100-x}Te_x$ films when they are modified with mercury.

Taking into account the results of structural studies of mercury-modified amorphous films of the Se-Te system, the authors of the works [1, 8, 10] assumed that a significant decrease in the electrical resistance and films of the Se-Sb and Se-As systems is caused by formation of crystalline inclusions of mercury selenide in amorphous matrices. To confirm the fact of formation of HgSe inclusions in mercury-modified amorphous films of these systems, direct studies of their structure are necessary.

This paper presents the results of studies of the structure of mercury-modified amorphous films of the Se-Sb system by using X-ray diffractometry and Raman spectroscopy.

I. Experiment methodology

$Se_{100-x}Sb_x$ (x = 3, 5, 7 and 9) amorphous films with a thickness of ~1 μm were prepared using the method of vacuum evaporation of glasses of the appropriate compositions from quasi-closed effusion cells on unheated glass substrates. Modification of $Se_{100-x}Sb_x$ films was carried out in special hermetic containers by exposing them in mercury vapor for 48 and 120 hours at room temperatures. At these temperatures (295–297 K), the values of saturated vapor pressure of Hg and its concentration are 0.193–0.228 Pa and 15.7–18.4 mg/m^3, respectively.

X-ray studies of amorphous and mercury-modified $Se_{100-x}Sb_x$ films were carried out on a modernized X-ray device "ДРОН-4" by using CuK_α radiation (λ = 1.5418 Å) and a nickel filter.

Raman spectra of nominally pure and mercury-modified films of the Se-Sb system were studied at room temperature T_{room} by using the Raman microscope Xplora Plus from Horiba Scientific (λ_{exc} = 532 nm). The measurements were carried out at different laser radiation powers and exposure modes: RI – (0.1 mW, 2 s, 100 pulses); RII – (10 mW, 2 s, 20 pulses).

II. Experimental results

X-ray diffractograms of amorphous $Se_{97}Sb_3$ and $Se_{93}Sb_7$ films exposed in mercury vapors for 48 and 120 hours are shown in Fig. 1. It can be seen that the diffractograms of mercury-modified films clearly show well-defined reflexes at 23.5, 25.27, 29.3, 41.9, and 49.6 degrees ($Se_{97}Sb_3$) and 24.0, 25.3, 29.6, 41.95, and 49.6 degrees ($Se_{93}Sb_7$). For other compositions of modified films, the position of these reflexes differs by no more than 0.5 degrees.

However, it should be noted that with increasing the content of antimony in the composition of films modified with mercury under the same conditions, the intensity of reflexes decreases. Note that the diffractograms of amorphous $Se_{100-x}Sb_x$ films, exposed in mercury vapor for a relatively short period of time (1-3 hours), practically do not differ from the diffractograms of nominally pure, in which there are no pronounced reflexes. With increasing the exposure time in mercury vapor, the reflexes become more pronounced. A similar picture was also found during the study of X-ray diffractograms of mercury-modified amorphous Se films [11-13, 15].

Weak reflex at 25.23 degrees in the diffractogram of a Se film with the thickness 700 nm was revealed only after its 6-hour exposing in mercury vapor at T = 290 K. After 48-hour exposure of the Se film in mercury vapor in the diffractogram the well-defined reflexes at 25.37, 29.33, 41.99 and 49.65 degrees were revealed [11, 13]. Even more pronounced reflexes at 25.2, 29.1, 42 and 49.6 degrees were revealed in the diffractogram of the Se film after its modification with mercury at 283 K for a week [12, 13, 15].

As follows from the above data, there is a certain discrepancy in the position of reflexes in the diffractograms of selenium films exposed in mercury vapor at different temperatures and exposure times. Such a discrepancy may be related both to the difference in the alignment of the samples and to the degree of deviation of the crystalline HgSe inclusions from stoichiometry.

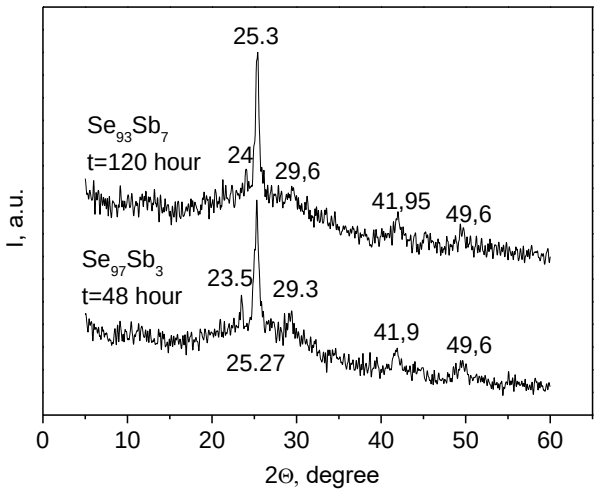

Fig. 1. X-ray diffractograms of amorphous $Se_{97}Sb_3$ and $Se_{93}Sb_7$ films, exposed in mercury vapor for 48 and 120 hours, respectively.

The diffractograms of trigonal selenium and HgSe, which were plotted according to the data of works [16] and [17], respectively, contain reflexes:

Se
23.51, 29.7, 41.33, 43.65, 45.36, 48.09, 51.71, 55.61 and 56.11 degrees.
HgSe
25.39, 29.73, 42.43, 50.12, 52.46, 61.48, 67.5, 69.5 degrees.

Reflexes at 23.59, 29.85, 41.54, 45.58, 48.27 and 51.72 degrees which are similar to those given for trigonal

selenium, were also detected in the diffractogram of the crystallized amorphous Se film [13, 18] (crystallization of the Se film with the thickness 1000 nm was performed by heating it to 423 K at a heating rate of 6 K/min).

The diffractogram of the mercury selenide film obtained by hydrochemical synthesis [19] with using an aqueous solution of $Hg(NO_3)_2$ and Na_2SeSO_3 contains brightly and weakly pronounced reflexes at 25.84, 29.73, 34.5, 42.51, 50.08, 61.37, 64.19, 67.56 and 77.11 degrees. At practically the same positions, reflexes were revealed in the diffractograms of crystalline mercury selenide obtained by hydrochemical synthesis with using aqueous solutions of $HgCl_2$, $SeCI_4$ i ($N_2H_4{\cdot}H_2O$) [20] and $Hg(CH_3COO)_2$ and $SeCI_4$ in the presence of $N_2H_4{\cdot}H_2O$ [21].

The analysis of the X-ray research data given above literature and obtained by us (Fig. 1) allows us to state that the structure of the phase formed in the matrix of amorphous $Se_{100-x}Sb_x$ films during their modification with mercury corresponds to the structure of mercuty selenide in cubic modification. This conclusion is confirmed by the results of Raman spectra studies of amorphous films of the selenium-antimony system exposed in mercury vapor.

Fig. 2 and 3 show the measured in the RI mode Raman spectra of $Se_{97}Sb_3$ and $Se_{93}Sb_7$ amorphous films that are nominally pure (curves 1) and modified with mercury for 48 hours (curves 2) and 120 hours (curves 3). The Raman spectrum of the nominally pure $Se_{97}Sb_3$ film contains an intense band at 254 cm^{-1} and less pronounced features at 237 cm^{-1} and 88–133 cm^{-1}. In the Raman spectrum of the nominally pure $Se_{93}Sb_7$ film, an intense band at 253 cm^{-1} and features at 237 and 86–130 cm^{-1} were revealed. In [14], when studying the Raman spectra of amorphous Se films, an intense band at 253 cm^{-1} and features in the spectral region at 87 – 143 cm^{-1} (87, 115, 135–143 cm^{-1}) and 236 cm^{-1} were revealed.

Comparing the Raman spectrum of an amorphous selenium film given in [14] with the spectra of glassy like, amorphous, polycrystalline, trigonal, and α-monoclinic selenium [22-25], it can be asserted that the bands at 253, 115, and 87 cm^{-1} can be attributed to the vibrations of atoms in fragments of Se_8, and features at 236 and 135-143 cm^{-1} – to vibrations of atoms in chain groups of Se_n. This allows us to conclude that the matrix of the amorphous Se film is built mainly by Se_8 structural groups and contains helical Se_n chains.

As can be seen from Fig. 2 and 3, the intense bands at 254 and 253 cm^{-1} and the feature at 237 cm^{-1} practically do not undergo changes in their position in the Raman spectra of films of the Se-Sb system when the content of Sb in their composition increases. At the same time, a certain shift of the Raman spectra regions of amorphous $Se_{100-x}Sb_x$ films to the region of smaller ν is observed. Thus, for the amorphous Se film, a number of features in the Raman spectrum were revealed in the region 87–143 cm^{-1} [14]. In the spectra of the $Se_{97}Sb_3$ and $Se_{93}Sb_7$ films, these regions are shifted in position to the short-wavelength region: from 87–143 for the amorphous Se film to 86–133 cm^{-1} and 85–130 cm^{-1} for the $Se_{97}Sb_3$ and $Se_{93}Sb_7$ films, respectively. These shifts, in our opinion, are caused by formation of $SbSe_{3/2}$ structural groups in the matrix of $Se_{100-x}Sb_x$ amorphous films, built mainly by selenium rings and chains. This process is most likely accompanied by a decrease in the length of selenium groups.

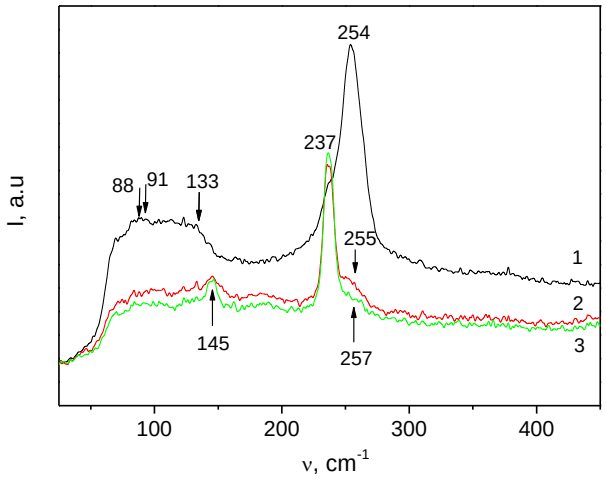

Fig. 2. Raman spectra of nominally pure (curve 1) and mercury-modified $Se_{97}Sb_3$ films for 48 hours (curve 2) and 120 (curve 3) hours.

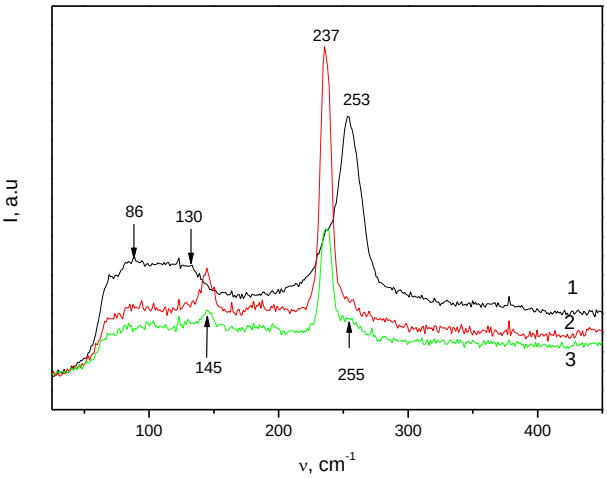

Fig. 3. Raman spectra of $Se_{93}Sb_7$ films, nominally pure (curve 1) and modified with mercury during 48 (curve 2) and 120 (curve 3) hours.

When the power of laser radiation increases (10 mW, RII mode), a significant change in the Raman spectra of $Se_{100-x}Sb_x$ films (Fig. 4) is observed, which is caused by their crystallization. Intense bands at 253-254 cm^{-1} in the spectra of amorphous films (Figs. 2 and 3, curve 1) degenerate into features at 254 cm^{-1}, and weak features at 237 cm^{-1} transform into intense bands at the same frequency. Peculiarities in the 85–143 cm^{-1} range of the spectrum are also more pronounced. We recall that the bands at 237 and 143 cm^{-1} are clearly revealed in the Raman spectrum of trigonal selenium, and other bands from the 85-143 cm^{-1} range are characteristic of the Raman spectrum of α-monoclinic Se. The obtained data allow us to conclude that during the crystallization of amorphous $Se_{100-x}Sb_x$ films, Se crystal inclusions of both trigonal and α-monoclinic modification are mainly formed in their matrix.

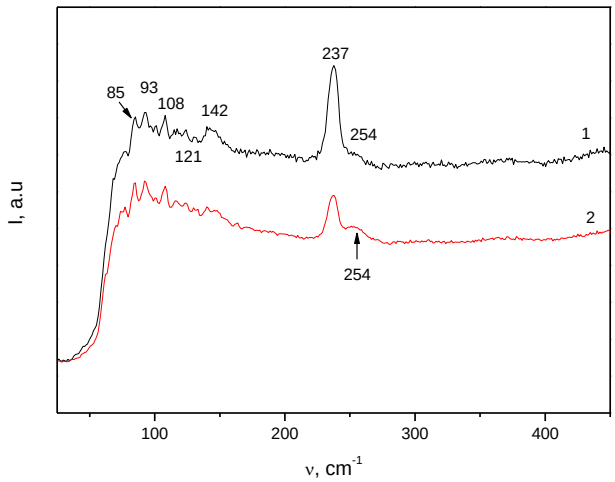

Fig. 4. Raman spectra of $Se_{97}Sb_3$ (1) and $Se_{93}Sb_7$ (2) films measured at a laser radiation power of 10 mW.

After exposure of amorphous $Se_{100-x}Sb_x$ films in mercury vapor, their Raman spectra underwent significant changes (Fig. 2 and 3, curves 2 and 3). The band at 237 cm^{-1} became dominant in the spectra, and the band at 253–254 cm^{-1} in the spectra of nominally pure amorphous films degenerated into a feature in the form of a shoulder. In the Raman spectra of the mercury-modified films, the well-pronounced bands at 145 cm^{-1} and certain features in the 160–200 cm^{-1} region of the spectrum were revealed. The similar features were revealed in the Raman spectrum of the mercury-modified amorphous Se film (weak bands at 168 and 194 cm^{-1}) [8] and in the spectra of HgSe nanoclusters formed in spherical pores (Nd-Y/HgSe) (168 and 192 cm^{-1}) and in the tubular pores (LTL/HgSe) (180 and 200 cm^{-1}) of the zeolite frame [26]. Similar bands (168–169 and 201–202 cm^{-1}) were also revealed in the mercury-modified $Se_{90}Te_{10}$ film [14]. A comparison of the data obtained in this work and the literature data makes it possible to assert that exposure of amorphous $Se_{100-x}Sb_x$ films in mercury vapor leads to formation of crystalline HgSe inclusions in their structural network.

As already noted above, the Raman spectra of mercury-modified $Se_{100-x}Sb_x$ amorphous films are characterized by the presence of intense bands at 237 cm^{-1} and less intense bands at 145 cm^{-1} (Fig. 2 and 3). We recall that these bands are dominant in the Raman spectrum of trigonal Se [22, 23]. This allows us to make an assumption that the exposure of amorphous films of the selenium-antimony system in mercury vapor leads not only to formation of crystalline HgSe inclusions in their near-surface layers, but also to the activation of the process of formation and growth of crystalline selenium inclusions of trigonal modification in the films bulk.

Conclusions

The structure of nominally pure and mercury-modified amorphous $Se_{100}Sb_x$ films (x = 3, 5, 7, 9) was investigated using X-ray diffractometry and Raman spectroscopy. It is shown that the matrix of nominally pure films is built mainly by elements of Se_8 rings and Se_n helical chains and contains a certain number of $SbSe_{3/2}$ structural groups. Exposure of the films in mercury vapor leads to formation of mercury selenide (HgSe) crystalline inclusions in the cubic modification in their near-surface layers, as well as to the activation of the process of formation and growth of Se crystalline inclusions of the trigonal modification in the film bulk.

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Структурні дослідження модифікованих ртуттю аморфних плівок системи селен-сурма

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З використанням методів Х-променевої дифрактометрії та раманівської спектроскопії досліджена структура модифікованих ртуттю аморфних плівок системи Se-Sb. Встановлено, що структура фази, яка сформувалася в аморфній матриці плівок після їх витримки в парах ртуті, відповідає структурі кристалічного селеніду ртуті в кубічній модифікації.

Методами Х-променевої дифрактометрії та раманівської спектроскопії досліджена структура номінально чистих і модифікованих ртуттю аморфних плівок $Se_{100}Sb_x$ (x = 3, 5, 7, 9). Показано, що матриця номінально чистих плівок побудована в основному елементами кілець Se_8 та спіралеподібних ланцюжків Se_n і містить певну кількість структурних угруповань $SbSe_{3/2}$. Витримка плівок в парах ртуті призводить до формування в їх приповерхневих шарах кристалічних включень селеніду ртуті (HgSe) в кубічній модифікації, а також до активування процесу утворення і росту в об'ємі плівок кристалічних включень Se тригональної модифікації в об'ємі плівок.

Ключові слова: халькогенідні аморфні плівки, Х-променева дифрактометрія, раманівська спектроскопія, структура.