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IMPURITY OF IRON ATOMS IN THE VITREOUS ARSENIC SELENIDE (As_2Se_3). ELECTRONIC EXCHANGE BETWEEN CENTERS OF IRON

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МАРГИМУШ СЕЛЕНИДИГА (As₂Se₃) КИРИТИЛГАН ТЕМИР АТОМЛАРИ.
ТЕМИР АТОМЛАРИ БИЛАН ЭЛЕКТРОН АЛМАШИНУВПРИМЕСЬ АТОМОВ ЖЕЛЕЗА В СТЕКЛООБРАЗНОМ ТЕЛЕ СЕЛЕНИДА
МЫШЬЯКА (As₂Se₃). ЭЛЕКТРОННЫЙ ОБМЕН МЕЖДУ ЦЕНТРАМИ ЖЕЛЕЗАIMPURITY OF IRON ATOMS IN THE VITREOUS ARSENIC SELENIDE (As₂Se₃).
ELECTRONIC EXCHANGE BETWEEN CENTERS OF IRON**Bobokhuzhaev Kudrat Umarovich***senior lecturer of the department of physics, doctor of physics and mathematics (PhD)**Chirchik State Pedagogical Institute,***Marchenko Alla Valentinovna***professor of the department of physical electronics, doctor of physics and mathematics**Russian State Pedagogical University named after A.I. Herzen,***Seregin Pavel Pavlovich***professor of the department of physical electronics, doctor of physics and mathematics**Russian State Pedagogical University named after A.I. Herzen,***Begisheva Nelia Rinadovna***senior lecturer, department of physics, Chirchik State Pedagogical Institute*

Аннотация. Темир атомлари ишиа кўринишидаги маргимуш селенидида бир электронли донор марказлар ҳосил қилади. Бунда киритилган темир атомлари концентрацияси ортиши билан Ферми сатҳи таъқиқ соҳа ўртасидан ўтказувчанлик соҳа туби томон силжийди, чунки бунда Ферми сатҳидан пастдаги акцептор сатҳлар темир атомнинг битта электрони билан тўлади. Мессбауэр спектроскопияси усули билан темирнинг нейтрал ва ишиа кўринишидаги маргимуш селенидидаги ионлашган марказлар ўртасидаги электрон алмашинув тадқиқ қилинган.

Таянч сўзлар: халькогенид ишиа, легирланган темир атомлари, электрон алмашинув, Мессбауэр спектроскопияси.

Аннотация. Атомы железа в стеклообразных пленках селенида мышьяка образуют одиночные донорные центры электронов, при этом уровень Ферми смещается от середины запрещенной зоны к дну зоны проводимости с увеличением концентрации железа за счет заполнения одиночных электронных состояний акцепторного типа, расположенных ниже уровня Ферми методом Мессбауэровской спектроскопии исследован электронный обмен между ионизированным и нейтральным центрами железа в стеклообразном селениде мышьяка.

Ключевые слова: халькогенидное стекло, атомы легирующего железа, электронный обмен, Мессбауэровская спектроскопия.

Abstract. Iron atoms in vitreous arsenic selenide films form single electron donor centers, while the Fermi level shifts from the middle of the band gap to the bottom of the conduction band with an increase in the iron concentration due to the filling of single electron states of the acceptor type located below the Fermi level. The Mössbauer spectroscopy method was used to study the electronic exchange between ionized and neutral centers of iron in the glassy selenide of arsenic.

Key words: chalcogenide glass, dopant iron atoms, electron exchange, Mössbauer spectroscopy.

Introduction. Mössbauer spectroscopy allows one to determine the charge state of dopant atoms, the symmetry and chemical nature of their local environment, and the kinetic parameters of the electron exchange processes between the neutral and ionized states of dopant atom sites [1]. In this work we have carried out studies of the mechanism responsible for an impurity conductivity arising in vitreous arsenic selenide As₂Se₃ films modified with iron. The present work also is

devoted to studies on the electron exchange process between the neutral and ionized states of iron dopant atoms in glass films of arsenic selenide, As_2Se_3 , modified by iron.

Experimental. The modification was carried out by a high frequency ion plasma sputtering of arsenic selenides and metaliron (enriched by ^{57}Fe isotope to 92 % with the summary content of impurities $< 10^{-4}$ at %) in an argon atmosphere at a pressure of 10^{-2} Torr at a frequency of 13.6 MHz with the subsequent annealing of the films at 150°C for 30 min, in order to stabilize their properties. The iron concentration, N_{Fe} , was varied from 0.5 to 4.0 at % and controlled by the method of X-ray fluorescent analysis with an error of ± 0.1 at %.. The amorphism of the films was checked by the X-ray phase analysis. The ^{57}Fe Mössbauer spectra in the range of temperatures from 80 to 400 K were recorded using a ^{57}Co radiation source in a palladium matrix. The electroconductance temperature dependence and the Seebeck coefficient sign were measured using standard methods. The error in determining the electroconductance activation energy E_σ amounted to ± 0.05 eV. The position of the optical absorption edge for pure and doped As_2Se_3 glass was determined in the range of 600–2500 μm at 20°C ; the error in determining the optical absorption edge was ± 0.02 eV. Since for chalcogenide glass an exponential dependence of the absorption coefficient α on the light frequency ω is typical in the region of the absorption (Urbach) edge, a linear dependence $(\alpha\hbar\omega)^{1/2} = f(\hbar\omega)$ was built and its extrapolation on $(\alpha\hbar\omega)^{1/2} = 0$ was made (here α is the absorption coefficient, and \hbar is the Planck constant), in order to measure the value of the optical band gap width E_{opt} .

Results and discussion. Electrical Activity of Iron Atoms. According to the Seebeck coefficient, the As_2Se_3 films, which do not contain iron, possessed a hole type of conductivity, while their modification results in an electron type of conductivity (at $N_{\text{Fe}} > 0.5$ at %). The introduction of iron leads to a substantial increase in the conductance σ (Fig. 1); the conductance activation energy E_σ decreases tending to a limiting value of 0.24 eV at $N_{\text{Fe}} = 4$ at% (Fig. 2). The optical band gap for the films E_{opt} decreases with the growth of the iron concentration (Fig. 3), but not as sharply as E_σ (Fig. 2).

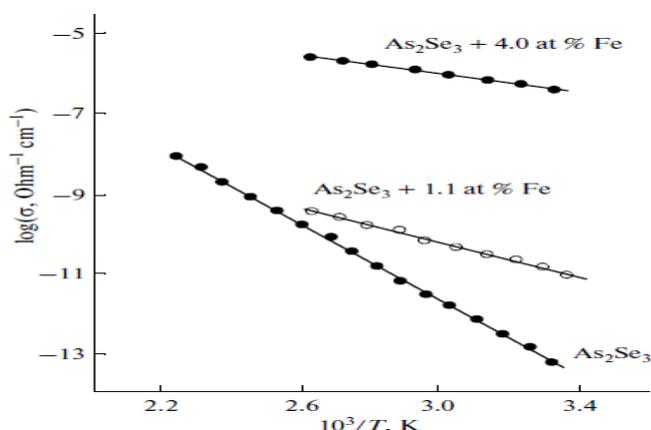


Fig. 1. Electroconductance temperature dependences for As_2Se_3 films and As_2Se_3 films modified with iron

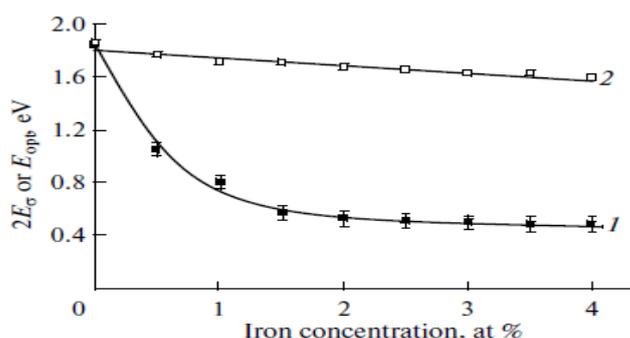


Fig. 2. Dependence of the electroconductance activation energy E_σ (1) and the optical band gap E_{opt} (2) of modified As_2Se_3 films on the iron concentration

The ⁵⁷Fe Mössbauer spectra in the modified As₂Se₃ films are shown in Figs. 4, 5. When $N_{\text{Fe}} \leq 2$ at %, the spectra at 295 K represent quadrupole doublets (the isomeric shift $IS = 0.22 \pm 0.02$ mm/s, quadrupole splitting $\Delta = 0.64 \pm 0.03$ mm/s, and the spectral line width at half height $G = 0.31 \pm 0.03$ mm/s; these values weakly depend on the iron concentration), which correspond to iron atoms Fe³⁺ in a distorted tetrahedral environment of selenium atoms (Fig. 4). When $N_{\text{Fe}} > 2$ at %, the Mössbauer spectra are a superposition of a quadrupole doublet referring to the already described state Fe³⁺ and a quadrupole doublet ($IS = 0.82 \pm 0.02$ mm/s, $\Delta = 1.82 \pm 0.03$ mm/s, and $G = 0.31 \pm 0.03$ mm/s; these values weakly depend on the iron concentration) corresponding to the iron atoms Fe²⁺ in a distorted tetrahedral environment of selenium atoms (Fig. 4). The fraction of iron atoms Fe²⁺ grows with the growth in the total iron concentration in the films (Fig. 6).

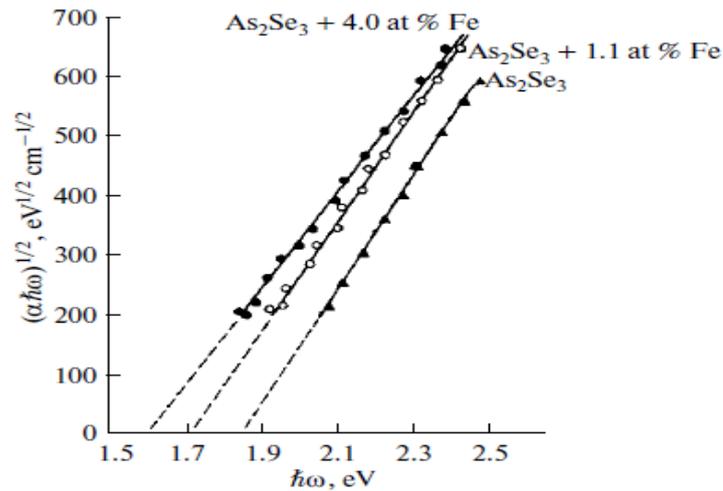


Fig. 3. Optical absorption edge of iron films

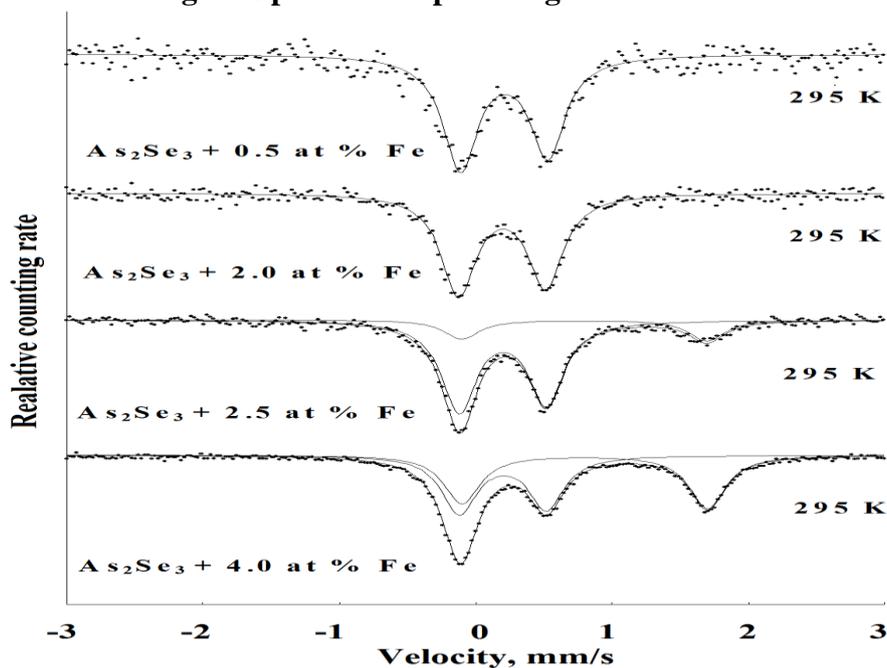


Fig. 4. Mössbauer spectra for ⁵⁷Fe atoms in modified As₂Se₃ films at room temperature in the range of concentrations of 0.5–4.0 at % Fe. The decomposition of the experimental spectra into two quadrupole doublets is shown, which correspond to the Fe³⁺ and Fe²⁺ states

Thus, an introduction of iron into vitreous arsenic selenide leads to a shift of the Fermi level from the band gap middle (for undoped glass) to the conduction band bottom (for doped glass). For undoped glass $E_{\text{opt}} \approx 2E_{\sigma}$ and the current transfer, according to the sign of Seebeck coefficient, is realized by holes. The introduction of iron into glass leads to a change in the sign of the current

carriers (the current transfer, according to the sign of the Seebeck coefficient, is realized by electrons) and to a decrease in the electro conductance activation energy.

Figure 7 presents the dependence of the shift of the Fermi level from the middle of the band gap $\delta E = (E_{\text{opt}})/2 - E_{\sigma}$ on the iron concentration. As can be seen from the figure, when the iron concentration increases, the Fermi level smoothly approaches its limiting position, which is distant from the middle of the band gap by a value of 0.57 ± 0.03 eV.

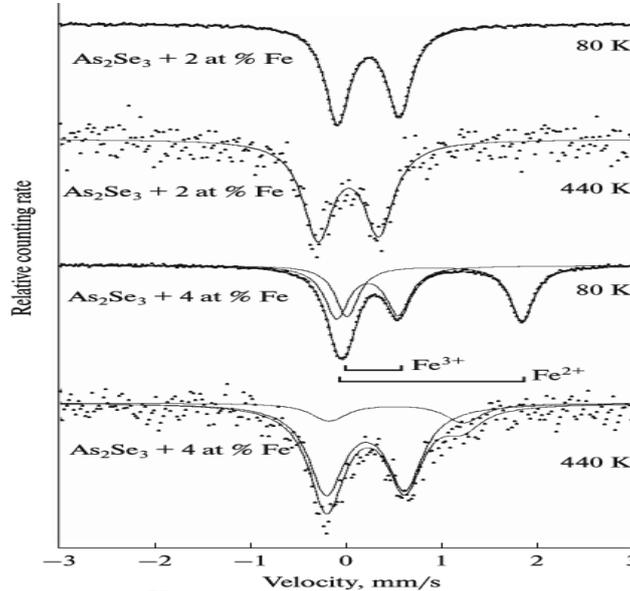


Fig. 5. Mössbauer spectra for ^{57}Fe atoms in modified As_2Se_3 films at 80 and 440 K at a concentration of 2 and 3 at % Fe. The decomposition of the experimental spectra into two quadrupole doublets is shown, which correspond to the Fe^{3+} and Fe^{2+} states

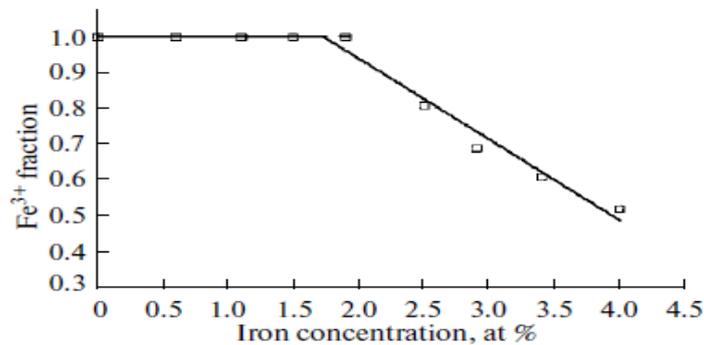


Fig. 6. Dependence of the Fe_t^{3+} fraction on the total iron concentration in the ^{57}Fe Mössbauer spectra of As_2Se_3

In order to explain the experimental data, we should assume that the iron atoms form a single-electron level of a donor type in the band gap. In other words, when an iron atom penetrates into a glass structural network, it forms chemical bonds with the chalcogen atoms using two valence $4s$ electrons coupled by the spin (i.e., the state Fe^{2+} of iron oxidation is formed), while one uncoupled valence $3d$ electron of an iron atom plays the role of a donor electron with an ionization energy of 0.24 ± 0.03 eV (the energy is counted from the bottom of the conduction band) (Fig. 2). If $N_{\text{Fe}} \leq 2$ at %, then all the donor iron centers are ionized and only the iron Fe^{2+} state is observed in the ^{57}Fe Mössbauer spectra (Fig.4). The fraction of the Fe^{2+} ions can be calculated by the following equation:

$$p = \frac{S_n^{80}}{S_n^{80} + S_i^{80}} \quad (1)$$

where S_n^{80} and S_i^{80} are the areas under the Mössbauer spectra recorded at 80 K and corresponding to the Fe^{2+} and Fe^{3+} ions, respectively. In other words, under the conditions of film

preparation, the concentration of acceptor type intrinsic defects in glass, which compensate the donor iron centers by transferring them from the Fe²⁺ state into the Fe³⁺ state, amounts to less than 2 at %. However, these electrons do not enter into the conduction band; instead, they fill local states having a continuous energy distribution from the top of the valence band to the bottom of the conduction band. This results in the shift of the Fermi level from the middle of the band gap to the bottom of the conduction band; this shift is larger the higher the iron concentration.

Naturally, when the Fermi level reaches an iron donor level (this happens at $N_{\text{Fe}} \sim 2$ at %), then a part of the iron centers turns out to be in a nonionized state, and the component corresponding to Fe²⁺ appears in the ⁵⁷Fe Mössbauer spectrum. The iron Fe²⁺ fraction at $N_{\text{Fe}} > 2$ at % will grow with the iron concentration growth, which is observed in the Mössbauer spectra (Figs. 4, 5).

The maximum shift in the Fermi level (~ 0.57 eV) corresponds to the energy position of the level of the iron donor to the middle of the band gap. The monotonic decrease in the conductance activation energy of the iron As₂Se₃ films by the increase of the iron concentration is explained by the shift of the Fermi level described above.

A small decrease in the optical width of the band gap for As₂Se₃ arsenic selenide modified with iron is explained by the formation of a solid solution in glass based on arsenic selenides and iron selenides with one of the solid solution components (iron selenide) being a narrow band gap semiconductor.

Electronic exchange between centers of iron. It is obvious that when the Fermi level is close to the level of iron, then the electron exchange between the two oxidation states of iron (Fe²⁺ and Fe³⁺) is possible, which should give rise to the convergence of the central shift values of the Mössbauer spectra of the Fe²⁺ and Fe³⁺ sites. In the limit of fast electron exchange, when the lifetimes of the Fe²⁺ and Fe³⁺ states are much shorter than the lifetime of the ^{57m}Fe nuclear isomer (~10⁻⁷ s), the Mössbauer spectrum of the averaged state of iron with the following central shift should appear:

$$S = \frac{S_i^{80} + PS_n^{80}}{P+1}, \quad (2)$$

where

$$P = \frac{N_{\text{Fe}^{2+}}}{N_{\text{Fe}^{3+}}} \leq 1, \quad (3)$$

and S_n^{80} and S_i^{80} are the central shifts of the Mössbauer spectra recorded at 80 K and corresponding to the Fe²⁺ and Fe³⁺ sites, respectively.

The temperature dependences of the central shifts of the Mössbauer spectra of Fe³⁺ and Fe²⁺ sites at $N_{\text{Fe}} = 2$ at % (when $P \approx 0$) and $N_{\text{Fe}} = 4$ at % (when $P \approx 1$) are shown in Fig. 8.

For the case when $P \approx 0$, only the quadrupole doublet corresponding to the Fe³⁺ ion is present in the Mössbauer spectrum, the central shift of which in the range of temperatures from 80 to 440 K only slightly decreases with an increase in the temperature, which can be explained by the manifestation of the transverse Doppler effect. In most cases, the central shift, S , of the Mössbauer spectra is determined by the isomeric shift, δ , and the squared Doppler shift, D .

$$S = \delta + D, \quad (4)$$

and the temperature dependence of the central shift is determined by the temperature dependence of the D value, which looks as follows in the Debye approximation [1]:

$$D = -\frac{3}{2} E_0 \frac{k\Delta T}{Mc^2} f\left(\frac{\Delta T}{\theta}\right), \quad (5)$$

where E_0 is the energy of the isomeric transition for the ^{57m}Fe isotope, k is the Boltzmann constant, M is the mass of the irradiating ⁵⁷Fe atom, c is the speed of light in vacuum, θ is the Debye temperature of the investigated material, $f\left(\frac{\Delta T}{\theta}\right)$ is the Debye function, and ΔT is the difference

between the temperatures at which the Mössbauer spectra of the sample are recorded.

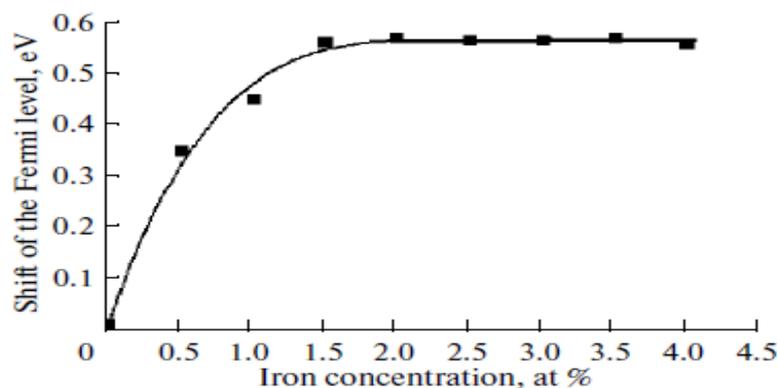


Fig. 7. Dependence of the shift of the Fermi level from the middle of the band gap $\delta E = (E_{opt})/2 - E_{\sigma}$ on the iron concentration

The theoretical temperature dependences of the squared Doppler shift of the Mössbauer spectrum of the ^{57}Fe isotope (the used Debye temperatures are 120 and 200 K) and the experimental temperature dependence of the central shift of the Mössbauer spectrum of the Fe^{3+} sites at $P \approx 0$ are shown in Fig. 9. As is seen from the plots, there is satisfactory agreement between the theoretical and experimental dependences.

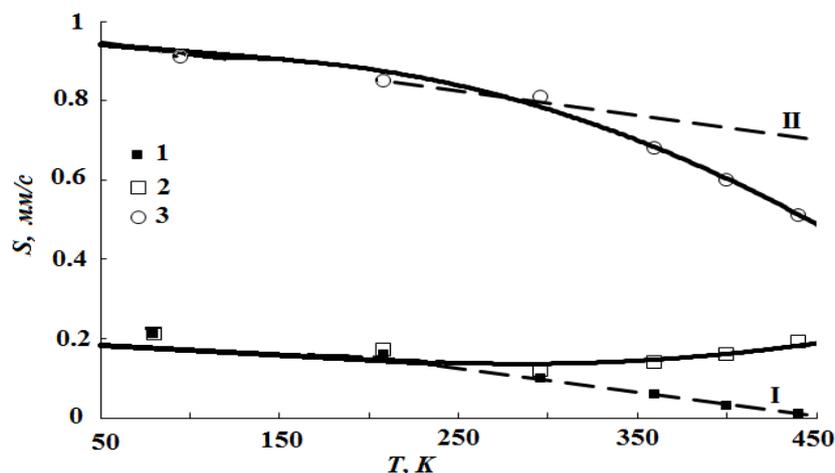


Fig. 8. Temperature dependences of the central shifts of the Mössbauer spectra of Fe^{3+} sites in As_2Se_3 films for an iron concentration of (1) 2 and (2) 4 at %, and (3) of Fe^{2+} sites for an iron concentration of 4 at %; the dashed lines correspond to the temperature dependences of the central shifts of the Mössbauer spectra of the (I) Fe^{3+} and (II) Fe^{2+} sites, which are determined from the temperature dependences of the squared Doppler shifts in the ^{57}Fe Mössbauer spectra

For the case when $P \approx 1$, two quadrupole doublets corresponding to the Fe^{3+} and Fe^{2+} sites are present in the Mössbauer spectrum, the central shifts of which become close to each other with an increase in the temperature (see Fig. 8), the spectral lines are broadened (Fig. 10), and the relative area under the spectrum corresponding to Fe^{2+} sites is decreased (Fig. 11).

The spectra in Fig. 5 and results of their processing in Figs. 8 and 10 illustrate the typical pattern of the electron exchange between the two iron oxidation states, Fe^{2+} and Fe^{3+} . A decrease in the relative area under the spectrum corresponding to Fe^{2+} sites does not indicate a decrease in the concentration of Fe^{2+} sites with an increase in the temperature but a more distinct dependence of the Mössbauer coefficient on the temperature for the compounds of doubly charged iron in comparison with the compounds of Fe^{3+} [1].

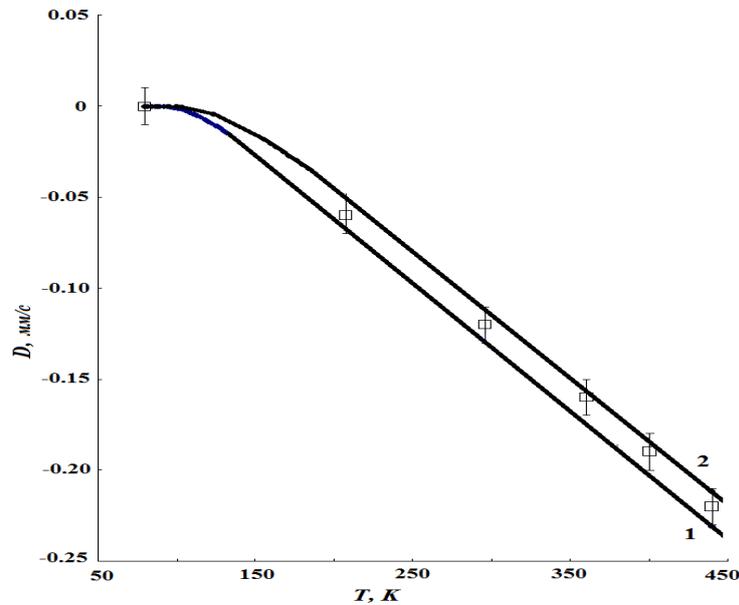


Fig. 9. Theoretical temperature dependences (solid lines) of the Doppler shift of the Mössbauer spectrum on the ^{57}Fe isotope for Debye temperatures of (1) 120 and (2) 200 K and the experimental temperature dependence of the central shift of the Mössbauer spectrum of the Fe_{3+} sites in As_2Se_3 films for an iron concentration of 2 at %

Taking into the consideration that the local symmetry of Fe^{2+} and Fe^{3+} sites is lower than cubic symmetry, it is reasonable to expect the electron exchange to be accompanied by a change in the tensor of the electric field gradient (EFG); i.e., the quadrupole splittings of the Fe^{2+} and Fe^{3+} Mössbauer spectra should change with an increase in the temperature.

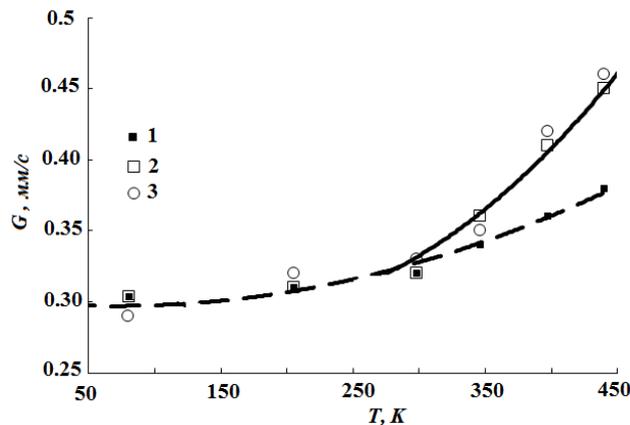


Fig. 10. Temperature dependences of the spectral line widths of the Mössbauer spectra of Fe^{3+} sites in As_2Se_3 films for an iron concentration of (1) 2 and (2) 4 at %, and (3) of Fe^{2+} sites for an iron concentration of 4 at %

Actually, if the local symmetry of the ^{57m}Fe Mössbauer probe is lower than its cubic symmetry, then the energy level of the ^{57m}Fe nucleus decomposes into two sublevels as a result of the interaction of the quadrupole moment, Q , of the iron nucleus with the EFG tensor on it, and the quadrupole splitting of the Mössbauer spectrum arises. The Laplace equation, $U_{xx} + U_{yy} + U_{zz} = 0$, is applicable for a diagonalized EFG tensor, and this EFG tensor is determined by the following two parameters: the main component, U_{zz} , of the EFG tensor and the asymmetry parameter, $\eta = \frac{U_{xx} - U_{yy}}{U_{zz}}$ (here x , y , and z

are the main axes of the EFG tensor, which are chosen based on the inequality $|U_{xx}| \leq |U_{yy}| \leq |U_{zz}|$). The Mössbauer spectrum is represented by a doublet with the following distances between the components:

$$\Delta = \frac{1}{2} eQU_{zz} \left(1 + \frac{\eta^2}{3} \right)^{1/2} \quad (6)$$

In general, there are the following two EFG sources on the atomic nuclei: the ions of the structural lattice of glass (they produce crystalline EFG) and no spherical valence electrons of the Mössbauer atom (they produce valence EFG). Accordingly,

$$U_{zz} = (1 - \gamma)V_{zz} + (1 - R_o)W_{zz}, \quad \eta = \frac{(1 - \gamma)V_{zz}\eta_{cr} + (1 - R_o)W_{zz}\eta_{val}}{U_{zz}}, \quad (7)$$

where U_{zz} , V_{zz} , and W_{zz} are the main components of the total, crystalline, and valence EFG tensors, respectively; η , and η_{cr} , η_{val} are the asymmetry parameters of these tensors; γ and R_o are the Sternheimer coefficients (they take into account the shielding and deshielding of EFG produced by the external charges and by the internal electron shells of the probe atom).

If the valence shells of the Mössbauer atom are completely or half-filled (the Fe^{3+} ion is a typical example), then only the charges of neighbor atoms should be taken into account for such an atom upon the calculations of EFG; the Δ value in this case is insignificant, and its temperature dependence is determined by the temperature dependences of the interatomic distances in the crystal and in the structural lattice of glass, and this dependence is very weak as a rule. However, if the valence shells of the Mössbauer atom are filled only partially (Fe^{2+} is a typical example), then the valence electrons are the main source of EFG; therefore the Δ value is significantly increased, and it noticeably decreases upon an increase in the temperature [1].

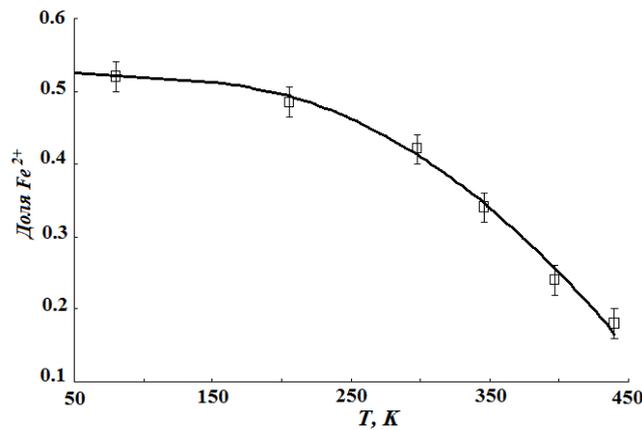


Fig. 11. Temperature dependence of the fraction of Fe^{2+} sites in the Mössbauer spectra of $\text{As}_2\text{Se}_3: {}^{57}\text{Fe}$ at $N_{\text{Fe}} = 4$ at %

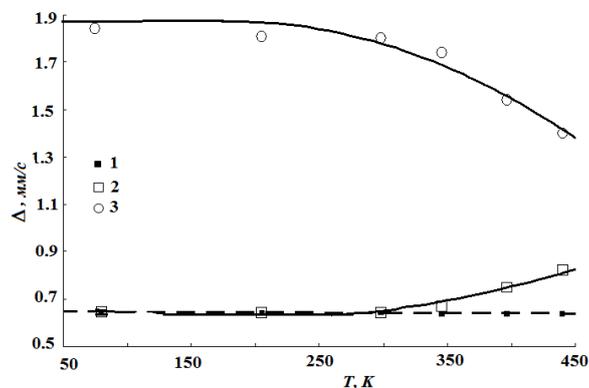


Fig. 12. Temperature dependences of the quadrupole splitting of the Mössbauer spectra of Fe^{3+} sites in As_2Se_3 films for an iron concentration of (1) 2 and (2) 4 at %, and (3) of Fe^{2+} sites for an iron concentration of 4 at % (3)

The temperature dependences of the quadrupole splittings of the ${}^{57}\text{Fe}$ Mössbauer spectra corresponding to the Fe^{2+} and Fe^{3+} sites are shown in Fig. 12. It is seen that a small value of the

quadrupole splitting, Δ_i , and its insignificant decrease with an increase in the temperature are observed in the range of temperatures from 80 to 400 K for the case with an iron concentration of less than 2 at % (iron is stabilized only in the Fe³⁺ state), as expected for the triply-charged Fe³⁺ state of iron. In the case with an iron concentration of 4 at % (iron is stabilized in the two equally populated Fe²⁺ and Fe³⁺ states), small values of the quadrupole splitting, Δ_i , and their decrease with an increase in the temperature are observed in the Mössbauer spectra of the Fe³⁺ sites in the range of temperatures from 80 to 350 K, whereas the Δ_i value starts to increase anomalously upon a further increase in the temperature. For the spectra of Fe²⁺ sites, the quadrupole splitting value, Δ_n , for the same samples is significantly larger, and the rate of its decrease upon an increase in the temperature has the following two areas: the area at the temperatures ranging from 80 to 350 K, in which the quadrupole splitting considerably decreases, and the area at $T > 350$ K, in which this decrease becomes more rapid.

Thus, the quadrupole splitting values of the Mössbauer spectra of the Fe³⁺ and Fe²⁺ sites in the films of arsenic selenide glass approach each other upon an increase in the temperature, which indicates that the EFG tensor parameters produced on the atomic nuclei of the neutral and ionized iron donor centers become closer.

Conclusions. In glass films of arsenic selenide modified by iron, the Fe²⁺ and Fe³⁺ sites corresponding to the neutral and ionized states of the one-electron donor iron center are detected. Both the central shift and quadrupole splitting values of the Mössbauer spectra of the Fe³⁺ and Fe²⁺ sites become closer upon an increase in the temperature, which indicates that the electronic densities and EFG tensor parameters on the atomic nuclei of the neutral and ionized iron donor centers are equalized.

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ЭЛЕКТР ТАЪМИНОТИ ТИЗИМИДА СТАБИЛЛОВЧИ ҚУРИЛМАЛАРНИ ТАҲЛИЛ
ВА ТАДҚИҚ ҚИЛИШ

АНАЛИЗ И ИССЛЕДОВАНИЕ СТАБИЛИЗИРУЮЩИХ УСТРОЙСТВ
В СИСТЕМЕ ЭЛЕКТРОСНАБЖЕНИЯ

ANALYSIS AND RESEARCH OF STABILIZING DEVICES IN THE
POWER SUPPLY SYSTEM

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Аннотация. Мақолада электр таъминоти тизимида электр энергиясининг асосий сифат кўрсаткичларидан кучланишининг огиши ва стабиллигини яхшилаш масаласи кўриб чиқилган. Шундан келиб чиқиб, кучланишининг огиши ва стабиллигини таъминлаш мақсадида стабилловчи қурилмаларнинг иши таҳлил ва тадқиқ қилинган. Стабилловчи қурилмаларнинг