
SIZE EFFECTS IN CHLORINE DOPED *PbSe* THIN FILMS

*The possibility of obtaining strongly degenerate ($\approx 3 \cdot 10^{20} \text{ cm}^{-3}$) *PbSe* thin films ($d = 5 - 220 \text{ nm}$) with *n*-type conductivity by thermal evaporation in vacuum of *PbSe* crystals doped with PbCl_2 , with subsequent condensation onto (001) *KCl* substrates was established. It was shown that the films had high homogeneity degree, no grain structure was observed. The thickness dependences of thermoelectric properties (the Seebeck coefficient S , the Hall coefficient R_H and the electric conductivity σ) of thin films were obtained. In the thickness range $d \approx 5 \div 30 \text{ nm}$, oscillation properties were observed with growth of d that are attributable to electron gas quantization. The calculation of $S(d)$ dependence on the assumption of size quantization with regard to contribution of several subbands and the thickness dependence of the Fermi energy was shown to be in agreement with the experimental data. In the region of $d > 30 \text{ nm}$ there was growth of S and σ with thickness, which is attributable to manifestation of classical size effect and interpreted in the framework of Fuchs-Sondheimer and Mayer theories.*

Key words: lead selenide, thin film, thickness, size effect.

Introduction

As is known, the range of application of IV-VI semiconductors is very wide: IR engineering [1], lasers [2], solar cells [3], thermoelectricity (TE) [4] and other fields of science and technology [5]. Lead selenide (*PbSe*) has proved itself as material used in thermoelectric power converters (thermal generators) [6]. Efficient use of *PbSe* is possible at high concentrations of charge carriers ($n \sim 2 \cdot 3 \cdot 10^{19} \text{ cm}^{-3}$) which do not seem possible to be obtained due to deviations from stoichiometry. In this connection, *PbSe* is doped with different impurities. Introduction of chlorine, iodine, sodium, bismuth into *PbSe* lattice allows achieving concentration values up to $\sim 10^{20} - 10^{21} \text{ cm}^{-3}$ [6].

Increase in TE figure of merit of material which is in a low-dimensional state was experimentally shown for superlattices based on IV-VI compounds [7, 8]. With practical application of thin films, it is important to take into account the influence of size effects (classical and quantum) that can change drastically the kinetic properties of material [9].

Quantum size effects (QSE) manifested in the oscillations of galvanomagnetic and TE properties with growth in film thickness, were observed in *PbX* films ($X = S, Se, Te$) with *n*-type conductivity and concentration $\sim 10^{18} \text{ cm}^{-3}$ [10-13] and in *PbTe* films with high degeneracy degree of the hole ($p \sim 10^{19} \text{ cm}^{-3}$) [14] and electron ($n \sim 10^{20} \text{ cm}^{-3}$) charge carrier gases [15]. For *PbSe* films obtained by thermal evaporation in vacuum of crystals of stoichiometric *PbSe* with hole concentration $p \sim 10^{18} \text{ cm}^{-3}$, there was increase in the electric conductivity and mobility with growth of film thickness, which was attributed to manifestation of classical size effect (CISE) [16, 17]. By example of *PbSe* films the possibility of a simultaneous investigation of CISE and QSE on the same object by changing film thickness was shown [17].

Few works are concerned with the investigation of the thickness dependences of TE properties of *PbSe* films with a high degree of electron gas degeneracy. In [18] we showed that chlorine-doped *PbSe* films show a complicated dependence of TE properties on film thickness, and an assumption was made on the superposition of QSE and CISE.

The purpose of this paper is to study in more detail the influence of thickness d of *PbSe* films with a high degree of electron gas degeneracy ($n = 3 \cdot 10^{20} \text{ cm}^{-3}$) on their TE properties at room temperature.

Procedure

PbSe films with thicknesses $d = 5\text{-}220 \text{ nm}$ were obtained by the method of thermal evaporation in vacuum ($10^{-5}\text{-}10^{-6} \text{ Pa}$) of *PbSe* crystals doped with 2 mol.% *PbCl₂*, with subsequent condensation onto (001) *KCl* cleavages, maintained at a temperature of $(520 \pm 10) \text{ K}$. Using electron beam method, the films were coated by *Al₂O₃* layer 20-25 nm thick. The thickness d of the films was controlled with the aid of precalibrated quartz resonator.

Surface morphology and chemical composition of the films were studied by means of electron probe microanalysis with the use of scanning electron microscope JSM-6390 LV (Jeol, Japan) with a system of energy-dispersive spectrometer X-Maxⁿ 50 (Oxford Inst., United Kingdom) with accelerating voltage 10 kV. Surface morphology and average roughness were studied by means of atomic force microscope (AFM) Solver Pro NT-MDT with Nova software, the images were obtained in the air in contact mode.

The electric conductivity σ and the Hall coefficient R_H were measured by standard *dc* method with an error not exceeding 5 %. As material for soldering of contacts, use was made of indium. The Hall concentration of charge carriers n was calculated on the assumption of one sort of carriers by the formula $n = r/R_H e$, where the Hall factor $r = 1$, e is electron charge. The Hall mobility μ_H was calculated as $\mu_H = R_H \cdot \sigma$. The Seebeck coefficient S was measured by the compensation method with respect to copper to an accuracy of $\pm 3 \%$. The type of charge carriers was determined by the sign of R_H and S .

Theoretical calculation for the description of oscillating character of $S(d)$ dependence was made with the use of mathematical package Maple 15; dependences of S and σ on the thickness of films were calculated in the framework Fuchs-Sondheimer and Mayer theories with the use of mathematical package MatLAB 6.5.

Results

Measurements of R_H and S have shown that the films possess *n*-type conductivity over the entire range of studied thicknesses, just as *PbSe*<*PbCl₂*> crystal which was used as a charge for preparation of films. For the crystal the following values of kinetic coefficients were obtained: $S = 20 \text{ } \mu\text{V/K}$, $\sigma = 4600 \text{ (}\Omega\text{m}\text{)}^{-1}$, $n = 3 \cdot 10^{20} \text{ cm}^{-3}$, $\mu_H = 100 \text{ cm}^2/(\text{V}\cdot\text{s})$. In the films one succeeded to reach 2-4 fold values of n and the values of mobility comparable to crystal μ_H . The electron conductivity type and high values of carrier concentration result from the introduction of chlorine into *PbSe* lattice and agree with the data reported in the literature [6].

Electron probe microanalysis of films surface in the mode of scanning over the area and probing by points has shown that the films possessed high degree of homogeneity on the microlevel (the ratio between the elements (*Pb*, *Se*, *Cl*) from one analyzed area to another was retained within the error of

the method). Studies in the mode of secondary electrons (resolution 10 nm) have shown that no grain structure was observed in the films and the surface was homogeneous (Fig. 1).

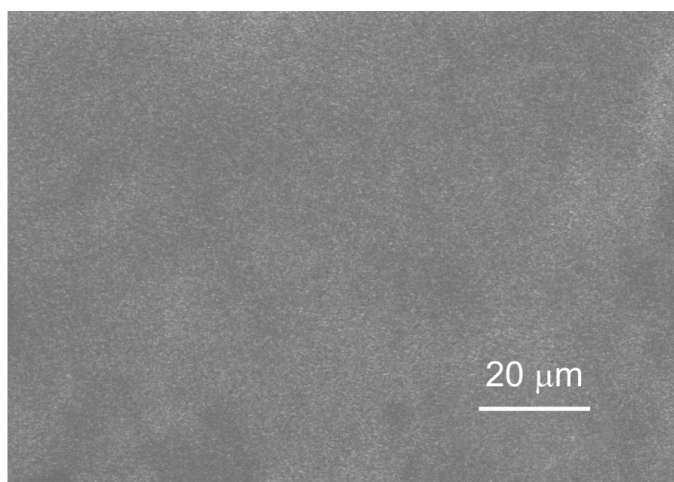


Fig. 1. Surface micro image of film with thickness $d = 220$ nm.

The results of AFM investigation of the films surface are given in Fig. 2. As is seen, the surface of the films is mostly smooth, though one can observe elongated asperities along $\langle 100 \rangle$ direction, with a flat apex (Fig. 2). The height of asperities was ≈ 12 - 18 nm, the base width did not exceed $40 \div 60$ nm, and the length – $170 \div 240$ nm. The average distance between the asperities was ≈ 350 nm. Note that the value of average roughness in the area between the asperities was $R_a \approx 1.03$ nm. One of possible reasons for the appearance of asperities can be inheritance by $PbSe\langle PbCl_2 \rangle$ layer of substrate surface, owing to which along the surface steps there appear dislocations penetrating the film and outcropping to the surface as asperities.

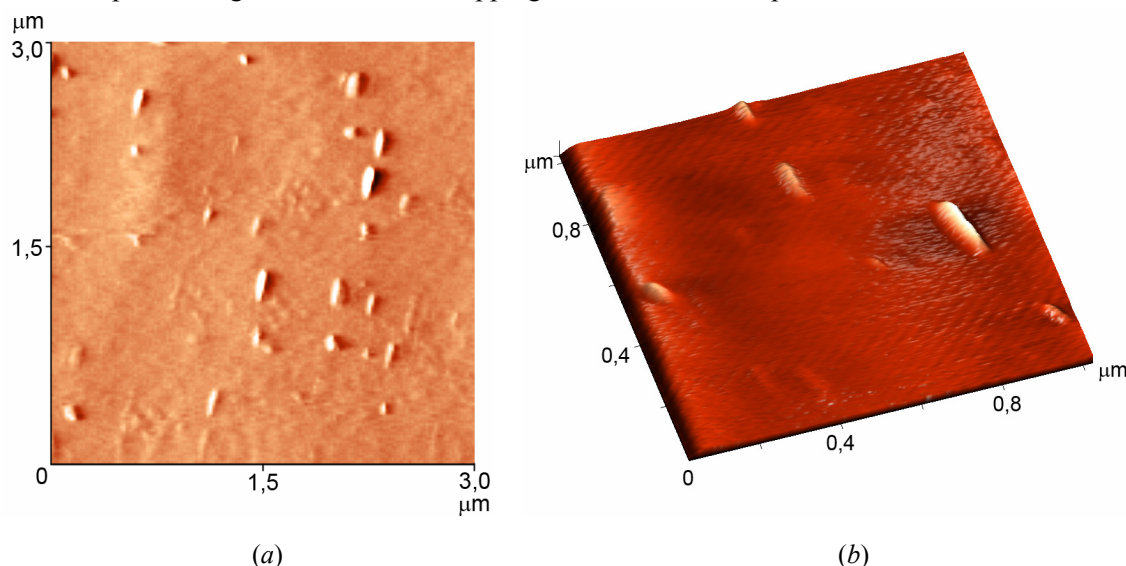


Fig. 2. AFM-topography (a) and 3D AFM surface image (b) of film with thickness $d = 56$ nm.

It is known that in lead chalcogenides films grown in alkali-halide crystals the dominant effects are dislocations sliding in (100) plane [19, 20]. According to the data from transparent electron microscopy, the dislocation density in $PbTe$ films grown on KCl cleavages is generally equal to $\approx 10^8 \text{ cm}^{-2}$ [19]. Assuming that elongated along $\langle 100 \rangle$ direction structures in Fig. 2, a are outcropping dislocations, one can determine their density. Evaluation of the density of dislocations outcropping to film surface yields the value $\approx 2.5 \cdot 10^8 \text{ cm}^{-2}$ which is in good agreement with the literature data [20].

Thus, it can be assumed that the asperities on the surface of films are caused by dislocations appearing along the surface steps due to inheritance of substrate surface, arranged at quite a distance from each other (≈ 350 nm). Nevertheless, $PbSe\langle PbCl_2 \rangle$ films are characterized by high level of homogeneity (Fig. 1), smooth surface and absence of macrodefects (cracks and pores, etc.).

Fig. 3 shows dependences of S , σ and R_H on the thickness of films at room temperature, black squares are used to mark the values of kinetic coefficients of crystal which served as a charge for preparation of films. On dependences $S(d)$, $R_H(d)$ and $\sigma(d)$ one can see extremes at thicknesses 8, 11, 15, 18 and 23 nm, the maxima on the curve $\sigma(d)$ corresponding to minima on dependences $S(d)$ and $R_H(d)$ and vice versa. The average distance between the two neighbouring minima or two maxima is $\Delta d = (7 \pm 1)$ nm. In the area $d > \sim 30$ nm the kinetic coefficients are monotonically increased with film thickness and tend to the values S , R_H and σ of the crystal.

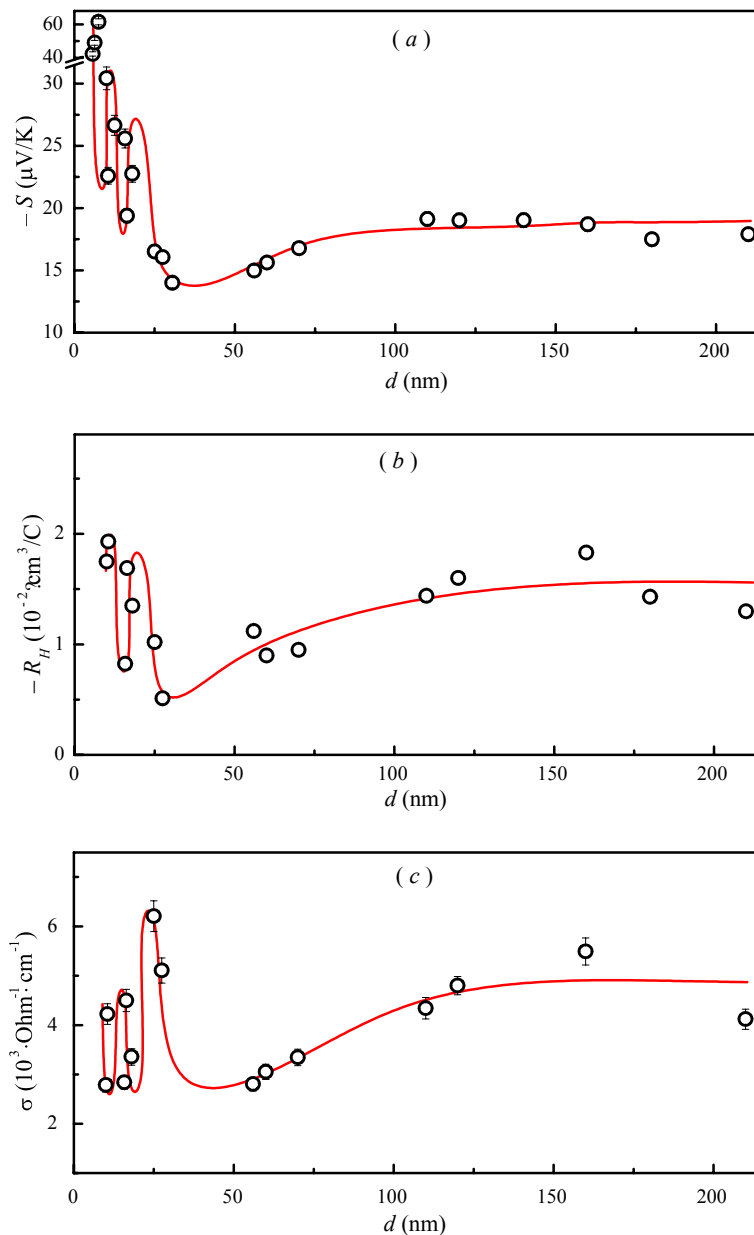


Fig.3. Dependences of the Seebeck coefficient S (a), the Hall coefficient R_H (b) and the electric conductivity σ (c) on the thickness of $PbSe\langle PbCl_2 \rangle$ films.

Rather high structural perfection of the films, their homogeneity and smooth surface suggest that the reason for oscillating character of change in galvanomagnetic and thermoelectric properties of films with growth of d can be QSE.

The $PbSe\langle PbCl_2 \rangle$ film can be presented as a quantum well located between two insulators – KCl substrate and Al_2O_3 protective coating. Electron movement in the direction perpendicular to film surface is restricted, which results in quantization of quasi pulse transverse component and formation of two-dimensional energy subbands. In the other two directions electron quasi-pulse is not quantized. On the assumption that all charge carriers occupy only the lower subband [21] in the approximation of effective mass, assuming square dispersion law, the energy of carriers in quantum size film can be written as follows:

$$E = \frac{\hbar^2 \pi^2}{2m_{\perp}^* d^2} n^2 + \frac{\hbar^2 k_x^2}{2m_x^*} + \frac{\hbar^2 k_y^2}{2m_y^*}, \quad (1)$$

where m_{\perp}^* is effective mass of charge carriers along the direction perpendicular to quantum well, k_x , k_y and m_x^* , m_y^* are components of wave vector and effective mass, respectively, with motion of charge carriers parallel to quantum well, n is quantum number ($n = 1, 2, \dots$). With increase in d , two-dimensional subbands will consecutively cross the Fermi level ε_F , which will involve the oscillating character of change in the density of states. Density of states oscillations will lead to the oscillations of kinetic properties with the period [9]:

$$\Delta d = \frac{h}{\sqrt{8m_{\perp}^* \varepsilon_F}} \quad (2)$$

Thickness d_1 whereby the first subband ($N = 1$) crosses ε_F , coincides with the value Δd [22], so to determine the oscillation period, it is enough to record the position of the first extremum on the thickness dependences of transport properties.

As is known, QSE can be manifested, provided the following conditions are met [9]:

- 1) the distance between the energy subbands ΔE , determined as $\Delta E = \frac{\hbar^2}{8m_{\perp}^* d^2} (2n+1)$, must exceed thermal blurring of the levels $\Delta E \geq k_B T$ (k_B is the Boltzmann constant);
- 2) charge carrier scattering partially blurs quasi-discrete spectrum, so to preserve the quasi-discrete character of spectrum, blurring \hbar/τ (τ is relaxation time) must be smaller than the distance between neighbouring subbands $\Delta E > \frac{\hbar}{\tau} = \frac{\hbar \cdot e}{2\pi \cdot m \cdot \mu}$;
- 3) it is necessary to have high quality of surfaces restricting the motion of carriers in quantum wells and reflection specularity, i.e. roughness dimensions must be smaller than the de Broglie wavelength λ_F ;
- 4) nanocrystallite size must exceed thickness d , otherwise charge carriers on grain boundaries will be scattered, and QSE observation will be impossible.

It can be easily demonstrated that in the films under study these conditions are realized. For instance, for n - $PbSe$ ($m_{\perp}^* = 0.04 m_0$ [6]) film of thickness $d = 10$ nm the value ΔE at $n = 1$ is $\Delta E \approx 283$ meV, and thermal blurring of subbands at room temperature $k_B T = 25.8$ meV, which indicates to fulfillment of the first condition. Rather high values of charge carrier mobility in thin films comparable to the values of μ in crystal, point to the possibility of fulfillment of the second condition. De Broglie wavelength ($\lambda_F = 2\Delta d$) exceeds the value of films roughness ($R_a \approx 1.03$ nm), determined by means of AFM, showing sufficiently high surface quality. The absence of grain structure,

confirmed by the results of probe microanalysis, also makes possible the fulfillment of the last condition. The foregoing indicates the possibility of consideration and interpretation of the observed oscillations within the concepts of QSE.

The calculation of quantum oscillation period by formula (2) with regard to the value of transverse component of electron effective mass in *PbSe* [6] and ε_F , determined by the R_H values of crystal, yielded the value $\Delta d = (3 \pm 0.5)$ nm. The resulting value Δd proved to be almost half the experimentally observed average distance between the nearest minima (or maxima) on d -dependences of kinetic coefficients ($\Delta d = 7 \pm 1$ nm). Such a difference can be due to simplifications used in the model of a rectangular potential well with infinitely high walls. Moreover, in the calculation it was assumed that the contribution to kinetic coefficients is only from charge carriers occupying the lower subband, and that the Fermi energy does not depend on the film thickness.

To specify the value Δd , calculation was made with regard to contribution of several energy subbands to the value of the Seebeck coefficient. Using the assumption of the energy independence of charge carrier relaxation time, the expression for the Seebeck coefficient S in a two-dimensional system can be written as follows [23]:

$$S = \frac{1}{eT} \left(\varepsilon_F - \frac{\sum_{n=1}^{E_n < \varepsilon_F} \int_0^{\varepsilon} E \varepsilon \left(\frac{\partial f}{\partial \varepsilon} \right) d\varepsilon}{\sum_{n=1}^{E_n < \varepsilon_F} \int_0^{\varepsilon} \varepsilon \left(\frac{\partial f}{\partial \varepsilon} \right) d\varepsilon} \right), \quad (3)$$

where f is the Fermi distribution function, $\varepsilon = E - E_n$, $E_n = \frac{\pi^2 \hbar^2}{2m_{\perp}^* d^2} N^2$. On rearrangement, the expression for S takes on the form:

$$S = \frac{k_B}{e} \left[\xi - \frac{\beta + \gamma}{\alpha} \right], \quad (4)$$

where $\alpha = \sum_{n=1}^{E_n \leq \varepsilon_F} (\ln z - y)$; $\beta = b \cdot \sum_{n=1}^{E_n \leq \varepsilon_F} n^2 \cdot (\ln z - y)$; $\gamma = \sum_{n=1}^{E_n \leq \varepsilon_F} \left(2 \cdot d i \log z + y^2 + \frac{\pi^2}{3} \right)$, $z = 1 + e^{bn^2 - \xi}$,

$y = bn^2 - \xi$, $b = \frac{1}{k_B T} \frac{\pi^2 \hbar^2}{2m_{\perp}^* d^2}$, $\xi = \frac{\varepsilon_F}{k_B T}$. Note that in Eq. (4) the summation is done over several energy

subbands, and it is also assumed that the value of ε_F does not depend on the film thickness.

An assumption of the constancy of ε_F with growth of d is not precise: according to [24], the Fermi energy in 2D-degenerate gas of charge carriers oscillates with thickness. Depending on d and the number of filled levels N in the potential quantum well, on fulfillment of condition $E_N \leq \varepsilon_F \leq E_{N+1}$ the value ε_F is of the form [25]:

$$\varepsilon_F = \varepsilon_F^0 \cdot \frac{4}{3} \left[\frac{d}{\lambda_F N} + \frac{\lambda_F^2 (N+1)(2N+1)}{32 d^2} \right], \quad (5)$$

where ε_F^0 is the value of Fermi energy in the bulk of the crystal, where dimensional quantization is inessential. Note that N is maximum number of a subband located below ε_F and depends on d and ε_F as

$$N = \frac{d \sqrt{8m_{\perp}^* \varepsilon_F(d)}}{h}.$$

The result of calculation of $S(d)$ dependence with the use of Eqs.(3)-(5) is given in Fig. 4, *a* (solid blue curve). As the input parameters, the values of effective masses of charge carriers in *n-PbSe*

and crystal electron concentration were used. As can be seen from Fig. 4, *a*, the value S oscillates with the period $\Delta d = (5 \pm 0.5)$ nm, which is close to experimental Δd . The insert in Fig. 4, *a* shows for comparison the experimental points and the theoretical curve. It is worthwhile to emphasize that account of all subbands located below ε_F , and of dependence $\varepsilon_F(d)$ in the calculation of the Seebeck coefficient yields a higher value of Δd , close to experimental, unlike the calculation that takes into consideration carriers that occupy only lower subband. Still, small number of experimental points in the area with $d < \sim 25$ nm prevents from speaking about full coincidence between calculation and experiment, though experimental results do not contradict theoretical calculation even in S value.

From Fig. 4, *a* it is evident that the amplitude of quantum oscillations S decays very fast with thickness, and at $d > \sim 25$ -30 nm one cannot describe dependence $S(d)$ in the approximation of dimensional quantization. Smooth growth of S and σ with thickness in the area of $d > 30$ nm can be related to manifestation of classical size effect. Taking into account high degree of electron gas degeneracy in the films, an attempt was made to describe the results in the framework of Fuchs-Sondheimer theory [26] and Mayer theory [27].

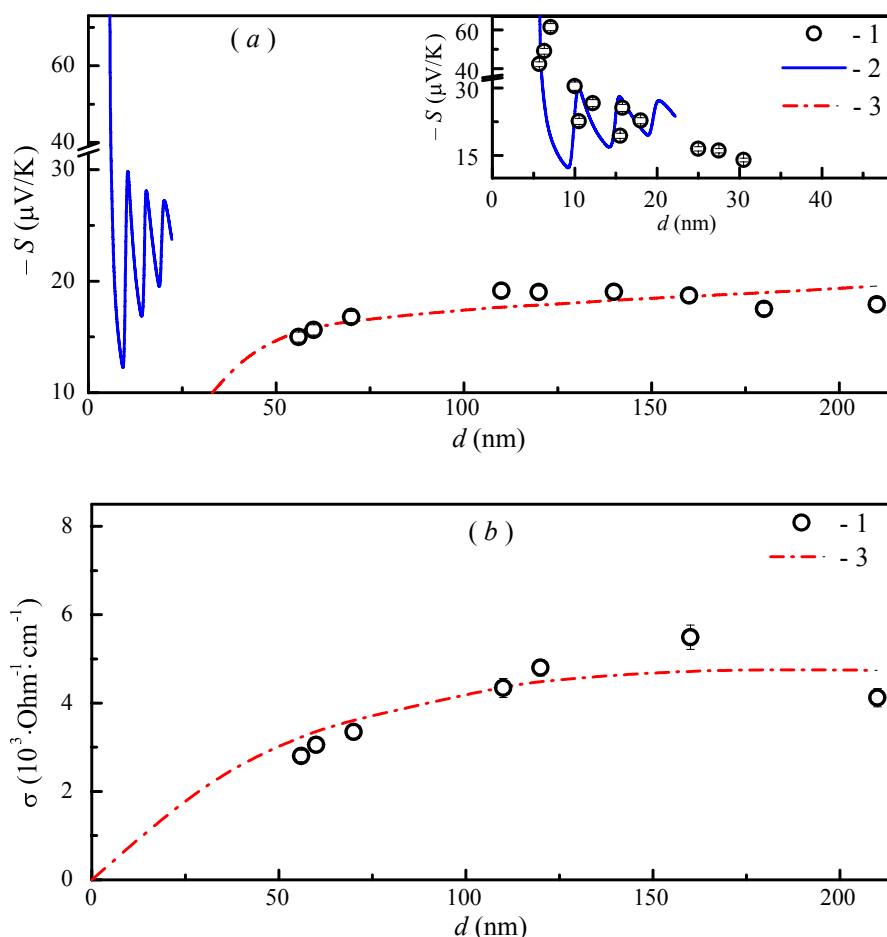


Fig. 4. Theoretical calculation of the dependence of the Seebeck coefficient S (a) and the electric conductivity σ (b) on the thickness of films: 1 – experimental data; 2 – calculation by formulae (3)-(5); 3 – calculation by formulae (6) and (7).

As a model, Fuchs-Sondheimer theory considers a metal with a spherical Fermi surface and isotropic carrier mean free path l independent of film thickness d . Specularity parameter p is introduced which determines the share of electrons elastically reflected from the surface, equal for

both surfaces, independent of d , the trajectory and incidence angle of electrons on the surface. With fully diffused scattering $p = 0$, and with fully specular $p = 1$.

In the approximation of thin films ($d \ll l$) the expressions for σ and S (at $p \sim 0$) are of the form [26, 27]:

$$\sigma_d = \sigma_\infty \cdot \frac{3}{4} \cdot \frac{1+p}{1-p} \cdot \frac{d}{l} \ln\left(\frac{l}{d}\right), \quad (6)$$

$$S = S_\infty \left(1 + \frac{U}{1+U} \cdot \frac{\ln(d/l) - 1.42}{\ln(d/l) - 0.42} \right), \quad p \sim 0, \quad (7)$$

where σ_∞ , S_∞ are the values of σ and S in a sample of infinitely large thickness, and parameter $U = \left(\frac{\partial \ln l}{\partial \ln E} \right)_{E=\varepsilon_F}$ characterizes the energy dependence of l . With a square dispersion law $U = 2$. As σ_∞ and S_∞ , we took the values of kinetic coefficients of the crystal of which the films were prepared. Using formulae (6) and (7), the theoretical dependences $S(d)$ and $\sigma(d)$ were constructed (Fig. 4, curve 3) and the values p and l were determined whereby the best agreement is observed between the experimental data and the theoretical curve, determined by the lowest value of mean-square deviation. It turned out that dependence $\sigma(d)$ is best described at $p = 0.58 \pm 0.02$ and $l = (490 \pm 40)$ nm, and dependence $S(d)$ – at $l = (380 \pm 40)$ nm. Note that for films of undoped *PbSe* the calculation of dependence $\sigma(d)$ by formula (6) yielded the following values of p and l : $p = 0.57 \pm 0.02$ and $l = (800 \pm 40)$ nm [17]. It can be seen that the calculated values of l in *PbSe*<*PbCl*₂> films proved to be considerably lower than for the undoped *PbSe* films which is naturally attributable to the presence of considerable amount of chlorine impurity in the films under study.

Conclusions

The method of thermal evaporation in vacuum of *PbSe* crystals doped with *PbCl*₂ on (001) *KCl* substrates was used to grow thin films of thicknesses $d = 5-220$ nm, having *n*-type conductivity.

By the methods of electron probe microanalysis and atomic force microscopy it was established that high degree of homogeneity is observed in the films, the surface is mostly smooth, grain structure is not manifested.

The oscillating character of dependences of the Seebeck coefficient S , electric conductivity σ and the Hall coefficient R_H on film thickness d was established in the range of $d \approx 5 \div 30$ nm, which is related to manifestation of QSE. The experimental data are in agreement with the theoretical calculation of dependence $S(d)$ on the assumption of dimensional quantization with regard to contribution of several energy subbands and dependence of the Fermi energy on d both in the value of oscillation period and the values of S . The possibility of observation of electron spectrum quantization in *n-PbSe* with high degree of charge carrier degeneracy at room temperature was shown.

Increase in S and σ with film thickness in the area $d > 30$ nm was discovered, which is attributed to manifestation of CISE. Interpretation of $S(d)$ and $\sigma(d)$ dependences was given in the framework of Fuchs-Sondheimer and Mayer theories.

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