## SIZE EFFECTS IN THIN PbSe FILMS

E.I. Rogacheva<sup>1</sup>, O.N. Nashchekina<sup>1</sup>, S.I. Ol'khovskaya<sup>1</sup>, M.S. Dresselhaus<sup>2</sup> (<sup>1</sup>National Technical University "Kharkov Polytechnic Institute", 21, Frunze Str., Kharkov, 61002, Ukraine; <sup>2</sup>Massachusetts Institute of Technology, 77, Massachusetts Ave., Cambridge, MA 02139, USA)

• The objects of the study are thin PbSe films with thicknesses d in the range of d = 5.5 - 410 nm, grown by thermal evaporation in vacuum of stoichiometric p-PbSe crystals on KCl substrates and covered with a EuSe layer. The room-temperature d-dependences of the Seebeck coefficient, Hall coefficient, electrical conductivity, charge carrier mobility, thermoelectric power factor are obtained. When d increases to ~ 20 nm, an inversion of the conductivity sign from p to n is observed. In the d-dependences of the transport properties one can isolate a monotonic and oscillatory components, whose presence is attributed to the manifestation of classical and quantum size effects, respectively. The oscillation periods  $\Delta d$  for electronic and hole gases are determined. Theoretically calculated  $\Delta d$ , assuming a size quantization of the electronic and hole spectra, and our estimate of the monotonic component of the electrical conductivity, using the Fuchs-Sondheimer theory, are in good agreement with the experimental data.

#### Introduction

It is known that in thin films the classical size effect (CSE) connected with the diffuse scattering of charge carriers at the surface and leading to a dependence of properties on the film thickness *d* is usually observed [1]. On the other hand, charge carrier confinement in the direction perpendicular to the film surface results in the quantization of the energy spectrum and leads to the quantum size effects (QSE) when *d* and the de Broglie wavelength  $\lambda_D$  of charge carriers are of the same order of magnitude. One of the possible manifestations of the QSE in 2D-structures is an oscillatory behavior of the *d*-dependences of the transport properties. Metallic films are not very suitable objects for studying QSE through measuring the transport properties because the oscillation period  $\Delta d$  is comparable to interatomic distances, which requires growing very thin films with a high degree of structural perfection. Semiconductors, which have significantly larger  $\lambda_D$ , represent more convenient systems for such studies; nevertheless up to now the number of investigations in that respect has been very limited. There are practically no studies in which the CSE and the QSE were investigated simultaneously.

A sufficiently small effective mass in the direction perpendicular to the direction of growth, with transport along the film surface, and high charge carrier mobility  $\mu$  [2] make IV-VI compounds suitable materials for studying QSE through investigating transfer phenomena. Earlier, for the first time we observed (see, for example, [3-7]) thickness oscillations of the galvanomagnetic and thermoelectric properties in IV-VI quantum wells (QW) and attributed them to the QSE.

Lead selenide, a representative of IV – VI compounds, is widely used in thermoelectricity and other spheres of science and technology [2, 8]. *PbSe* crystallizes in a *NaCl* structure and has rather wide two-sided homogeneity region (49.96 – 50.08 at.% *Se* at 1170 K) [9]. For thin film applications, it is necessary to take into account size effects, which can drastically change the properties of a material. In [3], we reported the observation of quantum *d*-oscillations in *n*-type thin *PbSe* films prepared from *n-PbSe* crystals and protected from oxidation by a *EuS* layer. In [10], where the objects of study were thin films grown from stoichiometric *p-PbSe* crystals on *KCl* substrates and covered by *EuSe*, the CSE was observed (with increasing *d*,  $\sigma$  and  $\mu$  also increased), and it was shown that the monotonic component of the  $\sigma$  (*d*)

dependence can be satisfactorily approximated by the classical Fuchs - Sondheimer theory [11, 12].

The goal of the present work is to conduct a detailed study of the thickness dependences of transport properties of thin films grown on KCl substrates by thermal evaporation of stoichiometric *p*-*PbSe* crystals in vacuum with a view to differentiating the influence of classical and quantum size effects.

As a result of this study, it was shown that the thickness dependences of the transport properties of *PbSe* films exhibit distinctly non-monotonic behavior, which is caused by at least three factors, specifically, the classical size effect, quantum size effect, and the structure factor. The thickness ranges were determined in which the influence of each of the above mentioned factors is dominating.

# **Experimental details**

*PbSe* epitaxial layers with various thicknesses (d = 5.5 - 410 nm) were prepared by thermal evaporation of stoichiometric *p-PbSe* crystals in vacuum  $(10^{-5} - 10^{-6})$  Pa and the subsequent deposition of *p-PbSe* onto a (001) *KCl* surface at  $(520 \pm 10)$  K. The condensation rate was 0.1 - 0.3 nm/s. Using electron-beam evaporation, the films were covered with a ~ 30 nm thick protective layer of a wide-gap semiconductor *EuSe*. This layer, on the one hand, protected *PbSe* thin layers from oxidation and, on the other hand, acted as a barrier layer, making a negligible contribution to the conductivity. The film thickness was controlled with the help of a quartz resonator calibrated either by using an interferometer (for greater *d*) or using small angle *X*-ray diffraction techniques (for smaller *d*). The Hall coefficient  $R_H$  and  $\sigma$  were measured using a conventional dc method with an error not exceeding  $\pm 5\%$ . The Hall carrier mobility  $\mu_H$  was calculated as  $\mu_H = R_H \sigma$ . The measurements of the Seebeck coefficient *S* were carried out in the plane of the *PbSe* layers relative to *Cu* with an accuracy of  $\pm 3\%$ . On the basis of the obtained *S* and  $\sigma$  values, the thermoelectric power factor  $P = S^2 \sigma$  was calculated. Transport measurements were carried out on freshly grown thin *PbSe* films at room temperature.

# Results

The electron microscopy and electron diffraction studies showed that *PbSe* films grow epitaxially on (001) *KCl* in the (001) *PbSe*||(001)*KCl* orientation. The mechanism of the film growth corresponds to the Volmer-Weber growth mode with the formation of three-dimensional islands, which increase in size as *d* increases. At  $d \sim 5$  nm, the films have a channeled structure and at  $d \sim 10$  nm, the film is practically continuous. *EuSe* grows on the *PbSe* surface epitaxially as (001)*EuSe*||(001)*PbSe*, in a layer-by-layer fashion (the Frank-Van der Merwe mechanism) with the formation of a square grid of the misfit dislocations with the Burgers vector (1/2)[110] on the interface. Under increasing *PbSe* layer thickness, the regularity of the misfit dislocation network becomes higher. Taking into account that the misfit dislocation network was regular over a large area of the interface and did not contain defects in the form of torn or shifted relative to one another misfit dislocation rows, one can conclude that there were no rough defects on the interface, and the interface was quite smooth. The electron diffraction patterns of *PbSe/EuSe* bilayers contained no reflections other than those from *PbSe* and *EuSe*.

In Figs. 1, the room temperature dependences of *S*,  $\sigma$ ,  $\mu_H$ , and *P* on the *PbSe* layer thickness are presented. In accordance with the results of the measurements of the Hall and Seebeck coefficients, at  $d \sim 20$  nm, the inversion of the dominant carrier sign from *p* to *n* occurs, and then in the processes of oscillations a periodic alteration of the conductivity sign is observed. Under further increase in *d* all films exhibit electron conductivity. It is also seen that in all the curves, one can distinguish at least two regions. In the first region of *d* (up to  $d \sim 80$  nm), there are oscillations in *S*,  $\sigma$ ,  $\mu_H$  and *P* with large amplitudes; in the second region (d > 80 nm), these parameters change monotonically up to  $d \sim 150 - 200$  nm, and above that film thickness remain practically constant.



Fig. 1. The Seebeck coefficient S, electrical conductivity  $\sigma$ , charge carrier mobility  $\mu_H$ and thermoelectric power factor  $P = S^2 \sigma$  as functions of the PbSe layer thickness d in the KCl/PbSe/EuSe structures. The EuSe layer thickness is ~ 30 nm.

In Fig. 2, the room temperature dependence of monotonic component of  $\sigma$  on the *PbSe* layer thickness in the *KCl/PbSe/EuSe* structures exhibiting electron conductivity (d > 60 nm) are shown. One can see that with increasing thickness  $\sigma$  gradually grow up to ~ 150 – 200 nm, and at large *d* the curve reach saturation, and  $\sigma$  remains practically constant.



Fig. 2. The room-temperature thickness dependences of the electrical conductivity  $\sigma$ in the KCl/PbSe/EuSe structures. The solid line is the result of theoretical calculations based on the Fuchs – Sondheimer theory [11, 12].

#### Discussion

1. It is known that *PbSe* crystals can exhibit either *n*-type or *p*-type conductivity depending on the character of deviation from stoichiometry [2, 9]. The stoichiometry of a thin film depends not only on the initial material composition but also on a number of additional factors. Under fixed technological conditions (the temperature and type of substrate, condensation rate, etc.), the film thickness can be one of such factors which can change the transport mechanisms. In [8], we showed that in thin *PbSe* films grown from *n*-*PbSe* crystals with an electron concentration of  $n \sim 10^{18}$  cm<sup>-3</sup>, *n*-type conductivity is observed over the entire d range. The fact that *PbSe* films grown from a stoichiometric *p*-*PbSe* charge (the present work), exhibit the *p*-type conductivity only at d < 20 nm (leaving aside the appearance of *p*-type conductivity in the process of oscillations), indicates that an increase in d leads to a shift in the film composition to the Pb excess side. The inversion of the conductivity type can be caused by a partial re-evaporation of Se from a heated substrate in the process of condensation, which results in the excess of Pb in the film. Under increasing d (and, consequently, increasing evaporation time) the proportion of the Pb atoms must increase. The inversion of the conductivity type with changing d can also be connected with a change in the thermodynamic equilibrium conditions in thin films as compared to bulk crystals, which in turn leads to a dependence of the thin film stoichiometry on d. The specificity of the thin film state is determined by a number of factors, which can lead to a change in the equilibrium defect concentration in thin films in comparison with bulk crystals. Among such factors are: an increase in the relative contribution of the surface energy to the total energy, the presence of strains in a thin film as a result of the mismatch between the substrate and the film crystal lattices, etc. Thus, it follows from the obtained data that by evaporation of the stoichiometric *p-PbSe* crystals, it is possible to preserve *p*-type conductivity in the films protected from oxidation if d is sufficiently small. One can suggest that thin *PbSe* films will exhibit a p-type conductivity over a wider interval of thicknesses if the hole concentration in the bulk PbSe crystal, which is used as the initial material for thin films growing, is higher. Such was the case observed for films of *PbTe* doped with *Na* [7]. It should be noted that the existence of *p*- and *n*-regions corresponding to different ranges of d makes it possible to study size effects, determined by the behavior of both electron and hole gases using a single type of initial material.

2. The existence of the dependence of kinetic properties of PbSe films on the film thickness indicates that there occur size effects which can be connected both with the diffuse scattering of

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charge carriers at the film surface (CSE) and with size quantization of the energy spectrum (QSE). As was mentioned before, in all the curves, one can distinguish subregions with different behavior of the transport coefficients under changing *d*. The oscillatory behavior of the dependences at d < 80 nm goes into a monotonic change in the properties up to ~ 150 - 200 nm, and after that the properties practically do not change. Such an analysis makes it possible to isolate a monotonic component from oscillatory one, whose presence is attributed to the manifestation of the CSE and QSE, respectively.

3. One should note however, that apart from the QSE and CSE, it is the structure factor that affects the electrical conductivity and other kinetic coefficients. The influence of the structure factor is explained by the fact that IV - VI films grow on halogen crystals in an island fashion [8] and under increasing film thickness a transition occurs from a film with isolated islands, formed as a result of nucleation and growth of separate particles of condensate, to a structure with channels linking islands and determining charge transfer along the entire film [9]. The increase in *d* leads to a gradual overgrowing of the spaces between islands and, accordingly, to the increase in  $\sigma$ , until the continuous film is formed. After the formation of the continuous film the influence of the structure factor on  $\sigma$  is significantly smaller, nevertheless, in the range of small thicknesses it is important to take into account the dependence of the film structure on *d*.

4. One can say with confidence that the oscillatory behavior of the observed *d*-dependences of the transport properties is due to the size quantization of the energy spectrum taking place in the 2D-structure *KCl/PbSe/EuSe*. The *PbSe* layer that is sandwiched between the substrate (*KCl*) and the covering layer (*EuSe*) forms a QW, within which carriers are effectively confined. Such a system can be approximated as a QW with infinitely high walls. Electron confinement in a QW leads to the quantization of the transverse component of the quasi-momentum and the formation of lateral subbands. For an infinite barrier height using the effective-mass approximation, the energy levels are given as [1, 13]

$$E = \frac{\hbar^2}{2m_z^*} \frac{\pi^2}{d^2} N^2 + \frac{\hbar^2 k_x^2}{2m_x^*} + \frac{\hbar^2 k_y^2}{2m_y^*},$$
(1)

where  $m_z^*$  is the effective mass for motion perpendicular to the QW, and  $k_x$ ,  $k_y$  and  $m_x^*$ ,  $m_y^*$  are the components of the wave vector and effective mass, respectively, for motion parallel to the QW. The observed oscillatory dependences of the kinetic properties can be interpreted in terms of quantummechanical concepts about a gradual filling of the lateral subbands with increasing *d*. As *d* increases, the number of populated subbands *N* in a 2D-system changes in a step-like fashion. Every time *d* increases by  $\lambda_{F}/2$ , where  $\lambda_F$  is the de Broglie wavelength at the Fermi level, an additional subband drops below  $\varepsilon_{F}$ , and contributes to electrical conduction. This interrelation between *d* and *N* leads to an oscillatory behavior in many physical properties and is the origin of the QSEs. The critical thickness  $d_1$ , at which the first subband passes through the Fermi level ( $E_1 = \varepsilon_F$ ), can be estimated as  $d_1 = h/\sqrt{8m_z^*\varepsilon_F}$ .

If we assume that a QW represents an ensemble of electrons with a fixed value of the chemical potential corresponding to  $\varepsilon_F$  in thick films, then with changing *d*, the carrier concentration *n* (or *p*) in the QW must oscillate relative to the constant value of  $\varepsilon_F$ . Within a single subband, *n* (or *p*) increases at first, then it reaches a maximum, after which it decreases. When the next subband (beneath the conduction band) intersects the Fermi level, a new wave function starts to contribute to the electron density and the carrier concentration *n* (or *p*) increases again. Oscillations in the density of states must cause oscillations in the transport properties.

One can make an estimate of the order of magnitude of the oscillation period  $\Delta d$  and N, using the following equations [1, 13]:

$$\Delta d = \frac{\lambda_F}{2} = \frac{h}{\sqrt{8m_z^*\varepsilon_F}},\tag{2}$$

$$N = \frac{k_F d}{\pi} = \frac{d}{\lambda_F / 2} = \frac{d\sqrt{8m_z^* \varepsilon_F}}{h}.$$
(3)

As is seen, an important parameter related to the oscillatory character of the *d*-dependences of the transport properties is the charge carrier concentration (or  $\varepsilon_F$ ), which determines  $\Delta d$  and the number of populated subbands N in (2) and (3). One can see that  $d_1 = \Delta d$  and one can measure  $\Delta d$  most precisely from the position of the first extremum.

Substituting the known values of the effective mass of electrons in *n*-*PbSe* ( $m_t^* = 0.04 m_0$ ,  $m_l^* = 0.07 m_0$  where  $m_t^*$  and  $m_l^*$  are the transverse and longitudinal masses, accordingly) [2] and the value of  $\varepsilon_F$  estimated from the average electron concentration ( $n \approx 2 \cdot 10^{18} \text{ cm}^{-3}$ ) in the *n*-region into Eq. (2), we calculated the oscillation period as  $\Delta d = 20 \pm 2$  nm. This value is in good agreement with the experimentally measured average distance between neighboring maxima in the *d*-dependences of *S*,  $R_{H_3} \sigma$ , and  $\mu_H$ , as well as the position of the first extremum in the *n*-region ( $d = 20 \pm 1$  nm).

As is seen from Fig. 1, in the *d*-dependences of all properties in the *p*-region, near d = 10 nm, one extremum is also observed (minima in *S* and  $R_H$  and maxima in  $\sigma$  and  $\mu_H$ ). The existence of this extremum can be attributed to the first hole subband's (n = 1) passing through the Fermi level. The estimation of the oscillation period for the hole gas based on the known value of the effective mass of holes in *p*-*PbSe* ( $m_t^* = 0.034 \ m_0, \ m_l^* = 0.068 \ m_0$ ) [2] and on the value of  $\varepsilon_F$  calculated from the average hole concentration in the *p*-region ( $p \approx 5 \cdot 10^{18} \text{ cm}^{-3}$ ), yields  $\Delta d = 10 \pm 2$  nm, which is in good agreement with the experimental observation of the extremum near d = 10 nm. It should be noted that in *PbSe* the transverse effective masses both of electrons and holes are sufficiently small and close to each other, to facilitate the observation of the oscillations for charge carriers of both types.

5. It is seen from Fig. 1 that the oscillations of the Seebeck coefficient at d > 20 nm are accompanied by a periodic change in the conductivity type from electron to hole conduction. A similar effect is observed for  $R_H$  also. The fact that an inversion of conductivity sign under the thickness oscillations occurs is connected, to all appearances, with a high sensitivity of the electron subsystem of the crystal to external influences at thicknesses close to the point of the conductivity sign inversion. One can expect that at the indicated thicknesses, the manifestation of the QSE will be specific, consisting in periodic changes in the conductivity sign due to the formation of standing electronic waves, when the size quantization conditions are fulfilled. This occurrence is confirmed by the fact that in [3, 4], where films of all thicknesses exhibited *n*-type conductivity, the oscillations occurred without any changes in the conductivity sign

6. The thickness oscillations, whose presence is attributed to the manifestation of the QSE, are observed practically only at *d* smaller than ~ 80 nm (Fig. 1). Further monotonic change in the kinetic coefficients with increasing thickness of *PbSe* layers in the range of  $d \sim 80 - 200$  nm indicates that the amplitude of quantum oscillations decreases, resulting in the predominant manifestation of CSE. Due to this, one can differentiate the influence of size effects of both types rather clearly. For the classical size effect, the Fuchs-Sondheimer theory [8, 9] predicts the following dependence of the electrical conductivity on the film thickness under the condition that  $d \ll l$ :

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

where l is the mean free path in the bulk material, p is the specularity parameter showing the proportion of electrons elastically scattered by the film surface;  $\sigma_d$  is the electrical conductivity of a film with a thickness d; and  $\sigma_{\infty}$  is the conductivity of an infinitely thick film (bulk crystal). The value of p lies between 0 (for entirely diffuse scattering) and 1 (for entirely specular reflection). In the latter case, the classical size effect will not be observed. Although in the Fuchs-Sondheimer theory, a number of simplifying assumptions are made, in particular a metal with a spherical Fermi surface and an isotropic l independent of d is considered; it is assumed that p is constant and similar for both surfaces, independent of d, the incidence angle on the surface, and the electron trajectories; even though the structure factor is not taken into account, for not very small thicknesses ( $d \ll l$ ), a good agreement between the theory and experiment is observed. In very thin films, even if they are continuous, conductivity usually decreases much faster than the theory predicts. One of the possible explanations is that in the region of very small thicknesses ( $d \le l$ ), the condition of the constancy of l is violated because of the above mentioned structure factor. In Fig. 2, the theoretical dependence  $\sigma(d)$ calculated using equation (4) for the thickness range d > 60 nm, where all films are of *n*-type and the influence of the structure factor and QSE is fairly insignificant, is shown as the solid line. Here  $\sigma_{\!\infty}$ corresponds to the value of electrical conductivity for films with d > 200 nm ( $\sigma_{\infty} = 250 \Omega^{-1} \cdot \text{cm}^{-1}$ ). The best match of the theoretically calculated results and experimental data was observed for  $p = 0.57 \pm 0.02$  and  $l = 800 \pm 40$  nm. The difference between this value and values of l obtained in [10]  $(l = 940 \pm 40 \text{ nm})$  can be explained as follows: in [10], l and p were calculated for a wider range of thicknesses (d > 30 nm), in which both the structure factor and QSE played certain roles.

7. It is seen that the thickness oscillations of the transport properties distinctly manifest themselves at room temperature, although in accordance with the commonly accepted theoretical views their observation is possible only at low temperatures. The fact that the QSEs are registered at room temperature may imply, on the one hand, that a rather high degree of structural perfection in the films is achieved experimentally, and, on the other hand, possible effects due to other still unknown factors may be important also.

8. It should be noted that the thickness dependence of another important thermoelectric parameter – the thermoelectric power factor  $P = S^2 \cdot \sigma$ , which determines to a great extent the efficiency of the thermoelectric conversion of energy, also exhibits an oscillatory behavior (Fig. 1). The observed oscillatory character of the *d*-dependences of the thermoelectric properties, as well as the classical size effect, should be taken into account for 2D-structure-related applications in thermoelectricity and other spheres of science and technology.

## Conclusions

1. An increase in the *PbSe* layer thickness in *KCl/PbSe/EuSe* quantum wells, prepared from the stoichiometric *p-PbSe*, leads to an inversion of the dominant charge carrier sign. At *d* smaller than ~ 20 nm the films exhibit *p*-type conductivity, in the thickness range 20 - 60 nm the dominant carrier sign alternates periodically with changing thickness, and at *d* > 60 nm the films become of *n*-type. The inversion of the conductivity sign is attributed to a change in the thermodynamic equilibrium conditions in thin films as compared to bulk crystals, and/or to the partial re-evaporation of *Se* from the heated substrate in the process of condensation. The dependence of the conductivity type on the *PbSe* layer thickness makes it possible to study size effects determined simultaneously by the behavior both of the electron and hole gases.

2. In the thickness dependences of the transport properties (*S*,  $\sigma$ , *R*<sub>H</sub> and  $\mu$ <sub>H</sub>), one can isolate a monotonic and oscillatory components, which are attributed to the manifestation of the classical and

quantum size effects, respectively.

3. The monotonic component of the thickness dependences of properties changes under *d* increasing up to  $\sim 150 - 200$  nm, after that the properties remain practically constant. The theoretical calculation of the monotonic component of the electrical conductivity dependence using the Fuchs - Sondheimer theory conforms with the experimental data fairly well.

4. At d < 80 nm, the dependences of the kinetic properties on the film thickness exhibit an oscillatory behavior. Extrema of the properties observed in the *p*-region (d < 20 nm) near d = 10 nm, are attributed to the size quantization of the energy spectrum of the hole gas in *PbSe* quantum wells, whereas oscillations in the thickness range d = 20 - 80 nm, accompanied by the periodic change in the dominant carrier sign up to  $d \sim 60$  nm – to the electron spectrum quantization. The theoretically estimated periods of quantum oscillations for electron and hole gases are in good agreement with the experimentally observed ones.

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