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SIZE EFFECTS AND THERMOELECTRIC PROPERTIES OF $BI_{0.98}SB_{0.02}$ THIN FILMS

The room-temperature dependences of thermoelectric properties (the Seebeck coefficient S, the electrical conductivity σ , the Hall coefficient RH, and the thermoelectric power factor $P = S2 \cdot \sigma$) on the thickness ($d = 5 \cdot 250$ nm) of the $Bi_{0.98}Sb_{0.02}$ solid solution thin films grown on mica substrates by thermal evaporation in vacuum from a single source were obtained. It is shown that the monotonic component of the $\sigma(d)$ dependence is well described within the framework of the Fuchs-Sondheimer theory for the classical size effect. The presence of an oscillating component in the d-dependences of σ , S, RH and S2· σ is attributed to the manifestation of the quantum size effect, and the experimentally determined period of quantum oscillations $\Delta d = 45 \pm 5$ nm is in good agreement with the Δd value calculated theoretically within the framework of the model of an infinitely deep potential well. Bibl. 77, Fig. 1.

Key words: $Bi_{0.98}Sb_{0.02}$ solid solution, thin film, thickness, thermoelectric properties, size effect, oscillation period.

Introduction

 $Bi_{I-x}Sb_x$ solid solutuions are well known as effective thermoelectric (TE) materials for refrigerating devices operating at temperatures below ~200 K [1, 2]. The growing interest in $Bi_{I-x}Sb_x$ solutions is due not only to the possibility of their practical use in TE power engineering but also to their unique properties associated with the characteristics of their energy spectrum and the possibility of its qualitative rearrangement with changing composition, accompanied by a number of phase transitions [3-7]. Bi exhibits semimetallic properties: the electron L_s — band and T — band of "heavy" holes overlap. As Sb is added to Bi, the distance between the electron L_s — band and L_α — band of "light" holes decreases and at $x = 0.023 \div 0.04$ (different authors report different values of x), the energy gap between them becomes zero, i.e. a gapless state occurs. With further increase in x, the gap between the L_s and $L\alpha$ bands increases again, simultaneously the

overlap of T and Ls bands decreases, and at $x = 0.06 \div 0.07$, a semimetal – indirect semiconductor phase transition takes place. Then at $x = 0.08 \div 0.09$, the tops of T and Ls valence bands coincide, and in the range $x \cong 0.09 \div 0.15$ the indirect semiconductor becomes a direct-gap one. In [8-12], it was shown that the concentration-dependent anomalies of the transport properties correspond to the critical compositions associated with electronic phase transitions and the percolation-type transition from dilute to concentrated solid solutions. According to [13,14], electronic phase transitions of such types take place in the $Bi_{1-x}Sb_x$ films.

Recently, interest in the properties of the $Bi_{1-x}Sb_x$ solid solutions has sharply increased after it was found that they exhibit the properties of topological insulators – the newest materials of quantum physics, in which the strong spin-orbit interaction leads to the appearance of topological metallic surface states with a linear Dirac E(k) dispersion [15-17]. The $Bi_{1-x}Sb_x$ solid solution with x = 0.1 was the first experimentally discovered 3D-topological insulator [16]. In recent years, after it was established that the best TE materials (including $Bi_{1-x}Sb_x$) belong to the topological insulators class, the possibility of using the unique properties of the topological surface layer to develop fundamentally new ways to increase in TE figure of merit ZT has been discussed.

The development of nanotechnologies and the possibility of enhancing ZT in lowdimensional structures [18] have stimulated studies of $Bi_{1-x}Sb_x$ thin-film structures. Besides, using thin films, in which the relative contribution of the metal layer to the conductivity is larger as compared to bulks, is beneficial for studying the properties of the topological layer. However, when studying thin films, it should be taken into account that in the thin-film state the manifestations of classical size effect (CSE) and quantum size effect (QSE) are possible, which can significantly change the properties of films in comparison with bulk crystals [19]. The CSE is caused by diffuse scattering of charge carriers at the film boundaries and manifests itself when the film thickness d is comparable to the mean free path l of charge carriers. The QSE results from the quantization of the charge carrier energy spectrum and is observed when the value of d becomes comparable to the Fermi wavelength, where h is Planck's constant, m^* is the effective mass, and EF is the Fermi energy. One of the manifestations of the QSE is an oscillatory behavior of the ddependences of the transport properties. Due to the extremely low m^* value and anomalously high mobility of electrons in Bi and Bi1-xSbx, these materials are very convenient for studying QSE. It is in thin Bi films that the d-dependent oscillations of the galvanomagnetic properties were observed for the first time [20] and explained theoretically [21].

After that a very large number of works dealing with size effects in Bi films have appeared (see, e.g. [19,22-51]. Based on the results of studies of the d-dependences of transport properties, some authors [23,27,31,38,39,43,44,49,50] determined l and the surface scattering coefficient p (the fraction of charge carriers which are specularly reflected from the surface) for Bi films using the Fuchs- Sondheimer theory (FST) [52,53] or other methods. It was found that at room temperature in monocrystalline Bi films l lies in the range of 600-1000 nm, whereas in polycrystalline films l = 100-250 nm, and p = 0-0.8. The discrepancy in l and p values obtained

by different authors was apparently connected with the fact that these parameters depend both on the film structure, which in turn is determined by the sample preparation technology, and on the method of l and p determination.

After the size-dependent oscillations of the kinetic coefficients had observed in Bi films in [20], the existence of such oscillations was confirmed in a large number of works [19,22-51] in which the oscillating dependences of electrical resistance ρ , the Hall coefficient RH, Seebeck coefficient S and magnetoresistance $\Delta\rho/\rho$ on d were also interpreted as a manifestation of the QSE. The oscillation periods Δd determined experimentally for Bi thin films by different authors were in the range of 25-45 nm. Some authors [54 – 60] even considered quantum corrections to conductivity, which are the result of quantum interference.

The next step was studying the transport properties of thin films of Bi1-xSbx solid solutions [61-74] in order to identify CSEs and QSEs. It turned out that, like in Bi films, the d-dependences of the kinetic properties in Bi1-xSbx exhibit CSE. The authors of [65] estimated l for epitaxial $Bi_{1-x}Sb_x$ films with x in the range of 0 - 0.142 from the d-dependences of electron mobility at 4.2 K using the FST theory. According to [65], 1 varies within the range of 1000 - 6000 nm. The authors of [71, 72] calculated l for polycrystalline $Bi_{1-x}Sb_x$ films from the room temperature $\sigma(d)$ and S(d) dependences under the assumption of total diffuse scattering of carriers (p=0) at x=0.07 [71] and x=0.12 [72], before and after annealing, and obtained l=60 and 180 nm, l=150 and 230 nm respectively.

The first studies [61 - 63] of the *d*-dependences of σ , *S*, and ρ of $Bi_{1-x}Sb_x$ films confirmed the presence of QSE in $Bi_{1-x}Sb_x$ thin films. The authors of [61,62] investigated the $\sigma(d)$ and S(d) dependences at 90 K for polycrystalline $Bi_{1-x}Sb_x$ films ($d \le 300$ nm) with x = 0.018 and 0.035 and found that as compared with Bi films Δd increases up to $\Delta d = 70$ and 90 nm, respectively. In [63], it was also shown that introduction of a small amount of Sb ($x \sim 0.04$) into Bi films with d = 420 - 570 nm prepared by thermal evaporation of Sb in vacuum and subsequent diffusion annealing, resulted in an increase in Δd from $\Delta d \sim 26$ nm for pure Bi to $\Delta d \sim 65$ nm for Bi1-xSbx at 4.2 K. Further more detailed systematic studies of the d-dependences (d = 400 - 200 nm) of the σ , R_H and $\Delta \rho/\rho$ at 4.2 K, carried out by the authors of [64-68], showed that as x increases to 0.059, Δd increases to ~110 nm, however, further increase in x to 0.142 leads to a decrease in Δd down to ~50 nm, which was attributed by the authors of [64-68] to a change in the parameters of the band spectrum of $Bi_{1-x}Sb_x$ under changing composition.

It can be seen that in various studies of the size effects in $Bi_{1-x}Sb_x$ films, films with different compositions, different thicknesses, prepared using different technologies were used and measurements were carried out at different temperatures. Meanwhile, the properties of $Bi_{1-x}Sb_x$ crystals and, to even greater extent, of films are very sensitive to various types of external influences, and therefore the various physical parameters of thin films should be compared for the same temperatures, the same intervals of the studied d's, and the same technologies of the film

preparation. In addition, in the available literature, either CSEs or QSEs were usually investigated, although both types of size effects can manifest themselves simultaneously.

In [73, 74] we reported the observation of oscillations with $\Delta d = 80 \pm 5$ nm [73] and with $\Delta d = 105 \pm 5$ nm [74] in the room-temperature d-dependences of σ , R_H , $\Delta \rho/\rho$ and S for $Bi_{I-x}Sb_x$ polycrystalline thin films (d = 5 - 400 nm) prepared by the thermal evaporation in vacuum of the crystals with x = 0.045 and x = 0.09 and the subsequent deposition on (111) mica substrates at $T_S = 380 \div 5$ K. The earlier determined at room temperature Δd for Bi films prepared under similar conditions was ~ 30 nm [46]. Simultaneously in [73,74] we investigated CSE and estimated I using the FST theory as 800 ± 40 nm (x = 0.045) and as 900 ± 50 nm (x = 0.09). The value of p practically did not depend on composition and was equal to 0.8. The composition x = 0.045 corresponds to the semimetallic state and lies between the critical compositions corresponding to the transition to the gapless state ($x \sim 0.03$) and the transition from the semimetal to an indirect-gap semiconductor ($x \sim 0.06$). The composition x = 0.09 corresponds to the direct-gap semiconductor region.

Thus, the compositions of $Bi_{1-x}Sb_x$ films studied in [73, 74] and films of pure Bi [46] were prepared and investigated using the same preparation and measurement techniques, and all those compositions lay outside the phase transition regions, whose presence could distort the manifestation of size effects. It was of interest to study under identical conditions thin films obtained from semimetallic Bi1-xSbx crystals with compositions between the critical compositions corresponding to the percolation transition ($x \sim 0.01$) and the transition to the gapless state ($x \sim 0.03$). The composition x = 0.02 belongs to such compositions.

The purpose of this work is to prepare semimetallic films of various d with composition x=0.02 using the technique similar to described in [46, 73, 74] and by measuring the d-dependences of TE characteristics to identify the manifestations of the classical and quantum size effects, estimate the values of l, p and Δd and compare them with those obtained in [46, 73, 74]

Results and discussions

The objects of this study are thin films of $Bi_{0.98}Sb_{0.02}$ solid solution with thicknesses d = 5 - 250 nm obtained by thermal evaporation in vacuum of $Bi_{0.98}Sb_{0.02}$ polycrystal and subsequent deposition on mica substrates at 380 K. The methods of preparing films, measuring their thickness, characterising the micro- and crystal structure, and measuring the TE properties are similar to those described in [46,73,74].

In Fig. 1,a,b,c,d, the dependences of σ , S, R_H and TE power factor $P = S^2 \sigma$ on d at 300 K are shown. A nonzero conductivity was observed starting from the critical thickness $d_c = 9$ nm, which corresponds to the transition from an island film to a channeled structure. According to earlier electron microscopic studies [46], the $Bi_{1-x}Sb_x$ films grow on mica substrates in an island-like

fashion in the epitaxial orientation (001) $Bi_{I-x}Sb_x \parallel$ (001) mica. BiI-xSbx films on mica represent mosaic single crystals with high-angle twin-type boundaries.

It is seen that all thickness dependences exhibit a distinctly non-monotonic behavior. With increasing film thickness, σ , S and R_H show a tendency to increase. However, along with the general trend to increase with increasing d, the kinetic coefficients exhibit an oscillatory behavior, too. For all d-dependences one can isolate a monotonic component and oscillatory one. We attribute the presence of the monotonic component to the CSE and the oscillatory component to QSE.

According to the FST of the CSE [52,53], the dependence of electrical conductivity on d, provided that $d \ll 1$, is described by the following equation:

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

where l is the mean free path in the bulk material, p is the surface scattering coefficient; Δd is the electrical conductivity of a film with thickness d; and σ_{∞} is the conductivity of a bulk crystal. The value of p lies between 0 (for entirely diffuse scattering) and 1 (for entirely specular reflection). In the latter case, the CSE will not be observed. The FST assumes that CSE occur when scattering is predominantly diffuse, i.e., at sufficiently low p values. It should be noted that the FST adopts a number of simplifying assumptions: it considers a metal with a spherical Fermi surface and isotropic l independent of d; p is assumed to be constant, i.e. the same for both surfaces, independent of d, the angle of incidence on the surface, and electron trajectories. Assuming the value of electrical conductivity for $Bi_{0.98}Sb_{0.02}$ films with a thickness of d=200-300 nm ($\sigma \approx 4000$ (Ohm cm)⁻¹) as σ_{∞} and varying the values of p and l, theoretical $\sigma(d)$ dependences were calculated. Comparing the experimental and theoretical $\Delta(d)$ dependences calculated within the framework of the FST, we established that the best match of the results of theoretical calculations and experimental data was observed for $p=0.5\pm0.05$ and $l=800\pm40$ nm. In Fig. 1,a, the theoretical dependence $\Delta(d)$ calculated using equation (1) is shown as the dotted line.

In Fig. 1,b, the S(d) dependence is presented. S has the negative sign, like in bulk Bi and Bi_{0.98}Sb_{0.02}. With increasing d, a tendency to an increase in the magnitude of the S values is observed. In Fig. 1,c, the $R_H(d)$ dependence is given. It can be seen that, in contrast to the sign of S in films and bulk crystal, the sign of R_H in films turns out to be positive. With increasing d, R_H exhibits a tendency to increase in magnitude. The oscillations are even more pronounced than in the $\sigma(d)$ and S(d) dependences.

The periods of oscillations observed in the dependences of σ , S, R_H on d are practically the same for all kinetic coefficients, amounting to $\Delta d = 45 \pm 5$ nm.

Using the data for σ and S, the TE power factor was calculated as $P = S^2 \sigma$. The behavior of the P(d) dependences is similar to that of the $\sigma(d)$ and S(d) curves (Fig. 1,d). The values of TE

power factor for "thick" films ($d\sim200\text{-}400 \text{ nm}$) were practically equal to those for bulk Bi_{0.98}Sb_{0.02} crystals, from which the films were obtained.

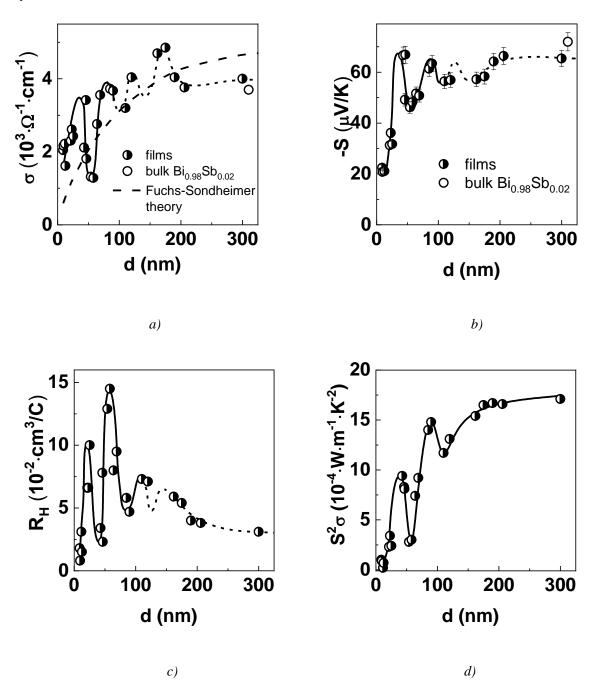


Fig. 1. Room-temperature dependences of the electrical conductivity σ (a), the Seebeck coefficient S(b), the Hall coefficient $R_H(c)$, and the thermoelectric power factor $P=S^2$ σ (d) on $Bi_{0.98}Sb_{0.02}$ thin films thickness d for $Bi_{0.98}Sb_{0.02}$ thin films prepared by thermal evaporation in vacuum on mica substrates.

In Fig. 1,a the theoretical dependence $\sigma(d)$ calculated using the Fuchs - Sondheimer theory (FST) is shown as a dotted line.

It is natural to assume that the oscillating behavior observed in the d-dependences of σ , $R_{\rm H}$, and S is the result of size quantization in the Bi_{0.98}Sb_{0.02} thin films. For observing QSEs in polycrystalline films, the formation of texture is required, though the film does not need to be monocrystalline. Due to the axial symmetry of the Bi band structure relative to the trigonal axis, oscillations of the electrical conductivity appear, if the trigonal axes are normal to the film surface in all crystallites.

A thin film of a semimetal is a quantum well, within which the movement of charge carriers is limited in one direction. Such a system is a convenient model object, since its properties are close to the model of a potential well with infinitely high walls. This model is simpler as compared to the finite width and height barrier model. The possibility of the observation of the oscillations in a film grown on an amorphous substrate is explained by the presence of texture in Bi_{0.98}Sb_{0.02} thin films in the direction of electron confinement [19].

Restricting the motion of electrons leads to the quantization of the transverse component of quasimomentum and, accordingly, the energy spectrum. For a barrier of an infinite height, using the effective mass approximation, the energy levels can be presented as:

$$E_{n} = \frac{\hbar^{2}}{2m_{z}^{*}} \frac{\pi^{2}}{d^{2}} n^{2} + \frac{\hbar^{2} k_{x}^{2}}{2m_{x}^{*}} + \frac{\hbar^{2} k_{y}^{2}}{2m_{y}^{*}}$$
(2)

where m_x^* , m_y^* are the components of the effective mass for motion parallel to the film surface and m_z^* is the effective mass for motion perpendicular to the film surface, n is the discrete quantum number. At fixed n values, the energy changes continuously as a function of κ_x and κ_y , corresponding to each subband n, and the energy spectrum consists of overlapping subbands. The $Bi_{0.98}Sb_{0.02}$ solid solution is a semimetal with degenerate charge carriers; therefore, the quantization condition for this material can be expressed in terms of the Fermi wavelength and the Fermi energy E_F :

Using this approximation, one can estimate the oscillation period Δd :

$$\Delta d = \frac{h}{\sqrt{8m_z^* \varepsilon_F}} \tag{3}$$

Knowing the effective mass of electrons and the Fermi energy for the Bi1-xSbx solid solution with x = 0.02 from the literature data [4] for bulk crystals ($m * x = 0.02 = 0.8 m *_{Bi}, m *_{Bi} = 0.012 \pm 0.002 m_o E_F = 22.7$ meV) and using equation (3), we calculated the theoretical period of quantum oscillations as $\Delta d = 52$ nm. It can be seen that the theoretical value is in good agreement

with the experimentally determined Δd . It should be noted that although σ , R_H and S were measured independently, in all d-dependent studies, distinct oscillations with approximately the same periods were observed for all measured characteristics. Comparing the value of Δd obtained in this work with the one we obtained earlier for Bi1-xSbx films prepared by a similar procedure but with different concentration x [46,73,74], we found that with increasing x, Δd increases, amounting to 30, 45, 80, and 105 nm for films with x equal to 0, 0.02, 0.045, and 0.1, respectively.

As follows from the data obtained, R_H for all $Bi_{0.98}Sb_{0.02}$ films has a positive sign, while S is negative. This difference can be explained by the strong anisotropy of Bi single crystals, which also occurs, but to a lesser extent, when Sb is introduced into Bi. According to [75], in Bi single crystals R_H has a positive sign only in the direction of a trigonal axis, while in the perpendicular directions the R_H sign changes to negative. In contrast to R_H , the Seebeck coefficient remains negative in any crystallographic direction [75]. That is why in all Bi films grown along the trigonal axis [001] a positive sign of R_H and a negative sign of S were observed [46]. However, let us note that we observed a change in the sign of R_H with increasing d in films with a high Sbcontent [73, 74] and explained this by the fact that for such samples the perpendicular orientation of the trigonal axis [001] relative to the substrate becomes less perfect. This explanation is confirmed by the results obtained in [29], where in polycrystalline Bi films the R_H sign, as well as the S sign, was negative, since there was no anisotropy in those films and the contribution of the bisector and bipolar components responsible for the negative sign of R_H was predominant. The fact that the positive sign of R_H in $Bi_{0.98}Sb_{0.02}$ films was observed for all d's indicates that upon the introduction of a small amount of Sb (2 at.%), the degree of texture of the film remains rather high.

It follows from the experimental data obtained in this work that the thickness oscillations of the transport properties of $Bi_{0.98}Sb_{0.02}$ are distinctly manifested even at room temperature, although it is expected that thermal smearing of energy levels increases with increasing temperature and can become comparable to the spacing between the quantized levels. In a number of works on Bi and Bi1-xSbx films, measurements were carried out at 4.2 K, however, the oscillations did not manifest themselves more clearly. In our works on IV-VI and V2VI3 films (see, for example, [76,77]) we reported that the amplitude of the quantum oscillations in the room temperature thickness dependences is quite large. That is why, perhaps, the weak temperature dependence of the oscillation amplitude is not determined by the specificity of $Bi_{0.98}Sb_{0.02}$ thin films but is common for films of different types and requires theoretical interpretation.

Conclusions

1. In the room-temperature dependences of the electrical conductivity σ , the Hall coefficient R_H , and the Seebeck coefficient S of $Bi_{0.98}Sb_{0.02}$ thin films on d (d = 5 - 250 nm), monotonic and oscillating components can be distinguished, which indicates the manifestation of classical and quantum size effects.

- 2. The monotonic component of is well described within the framework of the Fuchs Sondheimer theory with the mean free path of electrons $l \approx 800 \pm 40$ nm and the surface scattering coefficient $p \approx 0.5 \pm 0.05$.
- 3. The experimentally measured period of quantum oscillations ($\Delta d = 45 \pm 5$ nm) is in good agreement with that calculated theoretically within the framework of the model of an infinitly deep potential well. Comparing the value of Δd obtained in this work with those obtained earlier for Bi1-xSbx films prepared by a similar procedure but with different values of x, we found that with increasing x, Δd increases, amounting to 30, 45, 80, and 105 nm for films with x equal to 0, 0.02, 0.045, and 0.1, respectively.
- 4. In contrast to the previously investigated films with x = 0.045 and x = 0.09, in which the R_H sign depended on the film thickness, in $Bi_{0.98}Sb_{0.02}$ films, the R_H sign remained positive for all d's, like in pure Bi. At the same time, the sign of S was negative. The observed effect is associated with the fact that the sign of R_H is different in different directions and with the increasing degree of texture disorientation under increasing x.
- 5. The room-temperature d-dependence of the TE power factor P also exhibits an oscillatory behavior. The values of power factor for "thick" films (d~200-400 nm) were practically similar to those for bulk $Bi_{0.98}Sb_{0.02}$ crystals, which were used as a charge for thin film preparation.

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РОЗМІРНІ ЕФЕКТИ ТА ТЕРМОЕЛЕКТРИЧНІ ВЛАСТИВОСТІ ТОНКИХ ПЛІВОК ВІ0.98SВ0.02

При кімнатній температурі отримано залежності термоелектричних властивостей (коефіцієнта Зеєбека S, електропровідності σ , коефіцієнта Холла R_H і термоелектричної потужності $P=S^2\cdot\sigma$) від товщини (d=5-250 нм) тонких плівок твердих розчинів $Bi_{98}Sb_2$, вирощених на підкладках зі слюди методом термічного випаровування у вакуумі із одного джерела. Показано, що монотонна складова залежності $\sigma(d)$ добре описується в рамках теорії Фукса-Зондгеймера для класичного розмірного ефекту. Виявлена осцилююча складова d-залежностей σ , S, R_H та $S^2\sigma$ пов'язується з проявом квантового розмірного ефекту, і визначений експериментально період квантових осциляцій $\Delta d=45\pm5$ нм добре узгоджується зі значенням Δd , теоретично розрахованим в рамках моделі нескінченно глибокої потенційної ями. Бібл. 77, рис. 1.

Ключові слова: $Bi_{0.98}Sb_{0.02}$ твердий разчин, тонка плівка, товщина, термоелектричні властивості, розмірний ефект, період осциляцій.

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РАЗМЕРНЫЕ ЭФФЕКТЫ И ТЕРМОЭЛЕКТРИЧЕСКИЕ СВОЙСТВА ТОНКИХ ПЛЕНОК Віо,98 Sbo,02

При комнатной температуре получены зависимости термоэлектрических свойств (коэффициента Зеебека S, электропроводности σ , коэффициента Xолла R_H и термоэлектрической мощности $P = S^2 \sigma$) от толщины (d = 5 - 250 nm) тонких пленок твердых растворов $Bi_{98}Sb_2$, выращенных на подложках из слюды методом термического испарения в вакууме из одного источника. Показано, что монотонная составляющая зависимости $\sigma(d)$ хорошо описывается в рамках теории Фукса-Зондгеймера для классического размерного эффекта. Обнаружена осциплирующая составляющая d-зависимостей σ , S, R_H и $S^2\sigma$ связывается c проявлением квантового размерного эффекта, и определенный экспериментально период квантовых осципляций $\Delta d = 45 \pm 5$ нм хорошо согласуется со значением Δd , теоретически рассчитанным в рамках модели бесконечно глубокой потенциальной ямы. Библ. 77, рис. 1.

Ключевые слова: $Bi_{0.98}Sb_{0.02}$, твердый раствор, тонкая пленка, толщина, термоэлектрические свойства, размерный эффект, период осцилляций.

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