

Investigation of the growth mechanism, structure, and thermoelectric properties of thin PbTe films grown on mica

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The growth mechanisms, structure and thermoelectric properties of thin PbTe films prepared by thermal evaporation in vacuum and subsequent deposition on mica substrates at temperatures $T_s = 375, 525$ and 635 K were studied. The films were prepared from charge with different electron concentrations ($n \cong 10^{17}$ and $n \cong 10^{20}$ cm⁻³). The film thickness was varied in the range $d = 4-500$ nm. Electron microscopy study showed that PbTe grows on mica epitaxially in an island like fashion predominantly in the (111) orientation. It is established that in PbTe films there exists a critical thickness at which the transition from electron to hole conductivity with decreasing d is observed. Covering films with a protective layer, lowering the substrate temperature and increasing electron concentration in the charge result in narrowing of the thickness range corresponding to hole conductivity. It is shown that electron concentrations n in the charge and in thick PbTe films grown at the substrate temperature $T_s = 525$ K differ, the character and magnitude of this difference depending on n in the charge.

Исследованы механизм роста, структура и термоэлектрические свойства тонких пленок PbTe, полученных термическим испарением в вакууме на подложки из слюды. Варьировались толщина пленок ($d = 4-500$ нм), температура подложки ($T_s = 375, 525$ и 635 К) и концентрация носителей заряда в исходной шихте ($n \cong 10^{17}$ и $n \cong 10^{20}$ см⁻³). Методом электронной микроскопии установлено, что PbTe растет на слюде эпитаксиально по островковому механизму преимущественно в ориентации (111). Установлено, что в пленках PbTe существует критическая толщина, при которой наблюдается переход от электронной к дырочной проводимости при уменьшении d . Нанесение на пленки защитного покрытия, снижение температуры подложки и увеличение концентрации электронов в шихте приводят к сужению интервала толщин, соответствующих дырочной проводимости. Показано, что значения концентрации электронов n в шихте и толстых пленках PbTe, полученных при температуре подложки $T_s = 525$ К, различаются, причем характер и величина этого изменения зависят от n в шихте.

Lead chalcogenides belong to the well-known thermoelectric materials operated in the average temperature range (600 to 900 K) [1–3]. The interest to these materials has quickened recently, because it has

been predicted theoretically [3–4] and verified in practice [5–9] that the thermoelectric figure of merit could be considerably increased in thin film structures on the base of lead chalcogenide base. This stimu-

lates the complex research works on the influence of technological factors (the substrate material and temperature, the charge composition, etc.) on the growth mechanism, structure and thermoelectric properties of the thin film lead chalcogenide structures.

We shall study PbTe thin films grown on mica by thermal evaporation in vacuum. Lead telluride is widely used not only as a component in heterostructures and superlattices, but also in buffer layers [10–12]. PbTe has a narrow (no more than ~0.02 %) two-sided homogeneity region [13, 14] and, according to the deviations from stoichiometric composition, can display *n*-type (if there is a Pb excess) or *p*-type (in case of Te excess) conductance. The type and concentration of charge carriers in PbTe bulk crystals and thin films can be changed by doping or deviation from the stoichiometry [13, 14]. Both methods are accompanied by introducing defects into the crystal lattice, that, on the one hand, results, in changes of electronic properties and, on the other hand, should influence the thin film growth mechanism and its structure. The stoichiometry of a thin film depends not only on the composition of the charge used, but also on a number of additional factors (the temperature and the type of the substrate, the value of the binding energy between the film and the substrate, etc.), essential for the prediction and interpretation of thin film properties.

The existing works on structure and electro-physical properties of PbTe thin films report mostly about the films obtained on BaF₂, KCl, NaCl substrates and much less about the films grown on mica [10–12]. Meanwhile, mica belongs to rather cheap substrate materials, allowing to obtain epitaxial PbTe films with good electrophysical properties [15].

The aim of this work is to study the influence of substrate temperature, concentration of charge carriers in the charge and the film thickness on the growth mechanism, structure and thermoelectric properties of PbTe thin films.

PbTe films with thickness $d = 4\text{--}500$ nm were obtained by thermal evaporation of lead telluride crystals of nearly stoichiometric composition and with electron concentration $n \approx 10^{17}$ cm⁻³ from wolfram boats in oil-free vacuum of $10^{-5}\text{--}10^{-6}$ Pa, followed by condensation onto mica substrates heated up to temperature $T_s = 375 \pm 5$ K, 525 ± 5 K,

or 635 ± 5 K. At the substrate temperature $T_s = 525 \pm 5$ K, the extra second series of thin films with a higher charge carrier concentration was obtained. To that end, the charge consisted of PbTe enriched in lead and additionally doped with PbCl₂ (0.995 Pb₅₂Te₄₈ + 0.005 PbCl₂) with electron concentration $n \approx 10^{20}$ cm⁻³. The condensation rate was 0.1 to 0.3 nm/s. The film thickness was measured using a quartz resonator. In order to prevent the film exposure to air, those were coated in part with Al₂O₃ by electron beam evaporation.

The electron microscopy studies were carried out using a transmission electron microscope PEM-125K at 100 kV acceleration voltage. For the examination, the films were separated from mica using gelatin in the standard technique and picked out on object copper gauzes. The films less than 10 nm thick were secured with thin carbon film before separation from the substrate. The Hall coefficient R_H , electric conductivity σ and the thermo-e.m.f. coefficient S were measured at room temperature. R_H was measured in magnetostatic field of about 0.8 T, the sample carrying constant current. The material of contacts was indium. The electron concentration was determined using the formula $n = r(eRH)$, supposing the Hall factor $r = 1$. The error in R_H and σ measuring did not exceed ± 5 %. Electron mobility μ was calculated as $\mu = R_H \sigma$. The thermo-e.m.f. coefficient was determined by compensation method relative to copper within less than 3 %.

The measurements of lead telluride bulk crystal (used as charge) electrophysical properties show that PbTe crystals have electron conductivity with different sets of electrophysical parameters for doped and undoped charge. In the first case, $n = 1.0 \cdot 10^{17}$ cm⁻³, $\mu = 710$ cm²/V·s, $S = -440$ μ V/K, while in the second one, $n = 1.0 \cdot 10^{20}$ cm⁻³, $\mu = 145$ cm²/V·s, $S = -27$ μ V/K.

The electron microscopy shows that the films of effective thickness $d \leq 10$ nm have island structure, about 10 nm thick ones, channel structure (Figs. 1a, 1b) while those of about 20 nm thickness are already essentially solid (Figs. 2a, 2b). At all condensation temperatures, it is just the vapor→crystal growth mechanism that is realized. The nucleation of PbTe islands takes place in the orientations (111) PbTe (001) mica and (001) PbTe (001) mica (Fig.

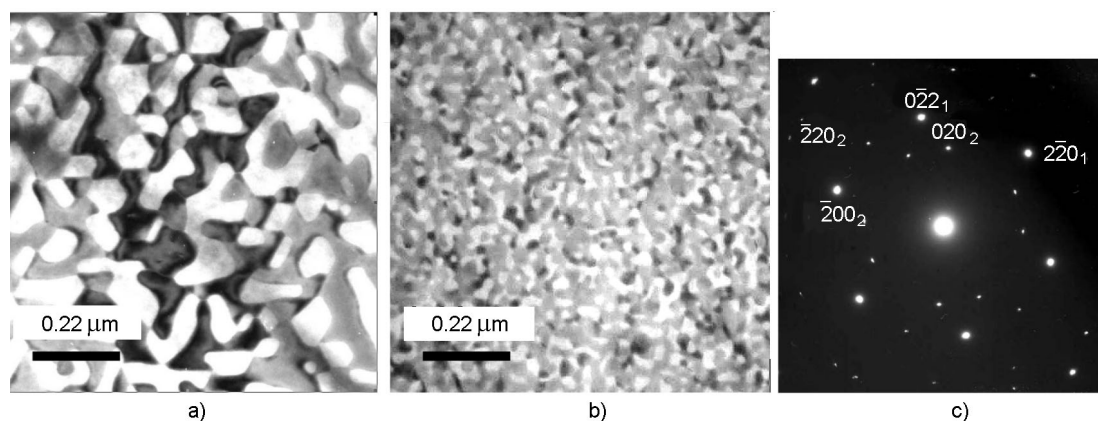


Fig. 1. The structure of 10 nm thick PbTe films grown on mica, $T_s = 525$ K: (a) the film grown from undoped charge; (b) the film grown from doped charge; (c) typical microdiffraction pattern of $d < 20$ nm thick PbTe films.

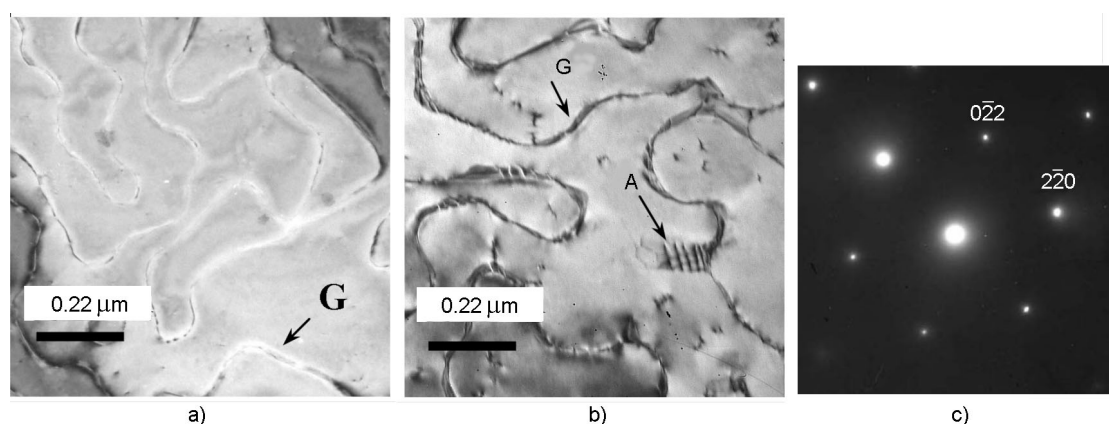


Fig. 2. Dependence of PbTe film structure on thickness d . The films are grown from undoped charge at $T_s = 525$ K: (a) $d = 20$ nm; (b) $d = 100$ nm; (c) microdiffraction pattern of the film (b).

1c). Most of the islands have (111) orientation. This can be observed at the evaporation of undoped as well as of doped raw material. Islands with orientation (111) take two positions, equal to one another in interphase conjugation ($[110]$ PbTe \parallel $[010]$ mica) rotated about each other by 180° about the axis $[001]$, on the mica surface (two-position nucleation). The islands of (001) orientation take on the mica surface three equivalent positions ($[110]$ PbTe \parallel $[010]$ mica) rotated by 60° about the axis $[001]$.

The conservation of islands faceting at the stage of their intergrowth and the faceting of the pores in the film at the stage of channel structure formation evidence a weak coalescence process as the condensate mass increases. It is found out by comparison of growth pattern for the films of the same thickness ($d = 10$ nm) prepared from doped and undoped charge that in the second type films the coalescence is more pronounced (Fig. 1a, 1b). This testifies an ac-

celerated surface diffusion stimulating the liquid-like confluence of the islands at their contact in the films prepared from the doped charge. One can suppose that in this case, the coalescence activation is caused by the high concentration of intrinsic and impurity defects in PbTe films, caused by introduction of over-stoichiometric lead and PbCl_2 added. A similar effect (the pronounced coalescence process) was observed in SnTe films and believed to be associated with a high concentration of cation vacancies in tin telluride [16]. As the film thickness increases, the number of (001) oriented crystallites decreases. It is so because of the recrystallization proceeds in continuous films. As a result, PbTe films prepared from stoichiometric charge at the substrate temperature $T_s = 525$ K, consist of crystallites with (111) orientation, if the film thickness exceeds 50 nm (Fig. 2c). However, even in 100 nm thick films, crystallites with (001) orientation sometimes are present.

Two-position nucleation of small crystals with (111) orientation causes a large number of twin boundaries to appear in the structure of continuous film (G in Fig. 2a,2b). Initially, the boundaries are oriented perpendicularly to the substrate and lie in the planes of (11 $\bar{2}$) type. Twin boundaries make closed figures, which at the initial stage are outlined with broken lines in the directions $\langle 01\bar{1} \rangle$ of PbTe. As the film thickness increases, the acute angles of the boundaries become rounded, and the boundaries become inclined to the film surface (Fig. 2b), leaving crystallographic planes of (11 $\bar{2}$) type. The boundaries inclined to the film plane are shown as bands and contain grain-boundary dislocations. It was observed that in about 100 nm thick films prepared from undoped charge, some twin type boundaries were split. In the place of splitting, a horizontal (111) twin boundary passes from the inclined boundary into the crystallite. Such region is indicated by arrow A in Fig. 2b. The (111) boundaries contain twinning dislocations. Similar effects have been considered thoroughly for silver films [17]. It was shown that twinning dislocations in the twin boundary (111) are generated by boundaries $\{11\bar{2}\}$. In $\{11\bar{2}\}$ boundaries, prismatic dislocation loops are formed at the expense of vacancies coming from the surface of the growing film. Some individual branches of these prismatic dislocations can emerge in the form of gliding twinning dislocations into the boundaries (111). A similar process seems to take place in PbTe films.

In the course of the collective recrystallization occurring in the films during the condensation with the increase of thickness, the inclined boundaries movement causes some boundaries to join and annihilate. On the annihilation spots, dislocation walls of lattice dislocations, that compensate small-angle off-orientation of the crystallites, can remain. We have evaluated the enlargement of the crystallite size caused by collective recrystallization by determining the length of twin boundaries per unit area of the film. This has been made with the intercept method, used as a rule to determine the dislocation density in compound dislocation accumulations [18]. It was found out that at $T_s = 525$ K, in the films prepared from undoped charge, the thickness increase from 20 nm to 120 nm is accompanied by the decrease of twin boundaries length from $10 \mu\text{m}/\mu\text{m}^2$ to $6 \mu\text{m}/\mu\text{m}^2$.

PbTe films prepared at the higher substrate temperature $T_s = 635$ K, had a more developed relief than those condensed at $T_s = 525$ K. In electron microscopy images thereof, numerous growth patterns in the form of dark triangles were observed on the surface of every crystallite (Fig. 3c). In the films condensed at $T_s = 525$ K, only flutes along the twin boundaries (light lines along the boundaries in Fig. 2a) were distinctly seen, the growth figures being much fainter.

The films condensed onto mica surface at $T_s = 375$ K were essentially epitaxial. There could be found one or two grains of (100) orientation in the area of $40 \times 40 \mu\text{m}^2$ (the cell area in the object gauze whereon the films were picked out for microscopic examination). In those films, however, there were a lot of very small pores or small dislocation loops (B in Fig. 3a) and the density of twin type boundaries was about twice higher than in the films prepared at $T_s = 525$ K. Another drawback of such films was a low reproducibility of structure characteristics: we have observed the appearance of numerous grains in the orientation (112) PbTe (001) mica in some films.

A distinctive feature of the films obtained from doped charge at $T_s = 525$ K was the higher mobility of the twin boundaries therein as compared to the films obtained from undoped charge at the same T_s . These boundaries become inclined at smaller d values and form a smaller angle with the film surface (in electron microscopy images, their plane projection is wider at the same film thickness (Fig. 3b)). Here one can observe much larger number of split boundaries with horizontal regions in (111) planes. The extension of the twin boundaries here is of the same order as in the films prepared from undoped charge. In the films of $d = 100$ nm, it is about $8 \mu\text{m}/\mu\text{m}^2$ at a more disperse initial structure than in those obtained from undoped charge. A higher diffusive mobility of twin boundaries in the films obtained from undoped raw material, as well as the trend to coalescence at the stage of island growth observed therein, must be caused by a high concentration of structure defects in these films resulting from stoichiometry deviations and doping.

The measuring of thermo-e.m.f. coefficient and Hall coefficient as a thickness function for the as-grown films without protective layer has shown that at d smaller than a certain critical value d_c , one can

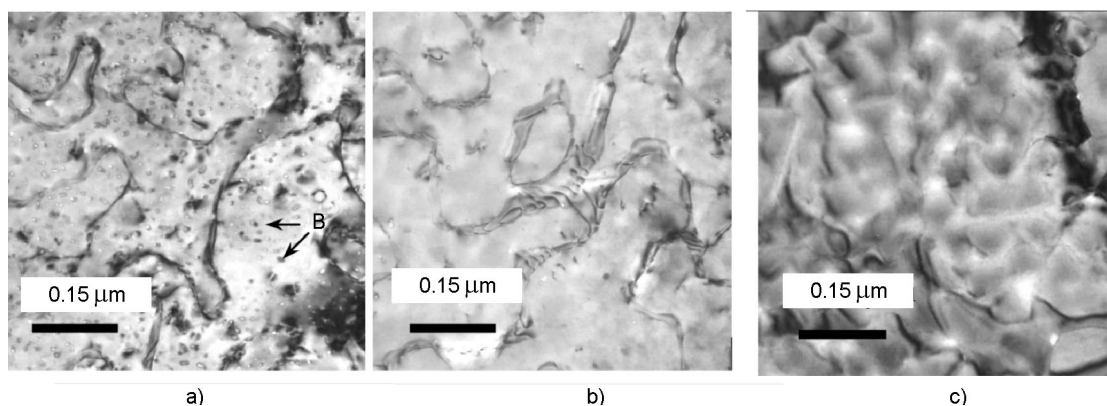


Fig. 3. Dependence of PbTe film structure on temperature T_s and the charge composition: (a) $T_s = 375$ K, $d = 65$ nm, undoped charge; (b) $T_s = 525$ K, $d = 75$ nm, doped charge; (c) $T_s = 635$ K, $d = 36$ nm, undoped raw material.

observe p -type conductivity in the films and as the electron concentration in the initial charge increases, the inversion point moves towards smaller d values. For instance, for the films obtained from doped and undoped charge, condensed at the substrate temperature 525 K, these values were $d_c \approx 110$ nm and $d_c \approx 25$ nm, respectively. Before, we have observed similar effects [19–21] in as-grown films of lead chalcogenides grown on (001) KCl: when the films were exposed in air, there appeared thickness dependence of thermoelectric properties and inversion of the conductivity type from n to p as d decreased.

When the films from undoped charge were condensed on the substrate at $T_s = 375$ K, the thickness interval where there was p -type conductivity became wider, and the critical thickness shifted towards larger values ($d_c > 250$ nm). If a protective coating (the about 20 nm thick Al_2O_3 layer) was applied to the films, the inversion point shifted towards smaller d . So, in the films obtained from undoped charge and condensed at $T_s = 525$ K, the inversion point was shifted to $d_c \approx 20$ nm, and in those obtained from doped charge, the p -type conductivity was observed at all thickness values. At $T_s = 375$ K, after applying the protective Al_2O_3 layer the critical thickness decreased to $d_c \approx 165$ nm. These facts indicate that, besides the surface oxidation of the film in open air, there are additional factors that stimulate the transition to p -type conductivity. The change of film stoichiometry with the T_s change can be considered to be among such factors.

It is known that the gas phase over PbTe crystals consists mainly of PbTe molecules,

but it is PbTe with excess tellurium that turns into vapor. The amount of free Te in the vapor over PbTe is small, but in time, when the stoichiometric composition evaporates, excess tellurium can accumulate in the condensate, resulting in the appearance of p -type conductivity. As the solid residue in this process must be enriched in lead, it becomes clear why one can usually find n -type conductivity in thick PbTe films regardless of initial charge composition [15, 19–21]. The conductivity sign in the films of any thickness will depend not only on d but also on many other factors, including the ratio of PbTe and Te condensation coefficients. One can expect this relation to be determined by T_s to a considerable extent.

The main stages of film formation process are adsorption, which results in nucleation, and the new phase growth because of surface atomic diffusion to the nucleation sites. T_s decreasing, the diffusion rate decreases and a part of the adsorbed atoms or molecules can re-vaporize before they attain the growing nucleus due to diffusion. If, at T_s decrease, the probability of Pb atoms re-vaporization increases, that is, their condensation coefficient decreases, there will appear Pb vacancies in the PbTe lattice. Such vacancies result in p -conductivity even in thin films with protective coating. Such situation is also likely in case $T_s = 375$ K.

On the other hand, the widening d interval where the conductivity is of p -type can be determined by purely thermodynamic factors. In fact, the change of condensation temperature must cause a change of thermodynamic equilibrium conditions and thus a change in the equilibrium concentration of defects in PbTe films. If the equilibrium becomes shifted towards the increase of Te

excess with respect to the stoichiometric composition, the number of cation vacancies, and so the concentration of *p*-type charge carriers, will increase. It is to note that we have also observed the increase of cation vacancies concentration at T_s decrease in the SnTe films grown on (001)/KCl crystals [22].

Comparing the electron concentrations and thermo-e.m.f. coefficient in the initial charge with average values of these parameters in "thick" PbTe films ($d = 400\text{--}600$ nm), when the contribution of oxidation processes to the kinetic coefficients can be ignored in the films without protective layer, we can see that in the films obtained from undoped charge, there is an increased electron concentration in comparison with bulk crystals ($n_b \approx 1.0 \cdot 10^{17} \text{ cm}^{-3}$ and $n_f \approx 7.0 \cdot 10^{17} \text{ cm}^{-3}$, respectively) and the decrease of thermo-e.m.f. coefficient ($S_b \approx 400 \text{ } \mu\text{V/K}$ and $S_f \approx 250 \text{ } \mu\text{V/K}$, respectively). On the contrary, for the films obtained from doped charge, the transition to thin-film state is accompanied by a decrease of electron concentration from $n_b = 1.0 \cdot 10^{20} \text{ cm}^{-3}$ to $n_f = 1.0 \cdot 10^{19} \text{ cm}^{-3}$ and the increase of thermo-e.m.f. coefficient from $S_b \approx 30 \text{ } \mu\text{V/K}$ to $S_f \approx 90 \text{ } \mu\text{V/K}$. So, the difference in n observed in doped and undoped bulk PbTe crystals used as charge (about 10^{17} and 10^{20} cm^{-3} , respectively) decreases in thin films (about 10^{18} to 10^{19} cm^{-3}).

In Fig. 4 the dependences of electron concentration n_f in "thick" films ($d = 400\text{--}600$ nm) obtained at $T_s = 525$ K on the electron concentration n_b in the bulk crystal used as the charge are shown. The data used here are taken from this work and the results from the works [19, 21], where PbTe films grown on (001)KCl substrates at $T_s = 525$ K were studied. The solid line in the Figure corresponds to the condition $n_b = n_f$. The dependence shown indicates that the best relation of n values in the film and in the charge is observed at $n_b \sim 2 \cdot 10^{18} \text{ cm}^{-3}$. This shows that in thin-film state at $T_s = 525$ K, the thermodynamic equilibrium conditions cause the equilibrium concentration of non-stoichiometry defects being close to the mentioned concentration value.

Thus, in this work, it has been established that at substrate temperature $T_s = 525$ K, PbTe growth on mica occurs epitaxially according to the island mechanism, mainly in the (111) orientation and the film becomes continuous at $d \approx 20$ nm. The two-position

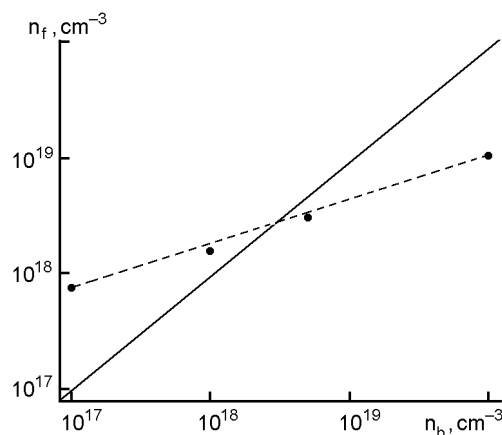


Fig. 4. Dependence of charge carrier concentration n_f in thick PbTe films on the current carrier concentration n_b in the charge. The straight line corresponds to the condition $n_f = n_b$ (the composition of the film and the charge coincide).

nucleation of PbTe crystallites of the (111) orientation causes the appearance of twin boundaries in the continuous film structure, the number thereof being increasing as the T_s decreases. In the films obtained from the doped charge, and containing charge carriers in a higher concentration, the trend to coalescence increases at the stage of island growth and the twin boundaries are more mobile, which is connected with the increase of surface diffusion rate as a result of the increase of structure defects concentration in the doped charge. It has been established that PbTe films prepared at $T_s = 625$ K exhibit a well-developed surface relief, which is undesirable for the preparation of heterostructures, and the films prepared at $T_s = 375$ K contain a great number of pores.

The influence of various factors (film thickness, substrate temperature, the charge carriers concentration in the charge, presence or absence of protective layer on the film) on the sign and concentration of charge carriers in PbTe thin films is revealed. It is established that for as-grown films, a certain critical thickness exists at which the transition from *n*-type to *p*-type conductivity is observed as the film thickness diminishes. Coating of the films with protective layer, lowering of the substrate temperature, and increasing of the electron concentration result in narrowing of the thickness range corresponding to hole conductivity. The effects observed are attributed to oxidation processes on the film surface exposed to air as well as to the changes

of condensation coefficients for molecules and atoms (PbTe, Pb, Te) in the condensate-vapor cloud system as the substrate temperature changes. It is shown that the difference in the electron concentration values in the charge (n_b) and thick PbTe films (n_f) grown at the substrate temperature $T_s = 525$ K depends on n_b and is maximum at $n_b \sim 2 \cdot 10^{18} \text{ cm}^{-3}$. This is attributed to the change of the thermodynamic equilibrium conditions in thin-film state compared with bulk crystals.

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Дослідження механізму росту, структури і термоелектричних властивостей тонких плівок PbTe на слюді

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Досліджено механізм росту, структура і термоелектричні властивості тонких плівок PbTe, одержаних термічним випаровуванням у вакуумі на підкладки із слюди. Варіювались товщина плівок ($d = 4\text{--}500$ нм), температура підкладки ($T_s = 375, 525$ и 635 К) та концентрація носіїв заряду у вихідній шихті ($n \cong 10^{17}$ и $n \cong 10^{20} \text{ cm}^{-3}$). Методом електронної мікроскопії встановлено, що PbTe росте на слюді епітаксіально за острівковим механізмом переважно в орієнтації (111). Встановлено, що у плівках PbTe існує критична товщина, при якій спостерігається перехід від електронної до діркової провідності при зменшенні d . Нанесення на плівки захисного покриття, зниження температури підкладки та збільшення концентрації електронів у шихті приводять до зменшення інтервалу товщин, який відповідає дірковій провідності. Показано, що значення концентрації електронів n у шихті і товстих плівках PbTe, одержаних при температурі підкладки $T_s = 525$ К, відрізняються, а характер і величина цієї відмінності залежать від n у шихті.