

Growth of Surface Micro- and Nanostructures During Depth Profiling of PbTe Crystals by Ar Plasma

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Peculiarities of depth profiling of PbTe crystals by Ar plasma with energy of 350 eV at the conditions of Secondary Neutral Mass Spectrometry originated from the crystal growth environment are presented. The crystals grown from vapor phase and from melt by the Bridgman method were studied. The natural faceted surface corresponding to the crystallographic plane of high symmetry (100), the natural lateral surfaces of crystal ingots, and the surfaces processed mechanically during cutting of the crystals were profiled. Nucleation, growth, and re-sputtering of the arrays of micro- and nanoscopic surface structures on the sputtered surfaces as a result of re-deposition of sputtered Pb and Te atoms were observed. It was determined that the growth environment of the PbTe crystal surfaces has a strong effect on nucleation and growth of the surface micro- and nanostructures in the conditions of continuous surface bombardment by Ar ions during depth profiling. This does not prevent the correct determination of the composition of the studied objects via the analysis of the composition of sputtered phase, if sputtering continues for at least 5-10 minutes.

Keywords: Secondary Neutral Mass Spectrometry, Depth Profiling, PbTe, Sputtering, Re-deposition.

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1. INTRODUCTION

IV-VI crystals and their solid solutions based on PbTe, SnTe and GeTe are widely used, in particular, for manufacturing of infrared photodetectors, lasers [1, 2], and spintronics structures [3-5]. Their physical properties are determined by both the intrinsic defects of crystal lattice and the foreign impurities. The impurities of rare earths elements are especially effective in controlling the system of intrinsic defects and other important parameters of these materials [6-11]. The use of doping processes requires control of the real distribution of the doping impurities in the doped crystals. Depth profiling with Secondary Neutral Mass Spectrometry (SNMS) is an efficient method for this. In particular, its use has allowed revealing the non-uniform segregation of the impurity of Europium at the PbTe crystals grown from doped melt by the Bridgman method [6]. More detailed studies of both the doped and undoped PbTe crystals by SNMS method recently allowed us to reveal the new phenomena of their sputtering by Ar⁺ plasma: aperiodical oscillations of Pb and Te sputtering; a huge preference of Te sputtering reaching more than two orders of magnitude at the beginning of sputtering process; and a significant excess of Te integrated sputter yield over that of Pb for prolonged sputtering by low energy plasma [14]. It was shown that these non-uniform sputtering phenomena are closely linked to the processes of re-deposition of the sputtered Pb and Te species on the PbTe crystal surface sputtering by Ar⁺ plasma. Such re-deposition leads to the formation of arrays of microscopic surface crystal structures on the sputtering surface in the conditions of its continuous bombardment by the ions of plasma during the depth profiling experiment.

In practice, there is a need for profiling of various structures with the surfaces of different quality. Taking

into account the features of profiling processes of the PbTe crystals by the SNMS method, mentioned above, one can suppose that the results of profiling of the structures with the same bulk properties may depend on the state of the surface of the investigated structure. To clarify this issue, we have studied two types of the PbTe crystals: grown from vapor phase and from melt by the Bridgman method. The different types of crystal surfaces were treated by Ar⁺ plasma: the natural faceted surface corresponded to the crystallographic plane of high symmetry (100), the natural lateral surfaces of crystal ingots, and the surfaces processed mechanically during cutting of the crystals. Results of the experimental studies are presented in this paper.

2. EXPERIMENTAL DETAILS

High-purity lead and tellurium were used for crystal growth. Despite a rather low content of background impurities, additional cleaning of the initial materials was performed before synthesis of the initial PbTe. Special attention was given to its clearing from oxygen, because both lead and tellurium oxidize easily. For this purpose, double vacuum distillation of tellurium was carried out. As to oxides of lead, which are formed at room temperature on its surface, they were completely removed by etching in aqueous solution of nitric acid (HNO₃:H₂O = 1:4) at 100 °C, followed by washing in chemically pure acetone.

The investigations were carried out on the PbTe crystal samples manufactured from two ingots. The first ingot was grown from melt by the Bridgman method using two quartz containers nested inside each other and evacuated down to 10⁻⁵ Torr. Before use both high-purity quartz glass containers were purified by the multi-stage procedure including washing with

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hot distilled water, steam cleaning and drying at 300 °C. In addition to this, the inner container was coated inside with a thin layer of pyrolytic graphite to avoid adherence of the grown ingot. To minimize adsorption of oxygen by the formed carbon coating, pyrolysis was carried out by vapouring of chemically pure acetone in an evacuated quartz container with subsequent burning at 1000 °C under vacuum pumping. The outer quartz container provided additional mechanical and vacuum protection in case of cracking of the inner container during the cooling of the ingot.

The second, completely single-crystal ingot was grown by method of free growth from the vapor phase, first proposed in [13] for preparation of II-VI single crystals from vapor phase. Its essence consists in removing of heat from the growing crystal by radiation through a light guide, which is the pedestal for crystal growth. Due to this, the crystal that is growing in the temperature gradient remains the coolest element of the system and the possibility of undesirable deposition of the initial PbTe both on the walls of the growth container and on the pedestal surface is completely excluded. Since the conditions for the formation of a block structure are absent, the resulting material can be up to 100 % single crystal. Crystallization takes place during the process of re-sublimation of the previously synthesized stoichiometric polycrystalline PbTe, and the solid phase is constantly in equilibrium with the vapor of the stoichiometric composition, which prevents the possibility of formation of the second phase inclusions. Moreover, absence of contact between the crystal and the walls of the growth container eliminates residual mechanical stresses. Due to this, we obtained a high quality single crystal of cylindrical shape with natural faceting by the crystallographic plane of (100) at the end of the ingot.

Sputtering experiments were carried out on INA-X type SNMS system produced by SPECS GmbH, Berlin. The surface morphology of samples after ion sputtering has been analyzed by SEM (Hitachi S-4300 CFE). The experimental conditions were the same as it is described in detail in [12].

3. EXPERIMENTAL RESULTS

Investigating the processes of depth profiling of PbTe crystals under SNMS conditions we noted [12] that the presence of oscillatory sputtering of PbTe components will affect the determination of concentrations of various elements in the crystal matrix. We concluded that to reduce this effect measurement is best done with energy of the Ar⁺ ions of about 300 eV or higher. Accordingly, the studies on the influence of PbTe crystal surface quality on the processes of re-deposition of the sputtered components were carried out with sputtering energy of 350 eV. The measurement duration was about 50 minutes.

The principal experimental results, which show the impact of sputtering on the state of the sputtered surfaces, are presented in Fig. 1-4.

Before we examine the obtained results, we have to note that the initial natural surfaces of the all crystal

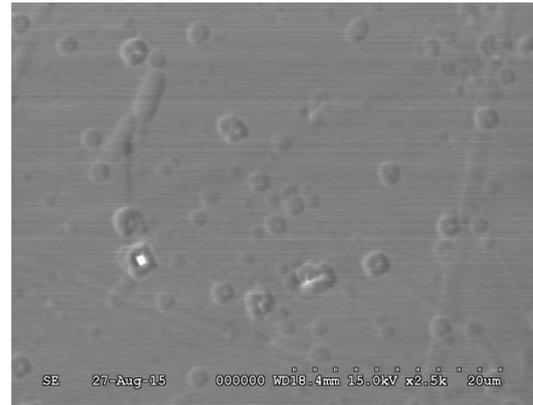


Fig. 1 – SEM image of (100) natural faceting surface of the PbTe crystal grown from vapor phase after sputtering by Ar plasma with 350 eV ions energy during 50 minutes

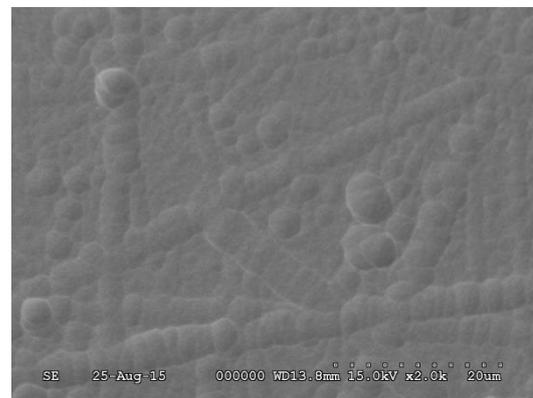


Fig. 2 – SEM image of lateral surface of the PbTe crystal grown from vapor phase after sputtering by Ar plasma with 350 eV ions energy during 50 minutes

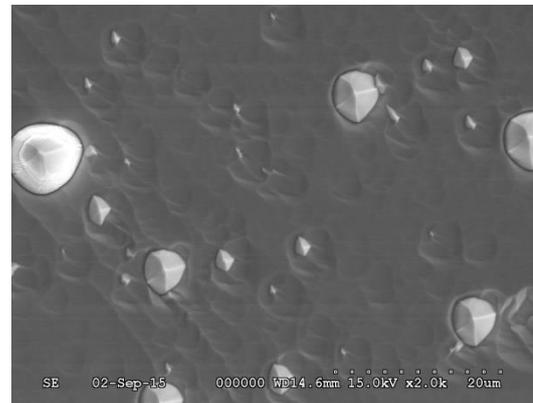


Fig. 3 – SEM image of lateral surface of the PbTe crystal grown from melt by the Bridgman method after sputtering by Ar plasma with 350 eV ions energy during 50 minutes

samples were smooth and had a characteristic uniform metallic gloss. The surfaces processed mechanically during the cutting of the crystals were uniformly dull. With this in mind it can be seen that morphology of the sputtered crystal surface is significantly modified under impact of Ar⁺ beams during the sputtering process, and the growth environment of the PbTe crystal surfaces makes a very strong effect on the character of those modifications.



Fig. 4 – SEM image of the surface processed mechanically during cutting of the PbTe crystal after sputtering by Ar plasma with 350 eV ions energy during 50 minutes

The most obvious common feature of the all modified surfaces is their dimple relief. However, the character of the relief significantly changes from surface to surface. Another feature is the formation of small surface structures from the sputtered phase on the sputtering surface under continuous bombardment with an Ar ions beam. These processes also proceed in different ways on the crystal surfaces of different origins and quality.

The free natural facet crystal surface is sputtered by Ar⁺ ion beam uniformly (Fig. 1). Here the dimple relief has a typical appearance of the dislocation pits of etching. Mostly the single etch pits are present. It is much rarer to observe separate dislocation chains. The shape of the etch pits is similar to square. The squares are uniformly oriented on the sputtered surface. The square shape of the etch pits is typical for the (100) crystallographic orientation of the PbTe crystal surface. Occasionally one can observe small crystal formations as the islands of re-deposited phase within the dislocation etch pits (the light spot in Fig. 1). For greater clarity one of them is shown in Fig. 5 at higher magnification in the geometry of back scattering of electron as the example. Identical color of both the sputtered surface and the microscopic pyramid on the bottom of etch pit shows that the re-deposited structure has the same composition as the sputtered surface.

Similarly, sputtering by Ar plasma modifies the lateral surface of the crystal grown from vapor phase

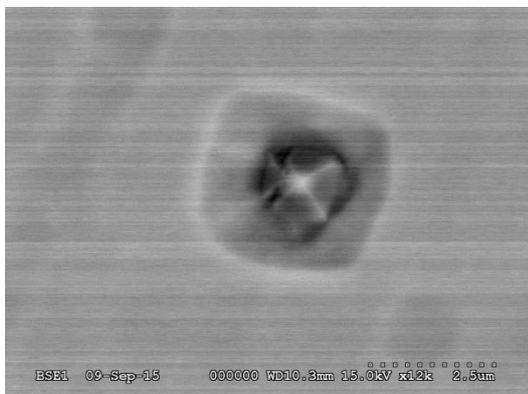


Fig. 5 – Backscatter electron image of the same PbTe crystal surface as in Fig. 1 at higher magnification (Fig. 2). But the density of dislocation etch pits on the

lateral surface is much higher than on the sputtered natural facet surface. Also it modifies the shape and the relative position of the etch pits. The shape becomes diffuse, the pits merge with each other and create a large number of dislocation chains of different space orientation. The situation with formation of the surface crystal structures from the sputtered phase is similar to that observed on the previous surface.

Both the surface of PbTe crystal grown from melt by the Bridgman method and the surface treated mechanically look quite different after sputtering by Ar ions.

In case of the Bridgman PbTe crystal the sputtering process forms the well-developed dimple relief on the sputtered lateral crystal surface (Fig. 3). There is a well-defined both the shape and the relative location of the dimples of the relief on the sputtered surface.

Appearance of the arrays of small microscopic structures on the sputtered surface as a result of re-deposition of sputtered Pb and Te atoms is another noticeable effect that accompanies the depth profiling of Bridgman PbTe crystal. As one can see the dimples of the sputtering surface relief are efficient sites for nucleation and growth of the surface crystal formations. The dominant forms of the re-deposited formations are pyramidal structures with natural faceting correlating with cubic structure of PbTe crystal lattice.

Mechanically treated PbTe crystal surface is the one most strongly modified during depth crystal profiling. Firstly, its dimple relief is extremely developed. At the same time there isn't any ordering neither in the form nor in the mutual arrangement of the dimples of the relief, which overlap and completely cover the entire sputtering surface. Secondly, the arrangement of the re-deposited surface structure, which covers the sputtering surface, radically changed. In addition to the pyramidal structures form on the sputtered lateral surface of the PbTe crystal grown from melt by the Bridgman method, typical for cubic crystal lattice, there are a large number of the big re-deposited structures without any defined shape and spatial crystallographic orientation (Fig. 4).

4. DISCUSSION

Formation and growth of the different crystal structures on the sputtered surface of solids in the conditions of continuous impact of sputtering ions is a well-known phenomenon [12, 14, 15]. In this study, it is of interest for us from the point of view of the influence of the method of crystal growth and surface treatment on the resulting state of the surface that is sputtered by the ion beams.

The first issue that needs to be explained is why the results of re-deposition of sputtered Pb and Te species on the sputtered surfaces of the PbTe crystals grown from vapor phase and from melt are different despite the conditions of the crystal sputtering being the same.

It is well known that deposition on the solid substrate from vapor phase depends on the conditions for nucleation of the new phase on the substrate surface and supersaturation of the vapor phase over the substrate. The conditions for nucleation depend first of all on the quality of substrate surface – the worse is surface quality, the better are conditions for nucleation.

Quality of the vapor phase crystals is higher than quality of the crystals grown from melt by the Bridgman method. This can be one of the reasons why re-deposition of sputtered species in the latter case is much more intense than in the former. Another reason can be the following. The resulting re-deposited surface structures are observed mainly within the dimples of the sputtered surface relief (Fig. 3, Fig. 5). Thus if a sufficient number of new phase nuclei have not formed at the initial stages of sputtering of the surface, it will certainly not be formed at later stages.

Let us now examine another factor, namely, supersaturation of the sputtered phase over the different sputtered surface. We cannot estimate the actual degree of supersaturation of the sputtered phase over each individual surface, but we can make their comparative evaluation through comparison of the profiling depth for different sputtered surface. For this purpose after sputtering process the shape of the sputtered crate was measured by AMBIOS XP-I type profilometer, used its in the line scan mode. By measuring the vertical stylus displacement as a function of position we were able to measure surface topography with nanometer vertical resolution with stylus load of 1 mg. One of these profiles for the crystal grown from melt by the Bridgman method as an example is shown in Fig. 6. The shape of the crater is close to square, which confirms the homogeneity of the lateral energy distribution of Ar^+ ions over the sputtered area. The process of sputtering and re-deposition of the sputtered species results in not smooth erosion area covered with pyramidal structures of different height and width, as it is confirmed by the results of scanning electron microscopy (e. g. Fig. 3).

The depth of the crater, as it can be seen in the Fig. 6, is about $8\ \mu\text{m}$. This allows estimating the rate of surface sputtering as $2.2\ \text{nm/s}$. Approximately the same rate was obtained for sputtering of the lateral surface of the PbTe crystal grown from vapor phase. At the same time for the (100) natural faceting surface of the PbTe crystal grown from vapor phase this value was almost two times lower.

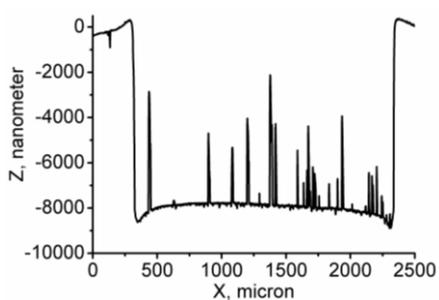


Fig. 6 – The profilogram of lateral surface of PbTe Bridgman crystal, sputtered about 50 minutes by Ar plasma with ions energy of 350 eV through the hole in Ta mask of 2 mm diameter

The rate of surface sputtering is one of the main factors determining the degree of supersaturation of the sputtered phase above the surface. So, one may think that for the (100) surface of the vapor phase crystal the last is about two times lower than for both the lateral surfaces. But there are no fundamental differ-

ences in the appearance of re-deposited structures on the sputtered (100) and lateral surfaces of the vapor phase crystal. They are practically absent there. On the other hand, the rates of depth profiling of the lateral surfaces of both the vapor phase and Bridgman crystals are very close, but the pictures of their re-deposited structures are drastically different. So, we have to conclude that the obtained difference in the formation of the arrays of the re-deposited surface structures for the vapor phase and Bridgman PbTe crystals is caused mainly by the difference in quality of the sputtered surfaces.

Another important question is how the processes of re-deposition impact the sputter yield of Pb and Te for the different PbTe crystal surfaces. For this purpose the sputtering spectra of the PbTe crystal samples were analyzed (Fig. 7 and Fig. 8).

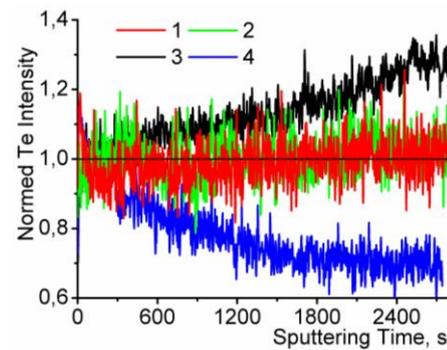


Fig. 7 – Te signal intensity normalized by the mean value over first 120 seconds sputtering vs. sputtering time for different PbTe crystal surfaces: 1 – (100) natural faceting surface (Fig. 1); 2 – lateral surface of vapor phase crystal (Fig. 2); 3 – lateral surface of Bridgman crystal (Fig. 3); 4 – mechanically processed surface (Fig. 4)

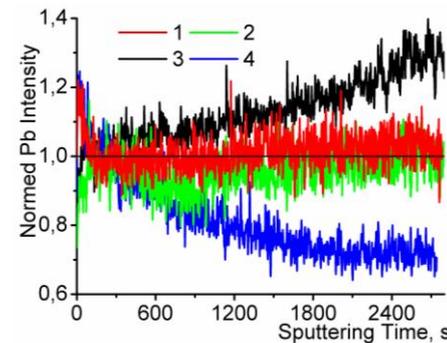


Fig. 8 – Pb signal intensity normalized by the mean value over first 120 seconds sputtering vs. sputtering time for different PbTe crystal surfaces. The designations are the same as in Fig. 7

Two fundamentally different behaviors can be seen.

One of them is independent of the average value of Te and Pb sputtering signal intensity from sputtering time in the process of crystal depth profiling not taking into account the initial 3-5 minutes of sputtering (the curves 1 and 2 in Fig. 7 and Fig. 8). That is the case for both the natural (100) faceting and the lateral surfaces of the crystal grown from vapor phase. It is those surfaces for which re-deposition of sputtered species during depth profiling is not observed. It must be so exact-

ly in the case when the macroscopic amount of matter is sputtered, and sputtering is the only process determining formation of the sputtered phase during the depth profiling [16, 17].

Another one is pronounced changes of the average Te and Pb sputtering signals over sputtering time (the curves 3 and 4 in Fig. 7 and Fig. 8). For the lateral surface of the PbTe crystal grown from melt by the Bridgman method the signal intensity significantly increases during crystal depth profiling, and for the surface processed mechanically, on the contrary, it decreases. Both of these surfaces are the surfaces for which there is an intense formation of surface structures in the conditions of continues bombardment by Ar ions (Fig. 3 and Fig. 4). This situation is similar to the one we observed for sputtering of PbTe crystal surfaces of the same type by Ar ions of different energies [12]. In the conditions of formation of the re-deposited surface structures on the sputtering surface the direction of the sputtering output changes over time as determined by the relation between the losses of sputtered phase because of the formation of the stable surface structures and its replenishing due to re-sputtering of the re-deposited structures. Explicitly, during sputtering the surface processed mechanically the losses dominate and the sputtering output decreases over time, while for the lateral surface of the PbTe Bridgman crystal the situation is reversed.

The last important issue which has to be considered is whether the sputtering spectra in the conditions of intense re-deposition of the sputtered species on the sputtering surface can be used for quantitative profiling of crystalline PbTe samples. To answer this question let's consider the behavior over time the ratio between the outputs of Pb and Te for different PbTe sputtering surfaces. The corresponding experimental data are shown in the Fig. 9.

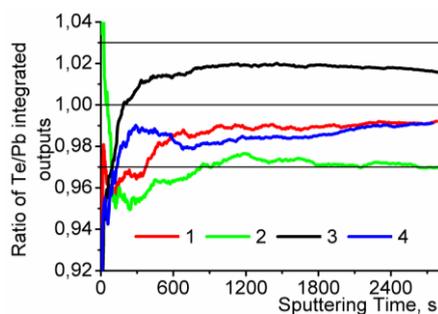


Fig. 9 – Ratio of integrated outputs Te/Pb vs. sputtering time for different PbTe crystal surfaces. The upper and lower horizontal lines denote a deviation of 3% of the unit. The designations are the same as in Fig. 7

According to Sigmund's linear collision cascade theory [22], if the beam energy is much more than the surface binding energy the ratio of partial sputter yields Y of 1 and 2 elements of diatomic system with the surface element concentrations N_i^S is $Y_1/Y_2 = f_{12} \cdot (N_1^S/N_2^S)$, where parameter f_{12} depends on the masses M_i and the surface binding energies U_i of the elements.

In a homogeneous sample the concentration of elements on the sample surface N_i^S is equal to their concentration N_i^b in the sample's bulk. Therefore, under preferential sputtering, when $f_{12} \neq 1$, at the beginning of sputtering the ratio of sputter yields of the elements does not equal the ratio of concentrations of the elements in the bulk of the sputtered sample. In accordance with the law of conservation of mass they must become equal at longer timescales, when the conditions of equilibrium sputtering are achieved [16]. Concerning PbTe this means that for large sputtering times, when the macroscopic amount of material is sputtered, the ratio must be equal to unity. As can be seen from Fig. 9, after 5-10 minutes of sputtering in the range of an error that does not exceed 3% this criterion is well satisfied for all sputtered surfaces, irrespective of how intense the re-deposition of sputtered species and the formation of re-deposited surface structures of the new phase was. Thus, despite the fact that the intense re-deposition of sputtered species and formation of the arrays of re-deposited structures strongly affects the average value of sputter outputs of Pb and Te, the sputtering spectra of the PbTe crystals can be used for quantitative profiling of crystalline PbTe objects if their sputtering is carried out for at least 5-10 minutes.

5. CONCLUSIONS

We investigated the processes of depth profiling of the PbTe crystals grown from vapor phase and from melt by the Bridgman method by Ar^+ ions plasma with energy of 350 eV through the surfaces of different origin and quality. We found that profiling of the crystal samples grown from melt or the samples with mechanically treated surfaces is accompanied by the intense growth of micro- and nanostructures on the sputtering surface. At the same time this effect is practically not observed for natural surfaces of the crystal samples grown from vapor phase. Intense re-deposition of the sputtered species and growth of the surface structures have a significant impact on time dependence of the average outputs of sputtering of Pb and Te. At the same time they do not actually affect the ratio of the outputs under sputtering of the macroscopic amount of material which, to the accuracy of 3%, corresponded to the stoichiometric composition of the elements in PbTe. Thus, despite the fact that the intense re-deposition of sputtered species and formation of the arrays of re-deposited structures strongly affects the average value of sputter outputs of Pb and Te, the sputtering spectra of the PbTe crystals can be used for quantitative profiling of crystalline PbTe objects if their sputtering is carried out by Ar plasma with energy of 350 eV for at least 5-10 minutes.

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Ріст поверхневих мікро- і наноструктур у процесі профілювання вглиб кристалів PbTe плазмою Ag

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Представлені особливості профілювання вглиб кристалів PbTe плазмою Ag енергії 350 eV методом вторинної нейтральної маспектрометрії, що спричиняються умовами росту кристалів. Дослідження проведено на кристалах, вирощених з парової фази та з розплаву методом Бріджмена. Різні поверхні були розпорощені: поверхня природної огранки, що відповідає кристалографічній площині високої симетрії (100), природні бокові поверхні кристалічних зливків та поверхні, оброблені механічно під час розрізання кристалів. На розпорощених поверхнях спостерігали зародкоутворення, ріст і повторне розпорощення масивів мікро- і наноскопічних поверхневих структур, переосаджених з розпорощених атомів Pb і Te. Встановлено, що ростові умови формування поверхонь кристалів PbTe дуже сильно впливають на процеси зародкоутворення і росту на них поверхневих мікро- і наноструктур в умовах неперервного бомбардування поверхні іонами Ag під час профілювання кристалів вглиб. Показано, що це не перешкоджає належній оцінці складу досліджуваних об'єктів шляхом аналізу складу розпорощеної фази, якщо розпорощення проводити щонайменше 5-10 хвилин.

Ключові слова: Вторинна нейтральна мас-спектрометрія, Профілювання вглиб, PbTe, Розпорощення, Переосадження.

Рост поверхностных микро- и наноструктур в процессе профилирования по глубине кристаллов PbTe плазмой Ag

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Представлены особенности профилирования по глубине кристаллов PbTe плазмой Ag энергии 350 эВ методом вторичной нейтральной масс-спектрометрии, порождаемые условиями роста кристаллов. Исследованы кристаллы, выращенные с паровой фазы и с расплава методом Бриджмена. Было проведено распыление различных поверхностей: поверхности естественной огранки, соответствующей плоскости высокой симметрии (100), естественных боковых поверхностей кристаллических слитков и поверхностей, обработанных механически во время разрезания кристаллов. На распыленных поверхностях наблюдали зарождение, рост и повторное распыление массивов микро- и наноскопических поверхностных структур, переосаждаемых с распыленных атомов Pb и Te. Установлено, что ростовые условия формирования поверхностей кристаллов PbTe очень сильно влияют на процессы зарождения и роста на них поверхностных микро- и наноструктур в условиях непрерывного бомбардирования поверхности ионами Ag во время профилирования кристаллов по глубине. Показано, что это не препятствует надлежащей оценке состава исследуемых объектов путем анализа состава распыленной фазы, если распыление проводить не менее 5-10 минут.

Ключевые слова: Вторичная нейтральная масс-спектрометрия, Профилирование по глубине, PbTe, Распыление, Переосаждение.

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