

Liquid Phase Epitaxy of Thin Isoperiodic Heterostructures of $\text{Pb}_{1-x}\text{Sn}_x\text{Te}_{1-y}\text{Se}_y$ Solid Solutions

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Results of the study of the initial stages of growth by the liquid phase epitaxy (LPE) method of isoperiodic structurally advanced heterostructures (HSs) based on solid solutions of the Pb-Sn-Te-Se system are presented in the paper. Theoretical analysis has shown that three principally different cases can be distinguished for the growth of $\text{Pb}_{1-x}\text{Sn}_x\text{Te}_{1-y}\text{Se}_y$ multicomponent solid solutions (MSS) on $\text{PbTe}_{0.88}\text{Se}_{0.12}$ and $\text{Pb}_{0.70}\text{Sn}_{0.30}\text{Te}$ substrates. It is shown that for $\text{Pb}_{0.70}\text{Sn}_{0.30}\text{Te}$ substrates in the case where the substrate and the epitaxial layer (EL) are isoperiodic at some average temperature between the growth temperature and the operating temperature of the device, it is possible to obtain ELs with the red photoelectric limit of 16-24 μm at operating temperatures, for which critical thicknesses $\geq 4 \mu\text{m}$, by the LPE method. The peculiarities of the LPE technology of quasi-isoperiodic HS obtaining, used in the work, equipment and methods of research are described. For example, at low cooling rates ($0.1 < v < 0.3 \text{ K/min}$), regardless of the temperature reduction range, which varied within $1.0 < \Delta T < 5.0 \text{ K}$, mirror-smooth ELs with a thickness of 1-3 μm and the surface dislocation density of less than $2 \cdot 10^5 \text{ cm}^{-2}$ were obtained. However, the study of the substrate/layer interface showed the formation, although thin $\sim 0.2\text{-}0.4 \mu\text{m}$, of transition layers. Further experimental studies have shown that a more promising LPE technology in this case is the method of pre-cooling of the solution-melt. At programmatic lowering of the temperature with velocity $v < 0.3 \text{ K/min}$ and initial cooling of 1-3 K, ELs were either mirror-smooth or had a light terraced structure, which may be due to the slight misorientation of the substrates relative to the growth plane.

Keywords: $\text{Pb}_{1-x}\text{Sn}_x\text{Te}_{1-y}\text{Se}_y$, Liquid phase epitaxy, Initial stages of growth, Cooling, Interface.

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1. INTRODUCTION

Issues related to environmental monitoring, the search for materials for high-effective thermo- and photoelectric converters are of particular relevance at the present stage of human activity. The main working materials of such devices are narrow-gap semiconductors based on solid solutions like A_4B_6 and A_2B_6 . The use of $\text{Pb}_{1-x}\text{Sn}_x\text{Te}_{1-y}\text{Se}_y$ multicomponent solid solutions (MSS), which allow to smoothly change the value of the band gap (E_g) and the lattice parameter (a) by changing the chemical composition is relevant. This property is promising for the production of isoperiodic epitaxial layers (ELs) – heterostructures (HSs) with a pre-set value of E_g in the working layer [1]. It is also important that we can change the width of the forbidden zone in such MSS, reducing it to almost zero, which makes it possible to use them in the process of designing devices for the far infrared region [2-4].

Among the current trends in the development of materials for electronic equipment, the relevance of thin-film technologies [3], which can be implemented by various modern technologies: thermal evaporation in vacuum, gas-phase epitaxy, epitaxy from molecular beams and others, should be noted. The well-known method of liquid-phase epitaxy (LPE) has several advantages compared to others: relatively simple equipment, high growth rates, the possibility of a wide selection of dopants, the simplicity and convenience of contacting the melt solution with the substrate, the ability to adjust the

deviation of the composition from stoichiometry upon receipt of epitaxial structures based on compounds, high industrial capacity and economy of the processes. However, the LPE of HSs of semiconductor materials takes place under non-equilibrium conditions due to the difference in chemical compositions and the mismatch between the periods of the crystal lattices of the substrate or the previous epitaxial layer with the liquid phase of the solid melt [5-6]. For this reason, a saturated multicomponent solution-melt, which is an equilibrium with a specific solid phase at liquidus temperature, according to the state diagram, is not equilibrium with the substrate or EL of a different composition at the same temperature. The lack of thermodynamic equilibrium of the solution-melt and the substrate (the previous EL) leads to its partial or even complete dissolution, which is unacceptable for instrumental HS [7, 8]. It is also well known the LPE method, which is aimed at the stability of a substrate to a nonequilibrium liquid phase – overcooling the growth solution [8]. Nevertheless, for the controlled conduction of LPE in the case of heteroepitaxy, it is necessary to know the amount of supercooling. This condition is due to the need to ensure the stability of the substrate, which in turn will not lead to an uncontrolled crystallization rate of the overcooled solution-melt and will ensure the structural perfection of the ELs.

Finally, it was shown [7] that for sufficient device efficiency, the consistency of the crystal lattices of the working layers should be at the level $\Delta a_0/a_0 \leq 2.5 \cdot 10^{-4}$.

Interest in studying the surface of epitaxial layers

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and interfaces between homo- and heterostructures of solid solutions like A_4B_6 appeared quite a long time ago, and in recent years has been stimulated by the development of micro- and nanotechnologies, the general trend towards miniaturization of electronic devices. It manifests itself in reducing the thickness of the working structures, the synthesis of complex multilayer and nanostructured compositions.

In this work, we investigated the initial stages of growth using the LPE method of isoperiodic structurally perfect HS based on solid solutions of the Pb-Sn-Te-Se system.

$$E_g = 0.32 - 0.03 \cdot y - (0.59 - 0.69 \cdot y) \cdot x \text{ (eV)},$$

$$a = 6.461 - 0.3345 \cdot y - 0.134 \cdot x - 0.0175 \cdot y \cdot x \text{ (\AA)}, \quad (1)$$

$$\partial a / (a \cdot \partial T) \cong (19.9 + 5.1 \cdot x - 1.3 \cdot y - 3.6 \cdot y \cdot x) \cdot 10^{-6} \text{ (K}^{-1}\text{)}.$$

Let us estimate the theoretical possibilities of obtaining structurally perfect HS of $Pb_{1-x}Sn_xTe_{1-y}Se_y$ MSS on $PbTe_{0.88}Se_{0.12}$ and $Pb_{0.7}Sn_{0.3}Te$ substrates. It is easy to show that if the EL is matched by the crystal lattice parameter at 300 K with these substrates, and then the temperature is raised to real epitaxy temperatures (~ 900 K), then there is a relative inconsistency in the parameters of the EL and substrate lattices ($\Delta a/a \leq 1.0 \cdot 10^{-3}$), which is greater than $\Delta a_0/a_0$. Moreover, this value grows as the difference in tin content in the EL and substrate increases. Thus, the maximum values are achieved with the use of $PbTe_{0.88}Se_{0.12}$ substrates, and the minimum values are achieved with $Pb_{0.7}Sn_{0.3}Te$. If the LPE processes are carried out at lower temperatures, for example, about 753 K, then the lattice parameter in the EL, as before, will be greater than that in the substrate ($\Delta a/a \leq 7.6 \cdot 10^{-4}$), that is already close to $\Delta a_0/a_0$. But the problem is that when the HS is cooled to 80 K (the actual operating temperature of the instruments), the substrate will already have a larger lattice parameter, with a relative inconsistency of approximately $\Delta a/a \leq -3.2 \cdot 10^{-6}$.

Thus, we can distinguish three fundamentally different cases:

1) Growth on the substrate, isoperiodic with ES at the LPE temperature. At the same time, during growth, there are no stresses in the layers, and accordingly, they do not affect the process of layer formation. But when cooling such HS to ~ 80 K, the total maximum discrepancies in the ES for epitaxy temperatures of 873 K and 723 K increase: $\Delta a/a \leq 1.0 \cdot 10^{-3}$ and $\Delta a/a \leq 0.7 \cdot 10^{-3}$ respectively.

2) The substrate and the EL are isoperiodic at 300 K. At the same temperatures of the epitaxy, we obtain the maximum inconsistency of the lattice parameters of the layer and the substrate ($\Delta a/a \leq 1.0 \cdot 10^{-3}$, $\Delta a/a \leq 1.0 \cdot 10^{-3}$) during the growth. It can significantly change the growth mechanisms and affect the quality of the layers. However, the stresses of the mismatch are partially compensated, and can practically be absent at operating temperatures, if they did not relax during growth.

3) The substrate and the EL are isoperiodic at some intermediate temperature between the temperature of the growing process and the temperature of 300 K. If we take a certain arithmetic mean temperature between the growth temperature and the operating temperature of

2. THEORY AND EXPERIMENTAL TECHNIQUE

2.1 HS Modeling in the Pb-Sn-Te-Se System

Most often, to create HSs based on solid solutions of the Pb-Sn-Te-Se system, substrates of PbTe and PbSe binary compounds are used, as well as $Pb_{1-x}Sn_xTe$ and $PbTe_{1-y}Se_y$ solid solutions, as more stable to a liquid melt solution than binary substrates. To calculate the band gap (E_g), the lattice parameter (a) and the dependence of the thermal expansion coefficient ($\partial a / (a \partial T)$), let us use the well-known relations [2, 5]:

the device, then the maximum value of the discrepancy will be approximately the same at both the growth temperature and the operating temperature. The calculation shows that in this case, the mismatches do not exceed the values $\Delta a/a \leq 5.0 \cdot 10^{-4}$ for the $PbTe_{0.88}Se_{0.12}$ substrate and $\Delta a/a \leq 2.8 \cdot 10^{-4}$ for the $Pb_{0.7}Sn_{0.3}Te$ substrate. These estimated results are commensurate with [7] for conditions, under which the high efficiency of working structures, both laser diodes and heterostructural receivers, remains.

According to [9], the critical thickness H , upon reaching which, the generation of misfit dislocations begins in the plane of the interface between the EL/substrate (that is, the EL can be elastically deformed only to a certain thickness, the excess of which leads to the formation of misfit dislocations):

$$H = (b / (8 \cdot \pi \cdot (1 + \nu))) \cdot \ln(H/b + 1), \quad (2)$$

where b , ν are the Poisson's index and Burgers vector, respectively. For the numerical evaluation, we used the typical values A_4B_6 of these quantities for compounds of the A_4B_6 type given in the literature [1].

Studies of a wide range of compositions of solid solutions of the Pb-Sn-Te-Se system show that with growth on the $Pb_{0.7}Sn_{0.3}Te$ substrates and the $Pb_{1-x}Sn_xTe_{1-y}Se_y$ EL for $x = 0.23-0.32$, thermal disagreement of lattice parameters, when the temperature changes by 100 K, does not exceed the estimated value. In this case, the maximum is reached at $x = 0.26$, which corresponds to the red border of the photoelectric effect in the EL for $17 \mu\text{m}$.

Moreover, using all possible compositions that are isoperiodic with the $Pb_{0.7}Sn_{0.3}Te$ substrate, one can obtain a $Pb_{1-x}Sn_xTe_{1-y}Se_y$ EL with a red photoelectric effect border at operating temperatures in the wavelength range of $16-24 \mu\text{m}$, for which critical thicknesses $\geq 4 \mu\text{m}$ (see Fig. 1), which may well be implemented by the LPE method.

2.2 LPE Technology of Quasi-isoperiodic HS

$Pb_{1-x}Sn_xTe_{1-y}Se_y$ ELs were grown by the LPE method on an upgraded "Epos" installation with vertical placement of the quartz reactor and horizontal arrangement of substrates in the graphite-rotating cassette [2].

Program control, monitoring and maintenance of the temperature-time regime in the growing zone, as well

as temperature recording, were carried out using a high-precision controller based on a personal computer. The program took into account the inertial characteristics of the heaters and the reactor. Digital filtering of the input signal from the thermocouples was carried out. Temperature characteristics were displayed in real time with the possibility of switching to manual control mode via the program interface.

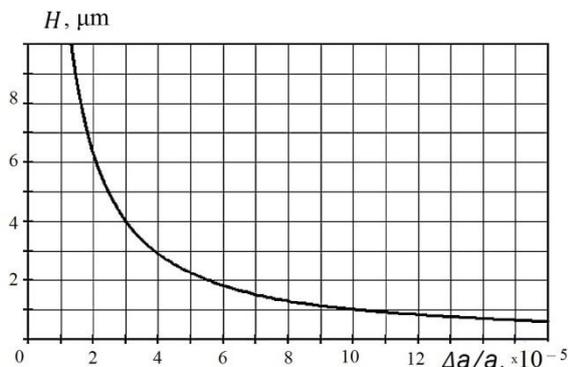


Fig. 1 – Dependence of the critical thickness on the mismatch between the periods of the $\text{Pb}_{1-x}\text{Sn}_x\text{Te}_{1-y}\text{Se}_y$ ELs and semiconductor substrates crystal lattices

The accuracy of maintaining the temperature in the working area was ~ 0.3 K, the cooling rate was regulated within 0.1-2.0 K/min. The growth processes were carried out in a stream of hydrogen purified through a palladium filter to a dew point no higher than 200 K. For the preparation of growth solutions, Pb, Sn, Te, and Se elements with the highest purity were used. Substrate material was washers with a diameter of 20 mm of $\text{Pb}_{0.70}\text{Sn}_{0.30}\text{Te}$ {100} single crystals obtained by the Czochralski method. The surface density of dislocations in the substrates was within $N_d = (2-14) \cdot 10^5 \text{ cm}^{-2}$. Preparation of the initial solution-melt components and substrates for LPE processes was standard. The initial epitaxy temperatures were within 840-820 K, and the temperature drop range did not exceed 5 K, which made it possible to obtain an EL with a thickness of 1-3 μm . Upon reaching the working temperature, the solution-melts were homogenized for ~ 2 hours. The growth of EL was carried out from a limited volume of solution-melt formed by two substrates with a distance of 1 mm, which made it possible to minimize the amount of consumed solution-melt. At the end of the growth of the EL, the solution-melt was removed from the surface of the substrates by centrifugation.

Particular attention was paid to the complete extraction of the growth melt, which has low enough coefficient of surface tension. It was possible to obtain a mirror-smooth surface of the EL without residual melt solution by intensive centrifugation (~ 1000 rpm) of the cassette with the substrates.

To calculate the composition of the liquid phase $(\text{Pb}_{1-v}\text{Sn}_v)_{1-w}(\text{Te}_{1-u}\text{Se}_u)_w$, we used the coherent state diagram of the Pb-Sn-Te-Se system. The composition of the equilibrium liquid phase $(\text{Pb}_{1-v}\text{Sn}_v)_{1-w}(\text{Te}_{1-u}\text{Se}_u)_w$ varied over a wide range: $0.163 \leq v \leq 0.371$ atomic parts (a. p.), $0.011 \leq u \leq 0.086$ (a. p.), $0.01 \leq w \leq 0.05$ (a. p.) to obtain a $\text{Pb}_{1-x}\text{Sn}_x\text{Te}_{1-y}\text{Se}_y$ solid solution along the isoperiodic line.

An “LCD Micro” microscope and a “REM-106I” electron scanning microscope with an energy dispersive microanalysis system were used to study the surface micro-relief. The latter is also used to measure the mass fraction of elements in the solid phase. For this purpose slanting grindings of the studied samples, polished at an angle of 3° , were prepared. The limits of the relative measurement error of the mass fraction of elements by an X-ray energy dispersive spectrometer were not more than 4 % for elements with a mass fraction of more than 10 % and not more than 20 % for elements with a mass fraction range from 1 % to 10 %. The resolution of the spectrometer on the Mn $K\alpha$ line was no more than 143 eV.

The boundary of the HS was detected by etching the chips and grinding sections in $\text{HNO}_3:\text{H}_2\text{O} = 1:4$ warm solutions. To study the density of dislocations we used boiling etchant: 50 g KOH + 10 ml of 0.5 % water solution KJO_3 .

3. RESULTS AND DISCUSSION

At low cooling rates ($0.1 < v < 0.3$ K/min) we obtained mirror-smooth 1-3 μm thick ELs with a surface density of dislocations less than $2 \cdot 10^5 \text{ cm}^{-2}$, irrespective of the temperature drop range, which was changed within $1.0 < \Delta T < 5.0$ K (see Fig. 2). However, the study of the substrate/layer interface showed the formation of transition layers (albeit thin $\sim 0.2-0.4 \mu\text{m}$), apparently due to the disequilibrium of the solid and liquid phases (Fig. 3a). This hypothesis is supported by the fact that with an increase in the amount of Se in the liquid phase from 0.011 to 0.086 (a. p.), the thickness of the transition layer increased. Such violations of the planarity of the heterointerface usually negatively affect the performance of devices based on them.

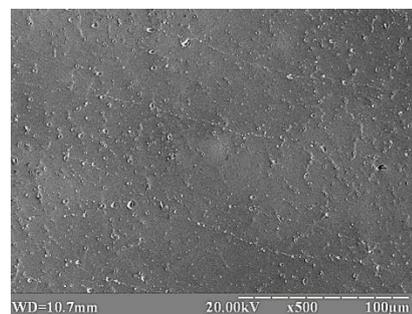


Fig. 2 – Etched surface of the $\text{Pb}_{0.749}\text{Sn}_{0.251}\text{Te}_{0.980}\text{Se}_{0.020}$ mirror-smooth layer prepared for the dislocation density calculation

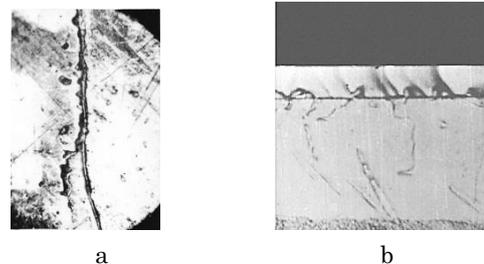


Fig. 3 – The substrate-layer interface at $0.1 < v < 0.3$ K/min without the initial supercooling of the solution-melt obtained by etching the slanting cut (140 \times) (a), planar interface of the $\text{Pb}_{0.749}\text{Sn}_{0.251}\text{Te}_{0.980}\text{Se}_{0.020}$ / $\text{Pb}_{0.7}\text{Sn}_{0.3}\text{Te}$ HS (in monochromatic light) obtained at the solution-melt cooling rate of 2 K/min (320 \times) (b)

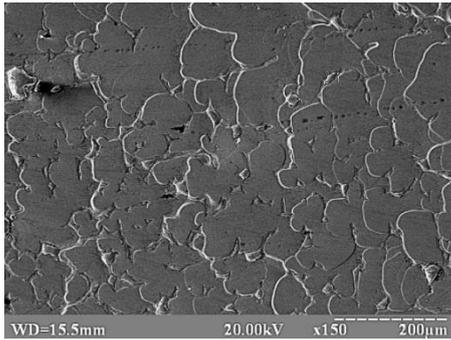


Fig. 4 – Terraced surface of the $\text{Pb}_{0.787}\text{Sn}_{0.213}\text{Te}_{0.960}\text{Se}_{0.040}$ EL obtained at the cooling rate of 0.6 K/min

If the rate of temperature decrease was changed in the range $0.5 < v < 1.0$ K/min, no transition layers were formed, the heterointerface was highly planar (Fig. 3b), but the surfaces of the layers were most often terraced (Fig. 4), and the surface density of dislocations increased to $(4-8) \cdot 10^5 \text{ cm}^{-2}$. To build up the next ELs or to make contacts to such layers, they need to be polished, which in many cases is unacceptable.

At rates of decreasing temperature $v > 1.0$ K/min, the surface of the EL had even more drawbacks: waviness, disoriented growth figures, dendrites, and solution residues.

The studies, conducted in this way, have stimulated further searches for technological methods aimed at improving the heterointerface structure. For this purpose, we used the method of preliminary supercooling of the solution-melt, known in the LPE technology [10].

Thus, some changes were made to the process: after homogenization of the solution-melt, it was cooled relative to the calculated liquidus temperature by ΔT , maintained for 10-20 min, and then poured onto the substrate. After holding at a fixed temperature, the program decrease of the temperature was conducted at a rate $v < 0.3$ K/min. At the initial supercooling of 1-3 K, the ELs were mirror-smooth or had a slightly terraced structure, which may be due to a slight misorientation of the substrates relative to the growth plane. In this case, the surface density of dislocations was $N_d \leq 2.3 \cdot 10^5 \text{ cm}^{-2}$. It is important that the

yield of such layers was 70-90 %, which is 10-15 % more than the results of previous experiments.

An increase in the supersaturation of the solution-melt by $\Delta T \geq 4$ K led to a fairly rapid crystallization of the intermediate layer of an imperfect crystal structure, and, accordingly, caused the formation of cracks in the EL, low-angle boundaries, and other structural defects. In this case, inclusions of metallic nature sometimes appeared in the grown layers (Fig. 5).

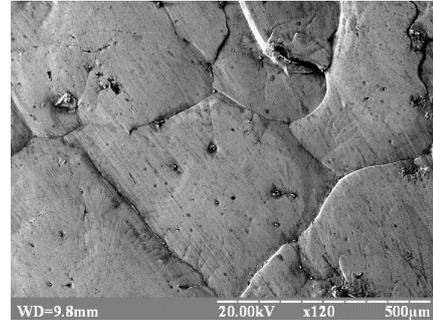


Fig. 5 – Imperfections of the $\text{Pb}_{0.749}\text{Sn}_{0.251}\text{Te}_{0.980}\text{Se}_{0.020}$ EL obtained during the initial supercooling of the solution-melt by 5 K

4. CONCLUSIONS

Theoretical estimations and experimental studies of the growth of thin ELs in the Pb-Sn-Te-Se system showed that $\text{Pb}_{1-x}\text{Sn}_x\text{Te}$ semiconductor substrates can be used to obtain active elements of IR optoelectronics using the LPE method with preliminary supercooling of the solution-melt and controlled temperature decrease rate. Such layers can be both active and buffer to obtain instrument HSs for the far infrared region of the spectrum.

Insufficient initial supercooling can affect the initial growth mechanism of the EL, which makes it impossible to obtain samples with a highly planar layer/substrate interface.

Of considerable interest is the continuation of the studies on the preparation of multilayer HSs in the Pb-Sn-Te-Se system with highly planar interfaces and high structural perfection.

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Рідиннофазна епітаксія тонких ізоперіодних гетероструктур твердих розчинів $Pb_{1-x}Sn_xTe_{1-y}Se_y$ О.В. Волчанський¹, Ю.Г. Ковальов², О.М. Царенко¹¹ *Центральноукраїнський державний педагогічний університет імені Володимира Винниченка, вул. Шевченка, 1, 25006 Кропивницький, Україна*² *Льотна академія Національного авіаційного університету, вул. Добровольського, 1, 25006 Кропивницький, Україна*

У роботі представлено результати дослідження початкових стадій росту методом рідинно-фазової епітаксії (РФЕ) ізоперіодних структурно досконалих гетероструктур (ГС) на основі твердих розчинів системи Pb-Sn-Te-Se. Теоретичний аналіз показав, що з ростом багатокомпонентних твердих розчинів (БТР) $Pb_{1-x}Sn_xTe_{1-y}Se_y$ на підкладках $PbTe_{0.88}Se_{0.12}$ та $Pb_{0.70}Sn_{0.30}Te$ можливо виділити три принципово різних випадки. Показано, що у випадку коли підкладка та епітаксійний шар (ЕШ) ізоперіодні при деякій середньоарифметичній температурі між температурою росту і робочою температурою приладу, то тоді використовуючи усі можливі склади, які ізоперіодні з підкладкою $Pb_{0.70}Sn_{0.30}Te$, можна отримувати ЕШ $Pb_{1-x}Sn_xTe_{1-y}Se_y$ з червоною межею фотоэффекту при робочих температурах в діапазоні довжин хвиль ≥ 4 мкм, що цілком може бути реалізовано шляхом РФЕ. Описано особливості технології РФЕ квазіізоперіодних ГС, що використовувалися в роботі, обладнання та методику проведення досліджень. При низьких швидкостях охолодження ($0.1 < \nu < 0.3$ К/хв), незалежно від діапазону зниження температури, який змінювали в межах $1.0 < \Delta T < 5.0$ К, отримували дзеркально-гладкі ЕШ товщиною 1-3 мкм з поверхневою густиною дислокацій меншою ніж $2 \cdot 10^5$ см⁻². Однак, дослідження межі розділу підкладка/шар свідчило про утворення, хоча і тонких ~ 0.2 - 0.4 мкм, перехідних шарів. Подальші експериментальні дослідження показали, що більш перспективною технологією РФЕ в цьому випадку є метод попереднього переохолодження розчину-розплаву. При програмному зниженні температури зі швидкістю $\nu < 0.3$ К/хв і початковим переохолодженням 1-3 К ЕШ були дзеркально-гладкими чи мали легку терасну структуру, яка може бути наслідком незначної дезорієнтації підкладок щодо площини росту.

Ключові слова: $Pb_{1-x}Sn_xTe_{1-y}Se_y$, РФЕ, Початкова стадія росту, Охолодження, Поверхня.