

Special regularities for lowering temperature during growth of high-quality CdTe semiconductor layers

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Abstract. To obtain epitaxial layers of A^2B^6 semiconductors with increased structural perfection from their own liquid phase, it has been proposed to use a technological process in which the synthesis temperature varies in such a manner that ensures a constant growth rate of layers during the whole process. The regularities of temperature variation with time for this process have been found on the basis of diffusion crystallization model. The developed model is realized by numerical methods and applied to description of the growth of cadmium telluride layers. Quantitative data on variations of synthesis temperature have been obtained, which can serve as a basis for choosing the temperature-time regimes of growth of cadmium telluride layers with a constant and required rate of solid phase formation.

Keywords: A^2B^6 solid solution, epitaxial film, CdTe compound, liquid phase epitaxy, kinetics of crystallization, supercooling technology.

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1. Introduction

Epitaxial layers of A^2B^6 semiconductors ($Cd_xHg_{1-x}Te$ and CdTe) are the main materials for photodetector matrices operating in the infra-red spectral range [1]. Monocrystalline layers of high optical and structural perfection and required thickness are grown from their own liquid phase by using epitaxial methods for a long period [2–5]. In these synthesis methods, crystallization of high-quality layers is ensured due to the proximity of the film growth conditions to the conditions of thermodynamic equilibrium in the whole growth system during the entire process time. Despite significant progress in obtaining the high-quality layers from its own liquid phase, it should be still admitted that this method of layers growth has not yet exhausted its potential for improvement to the end. The latter should include the possibility of developing more complex rules for changing the temperature of layer growth, which would provide, for example, constant kinetic conditions at the interface (constant crystallization rate). A constant and sufficiently low growth rate should ensure that the composition of the material and micro- and macrodefects in it will be distributed almost uniformly throughout the

layer thickness, even if the growth temperature is constantly decreasing to ensure the crystallization process.

The possibility of realizing a constant layer growth rate during the epitaxial process is confirmed by the experimental data adduced in Fig. 1, which presents (converted into other coordinate axes) the results of [5] for the dependence of thickness of $Cd_xHg_{1-x}Te$ grown layers on the crystallization time. For example, Fig. 1 shows nearly linear dependences of the thickness of grown layers on the time of growing even for a set of different cooling rates of the growth cell. These experimental data should be considered as a consequence of the approximately constant crystallization rate for this material. Indeed, the use of supercooling method [6], in which crystallization occurs from an initially supercooled liquid phase with its subsequent cooling at a constant rate, enables at an appropriate choice of a combination of the supercooling temperature and cooling rate to achieve an almost constant phase formation rate. This conclusion is a consequence of the dependence of the grown layer thickness on the growth time, theoretically substantiated in the model of diffusion-limited crystallization, when the discussed parameter corresponds to the dependence [5, 6]:

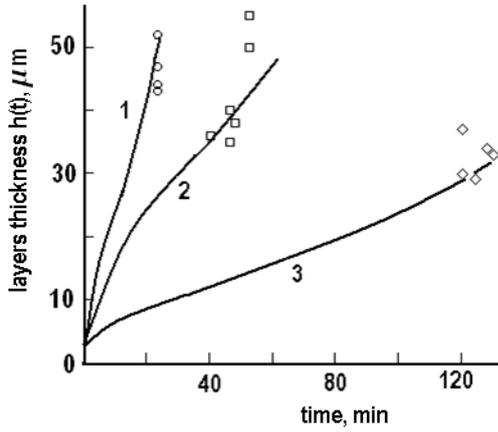


Fig. 1. Dependences of the thickness of $\text{Cd}_x\text{Hg}_{1-x}\text{Te}$ solid solution layers on growth time at various cooling rates: 1 – 0.25. 2 – 0.13. 3 – 0.05 K/min. The initial growth temperature is 825 K. The liquidus temperature is 827 K. The composition of solid solution to be grown is $x_{\text{CdTe}}^S = 0.23$ mol. frac. [4]. Solid lines are the result of numerical simulation of the growth process by the supercooling method in [5].

$$h(t) = K_1 \cdot \Delta T \cdot t^{1/2} + K_2 \cdot V_{\text{cool}} \cdot t^{3/2}, \quad (1)$$

where $h(t)$ is the time dependence of the thickness of grown layer, ΔT , V_{cool} are the initial supercooling of the melt and the rate of its cooling, respectively; K_1 and K_2 – constants depending on the parameters of phase equilibria in the system (partial slopes of the liquidus lines) and diffusion coefficients.

From the presented dependences, it follows that at certain supercooling of the system and a low rate of subsequent cooling of the growth cell, the total action of the two driving forces of the crystallization process will enable to obtain the dependences of grown layers thickness on time close to the linear ones. This fact is illustrated and confirmed by the data in Fig. 1, when the discussed dependences are close to straight lines, when using the supercooling method with $\Delta T = 2$ K during experiments [5].

It is extremely difficult to empirically find and substantiate the regularities of temperature variations in the growth cell, which would ensure a constant growth rate of the layer due to the need for a large number of high-precision technological processes under different conditions. At the same time, there are quite reliable theoretical models that simulate with high reliability the thermodynamic behavior of growth systems in the course of phase formation from the liquid one. These include, first of all, the model of diffusion-limited crystallization [6, 7]. It is the use of this thermodynamic approach that enables to predict with high reliability the parameters of layers grown from the liquid phase in a given temperature regime for a whole class of binary and multicomponent semiconductor materials [7].

2. Kinetic model of crystallization for the CdTe compound

The main assumption for constructing the diffusion-kinetic model is that the growth rate of a crystalline layer from a liquid phase is limited by diffusion flow of a substance directed to the interface. To calculate the concentration field in the bulk of the liquid phase under growth conditions, it is necessary to solve the following diffusion equation:

$$D^l \frac{\partial^2 x^l(z, t)}{\partial z^2} = \frac{\partial x^l(z, t)}{\partial t}, \quad (2)$$

where D^l is the intrinsic cadmium diffusion coefficient in liquid tellurium, $x^l(z, t)$ – concentration of the diffusing component (cadmium) in the liquid phase, z – coordinate directed along the normal to the interface into the bulk of the liquid phase.

From the known distribution of the component in the melt, it is possible to calculate the flow of matter to the interface and, therefore, the rate of layer growth. This velocity is calculated from the law of mass conservation of the components at the interface on the assumption that there is no accumulation and disappearance of matter at the interface. Mathematically, the relationship between the rate of the interface movement and the parameters of mass transfer is written in the form [6, 7]:

$$V(x_0^S - x_0^l) = D^l \frac{\partial x^l(0, t)}{\partial z}, \quad (3)$$

where x_0^S , x_0^l are the concentrations of the component at the interface from the sides of solid and liquid phases, $V(t)$ – growth rate, D^l is the cadmium partial diffusion coefficient in the tellurium melt at a given temperature.

The grown layer thickness $h(t)$ can be found by direct integration of the time dependence of the layer growth rate:

$$h = \int_0^t V(t) dt. \quad (4)$$

A fundamental assumption of the diffusion-limited growth model is the assumption of the presence of thermodynamic equilibrium at the solid-melt interface. It means that the concentrations of the components at the interface from the side of the solid x_0^S and liquid phases x_0^l are related to each other by the conditions of phase equilibrium at a given temperature.

It is shown in [8] that for theoretical description of the gas-melt-crystal phase equilibrium in A^2B^6 systems the model of associated or even polyassociated solutions, when the set of associates of different composition simultaneously exists in the melt, must be used. It means that the processes of matter transfer to the interface must be considered in relation to the transfer of each of the associates existing in the melt. However, the use of this model representations, the mathematical support of which is formed by a significant number of both

transcendental equations for finding boundary surface concentrations, and the need to solve a significant number of differential equations of mass transfer will overload the model unjustifiably. At the same time, the necessary relationship between the temperature and the composition of the equilibrium phases at the growth interface can be obtained using the well-known model of simple solutions [9]. The most powerful argument for using the simple solution model in this case is that the modelling of the crystallization process will be carried out in a rather narrow temperature range, when the assumptions about the power law for mixing energies are applicable [9]. In this work, the proposed thermodynamic model will be applied to predict the conditions for production of cadmium telluride, when the crystallization rate of the film is kept constant. According to the simple solution model, the equation for the liquidus line of the Cd-Te system has the form:

$$RT \ln \left[(1 - x_{Te}^l) x_{Te}^l \right] + 2\alpha_{Cd-Te}^l (x_{Te}^l - 0.5)^2 + \Delta S_{CdTe}^F (T_{CdTe}^F - T) = 0, \quad (5)$$

where x_{Te}^l is the concentration of tellurium in the liquid phase Cd-Te (at. fraction), α_{Cd-Te}^l – parameter of interaction in the liquid phase, ΔS_{CdTe}^F and T_{CdTe}^F are the entropy and melting point of cadmium telluride, R and T – universal gas constant and current temperature.

The boundary problem for searching the concentration fields during the growth of CdTe compound will be fully formulated using the following initial and boundary conditions. The boundary conditions adopted in this work are responsible both for the presence of thermodynamic equilibrium at the crystal-melt interface (6) and for the absence of evaporation of the components into the gas phase at the liquid-gas interface. The latter means the absence of a flux of solute at $z = L$ (7). As an initial condition (8), a uniform distribution of components in the initial liquid phase was taken, which corresponds to the crystallization of material from the melt homogeneous in composition x_{Cd}^{init} , the liquidus temperature of which is equal to T_l^{init} :

$$x_{Cd}^l(z=0, t) = x_{Cd}^0 \{T_l(t)\}, \quad (6)$$

$$\partial x_{Cd}^l(z=L, t) = 0, \quad (7)$$

$$x_{Cd}^l(z, t=0) = x_{Cd}^{init} = \text{const}(T_l^{init}), \quad (8)$$

where L is the thickness of the liquid phase, x_{Cd}^{init} – tellurium content in the initial liquid phase, $x_{Cd}^0 \{T_l(t)\}$ – cadmium equilibrium concentration depending on the current temperature (liquidus of the system at a given temperature). The presented equations of the diffusion-limited crystallization model are further applied to search for the temperature-time regimes of the growth of cadmium telluride high-quality layers.

3. Thermodynamic parameters of the Cd-Te system and mathematical support of the problem

To calculate the compositions of phases on the growth surface x_0^l that satisfy the conditions of liquid-crystal phase equilibrium, we used the thermodynamic representations of the model of simple solutions. According to this model (5), phase equilibrium in the Cd-Te system is determined by the excess energy of mixing the components in the liquid phase α_{Cd-Te}^l , as well as by the temperature and entropy of melting of cadmium telluride. If the values of the thermodynamic parameters of cadmium telluride are known with high reliability [6, 7]: $T^F = 1365.1$ K and $\Delta S^F = 36.81$ J/mol·K, then the parameter α_{Cd-Te}^l and its temperature dependence, as a rule, are found by processing experimental data on phase equilibrium in systems in accordance to the equations of the model of simple solutions. Due to the insignificant temperature interval in which the simulation is carried out, it was decided to restrict ourselves to a strictly regular approximation of the model of simple solutions. Our processing of the experimental data on the liquidus of the Cd-Te system within the indicated temperature range showed that these data are quite satisfactorily described by the value of $\alpha_{Cd-Te}^l = 7100$ J/mol.

The experience of calculating the A³B⁵ and A²B⁶ solid solutions crystallization kinetics of [5–7] showed that high reliability of the kinetic prediction of the growth process parameters can be achieved by setting the value of the partial diffusion coefficient of cadmium in liquid tellurium $D_{Cd} = 1.0 \cdot 10^{-5}$ cm²/s. This value is typical for materials of the classes under consideration, when these are synthesized within the temperature range 1000...1300 K.

The traditionally presented problem of a boundary value is used to describe the process of layer growth from a given composition of the liquid phase x_{Cd}^{init} at a selected growth temperature T_g . In this situation, the quantities T_g and x_{Cd}^{init} are assumed to be known, and the output data of the problem are the concentration profiles of the components $x_{Cd}^l(z, t)$, the rate of phase formation $V(t)$, and the thickness of the grown layer $h(t)$. According to this formulation of the problem, as a rule, the amount of computational work is not very significant and an explicit finite-difference scheme [8] is successfully used to solve the differential equation. In a situation where temperature-time growth conditions are necessary to find which provide a constant crystallization rate, the use of this software is not justified.

Indeed, a direct selection of the functions of temperature change in the simulated process will require a significant amount of computation, which is associated with a significant expenditure of computer time. Therefore, in this work, the differential equation of mass transfer was solved by an implicit scheme with a sweep [10]. Despite the use in the calculations of more complex software inherent to the implicit schemes, this more

complex algorithm was implemented and, thus, the search time for a solution of the boundary problem was significantly reduced [10].

The basis of the developed algorithm for solving the boundary problem was the block for solving the differential equation by an implicit scheme. In this case, the condition for the constancy of the growth rate in the simulated process was actually set by a constant value of the concentration gradient at the liquid-epitaxial layer interface. Then the value of the surface concentration itself served as the basis for calculating the current growth temperature in accordance to the formula (5). Obviously, this procedure was repeated for each time cycle, when integrating the differential equation. Conversion of the concentration data into the corresponding liquidus temperature of the system gave the final information about the desired function of temperature variations, which provided a constant and specified rate of layer growth.

4. Results and discussion

The main results of CdTe layers synthesis modelling are shown in Figs 2 to 4. So, Fig. 2 shows a set of curves that correspond to three different initial concentrations of cadmium in liquid tellurium with $x_{Cd}^{init} = 0.6, 0.7, 0.8$ at. frac. at the thickness of the liquid phase in the growth cell $L = 0.1$ cm. The calculations were performed for the range of growth rates from $V = 0.01 \mu\text{m/s}$ up to $V = 0.1 \mu\text{m/s}$. This interval includes the rates of phase formation most frequently used in practice. For all the calculated dependences, the total interval of cooling the liquid phase did not exceed 25 K, *i.e.*, did not exceed the value of supercooling of the melt, at which spontaneous crystallization in its bulk is possible [11]. Naturally,

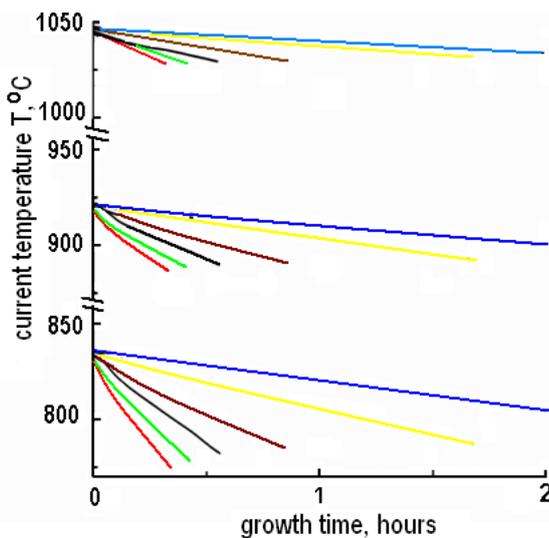


Fig. 2. Regularities of temperature decrease with time during the growth of CdTe layers for the liquid phase with different initial cadmium content: upper curves – $x_{Cd}^{init} = 0.6$; medium – 0.7, lower – 0.8 at. frac. The colors of the curves correspond to the following crystallization rates: red – $V = 0.1 \mu\text{m/s}$, green – 0.08, black – 0.06, brown – 0.04, yellow – 0.02, blue – 0.01.

the phenomenon of spontaneous crystallization of the melt substance is not taken into account in the model, and the modes in which it is possible should be excluded from consideration.

The use of a liquid phase with different initial concentrations of cadmium provides different liquidus temperatures of the initial melts. Therefore, the calculated temperatures of the synthesis begin to differ significantly from each other, which corresponds to different positions of the initial figurative points on the plane of the state diagram for the system. Obviously, with a decrease in the cadmium content in the tellurium-enriched melt, the liquidus temperature increases. It corresponds to the liquidus shape of the system in the tellurium-enriched part of its state diagram [8].

To ensure the necessary and constant growth rate of the layer during the entire synthesis time, the initial liquid phase, obviously, must be initially supercooled. In this case, a higher growth rate at the initial stage of synthesis can be provided only by increasing the supercooling of the initial liquid phase. Consequently, the calculated curves in Fig. 2 do not start at the same temperature, and the growth process begins at different temperatures. Further support of the required value of the growth rate is carried out by lowering the temperature in the growth cell in accordance with the data presented in Fig. 2.

We also point out that the values of the initial supercooling in the respective technology, which are calculated in the work, turn out to be insignificant and, as a rule, do not exceed 5 to 10 K. These values of supercooling can be realized in practice and correspond to the previously stated assumptions about the possibility of realizing the conditions for a constant crystallization rate during the entire synthesis process.

It is also necessary to point out that the calculated curves, in general, turn out to be close to linear ones. This result can be considered as the expected one, since for the majority of A^2B^6 systems at small intervals of cooling, no more than 30 K, the liquidus lines can be approximated by straight lines [6–8] with high reliability. Figs 3 and 4 shows the calculated regularities of temperature decrease for layers synthesis from melts with different initial cadmium contents and different thicknesses of the liquid phase in the growth cell. Data of Figs 3 and 4 show that variations of the composition of the initial liquid phase or, which is the same, the initial position of the figurative point on the Cd–Te phase diagram, as well as variations of the initial liquid phase thickness significantly affect on the shape of temperature functions which were founded. Therefore, the calculated dependences for various growth conditions differ significantly. Indeed, a decrease in the thickness (volume) of the initial melt also assumes a great rate of its depletion as the layer grows, and a change in the concentration of cadmium in the melt shifts the working point of crystallization along the phase diagram, which is reflected in the values of the slope of the liquidus line. It inevitably affects the growth rates of the layer [6, 7].

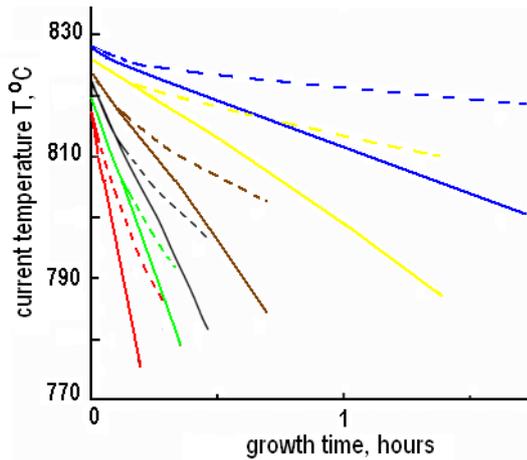


Fig. 3. Regularities of the temperature decrease with time during the growth of CdTe layers from the liquid phase with the cadmium concentration $x_{\text{Cd}}^{\text{init}} = 0.8$ at. frac. The colors of curves correspond to the following crystallization rates: red – $V = 0.1 \mu\text{m/s}$, green – 0.08, black – 0.06, brown – 0.04, yellow – 0.02, blue – 0.01. Solid lines correspond to the melt thickness $L = 0.1$ cm, dotted ones – 0.3 cm.

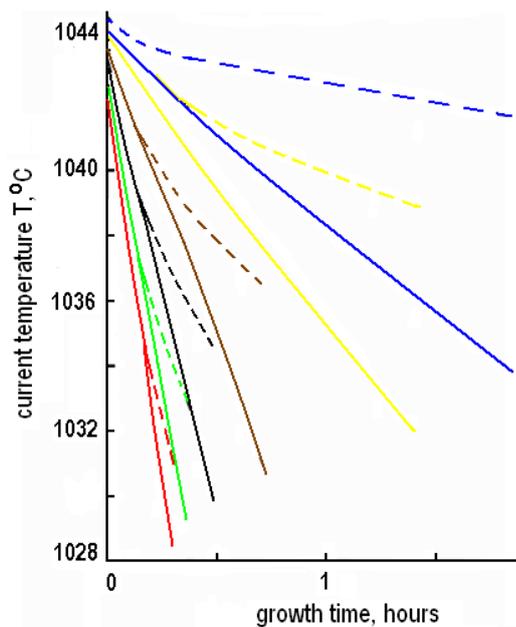


Fig. 4. Regularities of the temperature decrease on time during the growth of CdTe layers from the liquid phase with the cadmium concentration $x_{\text{Cd}}^{\text{init}} = 0.6$ at. frac. The colors of the curves correspond to the following crystallization rates: red – $V = 0.1 \mu\text{m/s}$, green – 0.08, black – 0.06, brown – 0.04, yellow – 0.02, blue – 0.01. Solid lines correspond to the melt thickness $L = 0.1$ cm, dotted ones – 0.3 cm.

Therefore, it is possible to provide a necessary growth rate from the liquid phase of a small thickness and at different temperature intervals of the phase diagram only by significantly varying both the initial supercooling of the system and the rate of its cooling during the synthesis.

Comparison of the data in Figs 3 and 4 allows us to assert that since the concentration of cadmium in the initial liquid phase decreases, which corresponds to an increase in the synthesis temperature of the compound, the influence of the considered process factors on the requirements of maintaining necessary regularities of temperature variation also change. So, if increasing the synthesis temperatures, it is possible to ensure the growth of sufficiently thick layers using short cooling intervals. This situation is a consequence of a decrease in the slope of the liquidus line on the phase diagram, because of increasing the equilibrium temperature [5, 6, 9]. With a small value of the slope inherent to the liquidus ($\partial x_{\text{Cd}}/\partial T$), it becomes possible, with the same supercooling of the growth system, to form a significant flux of matter to interface and, thereby, to provide a necessary growth rate. However, carrying out the synthesis under these conditions imposes its own limitations on the accuracy of precise maintaining the constancy of the growth rate. This is not always easy to implement it from the technical viewpoint, especially at high growth rates.

Less stringent requirements are imposed on the temperature control of the process, when a less concentrated melt is used, the liquidus temperature of which is lower. The quantitative data of Fig. 4, which shows the results of modelling of the growth from the liquid phase with $x_{\text{Cd}}^{\text{init}} = 0.6$ at. frac., confirm the fact known from the thermodynamics of crystallization processes that at low growth temperatures in classical epitaxial synthesis methods it is easier to control the growth process. To the above, it should be added that the use of low synthesis temperatures also provides a higher crystallographic quality of the structures.

The developed thermodynamic concepts and corresponding calculations allow the possibility of their generalization to the cases of epitaxial crystallizing the other solid solutions of A^2B^6 , A^3B^5 , A^4B^6 compounds [12].

5. Conclusions

1. To obtain epitaxial CdTe layers with increased structural perfection, it has been offered to apply the technological synthesis conditions, in which, due to the use of nonlinear laws of decreasing the temperature in the system, a constant growth rate of the layers can be ensured during the entire synthesis time.
2. Being based on the model of diffusion-limited crystallization, which has been applied to describe the growth process of cadmium telluride layers, numerical methods have been used to find the temporal regularities of lowering the temperature of layer synthesis, at which the layers growth rates remain constant and set.
3. Quantitative data on the regularities of temperature decrease in the synthesis process have been obtained, which are presented in graphical form. They can serve as a basis for choosing the temperature-time regimes for the growth of high-quality layers of cadmium telluride with a constant rate of phase formation.

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Особливі закономірності пониження температури росту високоякісних напівпровідникових плівок CdTe

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Анотація. Для отримання напівпровідникових епітаксialьних плівок A^2B^6 з підвищеною структурною досконалістю з власної рідкої фази запропоновано застосовувати технологічний процес, в якому температура синтезу змінюється таким чином, щоб забезпечити постійну швидкість росту плівок протягом всього часу процесу. Закономірності зміни температури з часом для такого роду процесів знайдено на основі моделі дифузійно-обмеженої кристалізації. Розроблену модель реалізовано чисельними методами і застосовано до опису процесу росту плівок телуриду кадмію. Отримано кількісні дані про закони зміни температури синтезу, які можуть служити основою для вибору температурно-часових режимів вирощування плівок телуриду кадмію з постійною швидкістю фазоутворення.

Ключові слова: епітаксialьний шар, твердий розчин A^2B^6 , сполука CdTe, рідинно-фазова епітаксія, кінетика кристалізації, технологія переохолодження.