

REVIEWS

STATUS AND PROBLEMS IN THE INVESTIGATION OF HEAT AND MASS TRANSFER PROCESSES IN GROWTH OF SINGLE CRYSTALS FROM MELT

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Single crystals are currently widely used in science and engineering. They are used in electrical energy conversion devices, in optics, quantum electronics, microelectronics, and other areas [1-5]. Crystal growth from a melt is a complex physicochemical process, in which heat and mass transport plays an extremely important role, often determining the quality of the crystals obtained. To obtain quality crystals, certain thermal conditions must be strictly observed. It is necessary to maintain a definite configuration of the temperature field with high stability of the process. Thus, for example, in many cases, to obtain constant gradients, the temperature must be maintained to within fractions of a degree for temperatures exceeding 2000°. If these conditions are not observed, then inadmissible mechanical stresses could appear in the crystals, giving rise to the formation of cracks, high dislocation density, and other imperfections in the structure.

Historically, the development of the technology for obtaining single crystals proceeded so that the methods for growing the crystals were created first and the process of obtaining crystals was mastered. The study of heat and mass transfer, as a rule, began when it became necessary to grow crystals with new properties or large dimensions, which could not be obtained by the previously developed technology.

Heat and mass transfer processes in the technology for obtaining single crystals from melt involve considerable difficulties. This is explained by the fact that in a rigorous formulation the problem must be viewed as a multidimensional problem in two contiguous regions (crystal and melt) with internal and external boundaries sought and a combined analysis of hydrodynamic, thermal, and diffusion processes. For the case of a crystal that is semi-transparent to heat rays, a complex heat transfer process will occur both in the crystal and in the melt.

Investigations of heat and mass transfer during crystal growth can be separated into two stages. The transport parameters (temperature, heat fluxes, impurity concentrations) and the general characteristics of the flow of the process in the growing setups are determined at the first stage. At the second stage, generalization of the data obtained permits introducing important adjustments to the technology and developing new methods and setups for obtaining single crystals. Here, in particular, the problem of the transition from temperatures to mechanical stresses in crystals and determination of the thermal conditions for obtaining single crystals with a defect-free structure is solved.

In connection with the complexity of the processes investigated, when describing them analytically, simplifying assumptions are usually introduced and the problems are viewed as coupled (combined analysis of heat and mass transfer) or uncoupled, multidimensional or single dimensional, nonstationary or stationary, in a nonlinear or linear formulation, in a coupled or uncoupled form, with the geometry being sought or given, etc. There are presently not enough complete generalizations indicating the cases in which the various simplifying assumptions used in analytical investigations of the process are applicable.

The number of theoretical and experimental papers concerned with the study of heat and mass transfer processes in the growth of crystals increases with each year. Experimental investigations permit determining the general characteristics of heat transport processes during crystal growth and creating mathematical models based on these investigations. The availability of models places new requirements on experiments, which confirms and refines the created model or corrects it [6-12].

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Let us illustrate this situation by examples. We shall examine some models for Czochralski's method. A two-dimensional stationary model of the heat-transfer process is presented in [13]. It is assumed that heat transfer in the crystal and in the melt occurs by means of heat conduction. The length of the crystal is fixed, the nonlinear boundary conditions on the surfaces of the crystal and melt are formulated, and the equation of heat balance is examined on the curvilinear crystallization front. In [14-17], the thermoelastic stresses in the crystals were calculated based on experimental data and calculation of the temperature distribution in the crystals. The analysis performed permitted determining the thermal conditions for obtaining perfect single crystals.

Heat- and mass-transfer processes are examined together in [18]. In [19, 12], a zonal method is used to investigate heat transfer in the setups. In all of the examples listed above, the diameter of the crystal is assumed to be fixed, while it is determined by the thermal conditions of growth, as shown, for example, in [20, 12]. This imposes definite restrictions on the boundary conditions in the models formulated. The mathematical model for Czochralski's method is improved by combining the analysis of hydrodynamics and heat- and mass-transfer processes in the melt-crystal system [21-23].

A one-dimensional nonstationary mathematical model of the process of obtaining single crystals by means of crucibleless zonal melting is presented in [24, 10]. A mathematical model of heat-transfer processes in obtaining single crystals using Stokbarger's method in an ampule is examined in [25, 26]. The problem is formulated as a nonstationary, one-dimensional problem with identical surfaces of the solid and liquid phase and a spatially stationary crystallization front, which is ensured by variation of the velocity of the ampule. The ampule velocity obtained from the calculation varies from 2 to 4 mm per hour, which is an order of magnitude lower than for the case in [13] with thermophysical parameters of the same order of magnitude. Evidently, this problem could have been examined as a quasistationary problem.

The demand for high-temperature single crystals made of semitransparent materials increased in recent years.

In semitransparent crystals, heat transport occurs via heat conduction and radiation, i.e., radiative-conductive heat transfer (RCHT) occurs. As is well known, the RCHT problem reduces to the solution of integrodifferential equations. Exact account of the radiation component of the heat transfer in crystal growth compared with nontransparent crystals with other conditions remaining the same greatly increases the complexity of the solution of transfer problems.

Analytic solutions are presently available only for one-dimensional RCHT problems with the simplest boundary conditions [27-29]. Programs for making numerical calculations have been developed practically also for one-dimensional problems [30].

In analyzing heat transport in shaped and cylindrical semitransparent crystals, obtained by Stepanov's method, in many papers the problem is viewed as a one-dimensional and stationary problem and Rosselund's approximation is used [31, 32]. In this case, the starting equation is written in the form

$$\frac{d}{dz} \left(\chi_m \frac{dT}{dz} \right) - \rho v c \frac{dT}{dz} - \frac{2}{r} q_h = 0. \quad (1)$$

It is assumed that $\chi_m \gg \chi_{mol}$. Equation (1) is solved with boundary conditions of the first kind, using the averaged value of the absorption coefficient and neglecting heat transport by moving crystal. In addition, when heat transfer from the lateral surface is neglected, the temperature field is found to be independent of the optical characteristics of the crystal. In [33, 34] the problem of heat transport in an optical crystal with large L/D is formulated in the light-guide approximation, i.e., radiation transport along the rod is examined taking into account the characteristic radiation and the external radiation incident on the surface of the crystal, as well as the presence of an angle of total internal reflection of the material.

This approach is a step forward compared to the Rosselund's approximation, but it is necessary to make more precise the problem of averaging the coefficient of absorption and taking into account the external radiation in formulating the problem, since, for example, for sapphire the coefficient of absorption is strongly temperature dependent [35]. In obtaining large single crystals of sapphire and yttrium-aluminum garnet (YAG) using the methods

of vertical (VDC) and horizontal directional crystallization (HDC), it is necessary to investigate in greater detail heat transport processes in the crystal, the starting material (grain), and the melt. For the HDC method, the problem of heat transport for the crystal, starting material and melt must be examined as coupled, multidimensional problems with the two boundaries of the phase transition being sought. The solutions of one-dimensional RCHT problems are presented in [30, 36, 37]. An attempt is made in [38, 39] to use the results of the solution of one-dimensional problem to analyze heat transport in a sapphire plate, obtained using the method of horizontal directional crystallization. Heat transport in a semi-transparent crystal, obtained by the method of vertical directional crystallization, is examined in [40, 41].

There are very few papers on transport processes in the melt in obtaining single crystals using the VDC and HDC methods, while for YAG there are no reliable data on the thermophysical properties of the melt. Free convection in the melt in obtaining a YAG single crystal using the VDC method is investigated in [42, 43]. In [42], it is shown based on the solution of the general equations that the reason for the mixing of the melt could be the deviation of the container axis from the vertical (beginning with 30-40') and unsymmetrical heating around the circle.

The main experimental problem, as a rule, reduces to finding the temperature distributions in the crystals and in the structural elements of the setups, as well as determining the configuration and position of the crystallization front during the crystal growth process.

Thermocouples are widely used to measure the temperature in the crystals, melts, and the elements of the setups used for growing single crystals. The complexity of measuring the temperature with the help of thermocouples lies in the fact that the growth process proceeds in small setups operating under a deep vacuum or excess pressure, while the crystal (and sometimes the melt) moves in space. This complicates the placement of the thermocouples and the output leads of the thermoelectrodes. The crystal and especially the melt, in many cases, due to its corrosiveness, have a destructive effect on the thermoelectrodes and the ceramic insulating stem of the thermocouples. The latter, at high temperatures, becomes electrically conducting, and it evaporates in a vacuum. As a result of the evaporation of structural materials (for example, molybdenum) and subsequent deposition of these materials on the structural elements and thermocouples, the thermoelectrodes are short-circuited. With prolonged action of high temperature, the characteristics of the thermocouples can change. All this requires taking special measures when using thermocouples in growing setups.

Introduction of thermocouples into the melt and their subsequent growth into the crystal with the crystallization of the melt are widely used [44-50, 12, 16, 34]. Before growing into the crystal, the thermocouple can measure the temperature of the melt [12, 16]. In order to fix the position of the thermocouple junction in the melt, it can be fixed to a special wire framework [46]. It is possible to grow the thermocouples into thin profiled sapphire crystals, obtained by Stepanov's method [34]. The thermoelectrodes of the thermocouples can be protected from corrosive media by depositing a lubricating layer [45] or by placing the junctions into a protective quartz sheath [12, 44]. Depending on the range of temperatures measured, Chromel-Alumel [12, 45], platinum-rhodium-platinum [45], and tungsten-rhenium [16, 46] thermocouples are used. Attempts have been made to grow ceramic tubes into the crystal and to move the thermocouples along them. The temperature of the external surface of the crystal can be measured by a mobile thermocouple [47]. Data obtained by measuring the temperature in setups for obtaining optical quality single crystals using the method of vertical directional crystallization are presented in [48]. In [50], a molybdenum-tungsten thermocouple, one of whose elements was a thin-walled molybdenum tube with an external diameter of 6 mm and closed at the end, was used to measure the temperature of a YAG melt. The second thermoelectrode consisted of a tungsten rod situated inside a tube and welded to its end.

In measuring the temperature in semitransparent crystals, the indications of thermocouples differ from the actual temperature of the medium due to a definite contribution of the radiation component of heat transfer. In this connection, negative arguments are presented in a number of papers concerning the use of thermocouples for the purposes indicated [51, 52]. In our opinion, thermocouples should be more widely used in measuring temperature fields in crystals, including also in semitransparent crystals, since in practice there is no other method for measuring the temperature in the volume of a medium. In this case, it is necessary to improve methods for estimating the errors both by experimental and computational methods [53]. At the same time, it is necessary to develop new methods for measuring and estimating the

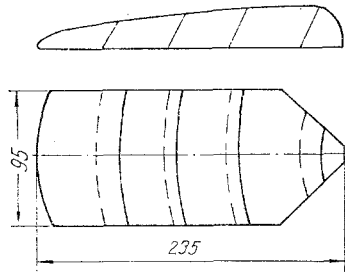


Fig. 1

Fig. 1. Distribution of lines of impurity capture on a grown crystal.

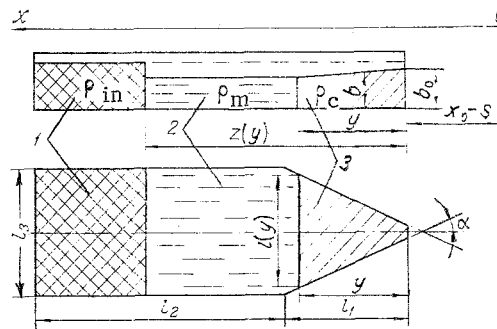


Fig. 2

Fig. 2. Diagram for formulation of the problem of the change in the height of the crystal for a one-dimensional model: 1) grain; 2) melt; 3) crystal.

temperature in semitransparent materials and their melts, using, for example, the dependence of the electrical resistance of the material on temperature as well as the dependence of the shape of the crystal on the average temperature of the melt.

IR partial radiation pyrometers, operating in the region of the fundamental absorption band of the object studied, have been developed to measure the surface temperature of semitransparent materials [51, 52]. For example, for leucosapphire, this wavelength interval lies in the range from 6.4 to 10.2 μm (the absorption coefficient is 10^{+5} - 10^{+6} cm^{-1} , while the reflection coefficient of the surface is of the order of 0.025-0.04). The use of IR spectrometers [54] requires optically transparent windows without the formation of any deposits on their surfaces.

To determine the configuration and position of the crystallization front during the growth of optical quality crystals, it is useful to use crystallophysical experiments, consisting of the formation of visible surfaces due to capture of mechanical particles and impurities with a jumplike displacement of the crystallization front (HDC method) [46]. The position of the boundary of the melt of the starting material in the boat can be determined from the rapid drop in intensity and termination of the process.

A considerable number of papers on the parameters of crystalline materials, their melts, and structural materials have recently appeared [55-59, 29]. However, the necessary data are not yet available for all materials and especially high-temperature melts. An estimate of the average value of the density of YAG near the melting point (3770 kg/m^3), the magnitude of the volume coefficient of thermal expansion, and change in the volume with solidification is presented in [43].

The basic problems that must be solved when investigating heat and mass transfer processes in growth of crystals, in our opinion, reduce to the following.

1. Conditions must be created for heat transfer into the growing crystal from the surrounding surfaces so that the temperature distribution in the crystal does not lead to the appearance of inadmissible mechanical stresses. It should be kept in mind that thermoelastic stresses in some cases relax via plastic deformation, while in others stresses relax primarily via the formation of cracks. This is confirmed by the fact that under otherwise identical conditions the density of dislocations in unfractured crystals is three to four times higher than in fractured crystals [60]. When inadmissible stresses are present in crystals, which could be determined either by calculation or experiment, the form, dimensions, and temperature regime of the heater, screening system, etc. must be changed so as to change the temperature field in the crystal (decrease the second derivatives of the temperature with respect to spatial coordinates). The expected effect of the structural changes must be first estimated from calculations using special programs constructed on the basis of simplified mathematical models of the heat transfer processes. It is presently impossible to obtain a unique solution of the problem examined.

2. The overall temperature interval and input of heat to the container prior to onset of crystallization must provide for melting of the seed crystal by a definite amount, which

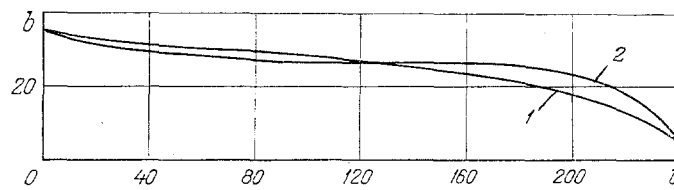


Fig. 3. Change in the height of the crystal b (mm) along the length l (mm), calculated using Eq. (2):
 1) $l_m = 150$ mm; 2) 50 mm.

is necessary in order to obtain a fixed crystallographic orientation. It should be kept in mind that the extreme melting of the seed crystal increases the duration of the growth process and, just as its inadequate suppression, can yield a crystal with a developed block structure.

3. The conditions for heat exchange near the crystallization zone must ensure a form for the crystallization front that does not allow the formation of crystalline defects, capture of impurities by the crystallization front, gas bubbles, etc. Thus, for example, in extruding single crystals from the melt, a plane crystallization front is strived after. In obtaining optical single crystals using the HDC method, the crystallization front must be inclined in the direction of motion of the boat, which is achieved by the more intense input of heat to the boat from above than from below.

4. Conditions must be provided that permit growing crystals with a given shape and dimensions, for example, the diameter of the crystals extruded from the melt, variation in the height of the single crystalline plate obtained by the HDC method along the length, etc. For this purpose, in the process of growing a crystal, it is necessary to maintain a definite temperature of overheating of the melt, dimensions of its zone, rate of displacement of the container, etc.

5. The given distribution of impurities over the length of the crystal must be secured by the rate of displacement of the container or seed, dimensions of the melt zone, and conditions for mixing it.

6. Mass and heat transfer processes in the setup must be examined together with shape-forming processes, since the latter greatly depend on the thermal regimes of operation of the setup, while the temperature distribution in its turn depends on the shape of the crystal.

7. Reliable means must be developed for experimental investigation of the thermal processes in growing setups.

Solution of the problems listed above will permit widespread use of **automatization** of growing setups.

To improve the technology for obtaining high-temperature optical single crystals, some specific problems arising from the conditions under which such crystals are grown must be solved. These problems include, for example,

a) investigation of optical and radiative characteristics of structural materials (molybdenum, tungsten, etc.) in the high-temperature range under real conditions of their operation in setups. Experience shows that in obtaining sapphire using the HDC method, after several crystallizations, deposits form on the surfaces of screens and the heater, and the power necessary to obtain the crystal decreases;

b) investigation of the transfer of heat in an optical crystal taking into account the radiation from the surfaces of the container and the crystallization front inside the crystal. Approximate calculations that have been performed show that in obtaining single crystalline sapphire plates using the HDC method, radiation transfer along the axis of the plate is more than an order of magnitude greater than the conductive component of the heat transfer;

c) an approximate estimate of the effective coefficients of transfer in order to make calculations using the conductive models. Thus, for example, the effective heat conduction coefficient for the YAG melt in obtaining crystals $\varnothing 14\text{--}30$ mm using the VDC method constitutes 130–140 W/m·deg [69].

As is evident from what was said above, it is necessary to expand the experimental and theoretical work on investigation of the characteristics of heat and mass transfer in obtaining

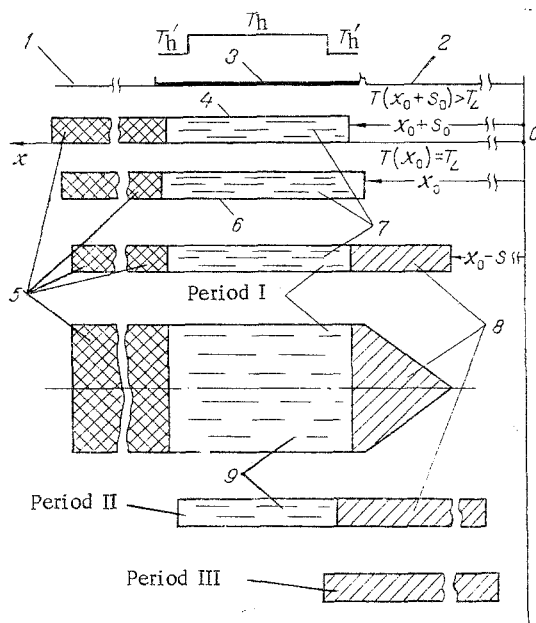


Fig. 4



Fig. 5

Fig. 4. Diagram for periodization of the process of obtaining a single crystal using the HDC method: 1) rear block of screens; 2) front block; 3) heater; 4) preliminary period; 5) grain; 6) initial state; 7) melt; 8) crystal; 9) melt.

Fig. 5. Boat containing crystal and starting material after drop in power.

single crystals. These investigations must be based on the physical and mathematical models of heat and mass transfer processes in setups for growing crystals. The general assumptions in physical and mathematical models and their roles in scientific investigations are presented in [61-66, 6-13, 49]. Physical models of industrial processes are examined in [7]. Thus their sphere of application for industrial purposes is increasing.

A characteristic of the cycle for obtaining single crystals from a melt is that in the growth process the number of regions with different aggregate states of the material, their position in space and heat exchange with the surrounding surfaces and medium, conditions for shape formation, the form of the crystal, etc. change. All this makes it unwise and in many cases impossible to create a general physical model for the entire cycle of obtaining a single crystal. For this reason, in creating physical models of heat- and mass-transfer processes in setups for obtaining single crystals from a melt, it is useful to separate the entire cycle for obtaining single crystals into separate periods and states between them and to examine an individual physical model for each period. Thus, for example, in the HDC cycle, it is useful to examine four periods and four states of the system: the preinitial state from the time the container begins to move to the beginning of crystallization of the melt; the first period, when the starting material, melt, and crystal are present; the second period, after melting of the entire starting material, when only the melt and crystal are present; the third period, during which the crystal cools after complete crystallization of the melt; the state in which the boat begins to move, when the entire melt is overheated; the initial state at the time crystallization of the melt in the boat begins, when the melt and starting material are present; the states of melting of the entire starting material and termination of crystallization of the entire melt.

The availability of a mathematical model permits developing computer programs for solving specific transfer problems for growing setups. When examining the periods of the cycle, it is necessary to solve the direct problem and to find the temperature distribution and the position of the phase transition boundary in the boat as a function of its position. In order to develop the technology for growing single crystals, it is useful to examine the problems of determining the fixed states of the system, which will differ from the states examined above by the fact that traditional requirements are given with respect to the temperature distribution in the boat and conditions for realizing them are determined. Thus, for example, for the

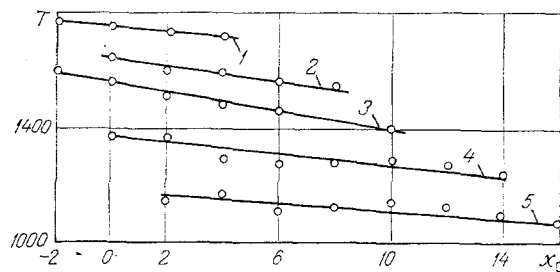


Fig. 6. Dependence of the temperature $T(K)$ at a fixed point in space on the position of the boat: 1) $x = 10$; 2) 14; 3) 16; 4) 18; 5) 20.

HDC method, the heater temperature and the position of the boat in space are determined from the solution of the problem for a conditionally fixed initial state of the system with a given length of the melt zone. This permits choosing the operational regime for the existing setup and planning the path for perfecting its design. The solution of the problem of the state of the system at the time of melting of the entire starting material permits solving the problem of the shape of the grown crystal. Models of transfer processes in obtaining single crystals from a melt, as a rule, can be viewed as quasistationary.

We shall illustrate some of what has been said above for examples of obtaining sapphire and YAG single crystals using the VDC and HDC methods. One-dimensional conduction models for the crystal growth process and the initial state of the system for VDC and HDC methods are examined in [67-69]. The results of calculations of the temperature in the boat containing the crystal and melt for a two-dimensional conductive model are presented in [54].

Figure 1 shows a diagram of the grown crystal and the position of lines of impurity capture on it, obtained as a result of fast motion of the boat during the crystal growth process (crystallophysical experiment). As can be seen from the figure, the height of the crystal changes along its length, which is caused by the different densities of the crystal, melt, and starting material. Figure 2 shows a diagram for formulating the problem of changing the height of the crystal when the crystal is obtained by the HDC method for a one-dimensional model. The approximate change in the height of the melt during the crystal growth process (as well as the height of the grown crystal along its length) for a one-dimensional variant can be obtained by solving the equation

$$\frac{db}{dy} + b \frac{l_3 \frac{dz}{dy} + l(y) \varepsilon_1}{[z(y) - y] l_3 - (l_1 - y)^2 \operatorname{tg} \alpha} - \frac{l_3 l_0 \frac{dz}{dy}}{[z(y) - y] l_3 - (l_1 - y)^2 \operatorname{tg} \alpha} = 0. \quad (2)$$

It is evident from the equation that the height of the crystal over its length b depends on the change, during the crystal growth process, in the position of the crystallization front y and the melting boundary of the starting material $z(y)$. Since the position of the boundaries is determined from a heat problem, the problems of heat transfer and formation of the crystal height along the length must be examined together. The change in the height of the crystal along the length can be regulated by changing the adjustable loading of the boat with the starting material and by introducing a mass source. Figure 3 shows the computed, based on the solution of Eq. (2), change in the height of the grown crystal along its length for different values of the length of the melt zone and values unchanged in magnitude up to the time of melting of the entire starting material. Figure 4 presents a diagram illustrating the periodization of the process for the HDC method. Figure 5 presents a photograph of the boat containing the crystal and the starting material after rapid drop in power and stopping of the boat, permitting determining the position of the melting boundary of the starting material. Figure 6 shows the dependences of the temperature at fixed points in space on the position of the boat, obtained with motion of the boat containing the starting material at temperatures up to the phase transition. From the figure we must draw the important conclusion that a separate temperature distribution must be examined for each position of the boat.

In conclusion, we note that it is difficult to find another area in technology where such a variety of complex heat and mass transfer problems in a single small aggregate could be found as is observed in growing single crystals out of melt. The solution of practical problems necessary for perfecting the technology for growing crystals requires further development of the theory of heat and mass transfer. This primarily involves developing methods for

solving problems of radiative-convective transfer, hydrodynamics, free and forced convection in high-temperature melts, and multidimensional problems with the phase transition boundaries being sought.

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