Influence of the temperature field in the energy-saving carbon-graphite thermal unit on configuration of the solidification front under different conditions of growing fluoride single crystals by the HDS method

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Abstract. Mathematical modeling of the influence of the temperature field created by the ECU on the position and on configuration of the solidification front under different conditions of growing single crystals of fluorides by the HDS method has been carried out. Data were obtained for different growing conditions for single crystals of fluorides and their effect on configuration of the solidification front in various modifications of the ECU of the first and second types. From a comparison of mathematical calculations, a conclusion was made about the most suitable conditions for the synthesis of these single crystals.

1. Introduction

The synthesis of single crystals from a melt is a complex physicochemical process in which the most important, often decisive, role is played by heat and mass transfer. Strictly specified temperature conditions must be observed to obtain structurally perfect single crystals of high optical quality. It is also necessary to maintain constant temperature gradients at the growth front of the single crystal and high stability of the process. The accuracy of maintaining the temperature for the synthesis of most single crystals of fluorides with the fluorite structure at a level of 1500°C reaches several fractions of a degree [1-3]. Disturbance of these growth conditions can lead to the appearance of various types of defects and high residual stresses in single crystals.

One of the most important tasks in the synthesis of single crystals is to control the shape of the solidification front, which is set by the thermal and kinetic growth conditions [4]. Thus, the mathematical calculation of the temperature field of the energy-saving carbon-graphite thermal unit (ECU) and its influence on the position and configuration of the solidification front is a non-trivial problem. This task requires strict knowledge of the boundary conditions of crystallization and the thermophysical properties of the system, including its aggregate states. The problems of the dependences of the elastic modulus of steels and alloys depending on high temperatures were discussed in detail in the articles [5-10].

The goal of this work is to study the processes of heat and mass transfer in various modifications of the ECU and their influence on configuration of the solidification front [11]. The possibility of varying the value of the temperature gradient in the zone of the crystallization front is shown by letting gas flow directly into this zone. The effect of the admitted gas flow on the shape of the solidification front and on the axial temperature gradient of the ECU has been studied. The most suitable flow rates of the admitted gas have been determined to achieve optimal conditions for the synthesis of a single crystal. The effect of the created temperature field in various modifications of carbon graphite heating units under different growing conditions on configuration of the solidification front of the synthesized single...
crystal and the temperature distribution in it is investigated. This work is a further development [12] on growing single crystals with a fluorite structure by the HDS method in a carbon-graphite thermal unit.

2. Experimental part

In addition to the basic modification of the carbon-graphite growth ECU described and calculated in [12], two additional modifications of the structures of growth units for growing fluoride crystals were developed.

Figure 1 shows a modification of the ECU of the first type. In this design, there is no system of replaceable heat shields of the external heat-shielding circuit on all three modules, instead of which thermal insulation made of graphite felt, such as a “sandwich panel”, is used. Thermal insulation is laid in several layers tightly adjacent to each other. Thus, the finished structure is a total thermal insulation volume like a muffle. The heating block of the central module also has additional thermal protection in the form of “sandwich panels”, made up according to the same principle into a common heat-insulating muffle. This modification of the ECU provides low-gradient zone (soft gradient zones) in the crystal growth zone and in the crystal cooling zone due to more efficient energy saving. This modification of the ECU allows to reduce the thermal inertia of the entire system, to reduce the thermal load on the inner walls of the crystallization apparatus (at the boundary of the furnace atmosphere and the channel heat exchanger). The power values on the heaters also decrease at the same melt temperatures in the crystal growth zone. The applied thermal insulation made of graphite felt, of the “sandwich panel” type, is a low-density carbon-carbon composite material based on carbon felt and carbon fiber, molded individually or in combinations in the form of layered structures compacted with pyrolytic carbon (SGL Carbon SIGRAFLEX, SIGRASIC, SIGRATHERM etc.).

A modification of the ECU of the second type is described [11] (Figure 2). In this modification, changes are present both in the design of the growth unit and in the crystallization apparatus. An additional unit for gas puffing is introduced into the design of the central chamber of the crystallization apparatus. This unit consists of a vacuum needle leak mounted on the outside of the central chamber of the crystallization apparatus, transition sleeves and nozzles made of graphite. In the design of the apparatus in the central module, the upper part of the inner contour of the right block has a central insulated gas supply channel with outlets into the holes of which nozzles are inserted. This adit has a single sealed flow channel with a vacuum needle leak. The tight connection of graphite couplings, branch pipes with each other, with the steel parts of the needle leak, as well as with the adit, is carried out with a special binding material at the joint points as a basis for graphite and graphite hardens during the start of operation of the furnace (that is, with an increase in temperature) [11]. During furnace operation (during crystal synthesis), an inert gas, Ar, N₂ or He, is continuously supplied through the gas supply channel directly to the crystal growth zone, namely, to the receiving area located immediately after the diaphragm. This contributes to a sharp change in the axial temperature gradient, while the temperature gradient is controlled directly by changing the flow rate of the supplied gas and the temperature on the heaters. Thus, this modification makes it possible to vary the value of the temperature gradient in the zone of the solidification front during the crystal synthesis process.

For each study, a semitransparent CaF₂ single crystal with dimensions of 300 mm x 198 mm x 40 mm, placed in a graphite crucible, which, in turn, was mounted on a tungsten drag, was considered in the calculation model. The thermal process was considered as quasi-stationary due to the low crystallization rates from 1 to 5 mm/h, [13, 14]. Thus, the crucible with the synthesized crystal was installed motionlessly in the central module through the gradient field from the melting zone (through the heaters) to the cold annealing zone, thereby undergoing asymmetric heating.

It is noteworthy that a distinctive feature of the HDS method from other melt ones is that the height of the melt in it is much less than its surface area, so the investigated crystal melt made up 2/3 of the crucible boat. So in this method, with a small height of the melt, large length and large surface area of the melt open in relation to the heater, temperature conditions of soft natural convection [2] of a laminar nature arise in the bottom part and along the surface of the melt without vortex flows. This fact excludes the formation of intense hydrodynamic and convective flows, which affect the degree of stability of the crystallization process and the morphological stability of the crystallization front. Thus, the velocity of the flow inside the melt can be neglected to simplify the problem being solved. And as a consequence, the investigated single crystal is considered in one state of aggregation - solid.
Figure 1. Layout diagram of the modified construction of the ECU of the first type placed in the crystallization apparatus "Rubitek - 1", where 1 - thermal insulation made of graphite felt, such as a "sandwich panel" of the loading module; 2.4 - thermal insulation made of graphite felt, such as a "sandwich panel" of the central module corridor blocks; 3 - additional external thermal insulation of the central module; 5 - thermal insulation made of graphite felt, of the "sandwich-panel" type of the receiving module;

Figure 2. The layout diagram of the modified construction of the ECU of the second type, placed in the crystallization apparatus "Rubitek - 1", where 1 is the crystallization apparatus; 2 - heat-insulating diaphragms; 3 - synthesized crystal; 4 - central module; 5 - upper slab of the inner contour of the right block of the corridor; 6 - vacuum needle leakage valve; 7 - transitional graphite couplings and branch pipes; 8 - gas supply channel (adit); 9 - taps with holes for supplying gas to the crystal growth zone; [5]
For these modifications of the growth nodes, various crystallization conditions were set. Thus, as the atmosphere inside the crystallization apparatus with the growth unit placed in it, a forevacuum of \(10^{-3}\) Pa and excess atmospheres of Ar, N\(_2\) or He gases were adopted, which were equal to 202.5 kPa. For the ECU of the second type, an additional series of studies was carried out with different rates of gas flow inlet into the chamber. Thus, these studies were also carried out in an excess atmosphere of an inert gas of argon, which was equal to 202.5 kPa, while it was supposed to relieve excess pressure through the overpressure relief valve. The calculation conducted out with a continuous supply of gas through the channel directly into the crystal growth zone for the main velocities of 0.1, 0.2, 0.5, 1, and 2 m/s. The main options for the calculation, including the object of study and its characteristics, block-structured grids, the temperature of the coolant at the inlet and its speed, etc. were similar to [11], both for the first and for the second modification of the ECU, with the exception of the temperature on the heaters in the modification ECU of the first type. So, in the course of research, due to the higher thermal performance of the thermal insulation of the graphite-graphite growth unit of the first type, the temperature of 1500 °C was set on the lower heater, and the temperature on the upper 1600 °C, respectively (the temperature on the heaters was selected and set lower than in the basic calculation based on the crystallization conditions test sample to avoid overheating).

The mechanism of heat transfer in a single crystal was carried out using the mechanisms of molecular heat conduction and radiation (radiation-conductive heat transfer). The single crystal itself was in a state of convective heat exchange with the medium in the thermal unit (only when using the technology of growing in an overpressure of an inert gas) and radiant heat exchange with the surrounding surfaces. In this study, it was necessary to determine the temperature distribution in the single crystal and the shape of the crystallization front.

3. Numerical procedure

The calculated mathematical model of this single crystal includes: the energy equation, the equations of heat and radiation transfer, the boundary conditions of heat conduction and radiation at the crystal boundaries. Also, this model is part of a complete heat and mass transfer model for a generalized cycle.

The energy transfer equation for the direction normal to the surface into the volume of the medium for this model was written as follows [13]:

\[
\frac{dE}{dt} = div q_E + e_\omega \epsilon = \epsilon_C + \epsilon_R, \quad q_E = q_C + q_R
\]

(1)

where \(q_E, q_C, q_R\) – are the vectors of total energy transfer, thermal conductivity and radiation, \(\epsilon, \epsilon_C, \epsilon_R\) – are the volumetric energy densities of the total, internal and radiation, \(e_\omega\) – is the volumetric power of energy sources.

The equation of energy transfer by thermal conductivity [13]:

\[
q_C = -\lambda grad t
\]

(2)

Radiation [7]:

\[
\frac{dI_S}{ds} = -kI_S + \eta
\]

(3)

where \(I_S\) – radiation intensity in the direction S; \(\eta\) – volumetric self-radiation coefficient of the medium. In this case, the values of \(\epsilon_R\) are omitted due to small values.

The components of the transfer vectors in the direction n have the form [13]:

\[
q_{Cn} = -\lambda \frac{\partial t}{\partial n}
\]

(4)

\[
q_{Rn} = \int_{4\pi} I_S \cos(n, s) d\omega
\]

(5)

In equations (1), (3), (5) the values of \(\epsilon_R, q_R, I_S, k, \eta\) refer to spectral and integral radiation or to the transparency zone of a single crystal.

The boundary conditions were set different, so on a fixed surface of the crystal for an elementary area \(dS_C\) centered at point M and were written with the direction of the normal n inside the crystal (Figure 3-a) [13]:

\[
\frac{dE}{ds} = \int_{4\pi} I_S \cos(n, s) d\omega
\]

(6)
\[
\frac{dt_{K}}{dn} = \frac{1}{\lambda_{K}} \left( \alpha \left( t_{K} - t_{f} \right) + E_{C} - aE_{i} - q_{R} \right)
\]

(6)

where \( \alpha \) - is the heat transfer coefficient; \( t_{K} \) – is the crystal temperature; \( t_{f} \) – is the temperature of the medium in the ECU; \( \lambda_{K} \) – coefficient of thermal conductivity of the crystal material; \( E_{C} \) and \( E_{i} \) – are the densities of the intrinsic and incident fluxes of the integral radiation; \( q_{R} \) – s the density of the resulting radiation flux in the range of partial transparency of the crystal \( \delta \lambda_{t} \) on the inner shell of the crystal; \( a \) – the degree of emissivity of the container wall radiation.

On the free surface in the opacity zone of the crystal for an elementary area \( dS_{K}^{F} \) centered at point N (Figure 3-b) [13]:

\[
\frac{dt_{K}}{dn} = \frac{1}{\lambda_{K}} \left( \alpha \left( t_{K} - t_{f} \right) + E_{C}'' - a''E_{i}'' \right)
\]

(7)

where \( E_{C}'' \) and \( E_{i}'' \) – are the intrinsic and incident surface radiation flux densities in the opacity zone of the crystal.

In the zone of partial transparency of the crystal (Figure 3-b) on the inner boundary of the area \( dS_{K}^{F} \)[7]:

\[
I_{1S(\varphi_{1})} = \bar{T}_{1S(\varphi_{1})}R + I_{2S(\varphi_{2})}(1 - R) \frac{n_{2}^{2}}{n_{1}^{2}}, \quad \text{при} \quad \varphi_{1} < \varphi_{BR}
\]

\[
I_{1S(\varphi_{3})} = \bar{T}_{1S(\varphi_{3})}, \quad \text{under} \quad \varphi_{3} < \varphi_{BR}
\]

(8)

on the outer edge of the site \( dS_{K}^{F} \)[7]:

\[
\bar{T}_{2S(\varphi_{2})} = I_{2S(\varphi_{2})}R + \bar{T}_{1S(\varphi_{1})}(1 - R) \frac{n_{2}^{2}}{n_{1}^{2}}, \quad \text{under} \quad 0 < \varphi_{2} < \frac{\pi}{2}
\]

(9)

where \( n_{1} \) and \( n_{2} \) – refractive indices of the crystal material and the medium in the thermal unit; \( I_{1S} \) and \( I_{2S} \) – radiation intensity in the interval \( \delta \lambda_{t} \) in the crystal and the environment of the thermal unit; the \( S \) direction is determined by the angle \( \varphi \); \( R \) – is the reflection coefficient of the free surface of the crystal.

**Figure 3.** a) - a diagram for the formulation of a mathematical model, where: 1 - heat system; 2 - heating unit; 3 - crystal; F - crystallization front; \( S, S_{K}^{F}, S_{K}^{F}, S_{K}^{F} \) - total surface of the thermal system, crystal surface: - total free and fixed; lines ab and bp conventionally represent free and fixed surfaces; \( t \) - normals to the surface; b) - Scheme for the formulation of the boundary condition on the free surface of the crystal: \( I_{1S}, I_{2S}, \varphi_{1}, \varphi_{2}, \varphi_{R} \) - radiation intensities and angles of their incidence in the crystal and the environment; \( n_{1}, n_{2} \) - refractive indices of the crystal of the environment: \( \varphi_{BR} \) – Brewster angle; \( S_{K}^{F} \) – free surface of the crystal; [13]
The Brewster angle was determined by the expression [7,9]:

\[ \varphi_{BR} = \arctan \frac{n_2}{n_1} \]  

(10)

The connection between the angles \( \varphi_1 \) and \( \varphi_2 \) is found from the relation [7,9]:

\[ \sin \varphi_2 = \sin \varphi_1 \frac{n_2}{n_1} \]  

(11)

Thus, the radiation descending from the single crystal to an elementary area within the Brewster angle minus the reflected one originated from the single crystal and was absorbed in the medium of the thermal node within the hemisphere. The radiation, descending to the site at an angle to the normal, was completely reflected by the Brewster angle. Radiation entering the elementary area from the thermal unit, minus the reflected one, entered the single crystal and was scattered in it within the Brewster angle. It can be seen from (6) that in the absence of a zone of opacity in the crystal and vacuum in the thermal unit, the following condition was met:

\[ \frac{d\lambda}{dn} = 0 \]

4. Results and discussion

The above-considered stationary, three-dimensional mathematical model of heat and mass transfer for a generalized cycle of directed crystallization gives a general idea of the course of heat and mass transfer processes in the ECU and a single crystal placed in it.

The main results of mathematical modeling of the temperature distribution of a solid modified by the ECU of the first and second types and the steel water-cooling jacket (SWJ) of the crystallization apparatus are presented in Figures 4 and 5. Figure 6 shows the results of calculations that give an idea of the temperature distribution in a single crystal and the effect of the temperature field on the shape of the solidification front at growing in ECU of various modifications.

It follows from Figure 4 that when growing single crystals in vacuum and an excess gas atmosphere, the design of the modified ECU of the first type and the low thermal conductivity of the graphite thermal insulation used in it make it possible to create a low-gradient region in the crystal growth zone and a low-gradient temperature region in the annealing zone with a step of changing the temperature of the solid bodies in the crystal growth zone 10 - 40°C/cm, in the annealing zone 15 - 40°C/cm (Figure 7-a).

To use the synthesis technology in an excess gas atmosphere, values of 20 - 30°C/cm were obtained in the crystal growth zone and 20 - 30°C/cm in the annealing zone (Figure 7-b).

For growing single crystals in the modified ECU of the second type, the values of temperature gradients similar to the values obtained in [12] for the basic ECU of the atmosphere with excess gas pressure were obtained (Figure 5). It should be borne in mind that with an increase in the gas feed rate, the axial temperature gradient becomes somewhat sharper. So, when blowing the model with flow rates of the admitted gas of 0.1, 0.2 and 0.5 m/s, the axial gradient changed insignificantly and looked like in Figure 7-c. And at speeds of the supplied gas 1 and 2 m/s, the temperature gradient took the form as in Figure 7-d. The average values in the region of the crystal growth zone were 70–50°C/cm and 20–40°C/cm in the crystal annealing zone at gas flow rates of 0.1, 0.2, and 0.5 m/s; for velocities of 1 and 2 m/s were equal to 75–60°C/cm and 20–40°C/cm in the growth and annealing zones of the single crystal, respectively.

It can be seen from Figures 6 (a-f) that the created thermal conditions in the modified ECU provide a slightly convex isotherm of the growth front and, thanks to the separate control of the heaters in terms of power, create the desired angle of inclination of the vertical gradient, which allows the growth of optically perfect crystals.

The symmetrical temperature field required to create a morphologically stable crystallization front was created using two graphite tape arc heaters with separate power control. In this case, the upper heater above the surface of the charge (and, accordingly, with the melt) had a flat L-shape, and the lower one had an inverted U-shape and was placed under the crucible (boat) and ensured uniform heating of both the bottom and side walls of the crucible.
Figure 4. * Results of mathematical modeling of the temperature distribution in the modified ECU according to the first type for the use of vacuum growing technology (a-d) and the technology of growing single crystals of fluorides at excess pressure (e-f), where: a, e) - in the longitudinal section (form side); b) - isotherm of temperature distribution along from the beginning of the loading zone to the end of the unloading zone; c) - in longitudinal section (top view); d) - in cross section; f-g) is an isotherm of the temperature (gas) distribution along from the beginning of the loading zone to the end of the unloading zone, while views (g) and (h) clearly display a high-gradient region in the crystal growth zone;

*- due to insignificant differences in the temperature distribution pattern in a solid for growing single crystals at excess pressure, the results are presented only for Ar
Figure 5. *Results of mathematical modeling the temperature distribution in the modified growth node according to the second type for the use of the technology of growing single crystals of fluorides under excess pressure and gas supply, where Figures a, c, e, g are for an inlet velocity of 0.1 m/s, and Figures b, d, f, h for 1 m/s, letters are designated: a, b) - in longitudinal section (side view); c, d) - isotherm of temperature (gas) distribution along from the beginning of the loading zone to the end of the unloading zone; e, f) - trajectory and temperature of the supplied gas; g, h) - velocity of the supplied gas and isotherm along the horizontal surface of the crystal;
* the pictures of the temperature distribution in the modified thermal unit according to the second type for the vacuum growing technology are similar to the pictures in [12]
Figure 6. * The result of computer simulation of the temperature distribution of a crystalline body along the growth axis of a single crystal along a longitudinal section, top and side view in a CaF$_2$ single crystal, where: a) - in the modification of the ECU of the first type (atmosphere - vacuum); b) - in the modification of the ECU of the first type (atmosphere - nitrogen); c) - in the modification of the ECU of the first type (excess helium atmosphere); d) - in the modification of the ECU of the first type (excess argon atmosphere); e) - in the modification of the ECU according to the second type (at a gas injection rate of 0.1 m/s - an excess Ar atmosphere); f) - in the modification of the growth unit according to the second type (at a gas injection rate of 1.0 m/s - an excess Ar atmosphere);

* - Figure 6 shows the main results of mathematical modeling for argon gas flow rates of 0.1 and 1 m/s, the rest of the gas inlet rates were excluded due to insignificant differences in the temperature distribution patterns in the crystalline body.
From Figure 6-a it follows that the preferred medium for growing single crystals was vacuum. Thus, a sharp temperature gradient of the crystallization front is created in the growth site, which is a necessary condition for the synthesis of single crystals of fluorides [12, 16-18]. During the synthesis of single crystals of fluorides in the ECU of the first type under conditions of an atmosphere of excess argon pressure at the same temperatures, a smoother temperature gradient is observed at the solidification front due to the increased thermal conductivity of the thermal system. In the case of an excess pressure of the atmosphere of \( \text{N}_2 \) and He, the crystallization front takes on a slightly diffuse shape as a result of the higher thermal conductivity of these gases in comparison with argon. Consequently, when using He or \( \text{N}_2 \) gases, a low-gradient region is created in the single crystal growth zone, which is the least preferable for the synthesis of fluoride single crystals from the point of view of thermal processes [16-18].

For the synthesis of a single crystal in the ECU of the second type using gas puffing at different rates, the obtained results of computer simulation (Figure 6 e, f) are almost identical to the results [12] for the basic ECU. The crystallization front has a sharp temperature gradient, as in the synthesis of fluorides in a vacuum atmosphere, which also ensures the high quality of the obtained single crystals [19-21].

5. Conclusion
The above-considered stat Mathematical modeling of the influence of the temperature field created by ECU on the position and configuration of the solidification front under different conditions of growing single crystals of fluorides by the HDS method has been carried out. The necessary data on the influence of temperature fields on the configuration of the solidification front have been obtained. It is found that the designs of the modified ECU create thermal conditions providing a slightly convex isotherm of the solidification front. It is shown that the most favorable thermal conditions for synthesis are observed in a modified ECU of the first type in a vacuum atmosphere (Fig. 6-a). Using an atmosphere of excess gas pressure, a smooth temperature gradient of the solidification front is observed due to the creation of a low-gradient region in the crystal growth zone (Fig. 6 b-d). For the synthesis of a single crystal in The ECU of the second type using gas puffing at different flow rates, the results of computer simulation are identical to those obtained using a vacuum atmosphere.

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