Journal of Engineering Physics and Thermophysics, Vol. 83, No. 5, 2010

CRYSTAL GROWTH IN TWO-LAYER FLUID SYSTEMS IN MICROGRAVITY

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UDC 532.5:532.78.538.4

A numerical study of the crystal growth in zero gravity with the use of an additional liquid layer for the ampoule-free variant of zone melting (floating-zone method) has been conducted. The crystallization process was investigated both without the magnetic-field action and with a static magnetic field at two directions of the magnetic field vector. A classification of the flow structure depending on the dominance of the thermocapillary effect on the free surface or at the interface between the liquids has been made. The radial microsegregation of the dopant for the above crystal growth methods with several combinations of physical parameters of the liquids has been investigated. The dependences of the radial microsegregation of the dopant on the flow conditions and the strength and sense of the magnetic field have been determined. It has been shown that the strongest effect of a decrease in the microsegregation of the dopant is achieved for flows with a dominance of the thermocapillary effect on the free outer boundary of the liquid two-layer system at a certain magnetic field strength.

Keywords: *fluid mechanics, crystal growth, encapsulation, dopant segregation, magnetic field.*

Introduction. Two-layer liquid systems have attracted increased interest in connection with the investigation of the stability of flows and the practical application of such systems for growing high-quality monocrystals. The stability of flows in two-layer systems began to be investigated in [1], where a linear method was used to analyze the stability of flows under the action of small perturbations on a liquid with an unlimited free boundary on which a thermocapillary effect takes place. Subsequently, a large number of works in which this method was used to investigate the stability of flows in two-layer systems appeared [2–5]. It should be noted that in [2, 4] oscillatory flow conditions were considered, also with account for the gravitational convection. In monograph [6], a systematization of such investigations with the use of the above-mentioned model was given. The results of numerical investigations of the stability of thermocapillary convection in bounded closed volumes under perturbing actions of various natures [7–9] made it possible to draw the conclusion that the flow stability in this case is determined by the interaction between the shear flow created by the thermocapillary effect and the flow near the solid boundaries. Numerical calculations [10–12] showed different regimes of thermocapillary convection in two-layer systems differing in layer size, physical parameters of the liquids, Marangoni numbers, and thermal boundary conditions. The liquid flow in two-layer systems at a combination of thermocapillary and thermal convections was considered in [13]. The results of the experiment in a three-layer liquid system in microgravity were described in [14].

In [15], the possibility of practical application of a liquid two-layer system in growing crystals by the floating-zone method (crystallizable liquid encapsulation method) was first discussed. It was suggested to surround the liquid zone from gallium arsenide by a boron oxide (B_2O_3) melt with the aim of eliminating arsenic vapors and maintaining the given stoichiometry of the GaAs crystal. In the inventor's certificate [16], it was suggested to use an additional liquid layer near the crystallization boundary to avoid contact with the ampoule walls and thus lower the thermal stresses in the growing crystal and their associated dislocation density in the process of crystal growth by the Bridgman–Stockbarger method. In the description of this inventor's certificate, it was noted that an additional liquid layer can also be used to lower the thermocapillary convection intensity in the crystallizing melt. The same description also gives some examples of semiconductor materials and liquids that can be used as an additional layer in growing semiconductor crystals and comply with the necessary conditions for their application: mutual chemical inertness of

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liquid pairs and preservation of the additional layer in the liquid state in the crystallization zone of the basic material. For gallium arsenide, it is suggested to use boron oxide, and for silicon and gallium phosphide — beryllium or magnesium fluoride. In the USA patent [17], it is suggested to use a two-layer liquid system in the ampoule variant of zone melting with the aim of maintaining the given stoichiometry and lowering mechanical stresses (decreasing the dislocations) in the growing crystal. In the description of this patent it is pointed out that in the future such systems can use a magnetic field to decrease the convection intensity in the melt. In [18], the first results of the investigation of the thermocapillary convection in liquid two-layer systems with a free outer boundary corresponding to the flows of the configuration of the floating-zone method were presented. It was revealed that there exists a real possibility of effective control of the thermocapillary convection in zero gravity due to the choice of a pair of liquids with suitable physical properties. In particular, it was noted that the effect of thermocapillary flow reversal in the internal (crystallizing) liquid layer with respect to the effective direction of the thermocapillary effect at the interface between the liquid layers is possible. Beginning with [19], the subsequent investigations [20, 21] were carried out for liquid two-layer systems and boundary conditions corresponding to the crystal growth by the floating-zone method. This is due to the clear understanding that two surfaces with a thermocapillary effect make it possible to investigate a much wider range of control actions on the liquid flow than in the presence of one surface with such an effect when two-layer liquid systems are used in growing crystals by ampoule methods. Of course, in this case the number of possible variants of investigation also increases considerably (threefold, as it turned out). In [19], it was shown for the first time that a marked decrease in the radial macrosegregation of the dopant can be obtained in growing crystals in two-layer liquid systems by the floating-zone method at a certain relation between the physical properties of the liquid pairs. Further investigations [20, 21] showed that a constant magnetic field can be used to control the process of dopant distribution in crystals grown in zero gravity. This conclusion turned out to be directly opposite to that drawn earlier for the one-layer variant of the floating-zone method [22]. In this case, the radial macrosegregation of the dopant always increases with increasing strength of the stationary magnetic field, acquiring a catastrophic character beginning with a certain value of this field strength. The liquid flows in two-layer systems belong to the class of multiparameter problems, which impedes investigations without a rational grouping of flows by some essential character. In [21], we proposed a classification of flows in liquid two-layer systems based on the intensity ratio between the thermocapillary effects on the free surface of the liquid and at the liquid–liquid interface.

Formulation of the Problem. Consider a rectangular two-layer liquid system of length $x = L$ and thickness $y = \delta = \delta_1 + \delta_2$. The plane geometry was chosen to show how a static magnetic field at two directions of magnetic induction influences the flow and the transport of the dopant in the process of crystal growth. At the lower boundary $y = 0$, the symmetry condition for the vertical velocity component, the temperature, and the dopant concentration $\partial u/\partial y = \partial\theta/\partial y = \partial C/\partial y = 0$ imitating cylindrical geometry (pseudocylinder) is used. The upper boundary $y = \delta$ is a free boundary of the liquid on which the distribution of the external heat flow is given in the form of an exponent with a maximum at the center of the liquid zone ($x = L/2$):

$$\frac{\partial\theta}{\partial y} = A_1 \exp\left[-A_2\left(x - \frac{L}{2}\right)^2\right].$$

At the ends of the region $x = 0$ and $x = L$, the melting (crystallization) temperature T_0 is held constant. The boundary $x = 0$ is a solid wall.

The static magnetic field is considered in the induction-free approximation and acts selectively only on one liquid layer (upper or lower). The second layer represents in this case an electric insulator, along with the side solid walls. It is assumed that the Joule heat liberated in the layer with an effective magnetic field is negligibly small.

At the crystallization boundary of the main liquid layer ($x = L$, $0 < y < \delta_1$), the thermodynamic condition for the dopant obtained for the condition of weak mixing in the liquid is set, but it is also suitable for the intermediate case where no complete mixing of the liquid occurs. The other boundaries of this layer are impenetrable to the dopant. The liquids do not mix and do not interact chemically. The thermocapillary effect is active at both the outer free boundary and at the interface between the two liquid layers. These boundaries do not deform under the action of the liquid flow, i.e., they are assumed to be planar. At the interface between the two liquid layers the conditions of heat-flow continuity and equality of viscous stresses are used.

TABLE 1. Parameters for Calculating the Crystallization Process in the Two-Layer System of the Floating-Zone Method

Regime	Ma_{12}	Ma_2	Pr_1	Pr_2	K_ν	K_λ	K_ρ	A_1	A_2
1	$2.8\cdot10^2$	$1\cdot10^3$	0.1	10	80	0.5	0.8	0.8	0.2
1a	$5.2\cdot10^2$	$3.74\cdot10^3$	0.1	10	80	0.5	0.8	15	10
2	$2.16\cdot10^3$	$7\cdot10^2$	0.01	0.021	2	1.5	0.4	0.8	0.3
3	$7.6\cdot10^3$	$2.35\cdot10^1$	0.02	1	30	0.5	0.3	0.8	0.2

The system of magnetic hydrodynamic equations and heat and mass transfer equations in the vector form of two viscous, incompressible, electrically conducting liquids has the following form:

$$\partial \mathbf{u}_i/\partial\tau + (\mathbf{u}_i\Delta)\,\mathbf{u}_i = -\,K_\rho^{-1}\,\nabla p_i + K_\nu\nabla^2\mathbf{u}_i + K_\beta^{-1}K_\nu\,Gr_{iT}\,\theta_i\mathbf{e}_g + K_\chi^{-1}\,K_\nu\,Ha_i^2\,(\mathbf{u}_i\times\mathbf{B})\times\mathbf{B}\,, \tag{1}$$

$$\operatorname{div}\mathbf{u}_i = 0\,, \tag{2}$$

$$\partial\theta_i/\partial\tau + (\mathbf{u}_i\Delta)\,\theta_i = K_\nu\,Pr_i^{-1}\nabla^2\theta_i\,, \tag{3}$$

$$\partial C/\partial\tau + (\mathbf{u}\Delta)\,C = K_\nu\,Sc^{-1}\nabla^2 C\,. \tag{4}$$

The boundary conditions are:

$$x = L:\ \ \partial C/\partial y = -\,Sc\,(1 - k_0C_s)\,Re_{cr}\,,\ \ u_i = \theta_1 = \theta_2 = 0\,; \tag{5}$$

$$y = 1:\ \ \partial u_2/\partial y = -\,Ma_2\,K_\nu\,Pr_2^{-1}\,(\partial\theta_2/\partial x)\,,\ \ v_2 = 0\,; \tag{6}$$

$$\partial u_1/\partial y = -\,Ma_{12}\,Pr_1^{-1}\,(\partial\theta_1/\partial x) + K_\nu\,K_\rho\,(\partial u_2/\partial y)\,,\ \ \partial\theta_1/\partial y = K_\lambda\,(\partial\theta_2/\partial y)\,, \tag{7}$$

$$y = \delta_1:\ \ \theta_1 = \theta_2\,,\ \ u_1 = u_2\,,\ \ v_1 = v_2 = 0\,.$$

Equations (1)–(3) pertain to the liquid layers (i = 1 and 2), and Eq. (4) — only to the first layer of the main liquid.

To solve system (1)–(7) numerically, we used a finite-difference scheme of a higher, third order of accuracy in spatial coordinates. This scheme and the computational algorithm has been used earlier in [7, 8, 18–22, 24].

All calculations were performed for complete weightlessness conditions, where Gr_{1T} = 0, and for one thickness of the second additional liquid layer $K_\delta = \delta_2/H$ = 0.2.

Results of the Calculations. With the use of the condition of classification of liquid flows in two-layer systems proposed in [21], the whole diversity of flows can be classified under one of the following three types: 1) with a dominance of the thermocapillary effect at the free boundary of the liquid (y = 1); 2) with an approximate equality of the thermocapillary effect at the free boundary (y = 1) and at the interface between the liquid layers ($y = \delta_1$); 3) with a dominance of this effect at the interface between the liquids ($y = \delta_1$). For the overwhelming majority of liquids, the surface tension increases with decreasing temperature and the liquid on the free surface or at the liquid–liquid interface moves under the action of the thermocapillary effect into a cooler region.

Table 1 shows the values of the dimensionless parameters of the problem for the above three types of flow conditions. For the first type of flow condition, calculations were made for two variants of heat-flow distribution at the outer boundary of the system: a wide heating zone (variant 1) and a narrow heating zone (variant 1a).

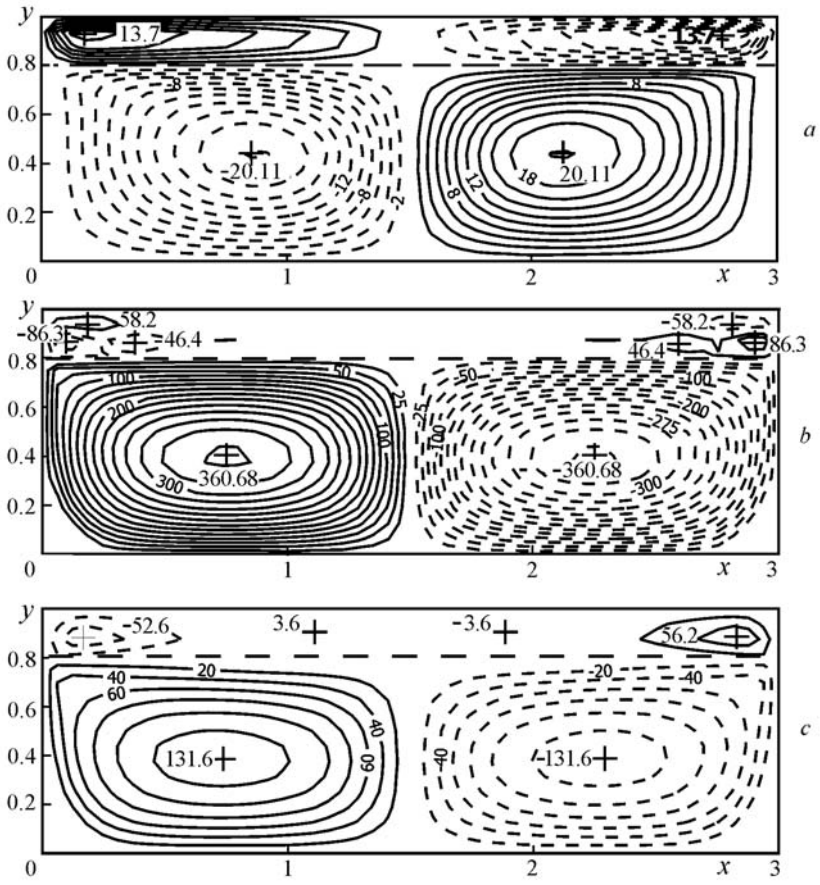

Fig. 1. Flow structure in the liquid layers for regimes 1 (a), 2 (b), and 3 (c).

Figure 1a gives an example of the liquid flow in a two-layer system (stream-function field) for the first type of flow condition. Hereinafter the clockwise flow of the liquid is marked by dashed lines and the counterclockwise flow — by solid lines. In the first type of flow condition, the liquid in the lower layer flows along the interface between the layers in the direction opposite to the acting thermocapillary effect at this interface. The second type of flow condition, where the action of the thermocapillary effect at the above boundaries is at some equilibrium, is shown in Fig. 1b. Along the boundaries with the acting thermocapillary effect the liquid in both layers moves in the direction given by this effect. As a result, in the additional upper layer, a flow with two circulation layers is formed. Such a flow structure in the upper layer is unstable, as a rule, and tends to go to the oscillatory regime. The third type of flow condition, where the thermocapillary effect predominates at the interface between the liquid layers, is shown in Fig. 1c. In this case, the liquid in the additional upper layer moves already counter to the direction of the thermocapillary effect at the outer free boundary.

The static magnetic field applied to an electrically conducting liquid offers an additional possibility of acting on the thermocapillary flow and the distribution of dopants. The Lorenz force (the last term in (1)) is bulk and, therefore, it practically does not act on the thin-surface monomolecular layer. As a result, the thermocapillary flow is pulled to the interface between the liquid layers. As was shown in [22], in the case of a thermocapillary flow in the one-layer variant of the floating-zone method, this effect leads to an intensification of the liquid mixing near the region of action of the thermocapillary effect and, conversely, to a weakening of such mixing at a distance from this region. As a result, the nonunformity of the dopant distribution along the crystallization boundary increases. In the case of a two-layer liquid system, the action of a static magnetic field on the thermocapillary flow has a more complex character. This is graphically demonstrated by the data presented in Figs. 2–5 for the three types of flow conditions under consideration. In these figures, the notations of the curves correspond to the variants of the flow conditions given in Table 1. The data for the radial inhomogeneity parameter ξ_c presented in these figures pertain to the instant of time at which the flow begins to go to the steady state.

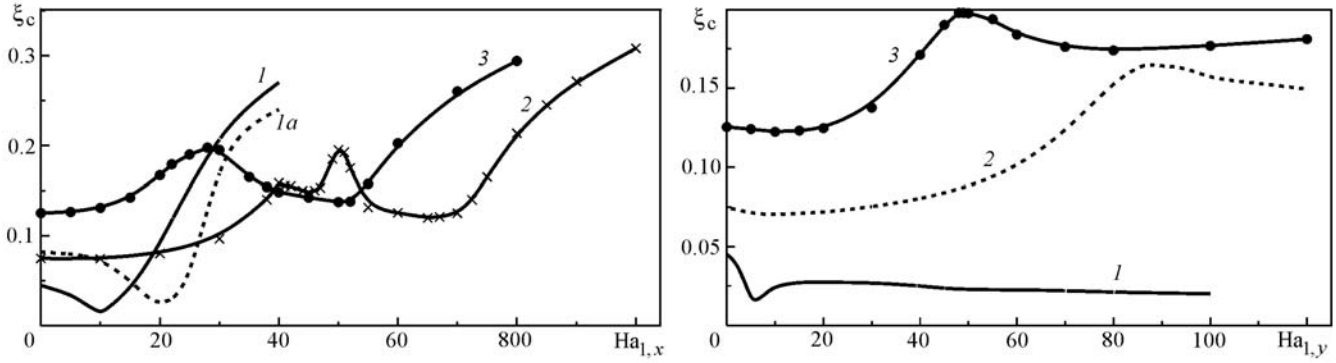

Fig. 2. Relative radial inhomogeneity of the dopant distribution versus the intensity of the magnetic field directed along the lower layer of the two-layer system for four variants of the floating-zone method.

Fig. 3. Relative radial inhomogeneity of the dopant distribution versus the intensity of the magnetic field directed across the lower layer for three variants of the two-layer system of the floating-zone method.

The dependence of the radial macrosegregation of dopants on the static magnetic-field intensity is different for the above types of flow conditions in two-layer systems. While for the first type of flow condition there exist zones of the magnetic strength value (Hartmann number) in which the radial macrosegregation of the dopant decreases with increasing magnetic field strength (Figs. 2, 3, and 5), for the other two types of flow condition (Fig. 4) this effect is practically absent. The maximum positive effect for regime 1 is obtained in the case of magnetic-field action along the x axis at Ha = 10, and for regime 1a at — Ha = 20 (Fig. 2). If the magnetic field in the lower layer acts along the normal to the layers (along the y axis), then the maximum decrease in the radial macrosegregation of the dopant is obtained for regime 1 at Ha = 7 (Fig. 3). For the other two variants of the flow, a positive effect is attained in this case at Ha = 10, but the effect itself is insignificant.

If the magnetic field is applied to the upper additional liquid layer, the action on the liquid flow and the dopant distribution in the lower crystallizing layer is due to the interaction of the layers. Naturally, a noticeable positive effect for the radial macrosegregation of the dopant is achieved in this case at a much larger magnetic field. Figure 4 shows the case where the magnetic field in the upper layer acts along the x axis. An insignificant positive effect takes place for regime 1 at the same Hartmann number (Ha = 10) as under the magnetic-field action on the lower layer (see Fig. 2). A strong positive effect of the magnetic field is attained (Fig. 5) when a very large magnetic field acts in the upper additional liquid layer along the normal to the layers (along the y axis). The minimal effect is attained in the case where Ha = 200 for variant 1 and Ha = 600 for variant 1a. Thus, if optimal parameters of the additional liquid and of the magnetic field are chosen, then the radial segregation of the dopant can be decreased by more than one-half. An important feature of the flow at which a considerable positive effect of the magnetic field is attained is the fact that the flow in the crystallizing liquid layer is always directed against the thermocapillary effect acting at the interface between the layers (regimes 1 and 1a). This condition, as well as the maximum values of the magnetic field, are important features used in the patent [23].

Under real conditions, the choice of physical parameters of suitable pairs of liquids (primarily density and viscosity) is quite limited. However, there exists a wide possibility of choosing the thickness of the liquid layers and the heat flow from the outside to the molten zone, and this should be done so that the above-mentioned necessary condition of positive use of the thermocapillary effect in two-layer liquid systems is realized. Under terrestrial conditions this choice should be made with account for the action of the gravitational convection. It should be noted therewith that in producing especially complex and expensive crystals the sizes of samples are usually small and the thermocapillary convection in this case is a dominant source of liquid motion.

In [24], the results of the numerical investigation of the liquid crystallization in two-layer systems for ampoule crystal-growth methods (Bridgman–Stockbarger and traveling heater methods) are presented. It was shown that in this case it is impossible to markedly decrease the radial segregation of dopants. This investigation was undertaken because

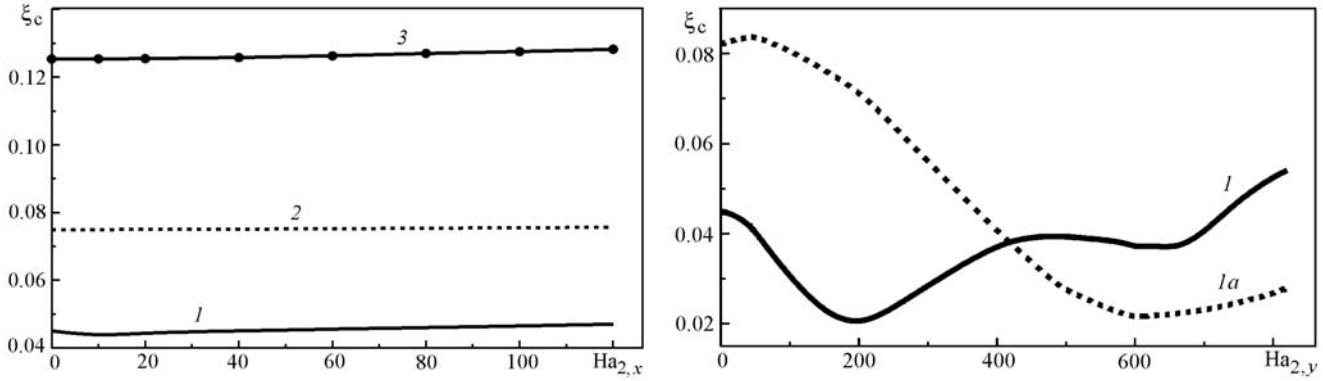

Fig. 4. Relative radial inhomogeneity of the dopant distribution versus the intensity of the magnetic field directed along the upper layer for three variants of the two-layer system of the floating-zone method.

Fig. 5. Relative radial inhomogeneity of the dopant distribution versus the intensity of the magnetic field directed across the upper layer for two variants of the two-layer system of the floating-zone method.

the majority of works, including expensive experiments in zero gravity, had been conducted for configurations of liquid two-layer systems pertaining to ampoule crystal-growth methods.

Conclusions. The investigation of two-layer liquid systems was stimulated by the aspiration to develop a technology of growing crystals having a perfect structure and a minimum of defects. For the model, we took natural diamond that has a perfect structure and is enclosed in a kimberlite tube. This points, apparently, to the fact that the diamond crystal grows in the midst of a liquid layer of another material but has no free outer surface of the liquid. The presence of a liquid layer surrounding the crystallizing liquid layer decreases the dislocation density in the crystal. Therefore, the majority of works on two-layer liquid systems [10–14, 16, 17] were performed for configurations and boundary thermal conditions corresponding to ampoule variants of crystal growth (Bridgman–Stockbarger and floating-zone methods). Besides the experiments on the Space Shuttle (in 1994) [14], experiments of this kind were performed as part of the Maxus program on a sounding rocket (in 1995) and during the flight of Spacelab (in 1996). As the results presented in the given paper show, two-layer liquid systems can be used successfully in the technology of crystals grown by the floating-zone method.

The present investigation completes the series of works devoted to modernization of the floating zone of crystal growth in microgravity. It was shown earlier that a rotating magnetic field [7] and capillary standing surface waves excited by axial vibration, separately and in combination with a static magnetic field [25], can be used to lower the macro- and microsegregation of dopants in microgravity. The method and the device with standing surface waves with a magnetic field and without it were issued a patent [26]. These methods can also be used in ground technology upon the necessary optimization of the parameters of external action on the crystallizing liquid.

NOTATION

A_1, A_2, dimensionless numerical parameters in the expression for the heat flow on the free surface of the liquid; a, thermal diffusivity, m^2/s; **B**, magnetic induction, T; c, dopant concentration in the liquid, kg/m^3; $C = c/c_0$, dimensionless dopant concentration; D, diffusion coefficient of the dopant in the liquid, m^2/s; $\mathbf{e}_g$, unit vector of gravitational acceleration; g, gravitational acceleration; $Gr_{iT} = g\beta_i \Delta T H^3/\nu_i^2$, Grashof number; $H = \delta_1 + \delta_2$, thickness of the two liquid layers, m; $Ha = BH(\chi/\rho\nu)^{1/2}$, Hartmann number; $K_\delta = \delta_2/H$, dimensionless thickness of the additional liquid layer; $K_\beta = \beta_2/\beta_1$, $K_\rho = \rho_2/\rho_1$, $K_\nu = \nu_2/\nu_1$, $K_\lambda = \lambda_2/\lambda_1$, $K_\chi = \chi_2/\chi_1$, dimensionless parameters composed of the physical properties of the liquid layers; L, liquid column length, m; k_0 = dimensionless equilibrium dopant distribution coefficient; $Ma = -(\partial\sigma/\partial T)H\Delta T/(\rho\nu a)$, Marangoni number; p, pressure, Pa; $Pr = \nu/a$, Prandtl number; $Re_{cr} = u_{cr}H/\nu$, dimensionless crystal growth rate; $Sc = \nu/D$, Schmidt number; T, temperature, K; t, time, s; **u**, velocity vector, m/s; u, v, projection of the velocity vector on the x and y axes, respectively, m/s; x, coordinate along the liquid

layers, m; y, coordinate across the liquid layers, m; δ, liquid-layer thickness, m; Δt, time step in numerical calculations; $\Delta T = T_{max} - T_0$, characteristic temperature difference in the system, K; $\Delta C_s = C_{s,max} - C_{s,min}$, radial difference of concentrations in the liquid; $\theta = (T - T_0)/\Delta T$, dimensionless temperature; Λ, specific heat conductivity, W/m·K; ν, kinematic viscosity, m^2/s; ρ, density, kg/m^3; $\xi_c = \Delta C_s/\overline{C}_s$, dimensionless relative radial difference of the dopant concentration; σ, surface tension, N/m; $\tau = t\nu/H^2$, dimensionless time; χ, conductivity, S. Subscripts: 0, initial value; g, gravitation; c, concentration; cr, crystallization; ζ, gravitational; i, liquid-layer number ($i = 1$ for the lower, crystallizing layer, $i = 2$ for the upper, additional layer); max and min, maximum and minimum values of a parameter; s, at the crystallization boundary; bar, mean value at the crystallization boundary.

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