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Analysis of the profile curves of the menisci for the sapphire tubes growth by EFG (Stepanov) technique

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This paper deals with investigation of the behavior of the profile curves of the melt menisci for the case of the sapphire crystal growth by EFG (Stepanov) technique. The cases of external and internal circular menisci of the crystal tube are considered. The cases of the positive and negative angles of the contact of the profile curve with the working surface of the shaper are considered. Features of the profile curves are used in the automated control systems of the crystal growth using of the weight sensor.

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1 Introduction

Shape and quality of crystals being grown by EFG (Stepanov) technique is defined significantly by the shape and position of the crystallization front and the shape of a liquid meniscus being located between a crystal and a shaper.

Analysis of menisci shapes and their influence on the crystallization processes always attracted investigators. There are a lot of publications devoted to this problem. For instance, an approximate expression for the meniscus height depending on the boundary conditions is determined in [1]. But this expression, widely being used in Czochralski technique, is difficult to applicate to EFG (Stepanov) technique because of interconnected boundary conditions. Distinctive analysis of this problem is given in [2]. An approximate expression describing profile curve of the meniscus for the Czochralski technique is found in [3]. Analysis of the influence of the negative external pressure in the meniscus on the meniscus height limits in the silicon crystal growth by the EFG technique is given in [4]. Distinctive investigation of the liquid menisci, mainly, for the positive values of the external pressure, is carried out in [2]. Menisci heights ensuring the fulfilling the growth angle constancy condition and depending on the external pressure for the growth of the sapphire crystals by the EFG (Stepanov) technique are described in [5]. Expressions for the program menisci masses (observation expressions for the weight sensor signal) for various shapes of the cross-section of the shapers and crystals being grown by the EFG (Stepanov) technique are found in [6] on the basis of integrating the Young-Laplace equation. Observation expressions of the menisci weight signals are often used in the automated systems controlling the shape and quality of the crystals in the course of the growth process [7]. These expressions are also used in the development and investigation of the dynamic models of the crystallization processes [8]. Non-cylindrical (quadratoïd) shapes of the meniscus specific for the growth of rare-earth molybdates by modified Czochralski and EFG techniques [9] are calculated on the base of numerical solution of the Young-Laplace equation represented in cartesian (non-cylindrical) coordinates system [10]. Thermo-capillary numerical models also require solution of the Young-Laplace [13-20]. In [21] the problem of mechanical stability of the liquid menisci is considered.

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The present paper deals with investigation of the profile curves of the liquid menisci (and their characteristics) specific for the growth of the sapphire crystals by the EFG (Stepanov) technique and depending on the various external parameters and boundary conditions defining a meniscus shape. Such parameters and boundary conditions are: external pressure in the meniscus, meniscus height, contact angles between meniscus and working edges of the shaper and edge of the crystal, dimensions of the crystal and shaper. Changes of the ranges of external parameters and boundary conditions ensuring optimal conditions of crystallization process are discussed.

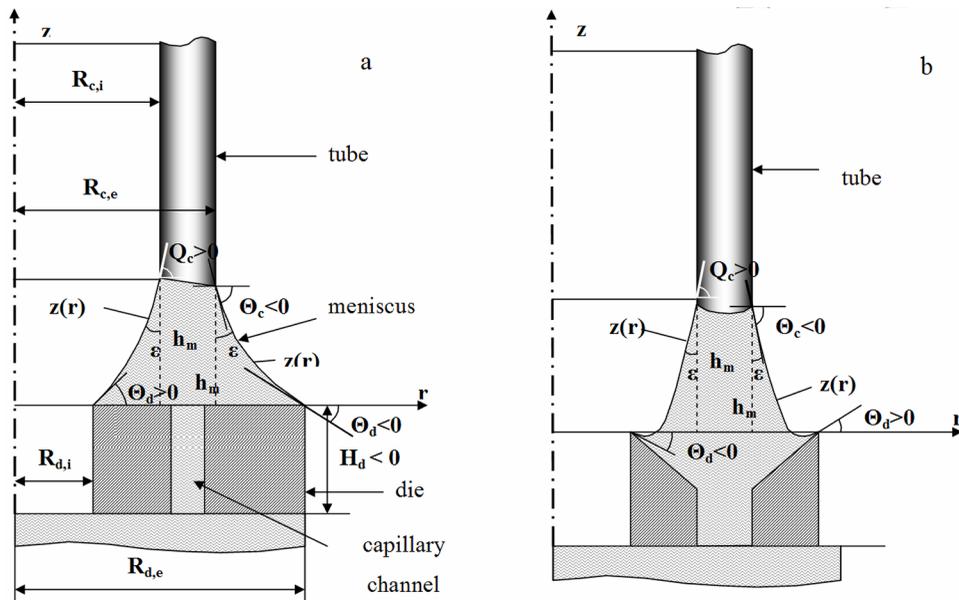


Fig. 1 Scheme of the crystal tube pulling, a – with ordinary die, b – with die having inclined working surface.

A scheme of the crystal tube pulling is shown in figure 1. The right side of the vertical cross-section of the growing tube is considered. This cross-section of the growing tube has to profile curves of the meniscus – right (external) and left (internal) profile curves. The angles θ_d and θ_c are the contact angles of the profile curve with the working edge of the die and crystal edge, respectively. Contact angle is defined as angle between a tangent and profile curve in the corresponding point. Two cases of the die shapes are considered: with horizontal working surface (Fig. 1a) and with inclined one (Fig. 1b).

Investigation of the profile curves of the liquid menisci was carried out on the basis of the numerical analysis of the capillary Young-Laplace equation with various parameters and boundary conditions close to the EFG (Stepanov) technique. Condition of gearing the meniscus with the working edge of the shaper was chosen for consideration among two possible conditions of contact between meniscus and shaper – gearing and wetting. Special consideration was carried out for menisci ensuring stationary isotropic growth of sapphire crystal, i.e. profile curve should be satisfied to the condition of the growth angle constancy at the edge of the crystal. Preset values of the boundary conditions for solving the Young-Laplace equation were close to the real ones produced by experimental processes of profiled crystal pulling.

Next items of research were carried out in the present paper:

- investigation of the profile curves of menisci depending on the type of the gearing with shaper, values of external pressure, dimensions of the cross-section of crystal and shaper;
- analysis of the menisci behavior under the conditions of negative and positive angles of contact of profile curves with working edges of the shaper;
- comparison of the outer and inner circular menisci for the case of crystal tube;
- comparison of the circular menisci and menisci forming near the lateral surface of crystal ribbon.

As it is known [11], profile curve $z(r)$ of liquid meniscus taking place between cylindrical crystal and shaper is described by capillary Young-Laplace equation in static consideration. This equation for circular meniscus should be written in dimensionless form as the next:

$$z''r + z'(1+z'^2) \pm 2(H_d - z)(1+z'^2)^{3/2}r = 0, \quad (1)$$

here r is radial coordinate, $z(r)$ is a function which describes the meniscus surface, H_d is external static pressure defining usually by a difference between levels of the melt surface in the crucible and the meniscus base at the working edges of the shaper.

Usually a surface of the melt in the crucible is lower than the meniscus base in considerable processes of sapphire crystal growth by the EFG (Stepanov) technique, i.e. $H_d < 0$. Therefore, present paper deals with negative or close to zero values of external pressure in the meniscus. Besides, profile curve description in a form of $z(r)$, not $r(z)$, is considered for solving Young-Laplace equation because we are not interested in menisci possessing ambiguity along horizontal coordinate r . Such menisci are absent during the stationary crystal pulling. At the same time analysis of the profile curves in $z(r)$ form allows to consider both positive, and negative values of the menisci angles θ_d between tangent to the profile curve and horizontal line of the working edges of the shaper, i.e. we can consider directly ambiguity along the vertical coordinate z (without sign change in the Young-Laplace equation for different branches of ambiguous profile curve). Positive contact meniscus angles θ_d (for example, in the case of right vertical cross-section of the external meniscus of the crystal tube) – when meniscus “goes” from the shaper at first downwards, and then, as usual, “goes” upwards – are possible in inclined working surfaces of the shapers. Such inclined shapers are widely used in the processes of sapphire crystal growth. The present paper deals with menisci relating to the stationary crystal growth and, hence, having no ambiguity along r – coordinate. Therefore, positive sign before last part is present in the Eq. (1).

Boundary conditions for solving Eq. (1) can be defined by different techniques. Our calculations operate basically with the next boundary conditions:

$$z(R_d) = 0, \quad -\arctg z'(r_c) = \pi/2 - \varepsilon, \quad (2)$$

here r_c is a crystal radius, R_d is a radius of the working surface of the shaper, ε is growth angle of material, which is approximately 13° for sapphire [5,12]. Thus, zero meniscus height is given at the working surface of the shaper. Upper edge of the meniscus contacting with the crystal should satisfy to the condition of the constancy of the growth angle.

2 External circular menisci

2.1 Investigation of the menisci profile curves for various values of the crystal radius Numerical results represented in figures 2 and 3 were produced for the external pressure $H_d = -4$ in dimensionless units. It should be taken in attention that capillary constant for sapphire is approximately 6 mm [12]. Thus, this pressure corresponds to the difference of -24 mm between heights of the melt surface in the crucible and working surface of the shaper. It is close to real data taken from the pulling process. External pressure is given to be negative, because the case of lower position of the melt surface in the crucible in comparison with location of the working surface of the shaper is considered. Crystal radius was being changed in the range from 0.95 to 0.995 with step 0.005. All linear sizes are represented in dimensionless units here and far in this paper. Radius of the working edges of the shaper was constant in this case.

Figure 2 shows profile curves of the menisci being calculated for the various radii of the cylindrical crystal, i.e. external right circular menisci of the crystal tube were analyzed. The angle θ_d was automatically adjusted as a result of iteration procedure in such a way that the angle θ_c of the meniscus near the crystal (in triple point) satisfied to the condition of the growth angle constancy. Taking into account the value of the growth angle for sapphire, the required angle of the inclination of the meniscus to horizontal line near the crystal edge should be equal to -77° with some sufficient accuracy.

The profile curves shown in figure 2a correspond to the negative angles θ_d , while in figure 2b – to the positive angles θ_d . One can see different forms of the menisci in these two cases.

Figure 2a shows that the height of the menisci decreases with increase of the crystal radius and approach of the crystal to the edge of the shaper. Profile curves “move” upwards from the shaper more “sharply”. Profile curves have some convexity upwards near the shaper, and then passing the excess point have convexity downwards.

As mentioned above positive angles θ_d are possible in the process with inclined working surfaces of the shaper. Figure 2b shows that profile curves “move” downwards at first, near the shaper, and then “move” upwards, as usual, to the crystal edge. Menisci behaviors in both cases, when crystal radius increases, are similar.

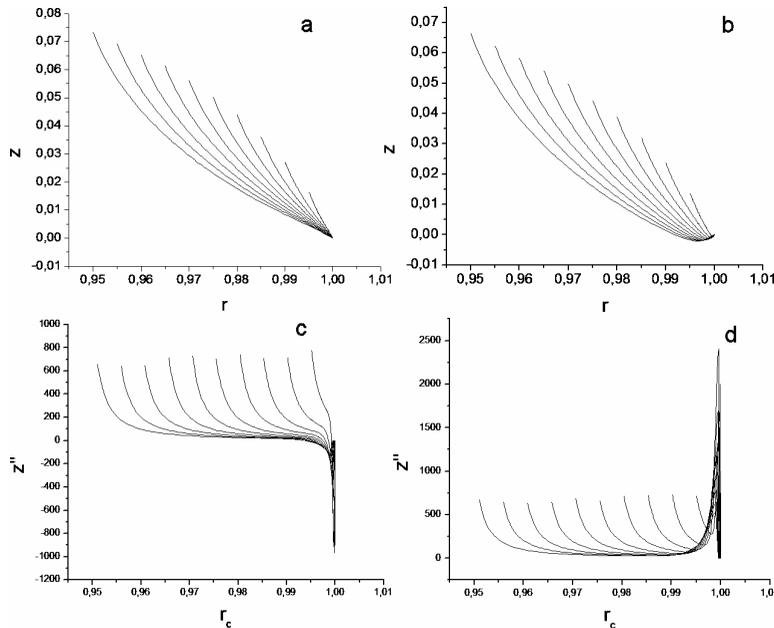


Fig. 2 a, b - Profile curves of the external menisci, a – for negative angles θ_d , b – for positive angles θ_d ; c, d – Second derivatives of profile curves of the menisci, c – for negative angles θ_d , d – for positive angles θ_d .

Thus, if to change the angle θ_d approximately from $-\pi/2$ to $+\pi/2$, two solutions of Young-Laplace equation correspond to the boundary conditions (2): one is in the area of the negative angles θ_d , second – in the area of positive angles θ_d .

Figures 2 c and d demonstrate second derivatives of the menisci profile curves characterizing direction of the curves convexity. Figure 2c corresponds to the negative values of the menisci angles near the shaper and shows that second derivative near the shaper is less than zero, i.e. curves at this place have convexity directed upwards. Then, second derivative changes its sign, becomes positive, with increase of the meniscus current height, i.e. curves have convexity directed downwards. Thus, second derivatives increase with approach of the profile curve to the crystal edge. This corresponds to “amplification” of the convexity of the profile curves near the crystal edge.

Second derivatives of the menisci profile curves for the positive angles θ_d shown in figure 2d are positive everywhere along the current menisci radii. Values of the second derivatives are significantly larger near the shaper than on removal from it. In the case of positive angles θ_d the excess point is absent, and menisci have convexity everywhere in melt direction.

Figures 3 a shows meniscus height dependence on crystal radius. Meniscus height decreases with crystal radius increase and its approach to the shaper edge. And meniscus height decreases faster, as crystal edge is closer to the shaper edge. Meniscus height decrease is a result of the decrease of a distance between working edge of the shaper and crystal edge. Smaller meniscus height and, hence, smaller crystallization front height correspond to more cold thermal zone of the system “melt-crystal”. In a pulling process it is necessary to choose some middle position of the crystallization front corresponding to the optimal conditions for crystal growth.

Behaviors of the meniscus height depending on crystal radius (Figs. 3a) are similar in both cases of negative (Fig. 3a, curve 1) and positive (Fig. 3a, curve 2) angles θ_d . One can only observe a little bit smaller heights at the same radii for the case of positive angles in comparison with the case of negative ones.

Figures 3b and c show dependence of the meniscus angle θ_d on the crystal radius. In the case of the negative area the angle decreases with the radius increase, i.e. its absolute value increases. And the angle absolute value increases faster if the crystal edge comes closer to the shaper edge. It should be explained by decrease of a distance between the edges of the crystal and shaper. In its turn, this explains more “sharp” behavior of the menisci shown in figure 2a near the shaper edge.

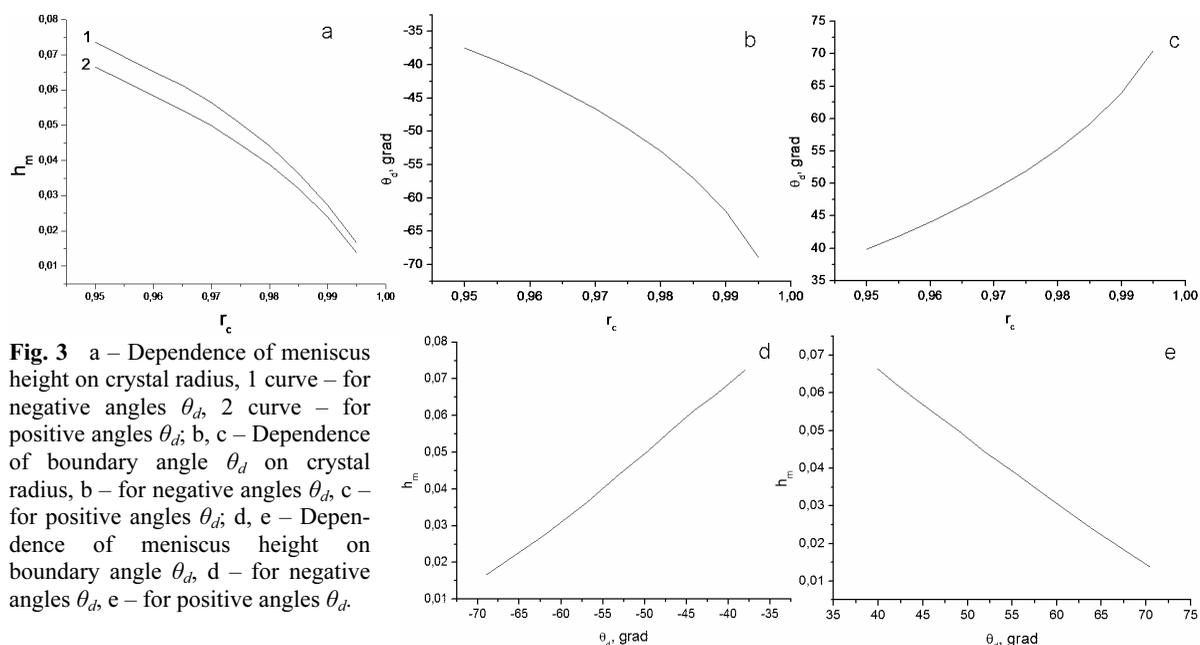


Fig. 3 a – Dependence of meniscus height on crystal radius, 1 curve – for negative angles θ_d , 2 curve – for positive angles θ_d ; b, c – Dependence of boundary angle θ_d on crystal radius, b – for negative angles θ_d , c – for positive angles θ_d ; d, e – Dependence of meniscus height on boundary angle θ_d , d – for negative angles θ_d , e – for positive angles θ_d .

Dependence for positive angles θ_d (Fig. 3c) is similar to the case of negative angles. The absolute value of this angle increases faster with closer crystal edge to the shaper edge. Difference between these two cases is in that meniscus “goes downwards sharper” at first, and then “goes upwards” in the case of positive angles near shaper edge, while in the case of the negative angles θ_d meniscus “goes upwards” at once from the shaper. The closer the crystal edge to the shaper edge, the smaller piece of meniscus “goes downwards” in the case of positive angles θ_d (Fig. 2b).

Figures 3d and e show dependence of the meniscus height on the meniscus angle θ_d . In the case of negative angles (Fig. 3e) meniscus height increases with the angle growth. Thus, the smaller an absolute value of this angle, the higher meniscus height and the more flat profile curve is. Figure 3d shows that the range of angles changing is small enough. In the crystal growth processes negative meniscus angle θ_d may be changed in the range from -70° to -30° for the chosen modeling parameters.

The case of positive angles shown in figure 3e is similar to the case of the negative angles: the larger an absolute value of the angle, the lower meniscus height. Both dependences are almost linear. Meniscus heights with positive angles θ_d are a little bit smaller than the same ones in the case of the negative angles.

2.2 Investigation of the menisci profile curves for various values of the external pressure The through calculation was carried out by different values of the external pressure which was changed in a range from -8 to 0 with step 1 in dimensionless units (capillary constants). The values of the crystal and shaper radii were the constant and equal to 0.97 and 1, respectively. At the point of contact of the meniscus with the crystal edge a condition of growth angle constancy was satisfied via the iterative changing of the meniscus angle θ_d .

Figures 4a and b show that meniscus height increases with increase of external pressure from $H_d = -8$ to $H_d = 0$. It can be explained so that decrease of absolute value of the external pressure the force “pressing”

meniscus down to the shaper also decreases. Second derivative of the meniscus profile curve characterizes well its form, all pieces of convexity and concavity. In the case of the negative meniscus angles near the shaper edge (Fig. 4c) absolute values of the negative second derivative are increasing with external pressure increase. In the same time positive second derivatives near the crystal edge decreases with external pressure increase. Increase of the absolute values of the negative second derivatives near the shaper edge should be explained by the common “lifting” of profile curves with approach of the external pressure to zero. Decrease of the second derivatives near the shaper edge is connected with “tendency of fluttering” of the profile curves with decrease of the absolute values of pressure. Fluttering of the profile curves is connected with decrease of the force “pressing” meniscus down to the shaper with the pressure growth.

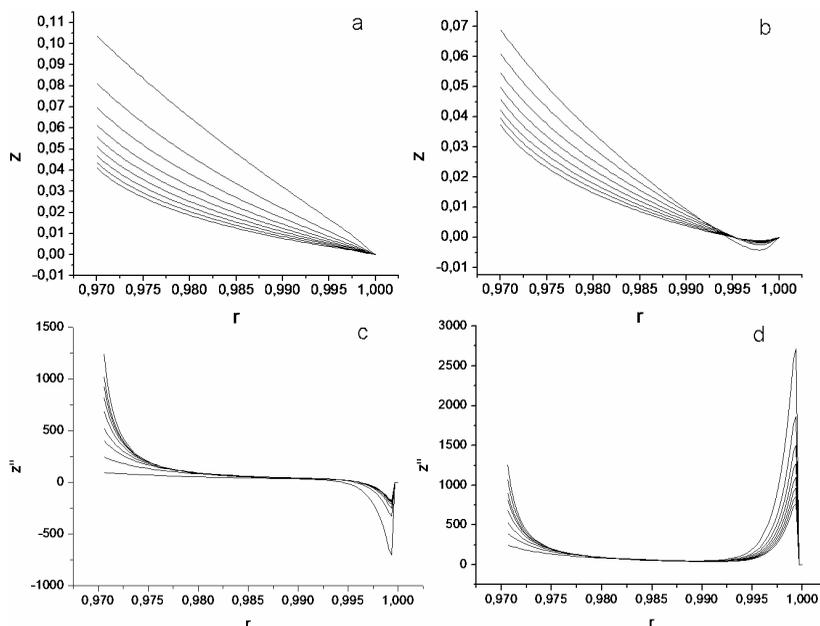


Fig. 4 a, b – Profile curves of the menisci for the various values of the external pressure, a – for the negative angles θ_d , b – for the positive angles θ_d ; c, d – Second derivatives of the profile curves of the menisci for the various values of the external pressure, c – for the negative angles θ_d , d – for the positive angles θ_d .

In the case of the positive boundary angles (Fig. 4d) the profile curves also become more flat near the crystal edge according to the reason described above, i.e. second derivatives decrease. Opposite case is near the shaper edge: second derivatives are growing with decrease of the absolute value of the pressure, i.e. menisci go downwards to the melt more “sharply”. It is connected with increase of the boundary angle θ_d with the decrease of the absolute value of pressure (Fig. 4b). Vice versa, increase of the boundary angle θ_d with approach of the pressure to zero is connected with that the larger this angle ensures, finally, higher “lifting” the profile curve near the crystal edge (satisfying to the condition of the growth angle constancy) – this is requirement of the pressure changing and approach to zero.

Figure 5a shows the growth of the meniscus height with the increase of the external pressure. And this is carrying out in both cases of negative and positive boundary angles θ_d . As mentioned above, it is connected with the decrease of the force “pressing” the meniscus down to the shaper. The closer the pressure to zero, the faster meniscus height grows. Corresponding profile curves for negative and positive boundary angles are almost similar. Therefore, figure 5 demonstrates characteristics of the meniscus profile curves only for negative boundary angles θ_d .

As figure 5b shows, absolute value of the boundary angle θ_d increases with decrease of the absolute value of the external pressure. By this, the closer pressure to zero, the faster growth of the boundary angle θ_d . For the providing the higher meniscus height (this is requirement of the pressure approach to zero), the larger absolute values of these angles θ_d are necessary (Fig. 4 a, b). Such behavior of the meniscus height and boundary angle θ_d influences on the process of crystal growth by EFG (Stepanov) technique. It is practically difficult to grow crystals with small absolute value of the negative external pressure. Large absolute values of the boundary

angles θ_d edge and necessity to maintain sufficiently high meniscus bring additional difficulties in the pulling process. In particular, these factors can be a reason of the overflow of the melt through the capillary over the shaper surface surrounding its working edges. Figure 5c demonstrates practically linear dependence of the meniscus height on the boundary angle θ_d .

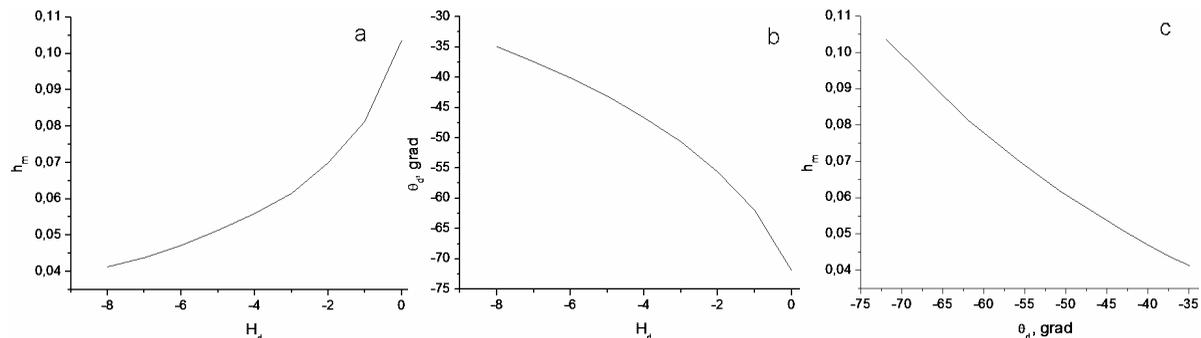


Fig. 5 Features of the profile curves of the menisci at the negative angles θ_d and satisfying to the condition of the growth angle constancy at the crystal edge, a – dependence of the meniscus height on the external pressure, b – dependence of the boundary angle θ_d on the external pressure, c – dependence of the meniscus height on the boundary angle θ_d .

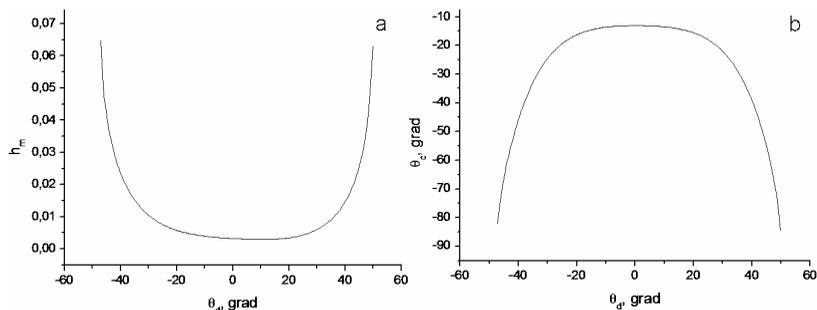
2.3 Investigation of the menisci profile curves for various boundary angles θ_d The calculation for the various values of the boundary angle θ_d was carried out for the constant values of the crystal and shaper radii. The crystal radius was equal to 0.97, shaper radius -1 and external pressure was equal to -4, in capillary constants. Condition of the growth angle constancy at the crystal edge was not required to be satisfied. Thus, instead of the boundary condition (2) there is used the next:

$$z(R_d) = 0, \arctg z'(R_d) = \theta_d, \tag{3}$$

Only menisci reaching the crystal edge with arbitrary contact angles θ_c were considered. The shapes of the meniscus profile curves both for negative, and positive boundary angles θ_d were similar to corresponding curves forms shown in figure 2.

As shown in figure 6a, the meniscus height is significantly higher under the large absolute values of the boundary angles θ_d than under small ones. Decreasing and increasing curve pieces (Fig. 6a) are corresponding to case above. Derivatives of the curve decrease and increase are significant. Hence, the range of the meniscus heights being sufficient for the stable growth and can be satisfied with the condition of the growth angle constancy is narrow enough. Figure 6b shows that the ranges of boundary angles being practically real are approximately: from -50° to -30° in the case of negative boundary angles θ_d and from $+30^\circ$ to $+50^\circ$ in the case of positive ones.

Fig. 6 a – Dependence of the meniscus height on the boundary angle θ_d , b – Dependence of the boundary angle θ_c on the boundary angle θ_d .



Profile curves reach very small (unreal in pulling processes by EFG (Stepanov) technique), meniscus heights near the crystal edge under relatively small absolute values of the boundary angles θ_d . Thus, small absolute values of the boundary angles θ_d (less than 30°) are not real during the process of sapphire crystal growth by

the EFG (Stepanov) technique. Relating to the behavior of the profile curves under the changing boundary angle θ_d – decrease of the absolute value of this angle result in lowering the profile curves in both cases of its negative and positive values.

Figure 6a shows that in the case of positive boundary angles θ_d menisci reach a little bit smaller height near the crystal edge than in the case of negative ones having the same their absolute values. It may be connected with a difference in profile curve shapes for the different cases. Under the positive angles θ_d meniscus profile curve at first “goes” downwards and it requires some “energy expenses” by reaching by the meniscus the crystal edge with its smaller height.

Figure 6b shows that absolute value of the meniscus profile curve boundary angle θ_c near the crystal edge decreases with decrease of the absolute value of the boundary angle θ_d – more sharply at the large absolute values. An area of almost constant boundary angles θ_c exists at small absolute values of the boundary angles θ_d . As it is shown in figure 6b, the approximate range – from -20° to -15° - of the angle θ_c changing corresponds to the range – from -20° to $+20^\circ$ - of the angle θ_d changing. But this area of the boundary angle θ_c values does not correspond to the real conditions of the crystal growth process because such angle θ_c values do not satisfy to the condition of the growth angle constancy. In addition, as it is shown in figure 6a, at such angles meniscus heights are unrealistic small.

Data have been used by figure 6b construction, allow still to narrow the range of the real values of the angle θ_d . In reality growth angle for the various materials is located in the range from 0° to 20° (for sapphire the growth angle is approximately 13° - 17° [5,12] which corresponds to the range – from -90° to -70° - of the boundary angle θ_c . As it is shown in figure 6b, next approximate ranges of θ_d changing correspond to this range of the angle θ_c (under the modeling parameters used): from -50° to -45° - in the case of negative angles θ_d and from 45° to 50° - in the case of positive angles θ_d .

Estimating the range of changing of the boundary angle θ_d is necessary for the calculation of the program mass of the growing crystal which includes the sine of this angle [6]. Numerical calculations represented in this paper allow to make such estimation which was described above. In addition, it is necessary to know the sign of the angle θ_d . This angle is negative at the usual shaper (with non - inclined working surfaces) and can be positive – at the inclined working surfaces of the shaper.

2.4 Investigation of the menisci profile curves for various values of the crystal and shaper radii with constant distance between them Mutual changing the crystal and working shaper surface radii was considered in the numerical calculations. The difference between these radii, i.e. distance between the edges of crystal and working surface of the shaper, was constant and equal to 0.03 (in capillary constants). The crystal radius was changing from 0.07 to 19.97 and shaper radius – from 0.1 to 20. It was equivalent for sapphire to the crystal radius changing from 0.42 mm to approximately 120 mm. Condition of the growth angle constancy near the crystal edge was taken into account.

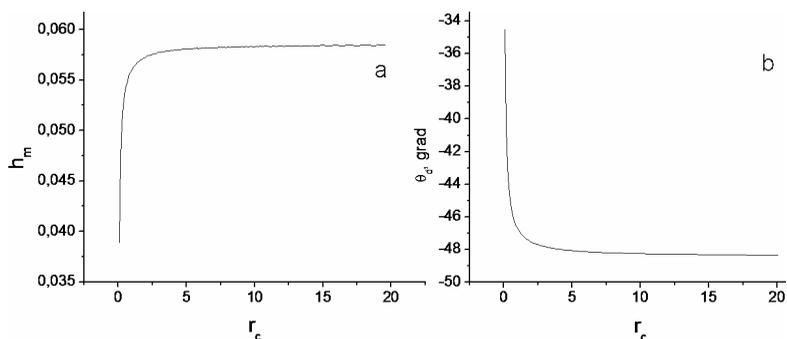


Fig. 7 a – Dependence of the meniscus height on the crystal radius and, correspondingly, on the shaper radius for the external menisci of the tube, b – Dependence of the boundary angle θ_d on the crystal radius and, correspondingly, on the shaper radius for the external menisci of the tube.

As it is shown in figure 7a, the main change of the meniscus height occurs at the crystal and shaper radii changes from approximately zero to the value of the capillary constant. The meniscus height practically does not change for further changes of the crystal and shaper radii. The boundary angle θ_d , as well as meniscus

height, changes significantly at the crystal and shaper radii change from approximately zero to the value of the capillary constant. As it is shown in figure 7b, absolute value of the boundary angle θ_d grows with increase of the crystal and shaper radii. It is connected with meniscus height increase requiring more “sharp” lifting the profile curve near the working surface of the shaper and, hence, larger absolute value of the boundary angle θ_d .

3 Internal circular menisci

Numerical analysis of the internal circular menisci was made for the cases similar to the investigation of external circular menisci. Calculation data being used in this part of the paper were similar to the data being used for the numerical analysis of the external circular menisci. Profile curves calculated for the internal circular menisci, in the whole, are similar to the curves of the external circular menisci shown in figures 2 and 4. Figure 8 demonstrates typical profile curves of the internal menisci. There are some differences connected with fact that azimuthal curvature (second part of the capillary equation (1)) has positive sign in the case of internal meniscus and negative sign in the case of external meniscus.

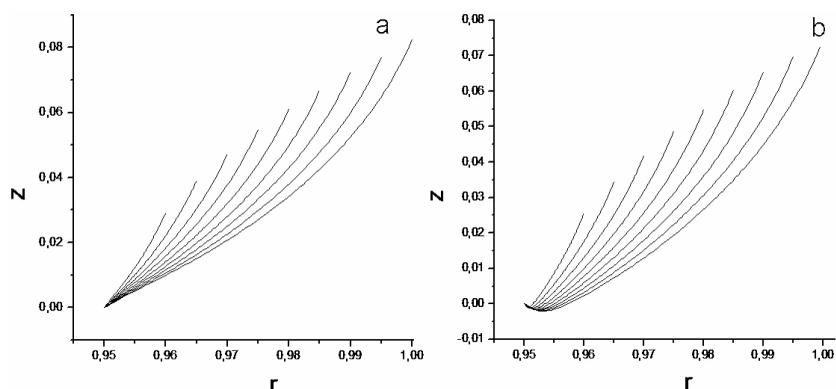


Fig. 8 a, b - Profile curves of the internal menisci, a – for the positive angles θ_d , b – for the negative angles θ_d .

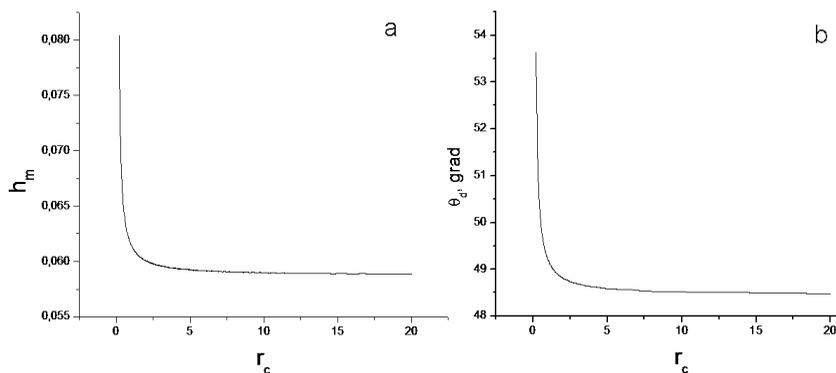


Fig. 9 a – Dependence of the meniscus height on the crystal radius and, correspondingly, on the shaper radius for the internal menisci of the tube, b – Dependence of the boundary angle θ_d on the crystal radius and, correspondingly, on the shaper radius for the internal menisci of the tube.

The value of this curvature increases the current meniscus height due to the positive sign of the curvature in the case of the internal meniscus. In the case of the external meniscus the value of this curvature reduces the current meniscus height. For the equal crystal radii and other modeling parameters profile curves of the internal menisci “go” a little bit higher than profile curves of the external menisci. Physically it looks like additional “compressing” the internal meniscus and “decompressing” the external meniscus.

There was also simultaneous changing the crystal and shaper radii in a way that difference between these radii was constant. External pressure was $H_d = -4$ in capillary constants. Condition of the growth angle constancy near the edge of the crystal was considered.

The dependence of the meniscus height on the crystal and shaper radii (Fig. 9a) differs from the one of the case of the external circular menisci and has opposite behavior. Dependence of the internal menisci height is

slightly decreasing, while external menisci height increases. It is connected with fact of different signs of the azimuthal curvature. For the small internal menisci this curvature is sufficiently large, its positive addition is very essential and physical “compressing” is significant. With increase of the crystal and shaper radii influence of the azimuthal curvature and “compressing” factor decreases and the internal menisci height decreases.

But such behavior of the internal menisci heights does not mean that menisci heights are growing without limitation with crystal and shaper radii decreasing and approach of them to zero. Small internal menisci have convexity upwards. As calculations have shown for small internal menisci angle θ_d increases and aspires to $\pi/2$ with approach of the crystal and shaper radii to zero. Thus, convexity upwards grows, and meniscus can not reach growth angle near the edge of the crystal. Namely, necessity of the reaching the growth angle near the crystal edge with growing convexity of the menisci requires increasing their height. As it was now described above, very small menisci at the constant difference between the crystal and the shaper radii can not reach growth angle, and further increasing the menisci heights does not occur, because there is no solution of the mathematical problem.

Therefore, it is necessary to lessen the difference between crystal and shaper by decrease of their radii to obtain the solution of the problem and to get a “real growth” meniscus. With decrease of this difference the meniscus height decreases and compensates increase of the meniscus height described above. As a result of the action of these two factors – decrease of the crystal and shaper radii and a difference between them – “real” meniscus height decreases and aspires to zero as in the case of the external circular menisci (Fig. 7a). In the case of external menisci azimuthal curvature has negative addition to the meniscus height. With increase of the crystal and shaper radii its influence decreases and the height of external menisci grows (Fig. 7a).

Main changing the boundary angle θ_d (as changing the internal menisci height) is focusing on the crystal and shaper change in the area of one capillary constant (Fig. 9b). The change of the boundary angle θ_d for internal menisci not so significant in comparison with corresponding angles in the case of external menisci, namely (Fig. 7b). By this, absolute value of this angle slightly decreases, while in the case of external menisci it increases. This is connected with necessity of the small decrease of the internal menisci height by the reasons described above. “Planar” menisci forming by wide side of the crystal ribbon were considered. As known [2], in the ideal case of the “planar” meniscus azimuthal curvature of the profile curve is equal to zero, and second part in the Young-Laplace equation (1) can be neglected. In some approximation “planar” meniscus can be considered as circular meniscus (external or internal) with the very large crystal radius – much more than capillary constant [2]. Therefore, calculations results are close to the results of modeling, for example, outer circular menisci with large crystal radii represented above.

Thus, profile curves of the “planar” menisci calculated for the various distances between crystal and shaper edges are similar to the profile curves shown in figures 2 and 4. By this, menisci heights of the ribbon a little bit higher of the corresponding heights of the outer circular menisci and a little bit lower of the heights of the internal circular menisci. As described above, it should be explained by no presence of the influence of the azimuthal curvature in the shape of “planar” menisci. The influence of the change of the external pressure on the shape and features of the “planar” menisci is analogous to its influence on the circular menisci. Meniscus height grows with decrease of the absolute value of the external pressure, and faster with approach of the external pressure to zero.

4 Use of calculations of the menisci profile curves in the automated process of the growth of the sapphire crystal tubes

Automated control system for the process of the crystal growth with use of the weight sensor requires the use of the program observation expression. The data of this expression at each period of time are being compared with real signal of the weight sensor. Deviation of the real signal from its program one forms then a control signal of the regulator.

The part of the program observation expression relating to the meniscus mass weighing by the weight sensor in the case of the stationary crystal tube pulling and in the case of planar interface boundary looks like

follows [6, 22]:

$$M_m = \pi\rho_L(r_{c,e}^2 - r_{c,i}^2)(h_{m,e} + h_{m,i})/2 - 2\pi\rho_L a^2 r_{c,e} \sin\theta_{c,e} + 2\pi\rho_L a^2 r_{c,i} \sin\theta_{c,i} + 2\pi\rho_L a^2 R_{d,e} \sin\theta_{d,e} - 2\pi\rho_L a^2 R_{d,i} \sin\theta_{d,i} - 2\pi\rho_L (R_{d,e}^2 - R_{d,i}^2) H_d, \quad (4)$$

here $r_{c,e}$ is a radius of the tube external surface, $r_{c,i}$ is a radius of the tube internal surface, $R_{d,e}$ is a radius of the die internal surface, $R_{d,i}$ is a radius of the die internal surface, $h_{m,e}$ is a height of the external meniscus, $h_{m,i}$ is a height of the internal meniscus, $\theta_{c,e}$ is an angle of the contact of the external meniscus with the crystal edge, $\theta_{c,i}$ is an angle of the contact of the internal meniscus with the crystal edge, $\theta_{d,e}$ is an angle of the contact of the external meniscus with the die edge, $\theta_{d,i}$ is an angle of the contact of the internal meniscus with die edge, a is a capillary constant, ρ_L is a melt density, H_d is an external static pressure.

The angle θ_c is related to the growth angle which is approximately known for the crystal material. The external static pressure can be estimated. The boundary angles θ_d and program meniscus heights h_m for the external and internal menisci should be calculated from a numerical solution of the Young-Laplace equation.

Results presented in the Section 2.3 allows to make estimations of the program values of the angle θ_d and meniscus height h_m using figure 6. For the known boundary angle θ_c (depending on the growth angle) the boundary angle θ_d should be found from figure 6b. Then using this value of the angle θ_d meniscus height should be found from figure 6a. This calculations should be fulfilled for both external and internal menisci.

To obtain the sapphire high quality tubes it is necessary to maintain the optimal thermal conditions on the crystal-melt interface, and hence to maintain optimal heights of external and internal menisci. Special thermal shields and die top design [23] are used for achievement of this purpose. Large-scale sapphire tubes of 55 mm in the external diameter (Fig. 10) have been grown with use of the automated control system with preset values of the meniscus heights and boundary angles as program values in the observation expression of the weight sensor.



Fig. 10 Sapphire tubes of 55 mm in external diameter grown with automated EFG (Stepanov) technique. (Online color at www.crt-journal.org)

5 Conclusions

Comparable analysis of the shape and behavior of the menisci profile curves for various signs of the boundary angle θ_d typical for the cases of ordinary (planar) and inclined working surfaces of the shapers was made on the basis of the numerical solution of the Young-Laplace capillary equation. Analysis of the second derivatives of the menisci profile curves for these cases showed that if the sign of the angle θ_d is such that profile curve at once goes upwards from the shaper, so meniscus has a region with convexity upwards, i. e. “from the melt”. Then, with approach of the profile curve to the crystal edge, passing the excess point, meniscus becomes concave to the melt side. If the angle θ_d at first directs the profile curve downwards (to the melt), the convexity of the profile curve has one sign for the whole curve – always to the melt side.

Such a difference should be explained by the different boundary conditions of contact of the meniscus and the shaper at its edge. Different heights of the external and internal menisci at the same other conditions of the modeling were calculated. For the external and internal menisci azimuthal curvature has different signs and, correspondingly, influences differently on the meniscus height in these different cases. The form of the Young-Laplace equation gives the result that the meniscus height is proportional to the sum of the main curvatures of the meniscus in the considerable point. The opposite behaviors of the heights of external and internal menisci depending on the simultaneous changing the crystal and shaper radii with a constant difference between them was found. Mainly, it can be also explained by different signs of the azimuthal curvatures of the external and internal menisci.

Similarity of the behaviors of the heights of the menisci with different signs of the angles θ_d depending on the external pressure was found. These dependences are non-linear, as was described in [5]. Increase of the meniscus height with decrease of the absolute value of the external pressure (negative in our case) should be explained by decrease of the force pressing the meniscus to the shaper surface. The dependences of the menisci heights and contact angles θ_c of the profile curves at the crystal edge on the boundary angle θ_d without requirement of the fulfilling the condition of the growth angle constancy were found. There was found sufficiently large “sensitivity” of the meniscus height and contact angle θ_c to the change of the large absolute values of the boundary angle θ_d and weak “sensitivity” of these parameters to the change of the boundary angle θ_d near zero.

For the modeling parameters considered for the growth of the cylindrical crystal tube the range of the boundary angles θ_d corresponding to the real conditions of the crystal shaping was found. According to this fact an absolute value of the real boundary angle θ_d can be changed from 30° to 50°. The knowledge of the value of this angle is important and necessary as input parameter in the automated system of control of the crystal shape and quality – for the defining the program mass weighing by the weight sensor.

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