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On the creation of a stable and convex static meniscus, appropriate for the growth of a single crystal ribbon, in strictly zero gravity by E.F.G. technique, with specified half thickness

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In this paper it is shown in which kind a stable and convex static meniscus, appropriate for the growth of a single crystal ribbon with specified half thickness, can be created in strictly zero gravity by choosing the pressure of the gas flow introduced into the furnace (for release the heat). The method is based on explicit formulas established for materials for which the contact angle α_c and growth angle α_g satisfy: $0<\alpha_c<\pi/2$; $0<\alpha_g<\pi/2$; $\alpha_c<\pi/2-\alpha_g$. The dependence of the obtained static meniscus shape and size on the shaper half thickness is also discussed. The procedure is numerically illustrated and the results are compared with those obtained on the ground.

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1. Introduction

The conventional melt growth techniques, as Bridgman growth [1-3] or Czochralski pulling [4-16] of single crystals typically produce ingots of circular or square cross-section which need to be cut into hundreds of slices to produce wafers for radiation detection, infrared optics, substrate for opto-electronics and micro-electronic applications. Using these processes, it is difficult to produce thin wafers from an ingot without wasting 40-50% of material as kerfs during the cutting process. For this reason the E.F.G. technology can be more appropriate to produce single crystals with prescribed shapes and sizes, which can be used without additional machining. Successful Si ribbon growth was reported in [17-43] and Ge ribbon growth was reported in [44].

A requirement of a successful constant half – thickness ribbon growth is that at the point where the solidification takes place the angle between the tangent line to the meniscus free surface and the vertical is equal to the growth angle α_g, i.e. the tangent line to the ribbon wall is vertical (Fig. 1). When this requirement is satisfied, then we say that the meniscus is appropriate for the growth of a ribbon with constant half-thickness. When the half-thickness x_1 of the ribbon, which has to be grown, is a priori given, then the x coordinate of that point (where the solidification takes place) has to be equal to x_1 and the following condition is satisfied: $z'(x_1) = -\tan(\pi/2 - \alpha_g)$.

During the growth on Earth, the temperature gradients in the melt generate buoyancy driving forces and thermal convection in the melt [45 – 47], which result in a “nearly completely” mixed melt [47]. Crystals grown from well mixed melts exhibit a nonlinear variation of the dopant concentration along the growth axis [48]. On the other hand, crystals grown from a quiescent melt, after an initial transient, exhibit a uniform axial dopant distribution [49]. Thus reduction of the magnitude of the buoyancy forces by processing semiconductors in a low gravity environment has been pursued over the past decades. The effectiveness of space processing for the growth of chemically uniform crystals is supported by experimental and theoretical studies. For example, the InSb crystal reported in [50] exhibits axial segregation profiles that are characteristic of diffusion – controlled mass transfer growth.

In [45-46] it is shown, based on modeling studies, that the low – gravity levels reached in space are sufficient to inhibit interference of thermal convection with segregation in small – diameter Ge and GeSi melts.

The above analysis has been performed using constant values of the gravitational acceleration. In [51, 52] there is a study of the influence of non steady gravity on the thermal convection during microgravity solidification of semiconductors. The study corresponds to the use of a low – duration low – gravity vehicle, such as sounding rockets and KC-135 aircrafts, for low gravity crystal growth experiments. In these vehicles, low – gravity periods of 20 seconds and 6 minutes, for KC-135 aircraft and sounding rockets, respectively, are achieved at cost that are at orders of magnitude smaller than space experiments.

The objective of this paper is to give a procedure for the determination of the pressure p_g of the gas flow, introduced in the furnace for release the heat, in order to obtain a meniscus, which satisfies the requirement $z'(x_1) = -\tan\left(\pi/2 - \alpha_g\right)$ in strictly zero gravity, when x_1 is a priori given. Moreover, to verify that for the obtained meniscus the energy of the melt column is minimum (i.e. the meniscus is static stable). The thermal problem concerning the setting of the thermal conditions, which assure that for the obtained meniscus the solidification conditions hold at the point $\left(x_1, z(x_1)\right)$, is not the subject of this paper. In the paper the dependence of the gas flow pressure p_g (which has to be used) on the shaper half thickness size is also analyzed.

2. The meniscus free surface equation

For single crystal ribbon growth by the edge-defined film-fed growth (E.F.G.) method in strictly zero gravity, in hydrostatic approximation the free surface of the static meniscus is described by the Young-Laplace capillary equation [53, 54]:

$$-\gamma \cdot \left(\frac{1}{R_1} + \frac{1}{R_2}\right) = p \qquad (1)$$

Here: γ is the melt surface tension; ρ denotes the melt density; $1/R_1$, $1/R_2$ denote the main normal curvatures of the free surface at a point M of the free surface and p is the pressure difference across the free surface:

$$p = p_m - p_g . \qquad (2)$$

Here: p_m denotes the pressure in the meniscus melt (under the free surface) and p_g denotes the pressure of the gas flow introduced in the furnace (above the free surface).

To calculate the meniscus free surface shape and size it is convenient to employ the Young -Laplace eq.(1) in its differential form:

$$\left[1 + \left(z_y\right)^2\right] \cdot z_{xx} - 2 \cdot z_x \cdot z_y \cdot z_{xy} + \left[1 + \left(z_y\right)^2\right] \cdot z_{yy} = = -\frac{p}{\gamma}\left[1 + \left(z_x\right)^2 + \left(z_y\right)^2\right]^{3/2} \qquad (3)$$

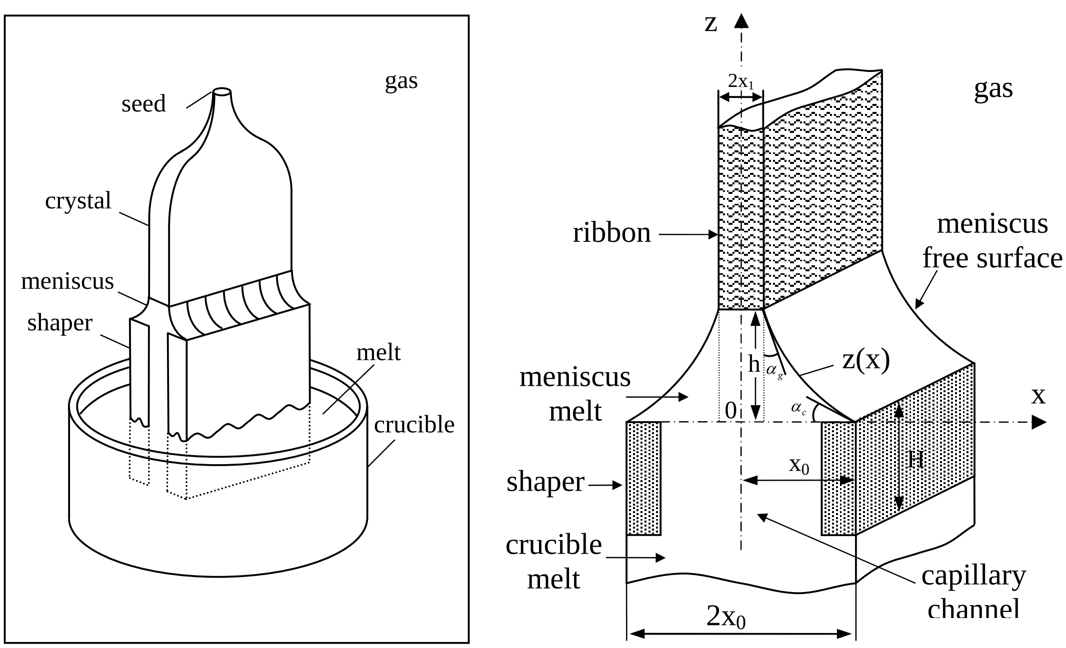

Fig. 1. Convex meniscus geometry in the ribbon growth by E.F.G. method. (3D representation).

This form of the eq.(1) can be obtained as a necessary condition for the minimum of the free energy of the melt column [53, 54].

For the growth of a single crystal ribbon of half thickness x_1, $0 < x_1 < x_0$ = the shaper half thickness; the differential equation for plan symmetric meniscus surface is given by:

$$z'' = -\frac{p}{\gamma}\left[1 + \left(z'\right)^2\right]^{3/2} \quad \text{for } 0 < x_1 \le x \le x_0 \qquad (4)$$

which is the Euler equation for the energy functional of the melt column [55]:

$$I(z) = \int_{x_1}^{x_0} \left\{\gamma \cdot \left[1 + \left(z'\right)^2\right]^{1/2} - p \cdot z\right\} dx \qquad (5)$$

$$z(x_1) = h > 0; \quad z(x_0) = 0;$$

When the meniscus is appropriate for the growth of a ribbon with constant half-thickness x_1, then the solution $z(x)$ of the eq.(4) satisfies the following conditions:

a. $z'(x_1) = -\tan\left(\pi/2 - \alpha_g\right)$

b. $z'(x_0) = -\tan\alpha_c$

c. $z(x_0) = 0$ and $z(x)$ is strictly decreasing on $[x_1, x_0]$,

Here: $x_0 > 0$ is the shaper half-thickness; α_g is the growth angle; α_c is the contact angle between the meniscus free surface and the edge of the shaper top and $0 < \alpha_c < \pi/2$; $0 < \alpha_g < \pi/2$; $\alpha_c < \pi/2 - \alpha_g$ (Fig.1).

Condition a. expresses that at the point $(x_1, z(x_1))$ which is the left end of the free surface, where the thermal conditions for the solidification have to be assured, the tangent to the crystal wall is vertical.

Condition b. expresses that at the point $(x_0, z(x_0 = 0))$, which is the right end of the free surface, the contact angle is equal to α_c.

Condition c. expresses that the right end of the free surface is attached to the outer edge of the shaper.

Usually eq.(4) is transformed into the system:

$$\begin{cases} \dfrac{dz}{dx} = -\tan\alpha \\ \dfrac{d\alpha}{dx} = \dfrac{p}{\gamma} \cdot \dfrac{1}{\cos\alpha} \end{cases} \tag{7}$$

for which conditions a - c become:

$$z(x_0) = 0; \ \alpha(x_0) = \alpha_c; \quad \alpha(x_1) = \pi/2 - \alpha_g; \ z(x) \text{ is strictly decreasing on } [x_1, x_0]. \tag{8}$$

3. The dependence of the meniscus shape and size on the pressure difference across the free surface

In [55] the following results were established:

Statement 1. [55] If $n > 1$ and for $x_1 = x_0/n$ there exists a solution $z(x), \alpha(x)$ of the eqs.(7) which satisfies (8) and $z''(x) > 0$ on $\left[x_0/n, x_0\right]$, then for the pressure difference p across the free surface the following inequality holds:

$$-\frac{n}{n-1} \cdot \gamma \cdot \frac{\pi/2 - (\alpha_c + \alpha_g)}{x_0} \cdot \cos\alpha_c \le p \le -\frac{n}{n-1} \cdot \gamma \cdot \frac{\pi/2 - (\alpha_c + \alpha_g)}{x_0} \cdot \sin\alpha_g \tag{9}$$

Comment: Statement 1 shows that in order to create an appropriate static meniscus with a convex free surface on $\left[x_0/n, x_0\right]$, the pressure difference p across the free surface has to be chosen in the range given by the inequality (9). Formula (9) can be useful for a rough evaluation of p when the ribbon half-thickness x_1 (which has to be grown) is a priori given by $x_1 = x_0/n$.

Statement 2. [55] If for $x_1 = 0$ eqs.(7) has a solution ($z(x), \alpha(x)$), which satisfies (8) and $z''(x) > 0$, then the pressure difference p satisfies the following inequalities:

$$-\gamma \cdot \frac{\pi/2 - (\alpha_c + \alpha_g)}{x_0} \cdot \cos\alpha_c \le p \le -\gamma \cdot \frac{\pi/2 - (\alpha_c + \alpha_g)}{x_0} \cdot \sin\alpha_g \tag{10}$$

Comment: Formula (10) can be used for a rough evaluation of the pressure difference p when the ribbon half-thickness has to be close to 0. It follows, for example, that if for p the following inequality holds:

$$p > -\gamma \cdot \frac{\pi/2 - (\alpha_c + \alpha_g)}{x_0} \cdot \sin\alpha_g. \tag{11}$$

then doesn't exist appropriate static meniscus having convex free surface.

Statement 3. [55] If the pressure difference p across the free surface satisfies:

$$p < -\frac{n}{n-1} \cdot \gamma \cdot \frac{\pi/2 - (\alpha_c + \alpha_g)}{x_0} \cdot \cos\alpha_c; \qquad n > 1 \tag{12}$$

then there exists x_1 in the range $\left[x_0/n, x_0\right]$ and a solution $z(x), \alpha(x)$ of eqs.(7) which satisfies (8) on $[x_1, x_0]$ and $z''(x) > 0$.

Comment: In other words, if the pressure difference p is chosen such that inequality (12) holds, then for a certain x_1 in the range $\left[{x_0}/{n} , x_0 \right]$ an appropriate static meniscus, having a convex free surface is obtained.

Statement 4. [55] If $p < 0$, then a solution $z(x), \alpha(x)$ of eqs.(7) which satisfies $z(x_0) = 0$ and $\alpha(x_0) = \alpha_c$ verifies $z''(x) > 0$ and vice versa.

Comment: In other words, potentially appropriate convex static menisci can be obtained only for $p < 0$. If $p > 0$, then the solution $z(x), \alpha(x)$ of eqs.(7) which satisfies $z(x_0) = 0$ and $\alpha(x_0) = \alpha_c$ verifies $z''(x) > 0$ and the meniscus is not appropriate for the growth of a ribbon with constant half - thickness.

Statement 5. [55] If for $1 < n' < n$ and p the following inequalities hold:

$$-\frac{n'}{n'-1} \cdot \gamma \cdot \frac{{\pi}/{2} - (\alpha_c + \alpha_g)}{x_0} \cdot \sin\alpha_g \le p \le \le \frac{n}{n-1} \cdot \gamma \cdot \frac{{\pi}/{2} - (\alpha_c + \alpha_g)}{x_0} \cdot \cos\alpha_c , \quad (13)$$

then there exists $x_1 \in \left[{x_0}/{n} , {x_0}/{n'} \right]$ and a solution $z(x), \alpha(x)$ of eqs.(7) which satisfies (8) on $[x_1, x_0]$ and $z''(x) > 0$.

Comment: Inequalities (13) are useful for locate the pressure difference when the half thickness x_1 of the ribbon which has to be grown is in the range $\left[{x_0}/{n} , {x_0}/{n'} \right]$.

Statement 6. [55] If a solution $z(x), \alpha(x)$ of the eqs.(7) which satisfies (8) is convex $(z''(x) > 0)$, then $z(x)$ is a minimum for the energy functional of the melt column, i.e. the static meniscus is stable.

4. Procedure for the determination of the pressure of the gas flow

Assume that in strictly gravity the ribbon half-thickness which has to be grown (from a specified material by E.F.G. technique) is equal to x_1 and for this purpose we have chosen a shaper of half-thickness x_0. In order to find the pressure difference p across the free surface which has to be used for the creation of an appropriate meniscus, the following limits, presented in the previous section, are considered:

$$\underline{L}(n) - \frac{n}{n-1} \cdot \gamma \cdot \frac{{\pi}/{2} - (\alpha_c + \alpha_g)}{x_0} \cdot \cos\alpha_c$$

$$\underline{L}(+\infty) = -\gamma \cdot \frac{{\pi}/{2} - (\alpha_c + \alpha_g)}{x_0} \cdot \cos\alpha_c$$

$$\overline{L}(n) - \frac{n}{n-1} \cdot \gamma \cdot \frac{{\pi}/{2} - (\alpha_c + \alpha_g)}{x_0} \cdot \sin\alpha_g$$

$$\overline{L}(+\infty) - \gamma \cdot \frac{{\pi}/{2} - (\alpha_c + \alpha_g)}{x_0} \cdot \sin\alpha_g$$

Now consider $n_1 = {x_0}/{x_1}$ and $\underline{L}(n_1), \overline{L}(n_1)$.

- For p in the range $(-\infty, \underline{L}(n_1)]$ the obtained convex meniscus is not appropriate because its half-thickness is more than the desired half-thickness $x_1 = {x_0}/{n_1}$, according to the Statement 3.
- For p in the range $(\overline{L}(n_1), \overline{L}(+\infty))$ the obtained convex meniscus is not appropriate because its half-thickness is less than the desired half-thickness $x_1 = {x_0}/{n_1}$, according to the Statement 3.
- For p in the range $(\overline{L}(+\infty), 0)$ the obtained convex meniscus is not appropriate because the growth angle is not reached on it, (i.e. the obtained half-thickness is negative) according to the Statement 2.
- For p in the range $(0, +\infty)$ the obtained meniscus is concave and the growth angle can not be reached on it, according to the Statement 4.
- For $p = \underline{L}(n_1)$ solving equation $\overline{L}(n) = \underline{L}(n_1)$ we find $n_1' < n_1$ and the fact that for this particular value of p the obtained meniscus half-thickness is in the range $\left[{x_0}/{n_1} , {x_0}/{n_1'} \right]$, according to the Statement 5. This meniscus is not appropriate since its half-thickness is more than the desired one.
- For an appropriate convex meniscus, according to the Statement 1, the pressure difference $p = p_m - p_g$ has to be searched in the range $[\underline{L}(n_1), \overline{L}(n_1))]$. A particular value of the pressure difference $p = p_m - p_g$ in this range is $\underline{L}(+\infty)$. If for $p = \underline{L}(+\infty)$ the computed ribbon half-thickness (obtained by numerical integration of the eqs.(7) for $z(x_0) = 0$ and $z'(x_0) = \alpha_c$) is less than the desired half-thickness x_1, then p has to be in the range $[\underline{L}(n_1), \underline{L}(+\infty)]$. If the computed half-thickness is more

than x_1, then p has to be in the range $\left[\underline{L}(+\infty), \overline{L}(n_1)\right]$. The exact value of $p = p_m - p_g$ in the range $\left[\underline{L}(+\infty), \overline{L}(n_1)\right]$ or in the range $\left[\underline{L}(n_1), \underline{L}(+\infty)\right]$ respectively, is found by numerical integration of the eqs.(7) for $z(x_0) = 0$ and $z'(x_0) = \alpha_c$) for different values of p in the considered ranges.

For the numerical data:

$$x_0 = 4 \cdot 10^{-4}[m]; \alpha_c = 0.814\,[rad]; \alpha_g = 0.209\,[rad]; \rho = 5.538 \cdot 10^3\,[kg/m^3]; \gamma = 622 \cdot 10^{-3}\,[N/m]$$

using the soft MATHCAD Professional 13, the procedure was applied in two cases:

$x_1 = 2 \cdot 10^{-4}[m]$ $(n_1 = 2)$ and

$x_1 = 10^{-4}[m]$ $(n_1 = 4)$, respectively.

The possible pressure ranges, which were investigated, can be seen in Fig.2. on which the limits as functions of $n > 1$ are represented.

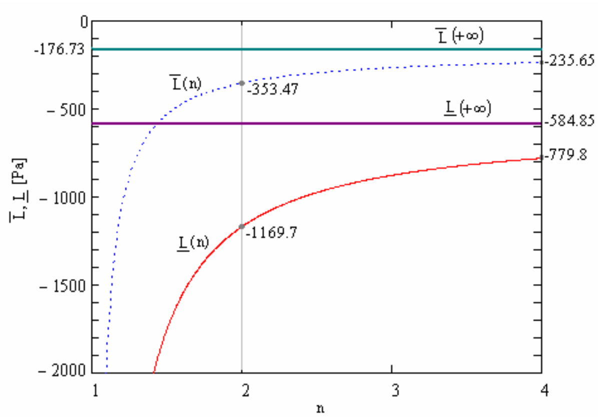

Fig. 2. The possible p ranges which have to be investigated for

$$x_0 = 4 \cdot 10^{-4}[m] \text{ and } n = 2; 4.$$

For $n_1 = 2$ $(x_1 = 2 \cdot 10^{-4}[m])$ the range $\left[\underline{L}(n_1), \overline{L}(n_1)\right)]$ is equal to $[-1169.7; -353.47][Pa]$.

The pressure $\underline{L}(+\infty)$ is equal to $-584.85[Pa]$ $\underline{L}(+\infty) = -584.85[Pa]$

The pressure $\overline{L}(+\infty)$ is equal to $\overline{L}(+\infty) = -176.73[Pa]$.

The enumerated ranges of p for which the obtained meniscus is not appropriate are:

$\left(-\infty, \underline{L}(n_1)\right] = \left(-\infty, -584.85\right]$;

$\left(\overline{L}(n_1), \overline{L}(+\infty)\right) = \left(-353.47, -176.73\right)$;

$\left(\overline{L}(+\infty), 0\right) = \left(-176.73, 0\right)$; $(0, +\infty)$.

The range of p for which the obtained meniscus can be appropriate is: $\left[\underline{L}(n_1), \overline{L}(n_1)\right] = \left[-1169.7, -353.47\right][Pa]$.

The computed ribbon half thickness for $p = \underline{L}(+\infty) = -584.85\,[Pa]$ is equal to $1.32 \cdot 10^{-4}[m]$. Since this half thickness is less than the desired half thickness $x_1 = 2 \cdot 10^{-4}[m]$, the right p_1 value has to be searched in the range $\left[\underline{L}(n_1), \underline{L}(+\infty)\right)] = [-1169; -584.85][Pa]$.

Integrating, for different p values from this range, eqs.(7) for $z(x_0) = 0; z'(x_0) = \alpha_c = 0.814[rad]$ it is found that for $p_1 = -784[Pa]$ the computed half thickness is equal to the desired half thickness $x_1 = 2 \cdot 10^{-4}[m]$.

For $n_1 = 4$ $(x_1 = 1 \cdot 10^{-4}[m])$ the range $\left[\underline{L}(n_1), \overline{L}(n_1)\right]$ is equal to $[-779.81; -235.65][Pa]$. The pressure $p = \underline{L}(+\infty)$ is the same as above, $-584.85[Pa]$, and the ranges $\left[\underline{L}(n_1), \underline{L}(+\infty)\right)]$ and $\left[\underline{L}(+\infty), \overline{L}(n_1)\right]$ are $[-779.81; -584.85][Pa]$ and $[-584.85; -235.65][Pa]$, respectively. Since for $p = \underline{L}(+\infty) = -584.85\,[Pa]$ the computed ribbon half thickness is equal to $1.32 \cdot 10^{-4}[m]$, which is greater than the desired half thickness $1 \cdot 10^{-4}[m]$, the right p_1 value has to be searched in the range $\left[\underline{L}(+\infty), \overline{L}(n_1)\right] = [-584.85; -235.65][Pa]$. For different p values in this range, integrating the eqs.(7) for $z(x_0) = 0; z'(x_0) = \alpha_c = 0.814[rad]$ it is found that for $p_1 = -522[Pa]$ the computed half thickness is equal to the desired half thickness $x_1 = 1 \cdot 10^{-4}[m]$.

Since the p_1 value obtained for x_1 verifies: $p_1 = p_m - p_g^1$, neglecting p_m (the thermodynamic pressure due to the thermo-convection in strictly zero gravity is neglectible), it follows that $p_1 = -p_g^1$. For the considered numerical values the results, including the meniscus heights h_1, are summarized in Table 1.

Table 1.

x_0	n_1	$x_1 = x_0 / n_1$	h_1	p_1	p_g
[m]		[m]	[mm]	[Pa]	[Pa]
4×10^{-4}	2	2×10^{-4}	0.38	-784	784
4×10^{-4}	4	1×10^{-4}	0.57	-522	522

4. The dependence on the shaper size

In order to analyze the dependence on the shaper size, the procedure described in the sequence 3 was applied for $x_0 = 8 \cdot 10^{-4} [m]$ in order to create appropriate menisci for the same x_1 values: $x_1 = 2 \cdot 10^{-4} [m] \Leftrightarrow n_1 = 4$ and $x_1 = 10^{-4} [m] \Leftrightarrow n_1 = 8$. The possible pressure ranges, which were investigated, are represented in Fig. 3.

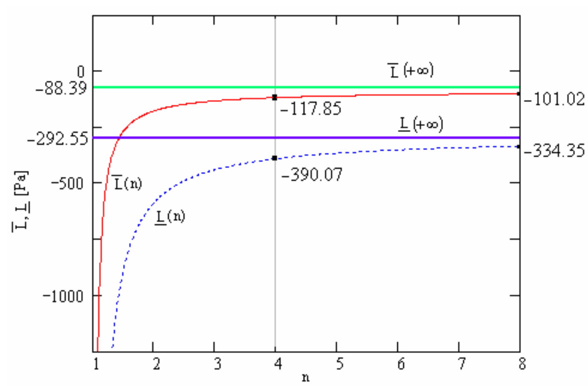

Fig. 3. The possible p ranges which have to be investigated for $x_0 = 8 \cdot 10^{-4} [m]$ and $n = 4; 8$.

The numerical results, including the meniscus heights h_1, are given in Table 2.

Table 2.

x_0	n_1	$x_1 = x_0 / n_1$	h_1	p_1	p_g
[m]		[m]	[mm]	[Pa]	[Pa]
8×10^{-4}	4	2×10^{-4}	1.13	-260.5	260.5
8×10^{-4}	8	1×10^{-4}	1.33	-223.5	223.5

5. Conclusions

The Young-Laplace equation permits to determine the pressure of the gas flow introduced into the furnace (for release the heat) in order to create a convex static meniscus, appropriate for the growth of a single crystal ribbon with specified half thickness in strictly zero gravity.

The numerical analysis reveals that the pressure and the meniscus size are highly dependent on the shaper half thickness. The obtained pressure differences, which have to be used in strictly zero gravity, are similar to those which have to be used on the ground [56]. The main difference is that in strictly zero gravity these differences have to be realized only by the choice of the pressure of the gas flow.

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