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Simulation of Single Crystalline CdZnTe Solidification Process

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Abstract Single crystals of $Cd_{1-x}Zn_xTe$ ($0 \leq x \leq 0.1$) (CZT/CdZnTe) are used in manufacture of gamma and X-ray detectors and as substrates for epitaxial growth of HgCdTe. Computer simulation for the solidification of CZT was performed using finite elements. The simulation results indicate that a lower translation speed of the quartz ampoule within the Bridgman furnace determines a lower concavity of the liquid interface which assures a good crystalline quality. When the rate is 3.32 mm/h the concavity is 58% greater than for a speed of 0.50 mm/h. It was experimentally found that when growing at low speed, 1.66 mm/h, the process is more stable and improves the crystalline quality due that only two grains were generated in CZT ingots. Meanwhile a faster growth speed- 3.32 mm/h- generates a large amount of grains in the CZT ingot.

Keywords: *CdZnTe, Bridgman method, numerical simulation, finite element method, single crystal growth, II-VI Semiconductors*

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1. Introduction

The CdZnTe is a semiconductor employed in the manufacture of x and gamma (γ) ray detectors that may be used at room temperature. It is also used as a substrate for epitaxial growth of sensitive films for IR radiation. This semiconductor can be grown by the Bridgman method that consists of the fractional solidification of the alloy previously melted while passing through a temperature gradient, in our case of 10°C/cm [1,2,3] into a tubular and vertical furnace. This technique requires an appropriate temperature profile and a strict control of the growth conditions [4,5,6]. The elements (Cd, Zn and Te) are placed in a quartz ampoule in stoichiometrical relationship which is then sealed in vacuum. The ampoule is vertically moved along the furnace axis at a very slow rate and the semiconductor solidification is slowly performed and carefully controlled [4,6].

Semiconductors used to build optoelectronic devices should be single crystals because crystalline discontinuities as boundary grains, inclusions and dislocations damage device sensitivity. In consequence, the number of grains has a strong influence in the electronic properties and the final quality of the CZT crystalline ingot [4]. Several parameters might influence the number of grains present in the final CZT obtained ingot, as the growth rate and the doping composition [4]. The form of the solid-liquid interface (SLI) has also a strong influence in the final number of grains, for that reason it is an important characteristic to study during the solidification process. It is already known that at low translation speed of the ampoule the form of the interface will be more favourable in the reduction of the number of grains because it will be more horizontal [5]. Besides, it is important to control the form of the SLI because it also influence in the homogeneity, composition and number of crystalline defects presents in the material.

Theoretically, the ideal interface should be convex with regards to the liquid phase, in that form only one grain will be grown because spurious grains will be pressed against the wall of the ampoule melting again. However experimental observation and theoretical calculation [5] indicate that this system usually presents a concave interface to the liquid promoting the growth of several grains.

Computer modelling and simulation are powerful techniques used for analysis and design of industrial products as well as in the field of materials science. Simulation could be used to get the best growth conditions for CZT semiconductor to obtain good crystalline quality to be used as electronic sensor of radiation. The main goal in this work is to analyse the effect of the translation velocity in the form of the interface during CZT growth. The modelling during the semiconductor solidification into the furnace could be used to optimize the general process and to plan further modifications of the process itself.

2. Mathematical Model

2.1. System Domain and Geometry

In the mathematical model used the domain includes the Bridgman vertical furnace and a double-wall quartz capsule. In this case the inner wall is in contact with the CZT semiconductor and the outer wall is a protection in case of failure of the inner one. Both capsules are cylindrical with curved ends. Both of them are sealed in high vacuum (Figure 1a). The cylindrical symmetry was used to simplify the mathematical domain, then a tridimensional model with axial-symmetry was used (Figure 2b). In that form the number of used nodes could be reduced, which in consequence might also diminished the calculation time and processing memory. The finite element method was used to solve the heat transfer equations [7].

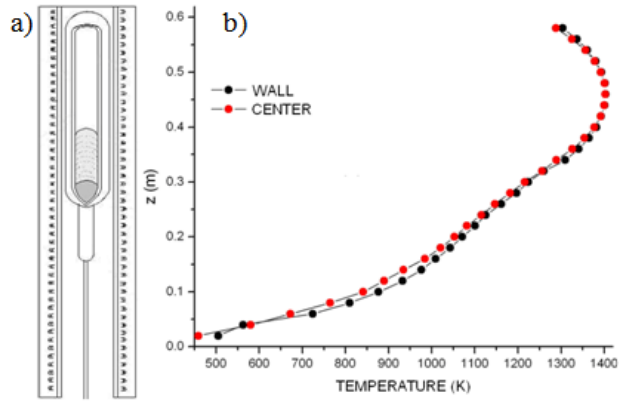

Figure 1. (a) Scheme of the double-wall quartz ampoule with CZT in the conical part of the quartz ampoule, placed into the Bridgman furnace, (b) Temperature profile on the ampoule inner wall and the center of the empty Bridgman furnace

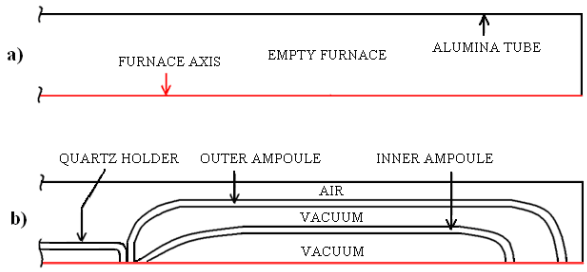

Figure 2. Different disposition of the Bridgman furnace (a) empty furnace, (b) furnace with double-wall quartz capsule. The orientation of the draw is rotated 90º to the right with respect to the actual position

2.2. Calculation Strategy

The system integral calculation is very complex (Figure 2b), because it involves the heat transmission from the furnace wall to the outer wall of the capsule (by convective, radiative and conductive processes), through the capsule wall (by conductive process), from the outer wall to the inner wall (by radiative and conductive processes), through the inner capsule wall (by conductive process) and finally the transmission to the material (by radiative, conductive and convective processes). Otherwise, it has also been considered the heat of the material solidification. For that reason the whole process was split in three simpler ones.

In the 1st level, it is only modelled the empty Bridgman furnace (Figure 2a), the purpose of this level is to perform a general validation of the model with available experimental results. Convection, radiation and conduction are modelled in this level.

In the 2nd level it is modelled the Bridgman furnace with the double-wall quartz capsule without the material (Figure 2b). The heat transfer between furnace and capsule is by conduction, convection and radiation.

In the 3rd level it is meticulously modelled the quartz capsule with the material into it. The material is assumed partially solidified, i.e., into the capsule there are three phases: solid, liquid and vacuum (Figure 3), the solid and the liquid phases are using the same mesh; these phases are differentiated by the value of the physical properties values as a function of the temperature, all the nodes of the mesh with a temperature lower than the melting point have the properties of the solid phase and the nodes with a temperate larger than the melting point have the properties of the liquid phase. The temperature of the 2nd level are the boundary conditions of the 3rd level. For that reason, the calculation is focused in the solid-liquid interface. The heat transfer into the capsule is by conduction and radiation. The capsule is static and the considered boundary temperature function simulates the movement of the capsule into the furnace.

There is a different mesh for each level. In the 1st level the mesh is structured and composed by isoparametric quadrilateral elements with 9 nodes, it was used 18636 elements and 65453 nodes; the nodes in the mesh are separated 0.99 mm. In the 2nd level the mesh is non-structured with isoparametric quadrilateral elements with 9 nodes, it was used 31230 elements and 99799 nodes, the nodes in the mesh are separated 0.80 mm. In the 3rd level the mesh is non-structured with isoparametric quadrilateral elements with 9 nodes, it was used 3200 elements and 13700 nodes (Figure 3). In that level the nodes are respectively separated 0.70 mm and 0.15 mm, in the liquid near the axis and in the liquid near the capsule wall.

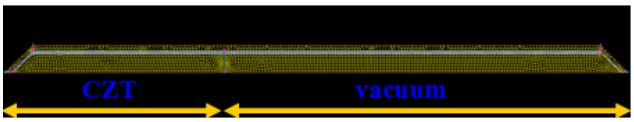

Figure 3. Mesh used in the inner ampoule containing CZT at the left and vacuum at the right (3rd level)

2.3. Material Properties and Boundary Conditions (domain properties and model characteristics)

- Laminar flow,
- Constant density fluid,
- Newtonian fluid,
- The change of phase heat is included in the specific heat by the enthalpy–temperature function [8,9].
- Constant viscosity fluid non temperature dependant.
- The solid phase is modeled as a very high viscosity fluid, 10^6 larger than the liquid phase viscosity.
- As the experimental velocity is very low, in the order of 1 mm/h, the simulation was performed as a quasi-steady state [6,8,10,11].

2.4. Boundary Conditions

- The Bridgman furnace top is isolated.
- The furnace bottom is opened to the atmosphere at 293 K.
- The heat flow is zero through the symmetry axis.
- The solid-liquid interface has a surface tension [12,13].

The furnace wall temperature (Figure 1b) is used as boundary condition in the model, this experimental data is a polynomial function of the furnace height (z), which has the following form (1):

$$T = 296.485 + 10004.8 * Z - 59468.3 * Z^2 + 198817 * Z^3 - 308627 * Z^4 + 172850 * Z^5 \quad (1)$$

T is the furnace wall temperature in Kelvin, and z is the furnace height in meters.

As the translation speed of the ampoule is considered into the boundary condition, this equation should be given as a function of the time with the following form (2):

$$T = 296.485 + 10004.8 * (x - v * t) - 59468.3 * (x - v * t)^2 + 198817 * (x - v * t)^3 - 308627 * (x - v * t)^4 + 172850 * (x - v * t)^5 \quad (2)$$

x is the furnace height in meters, v is the translation speed in meters per second, t is the process time in seconds and T is the furnace wall temperature in Kelvin.

Ampoule temperature values are calculated for three different rates: 0.50 mm/h , 1.00 mm/h and 3.32 mm/h. In all cases the x takes values from 0 to 0.61 m. The estimated growth time for a 6 cm ingot are: 18 h, 60 h and 120 h for translation speeds of 3.32 mm/h, 1.00 mm/h and 0.50 mm/h respectively.

The measured temperatures in the furnace axis were fit with a fifth grade polynomial which was used to validate the model by comparison with the temperature simulation results. The polynomial function obtained which gives the temperature in the furnace axis as a function of the furnace height z, in meters, is the following equation (3):

$$T = 288.953 + 9089.87 * z - 50463.8 * z^2 + 164745 * z^3 - 250568 * z^4 + 135824 * z^5 \quad (3)$$

2.5. System Parameters

The Table 1 shows the most important system parameters

Table 1. Growing system parameters

Geometrical parameters	Values
Length and inner diameter of furnace	61 cm x 2.64 cm
Axial temperature gradient at the solidification point	10.7 °C/cm
Internal ampoule dimensions	
internal diameter	1.25 cm
wall ampoule thickness	0.15 cm
ampoule length	17.5 cm
ingot mass	25 g
External ampoule dimensions	
internal diameter	1.7 cm
wall ampoule thickness	0.15 cm
ampoule length	20 cm
ampoule translation speeds	3.32, 1.0 y 0.5 mm/h.

2.6. Physicochemical Properties Used in the Simulation

Solid and liquid CZT physicochemical properties are scarcely present in the literature. However, it was possible to find some data, such as the phase diagram of the pseudo-binary ZnTe ± CdTe system and the heat of fusion. All other properties have a serious lack of experimental data. In those cases where there are no CZT properties values**,** some authors chose to use an interpolation between the properties of CdTe and ZnTe [14]. Although, in cases that ZnTe properties values are not known then CdTe values are directly used. Another different approach is to be consistent with the material and use all the same intrinsic properties. In this particular case, the values correspond to all the properties of CdTe as there are no ZnTe properties values. CdTe properties can directly be used to model CdTe alloyed with 2% and 5% at. Zn [8], [12-27], assuming that the Zn low concentration has no appreciable influence on the values of the properties and it can be used as a first approach [8,9]. In this work we took the last method described above.

3. Results and Discussion

3.1. Validation of the Mathematical Model

The mathematical model was validated by comparing the simulation results with the experimental axis temperature profile of the empty furnace [28]. In this stage the results of the 1st level calculation were used, i.e. the furnace without ampoule. The temperature profile in the furnace wall was imposed in the model as boundary condition. It was used a high density mesh to compare with the experimental profile, it was employed a mesh with 18636 elements and 65453 nodes and the computation time was 1.67 hours.

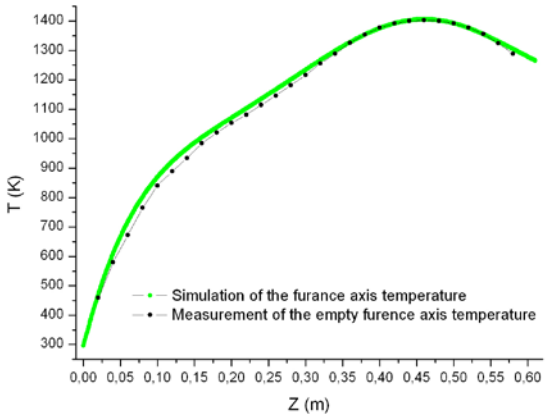

Figure 4. Comparison of the measured temperature profile along the axis of the furnace with the result of simulation

Figure 4 shows that there is a very good fit in the temperatures values between 32 and 54 cm where the maximum difference is 10°C (0.10 %) at the beginning of the range, however, at the point of solidification (1365 K) the temperature difference between the experimental and simulated curve is less than 1°C. In the range between 0 and 32 cm the maximum difference is 50°C, at 6 cm. In the range between 54 cm and 61 cm the maximum difference is 16°C at the end of the range. The adjustment

obtained in the range between 32 cm and 54 cm indicates that the model is acceptable for use in the simulation of CZT growth since the principal solidification process is located between 38 cm and 40 cm, area in the range where the model was satisfactory. The temperature difference is only located in a region where the ingot is already completely solidified and it is not significant for the solidification process.

3.2. Model Sensitivity to Meshing Refinement

It was used three different mesh densities to examine the model sensitivity to grid size in the empty Bridgman furnace: COARSE density was used whit 325 nodes, INTERMEDIATE density with 18067 nodes and FINE density with 65453 nodes. Comparing the simulated temperature profiles with the experimental axis temperature profiles in the Bridgman furnace, it was possible to analyze if the used mesh in the model satisfactorily reproduces the experimental profile. The mesh refinement results were:

The COARSE mesh gives temperature values with a relative error of 0.1% at the solidification point of CZT and fits the experimental temperature profile from 38 to 44 cm in height of the furnace around the solidification temperature. In that region the grid points are spaced 1.91 mm and calculation time was 1.17 s.

The INTERMEDIATE mesh also has temperature values with a relative error of 0.1% at the solidification point of CZT and fits the experimental temperature profile in the same region than before. The grid points are spaced 0.99 mm and the calculation time was 33.64 s.

The FINE grid, as in the prior case, has temperature values with a relative error of 0.1% in the solidification point of CZT and fits the experimental temperature profile in the region just mentioned. The grid points are spaced 0.70 mm and the computation time was 1.67 hours.

The curve obtained from simulation fits the experimental data in identical way with the three meshes. Therefore, the selection is based on the calculation time and the proper spacing of the nodes to the next level in the model.

It was found that the INTERMEDIATE mesh is optimal as it represents the temperature values with a spacing of 0.99 mm , which is satisfactory for later use in modelling the ampoule of 200 mm in height. Time calculation has a value of 33.64 s which is acceptable, especially considering that the FINE mesh needs 180 more time.

3.3. Results and Discussion of the Solid-Liquid Interface Simulation

In Table 2 are listed the total simulation time and the calculation times used by the simulations to get the solidification interface. In the two faster cases the solidification was simulated up to a height of 57 mm, and for the slowest case it was simulated up to a height of 47.5 mm. In all cases the same mesh was used (Figure 3). The boundary condition of the wall of the ampoule was obtained from the results of the 2nd level of the model, assuming the temperature profile on the wall of the furnace as a function of time.

The Rayleigh number was calculated using CZT properties. It was used the correlations for natural convection [29], obtaining a value of 8921, which indicates a laminar flow regime.

For that reason the whole process was split in three simpler ones.

Table 2. Simulations performed to analyze the solidification interface

Translation speed [mm/h]	Total number of simulations, one simulation for each hour	Average solving time per simulation[h]	Overall solving time [h]	Figure
3.32	17	10	17 *10 = 170	5.a
1.00	57	14.75	57 *14,75 = 840.75	5.b
0.50	95	32.49	95 * 32,49 = 3086.55	5.c

Figure 5 shows that for the three translation speeds of the ampoule the solidification isotherms of CZT are smoothly concave towards the liquid phase. Figure 5b and Figure 5c show two planar interfaces (at half height of the specimen), which might be caused by calculation errors that could not be identified so these data will not be considered in the analysis. The experimental conditions of CZT single crystal growth [30,31] were used in finite elements simulation. The isotherms of Figure 5a, Figure 5b and Figure 5c are shown up to the 17, 57 and 95 h of growth, as indicated in Table 2. The typical concave shape of the solidification isotherms obtained in the simulation (Figure 5) are similar in shape to those obtained by other authors [4,5,10].

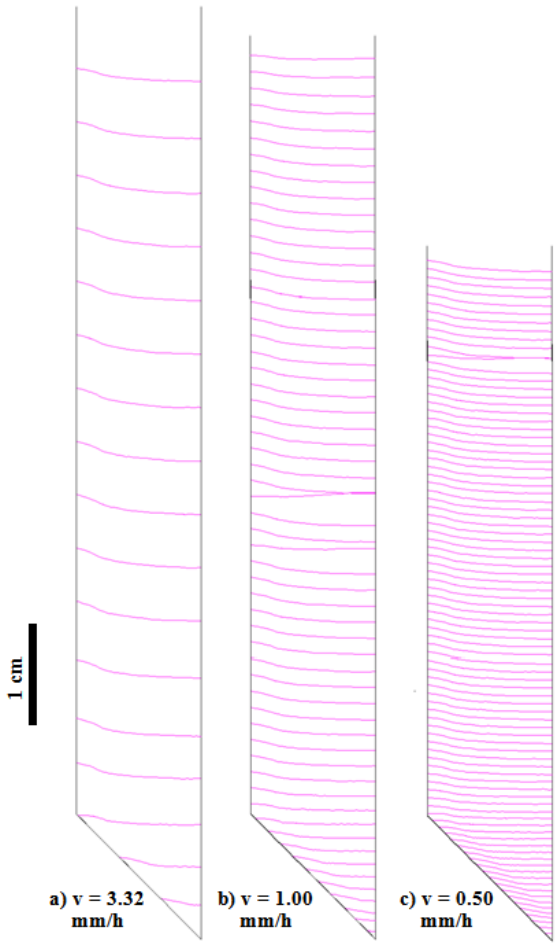

Figure 5. Interface shape per hour for the three simulated translation speeds

Table 3. Interface curvature calculataed from the simulations

Translation speed (mm/h)	Average interface curvature in the conical region.	Average interface curvature in the cylindrical region.
3.32	0.085	0.157
1.00	0.065	0.106
0.50	0.060	0.095

The interface curvature parameter is used to analyze the obtained interface shape. It is defined as δ= h/r where h is the height of the interface and r is the radius of the ampoule, then the curvature could be calculated on the axis of the ampoule. A comparison is made between the CZT interface curvatures for the three translational speeds used in the simulation, to see in detail the differences between them. In Table 3 are listed the average values of the interface curvature split in two different regions, one at the conical tip of the ampoule and another one at the cylindrical body of the ampoule.

In Table 3 it is observed in both regions that the average value of the interface curvature decreases when the translation speed decreases.

The Figure 6a shows the interface curvature for the three growth rates. In this graph it is observed that in all cases at the beginning of the solidification, located in the conical region, the interface curvature values are lower than the average values of the cylindrical zone. These indicate that interfaces are flattened in the conical region more than in the central part of the ampoule, which is favourable for single crystal growth.

At the end of solidification, the interface curvature is also below the average value of the cylindrical region (this not shown in the graph for 0.5 mm/h due to that the curvature values were not obtained for the entire range of time). The ingot grown at 0.5 mm/h starts the growing process with an interface more flattened than the other cases. In all three cases it is observed that the interface curvatures have a local minimum for the value corresponding to the upper end of the conical region of the ampoule (Figure 6a).

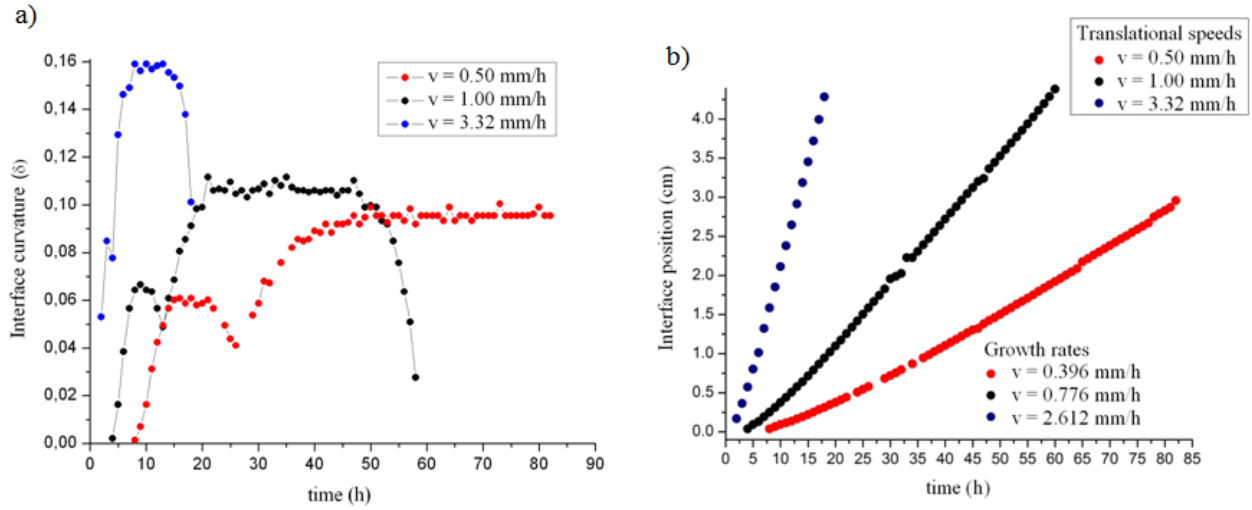

Figure 6. (a) Interface curvature as a function of the simulation time, for the three simulated translational speeds; (b) Interface position as a function of the simulation time for the three simulated translational speeds

Based on the graphs of Figure 5, it was determined the position of the interfaces along the axis of the ampoule as a function of time for the three analyzed translational speeds. These values are shown in Figure 6b, where it can be observed the differences in slopes of the curves, which represent the growth rate (v_c) for each translation speed used in the simulation:

$$v_c\left(\text{for a travel speed of } 3.32 \text{ mm / h }\right) = 2.61 \text{ mm / h}$$

$$v_c\left(\text{for a travel speed of } 1.00 \text{ mm / h }\right) = 0.78 \text{ mm / h}$$

$$v_c\left(\text{ for a speed of } 0.50 \text{ mm / h }\right) = 0.40 \text{ mm / h}$$

The heat transfer through the ingot is affected by the furnace thermal profile and the ingot translation speed, so the shape of the solid-liquid interface could be modified by changes in the thermal profile of the furnace and ingot translation speed. A small translation speed of the CZT ingot, keeps the heat balance in the solid-liquid interface, since the thermal conductivity of solid CZT is lower than the liquid CZT.

It was experimentally observed that the effect on reducing the growth speed produced a composition more homogenous and improved CZT crystal quality, since that for a translation speed of 1.66 mm/h are two grains in the central part of the ingots (with a larger grain size it might be used 75% of the surface of the wafer and therefore allows a better use of the available material for employment in devices). In contrast, when growing at a greater speed (3.32 mm/h) five grains were generated [30,31]. By comparing simulations with experimental results [30,31], it could be realized that computer simulation calculations acceptably reproduce the experimental results. When the ingot is grown at 3.32 mm/h it was found a pronounced concave interface, 58% higher than for a speed of 0.50 mm/h, which promotes the generation of more grains and crystalline defects.

4. Conclusions

From the analysis of the results of the solidification process simulation of CZT growth through finite elements it can be concluded that there are significant differences in the curvature of the interface when using different ampoule translation speeds. The conditions that offer

lower interface concavity to liquid phase of the interface are those that correspond to slower ampoule translation speed. In consequence, a slower rate of translation of the ampoule in the furnace determines lower concavity of the solid-liquid interface respect to liquid and thus better crystal quality.

It was found that when the travel speed is 3.32 mm/h the concavity of the solid-liquid interface is 58% greater than for a translation speed of 0.50 mm/h.

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