Model Experiments for Flow Phenomena in Crystal Growth

Kaspars Dadzis,* Olf Pätzold, and Gunter Gerbeth

The concept of a physical model experiment is introduced and discussed in the context of melt and gas flows in bulk crystal growth processes. Such experiments allow one to "extract" selected physical phenomena from the full complexity of a real crystal growth process and "transfer" them to material systems with an easier access for experimental measurements. Model experiments for the main techniques of melt growth are summarized in a literature review, and the applicability of the results to real crystal growth systems is analyzed. Recent examples of model experiments for melt and gas flows in Czochralski growth of silicon are used to demonstrate the state of the art and show the potential of such experiments to improve the understanding of complex multi-physical multi-scale phenomena occurring in every crystal growth process.

1. Introduction

Theoretical models are a fundamental part of the description and analysis of crystal growth processes, and numerical simulations in particular have become indispensable for process development and equipment design. Due to the coupling of multi-physical phenomena from thermodynamics, solid state physics, fluid dynamics, and other fields on various length and time scales, models for crystal growth require a practical balance between physical simplifications and computational power. Consequently, experiments with measurements of sufficient resolution are needed to validate the theoretical assumptions and approximations. We distinguish between validation and verification, where the latter addresses the mathematical accuracy.[1,2]

Crystal growth processes often take place at high temperatures and elevated pressures as well as under aggressive atmospheres, which significantly limits experimental in-situ observations. Dedicated model experiments can be used in two ways:

- Transfer the materials of interest to an experiment where their properties (e.g., thermal conductivity, fluid viscosity) can be measured under the relevant conditions
- Transfer the physical phenomena of interest to a model system (using a model material) with an appropriate in situ experiment access

While the former category has been realized in various standardized methods, the latter one must still convince crystal growth practitioners of its advantages when compared to a theoretically simpler trial-and-error approach in the development of crystal growth techniques and their numerical models. The present paper will review the use of physical model experiments to investigate flow phenomena in crystal growth processes. We focus on the growth of bulk crystals from the melt, but the presented methodology is general.

2. Flow Phenomena in Crystal Growth from Melt

The main methods for melt growth are Czochralski (CZ) and Floating Zone (FZ) as well as a group including Directional Solidification (DS), Vertical–Gradient–Freeze (VGF) and Bridgman methods. Flows in the melt and in the gas atmosphere (e.g., argon) play an important role for the transport of both heat and mass (impurities, dopants, etc.). The typical geometries are sketched in Figure 1 considering silicon crystal growth as an example. Note that these setups may differ significantly for other materials, for example, oxide crystals are usually grown using optical heating in FZ and induction heating in CZ. Cylindrical crucibles with a conical bottom are applied for the VGF growth of compound semiconductors. Melt flows in the geometries shown in Figure 1 usually take place in a single, closed domain and are driven by buoyancy and Marangoni forces due to thermal gradients, rotation of solid boundaries as well as electromagnetic forces in some cases (e.g., from AC heaters). Gas flows are determined by buoyancy as well as conditions at inlets and outlets. The entire growth furnace must often be considered as the relevant geometry.

There are only a few publications about experimental measurements of flow phenomena in real crystal growth systems. Information about flow oscillations in silicon melts has been deduced from temperature measurements using a pyrometer with a wave
guide in FZ systems\cite{3} and encapsulated thermocouples in CZ systems.\cite{4,5} X-ray visualization of the transport of tracer particles has been demonstrated for a small silicon melt.\cite{6} There are no dedicated measurements of gas flows in crystal growth furnaces known to the authors.

Consequently, numerical simulation can be considered as the main tool for the analysis and design of flows and related processes in CZ,\cite{7} FZ,\cite{8} and DS\cite{9} growth (representative references are given). These simulations are usually based on the following fundamental physical models:

- Navier–Stokes and continuity equations (mostly the incompressible versions)
- Heat equation including convection
- Additional models for volume and surface forces as well as for interaction processes at the boundaries

Although the specification of these equations and conditions may seem fundamentally valid and complete, one must be aware of many inherent assumptions on various levels. In practice, a limited geometric region with limited resolution in length and time is considered for all equations. This often requires to introduce additional models, for example, for turbulent flows. Furthermore, the models for forces and boundaries rely on many specific assumptions.

The validation of these physical assumptions is the key task of model experiments, to which the physics of flow phenomena can be "transferred" using the theory of scaling. If all relevant equations (including boundary conditions) are formulated in a dimensionless form, several characteristic numbers are obtained such as the Reynolds number $Re = \frac{u_0 L_0}{\nu}$, Grashof number $Gr = \frac{\Delta T_0 L_0^3 g \nu^3}{\eta^4}$, Prandtl number $Pr = \frac{\nu c_p}{\lambda}$, general electromagnetic forcing parameter $F_{em} = F_0 L_0^2 \frac{\sigma}{\eta}$, and Hartmann number $Ha = \frac{B_0 L_0}{\sqrt{\sigma \eta}}$ (characteristic values for velocity $u_0$, length $L_0$, temperature difference $\Delta T_0$, gravity $g$, volume force density (in N m$^{-3}$) $F_0$ for AC magnetic fields, and magnetic flux density $B_0$ are separated from material properties such as density $\rho$, viscosity $\eta$, volumetric thermal expansion $\beta$, heat capacity $c$, heat conductivity $\lambda$, electrical conductivity $\sigma$). Frequently used combinations are the Peclet number $Pe = Re Pr$ and the Rayleigh number $Ra = Gr Pr$. Further dimensionless parameters arise from various types of external magnetic fields applied for melt flow control or from specific boundary conditions. Model experiments should attempt to keep these numbers (i.e., a complete set of such numbers) similar to the real case (see Table 1 for an example) to obtain “similar physics.” Note that several dimensionless numbers contain characteristic values which are not known a priori. For example, $u_0$ obviously depends on the resulting flow, $F_0$ may depend on a complex inductor geometry. To compare different systems, it is important to evaluate (measure or calculate numerically) such characteristic values exactly in the same way in each system.

A review of the literature revealed a number of comprehensive model experiments devoted to melt flows in crystal growth, but none for gas flows. A selection of these studies is summarized in Table 2. The applied model materials can be divided into two groups:

- With $Pr \ll 1$, suitable to model semiconductor melts: for example, Hg (melting point at –39 °C), GaInSn (10.5 °C), Ga (29.8 °C), BiPbCdSn (Wood’s metal) (72 °C)
- With $Pr \gg 1$, suitable to model oxide or fluoride melts: for example, silicone oils, NaNO$_3$ (307 °C)

| Crystal/melt diameter, mm | Melt flow | Gas flow |
|--------------------------|-----------|---------|
|                          | $Re$      | $Gr$    | $F_{em}$ | $Re$ | $Gr$ |
| CZ 300/700               | $8 \cdot 10^4$ | $4 \cdot 10^{10}$ | $3 \cdot 10^{10}$ | $2 \cdot 10^3$ | $1 \cdot 10^7$ |
| FZ 200/200               | $2 \cdot 10^4$ | $6 \cdot 10^8$ | $8 \cdot 10^9$ | $5 \cdot 10^2$ | $3 \cdot 10^5$ |
| DS 1000/1000             | $2 \cdot 10^5$ | $8 \cdot 10^{10}$ | $1 \cdot 10^{11}$ | $2 \cdot 10^3$ | $4 \cdot 10^7$ |

Figure 1. Simplified sketches of typical geometries for melt (bottom) and gas (top) flows in various crystal growth processes (based on industrial Si growth).
Table 2. Literature review of model experiments for melt flows. Note that for each case only selected (usually—most recent) references are given here, which represent the corresponding research group. \( Re \) and \( Gr \) numbers are estimated by the authors. Abbreviations: Ox/Fx—oxides and fluorides, Intermet—intermetallic compounds.

| Growth method | Target material | Max. crystal (melt) diam., mm | Model material | Phase change | Magnetic fields | \( Re \) \([\text{max. } u_c]\) | \( Gr \) \([\text{max. } \Delta T_s]\) | Measurement techniques | Selected references |
|---------------|-----------------|-----------------------------|---------------|-------------|----------------|------------------|------------------|---------------------|---------------------|
| CZ O\(_x\)/Fx | 38 (76)         | NaNO\(_3\)                | +             | 5·10\(^3\)  | 2·10\(^7\)   | TC, PIV          | [14]             |
| CZ O\(_x\)/Fx | 22 (40)         | Oil                        | +             | 6·10\(^3\)  | 2·10\(^6\)   | TC, PIV          | [15]             |
| CZ Si         | 64 (160)        | Hg                         | DC            | 5·10\(^4\)  | 8·10\(^8\)   | TC               | [16]             |
| CZ Si         | 165 (500)       | GaInSn                     | AC, DC        | 1·10\(^4\)  | 5·10\(^9\)   | TC, potential probes | [17,18]         |
| CZ Si         | 300 (800)       | BiPbCdSn                   | AC, DC        | 1·10\(^4\)  | 1·10\(^10\)  | TC, potential probes | [19]             |
| CZ Si         | 70 (178)        | GaInSn                     | AC, DC        | 2·10\(^3\)  | 2·10\(^9\)   | TC, UDV          | [20,21]          |
| FZ (high Pr) | 6               | NaNO\(_3\) etc.            | +             | 2·10\(^1\)  | 2·10\(^4\)   | TC, PIV          | [22,23]          |
| FZ Internet  | 50              | GaInSn                     | AC            | 8·10\(^3\)  | 0             | UDV              | [24]             |
| VGF (low Pr) | 60              | Ga, GaInSn                 | AC, DC        | 9·10\(^2\)  | 6·10\(^6\)   | TS, UDV          | [25,26]          |
| VGF GaAs, Ge | 73              | GaInSn                     | AC, DC        | 7·10\(^2\)  | 5·10\(^6\)   | TC, UDV          | [27,28]          |
| DS Si         | 100             | Ga, GaInSn                 | +             | 3·10\(^3\)  | 1·10\(^7\)   | TC, UDV          | [29,30]          |
| DS Si         | 220             | Ga                         | +             | 3·10\(^4\)  | 1·10\(^8\)   | TC, UDV          | [31,32]          |
| DS Si         | 420             | BiPbCdSn                   | AC            | 6·10\(^3\)  | 4·10\(^9\)   | TC, UDV          | [33]             |

Note that simple isothermal flows depend only on the Reynolds number, so that even water may be suitable to model a semiconductor melt.\(^{[10]}\) As can be seen in Table 2, for CZ and FZ growth of oxides and fluorides, the role of Marangoni forces, buoyancy forces and crystal rotation have been studied in detail, including also the effect on the crystallization front. A significant amount of results has been published by D. Schwabe at the University of Giessen (Germany) since 1970s.\(^{[11]}\) The inductively heated FZ process has not been investigated much so far. The research for CZ and VGF growth of semiconductors has been focused on the application of steady (DC) and AC external magnetic fields of various types and has been particularly pushed since 1980-ies by a team at the Institute of Physics (Salaspils, Latvia) headed by Y. Gelfgat.\(^{[12]}\) It should be noted that the industrially relevant flow regimes have not been always reached, especially for buoyancy forces defined by the \( Gr \) number (compare Table 1 and Table 2). An important difference in DS growth of Si compared to all other methods is the square shape of the melt. A series of interesting flow patterns including effects on the crystallization front have been observed in model experiments.

The temperature distribution in model melts is typically measured using miniature thermocouples (TC) or thermistors (TS). Transparent model fluids such as oil and NaNO\(_3\) enable 2D flow measurements using various optical techniques such as Particle Image Velocimetry (PIV). But also for metallic melts, today a variety of ultrasonic (single Ultrasonic Doppler Velocimetry (UDV) sensors and transducer arrays) and electromagnetic (local potential probes and global contactless inductive tomography) measuring principles and sensors allow an almost complete measurement of the velocity field in the melt, including turbulent fluctuations. With measuring capabilities of sufficient spatial and temporal resolution, it is possible to provide experimental data reaching the quality of computational fluid dynamics. For a recent review we refer to Ratajczak et al.\(^{[13]}\)

The design of model experiments taking into account the scaling from the real growth system and the application of modern measurement techniques will be discussed in the following two sections for CZ growth.

3. Model Experiments for Melt Flow in CZ Growth

Recent model experiments at the Helmholtz–Zentrum Dresden–Rossendorf for the CZ process follow an approach of increasing complexity as shown in Figure 2. The simplest model of a CZ growth process considers a Rayleigh–Bénard (RB) configuration, that is, a cylindrical cell characterized by a height \( H \) and diameter \( 2R \) with adiabatic side walls. The cell is heated from the bottom and cooled at the top. The temperature difference \( \Delta T \) between the bottom and top walls generates a buoyant convection. Besides the usual control parameters \( Gr \) and \( Pr \), a third control parameter, the aspect ratio \( a \equiv H/2R \) concerns the geometry of the setup and affects crucially the developed convective pattern inside the melt. The initial filling level in the real industrial CZ facility is lower than \( a \approx 0.5 \) and decreases continuously during the process. Starting with the generic RB case,\(^{[14]}\) a modified RB case was then considered (see Figure 2a,b), with an upper cooled wall smaller than the melt cross-section modeling the solidified crystal.\(^{[35]}\) Next, a setup with a realistically shaped, double-walled glass crucible, flown through by a heating fluid, and a water-cooled crystal model was installed, both differentially rotatable.\(^{[21]}\) A surface cooling system can be added in order to provide realistic heat transfer conditions at the upper free surface of the melt (see Figure 2c), that is, adapting the Biot number as additional control parameter to the real CZ case.\(^{[21]}\) In addition to purely buoyant flows, the influence of various types of magnetic fields using the MULTIMAG setup\(^{[36]}\) has been investigated. It covered DC (uniform vertical or horizontal, or cusp-type fields, each characterized by the dimensionless Hartmann number \( Ha \)) as well as AC magnetic fields with a certain flow driving action such as for example, the rotating magnetic field (RMF, characterized by the non-dimensional Taylor number \( To \)). The following...
Figure 2. CZ model experiments of growing complexity: a, b) modified Rayleigh–Bénard configuration with a cooled crystal model on top inside the MULTIMAG coils; c) CZ-like glass crucible with additional surface cooler. References are given in the text.

Figure 3. Sketch (left) and measured (right) velocity field for the modified RB case with $a = 0.3$ and different $Gr$ numbers. Measurements were done with an UDV array of 25 transducers located in the upper melt part.

paragraphs summarize selected results for the modified RB and CZ-like cases in particular.

The pure buoyant flow in the modified RB case consists of low $\Delta T$ of an axisymmetric torus, which has been measured in a GaInSn model experiment (crucible diameter 178 mm, $a = 0.3$, crystal diameter 70 mm) only for a low temperature difference of $\Delta T = 1$ K, which corresponds to $Gr = 1.9 \times 10^6$ (Figure 3a). If $\Delta T$ exceeds about 3 K, the axisymmetric flow becomes unstable and for further increased $\Delta T$ the flow develops the so-called “wind structure” which is a single 3D roll inside the crucible (Figure 3b). These model experiments were performed up to $\Delta T = 51$ K corresponding to a maximum Grashof number of $Gr \approx 10^8$, which is in the lower range of real Si-CZ crystal growth cases.\footnote{37} If an RMF is applied here, the axisymmetric flow at lower $Gr$ is deformed as the meridional RMF flow supports it only in the upper part whereas it counteracts the toroidal flow in the lower part. Application of the RMF to the “wind” at larger $Gr$ leads to the rotation of the “wind” until a transition to an RMF-driven small-scale turbulence occurs at higher RMF forcing.

Application of an increasing RMF to the generic RB case showed a transition from a low-frequency large-scale turbulence to a high-frequency small-scale turbulence accompanied by a significant reduction of temperature fluctuations in the melt (reduction by factor of about 10 at the rim, and by a factor of about 30 in the bulk of the melt). The transition appeared up to $Gr \approx 1.1 \times 10^9$ at a well-defined threshold $T_{at} \approx Gr$.\footnote{34} This damping of temperature fluctuations was also obtained for model experiments in the CZ-like setup, though not that expressed as in the generic RB case. Interestingly, these experimental results have not yet been reproduced by numerical simulations. If such a reduction for the real CZ case could be shown, it would allow running the process with a much higher initial filling level of the melt.

Application of a horizontal DC magnetic field to the modified RB case showed that the action of the magnetic field does not purely consist in the expected damping of the mean flow and the turbulent fluctuations. Instead, low-frequency oscillations with large amplitudes came up for increasing $Ha$ numbers, and in some parameter ranges a decrease of the integral heat flux as well as an increase of the vertical melt velocity was observed.\footnote{20,38} Again, these phenomena have not yet been reproduced by numerical simulations. Note that the $Ha$ numbers in the model experiments of $Ha = 300...1200$ correspond to the real Si-CZ case.

4. Model Experiments for Gas Flow in CZ Growth

As noted in Section 2, gas flow model experiments represent a new concept of modeling crystal growth processes, which has
not been studied in detail so far. Such experiments are particularly relevant for growth techniques with a gas flow in permanent contact with crystal and melt surfaces, such as the CZ and FZ techniques (see Figure 1). The flow in the hot atmosphere of industrial furnaces can be modeled by a transparent model fluid in a static lab-scale setup at ambient temperature. The velocities in the model fluid can be measured by the well-known PIV method,\cite{PIV} which is a non-intrusive method to determine 2D velocity maps by recording the time-correlated movement of tracer particles.

For an upscaling of the model experiments, the similarity between the model and growth setups in terms of the Reynolds number has to be fulfilled (see Section 2). Hence, the scaling can be done by adjusting the viscosity of the model fluid, the dimensions of the model setup, and the volume flow rate giving the characteristic flow velocity in the relevant domains of the setup. The above approach implies the following simplifications:

i. The compressibility of the gas phase is neglected.
ii. Melt surfaces are considered as solid walls.
iii. The effects of buoyancy and of moving components on the gas flow are neglected.

The assumptions (i) and (ii) are reasonable, because typical gas flow velocities in growth processes are below 1 m s\(^{-1}\). The latter approximations (iii) result from the concept of a static, isothermal setup, where the model fluid is driven by an externally generated forced flow. Consequently, such model experiments are limited to situations, where the influence of the forced flow dominates.

Figure 4 shows the scheme of a simplified CZ-type setup made of plexiglass, which was developed at the TU Bergakademie Freiberg to demonstrate the potential of the experimental gas flow modeling for crystal growth processes. The setup consists of an outer vessel with plane walls and a cylindrical inner vessel. The vessels are filled with a mixture of Diesel and \(\alpha\)-Methylnaphtalene to achieve a refraction index matching between the plexiglass components of the setup and the model fluid, which is a necessary condition for recording undisturbed PIV images. The internal design of the setup refers to the CZ configuration described by Kalaev et al.\cite{Kalaev} The main components are the cylindrical body with a cone-shaped tail end mounted in the center as model for the crystal and the funnel-shaped model of the heat shield, whereas the melt surface is formed by the base plate of the inner vessel.

The model fluid is fed through a central inlet pipe with a diameter of 5 mm. The inlet is connected to an external fluid reservoir via an outer flow cycle, which is equipped with a circulation pump and a mass flow controller to maintain a continuous feeding with a constant volume flow rate of up to 2 l min\(^{-1}\). Spherical hollow glass particles with a mean diameter of about 60 µm are used as tracers.

PIV images are produced by a Continuous-Wave-Laser (wavelength 532 nm) and an optical camera in a perpendicular configuration (Figure 4). The laser light passes a light sheet optics before entering the plexiglass setup. The camera is equipped with an electronic shutter to record the scattered light from moving tracer particles consecutively with up to about 160 fps (frames per second) corresponding to a time resolution of 6.25 ms. The open source software package Fluere is used to transform the particle image data into a 2D map of the flow velocity.

First results show that the flow modeling using the present setup works well. It is possible to detect complex, unsteady flow.
structures with sufficient spatial and time resolutions. As an example, an image of accumulated particle traces and the corresponding vector plot of the mean flow velocity around the heat shield are shown in Figure 5. The results reveal a distinct recirculation of the flow behind the shield, which is due to the high-speed, jet-like flow between the shield and the base plate of the inner vessel.

One of the challenges of this type of model experiments results from the inflow of the fluid at the top of the setup, which can produce asymmetric and time-dependent flow patterns in the free jet-like region below the inlet. This is of particular relevance for the validation of numerical flow simulations that require well-defined starting and boundary conditions. Therefore, in the development of gas flow model setups, special attention has to be paid to the inlet design to establish stationary and symmetric inflow conditions.

5. Conclusions and Outlook

Model experiments have enabled new insights into melt flows during crystal growth processes. In complex systems driven by buoyancy, Marangoni, and electromagnetic forces as well as boundary rotation, many previously unknown flow patterns and regimes have been identified and their stability properties determined. In several cases, phenomena not expected from the general theory or previous numerical models have been observed, for example, increased flow damping with an AC magnetic field and increased flow oscillations with a DC magnetic field when applied to a buoyant flow in a CZ melt. Consequently, a complementary application of model experiments and numerical models allows one to achieve a new level of understanding.

It should be noted that model experiments always address (actually—propose) only a subset of physics (e.g., certain flow phenomena) present in the real growth process. For example, in several cases, buoyancy forces have been neglected or reduced, free surfaces have been replaced by solid walls. These results cannot be transferred directly to the real growth process and must be supplemented with numerical simulation. Here it is important to note that even if the numerical models have been experimentally validated only on a subset of relevant phenomena, they can be used with a much higher confidence than before the validation.

In order to further improve the understanding of modern crystal growth processes including their multi-scale and multi-physics nature, the following aspects could be addressed in model experiments in the future:

- Use of large melt sizes to obtain good scaling of buoyancy forces at realistic temperature gradients ($Gr \sim \nu^3 \Delta T_0$).
- Analysis of gas flows, which have mostly been neglected in the literature so far, using modern measurement techniques.
- Investigation of flows driven by high-frequency electromagnetic forces as in the inductively heated FZ growth.
- Interaction of two or more coupled physical phenomena such as melt flow and crystallization front motion (which is a key part of every crystal growth process).
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Conflict of Interest

The authors declare no conflict of interest.

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