Model experiments for Czochralski crystal growth processes using inductive and resistive heating

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Abstract. The Czochralski (CZ) growth technique is widely applied in crystal growth, using both induction and resistance heaters. In this work, a novel model experiment platform with comprehensive in-situ measurement capability is introduced. Growth experiments with the model material tin applying both heating concepts are performed and analyzed, e.g., in terms of the maximum achievable crystal diameter. Strong asymmetries in the magnetic field of the induction heater are measured and temperature distribution on the resistance heater is found to be non-uniform. Furthermore, significant losses are observed in the power supplies of the resistance heater. The heating efficiency of both concepts is compared considering different insulation geometries. The obtained results show the capability of model experiments for design optimization and will provide valuable input for further validation of numerical simulations.

1. Introduction

Crystals are an indispensable part of modern electronics. One of the main methods for crystal growth from the melt is the Czochralski (CZ) technique. Generally, the CZ process starts with the melting of the raw material in a crucible and continues with contacting of a single crystal seed to the melt surface. Under rotation and simultaneous upward pulling, the melt solidifies at the seed and a crystal grows. Detailed descriptions can be found in [1,2] and the references therein. Nowadays, the CZ technique is widely established to produce many types of bulk single crystals, e.g. Si or Ga2O3.

Both induction heating [3,4] and graphite resistance heaters [5,6] are applied in the CZ growth technique, selected based on various requirements such as cost efficiency, electromagnetic forces, or degree of purity inside the furnace. Numerical simulations have been applied for investigation of both concepts [4,6]. However, due to various simplifications in the numerical models as well as due to often uncertain or unknown material properties, validation is required to give reliable simulation results [7].

Setups on laboratory scale with low working temperatures, relaxed vacuum-sealing requirements and hence with easy access for various in-situ measurement techniques allow to investigate selected physical phenomena extracted from the complex crystal growth process. Such “model experiments” are, therefore, predestined for validation of numerical simulation and are of growing interest, e.g. for investigation of flow phenomena [8-10], process control [11,12], or phase boundary stability [13]. Within the NEMOCRYS (Next Generation Multiphysical Models for Crystal Growth Processes) project, we are developing a new generation of multiphysical models [14,15] and model experiments for crystal growth processes, where multiple physical phenomena can be observed simultaneously and hence validated in a coupled manner. The current experimental basis for model experiments is the Test-CZ setup – a multipurpose system with improved similarity to real CZ growth furnaces. For the present study, the Test-CZ setup can be optionally equipped with an induction or a graphite resistance
heater. Tin (Sn) is applied as a model material, selected due to its low melting point of 232°C and positive results in previous studies, e.g. for investigation of diameter control [12].

2. Experimental setup and measurement techniques
The Test-CZ setup consists of a steel furnace equipped with a top and bottom axis, a surrounding support structure, and a control unit for various electronic systems (see figure 1, left). The furnace is made of stainless steel and has an inner diameter and height of 263 mm and 383 mm, respectively. A large front door allows convenient in-situ access while various flanges are available, e.g., for connection of various sensors or heater power supplies. Different atmospheric conditions such as air (with open / closed door), vacuum, inert N₂ / Ar gas, or air cooling from the side using a fan can be applied in the experiment. Both furnace and axes are water-cooled, enabling the application of model materials with higher melting points in future.

The furnace can be equipped with two different heating systems. The induction heater consists of a copper coil with three windings and an inner diameter of 140 mm, that is internally cooled with water. It is powered by a generator (Trumpf, TruHeat MF 5030) delivering up to 30 kW power at frequencies between 5 to 200 kHz. The meander-shaped resistance heater is made of graphite, has an inner diameter of 110 mm and a resistance of 125 mΩ at room temperature. It is supplied from a transformer and thyristor power controller with a maximum of 4.2 kW at a voltage of 22 V and a frequency of 50 Hz. The latter setup can be optionally equipped with an insulation made of a carbon felt that can be further varied in its shape (without, bottom-only, side / different heights, complete including a cap). Due to the continuous development of the experiment the graphite crucibles used in the two setups are different: an inner diameter / height of 100 mm / 40 mm is used in the induction heating, and of 50 mm / 20 mm in the resistance heating setup.

![Figure 1. Picture of the Test-CZ laboratory setup: overview (left), cases with the induction heater (center) and the graphite heater (right). In the latter case, a partial insulation up to half of the total available height is used.](image)

The Test-CZ setup is equipped with different measurement instruments and sensors for in-situ observation, ranging from optical images through temperatures to voltages, currents and magnetic fields. Some of the applied sensors are highlighted in figure 1 (center / right). In the following, the most important measurements for the present study are discussed in more detail.

Direct contact temperature sensors [16] of resistance type (4-wire Pt100) and sheath thermocouples (TC; type K, J) are mounted in the sidewall of the crucible (and insulation) to monitor its temperature distribution. Two temperature sensors are immersed directly into the melt with a distance of approx. 5 mm to the crucible bottom and wall: a Pt100 is applied for the temperature control using an external device (Eurotherm, model 902), while a TC at the same position serves for monitoring purposes. For
high-accuracy measurement, an external cold junction unit (Isotech, model TRU 937/50) is used in the connection of the TC’s to the measuring instrument (Keithley, DAQ 6510 with 7710). Furthermore, all TC’s and Pt’s are calibrated in an external dry calibrator (Isotech, model Jupiter) in the range between 50°C and 400°C in steps of 50°C, which allows to achieve an accuracy of at least 0.2°C.

For contactless temperature measurement [16], various pyrometers are applied: short wavelength (2.0 - 2.6 μm, Lumasense, IGA 6-23 advanced), short wavelength ratio type (1.5 - 1.6 and 2.0 - 2.5 μm, Lumasense, IGAR 6 advanced), and a pyrometer array (Lumasense, series 600) equipped with 2 long wavelength (8 - 14 μm) receivers. The availability of different devices/wavelengths gives a greater scope for measurements of various materials in a broad range of temperatures; furthermore, the ratio pyrometer enables emissivity-independent temperature measurements under specific conditions such as insignificant wavelength dependency of the emissivity. In some cases, stickers with a known emissivity of 0.95 (8 - 14 μm) are applied. Additionally, an infrared (IR) camera [17] (Optris, PI-640) is used for recording of thermal images at 640x480 pixel resolution and wavelength of 8 - 14 μm. An optical camera (Basler acA2500-20gc, Kowa LM50HC lens) is used for documentation purposes.

For the analysis of the effective heater power, the simultaneous measurement of the heater current and voltage is required. In both setups, the current is measured using a Rogowski coil (Chauvin Arnoux MA200). In the resistance heating setup, measurement of the voltage drop on the graphite heater is sufficient; it is recorded using a multimeter. In contrast, in the induction heating setup, the phase shift between the current and voltage is required in addition to the voltage; respective measurements are under preparation. The magnetic field of the induction coil is measured using a Teslameter (Lakeshore, model F41) with a high-frequency probe.

Data acquisition is realized by a self-developed python program. For most of the measurement devices, such as the multimeter, the pyrometers, or the optical camera, separate interface libraries according to the respective connection (Serial-232 or RS-485 using an ethernet-to-serial converter Moxa Nport 5650-8-DT, Ethernet, USB) are implemented. A graphical user interface for process observation and visualization is realized using the PyQt5 library. The code is available under a GPLv3 open source license and can be found on GitHub [18].

3. In-situ analysis of the heaters
A series of experiments is performed to investigate the thermal and electromagnetic behavior of both heaters (cf. [3,5]), to get input data for validation of numerical simulations, as well as to analyze the advantages and disadvantages of both heating concepts applied in a similar setup.

A typical IR camera snapshot during a growth experiment with induction heating is shown in figure 2 (left). It can be seen that heat is directly generated in the crucible – where it is needed – while the coil itself remains cold. The melt temperature is controlled at 236°C by an applied current of I = 162.3 A at f = 13.5 kHz. Because of the missing voltage measurement, a 2D numerical simulation is used for power estimation [14,15]; it yields a heater power of 105 W. TC measurements in the crucible wall are indicated by arrows in figure 2 (left), showing a temperature asymmetry of 1.0°C.

The measured vertical component of the magnetic field along the inductor diameter is shown in figure 2 (right). To give a better comparison to a 2D simulation, the idealized symmetric distribution is indicated. Comparison of the two lines indicates a strong 3D character, as observed also in [4], thus the accuracy of a 2D simulation is limited. The asymmetric magnetic field is assumed to be caused by the influence of the current supplies at one side of the coil and by the spiral shape of the windings; further investigation using both additional measurements and 3D simulations are under preparation. A correlation between magnetic field and the temperature asymmetry is assumed, but cannot be assessed clearly with the available data.
Figure 2. Experimental results for induction heating: thermal image during a growth process with TC measurements indicated by arrows (left); measurement of the vertical component of the magnetic field of the induction coil supplied with a current of 100 A (right).

An IR image from a growth experiment with the resistance heater is shown figure 3 (left, center). Only bottom insulation is used for better observability of the heater. Melt temperature is controlled to be 236°C. In contrast to the induction heating case, heat is produced in the heater volume from where it is transferred to the crucible by radiation. Therefore, the heater has an approximately 150°C higher temperature than the crucible.

Evaluation of heater temperature at different positions in figure 3 (left) indicates variations of 8 and 12°C between two adjacent curves of the meander and, respectively, two points in the center and at the edge of the same curve. Whether these variations are caused by local differences in heat generation, convective surface cooling or reflection artifacts caused by the steel furnace has to be investigated in future. At the inside of the heater, a temperature drop from 40°C to 352°C is observed next to the power supply connection.

Figure 3. Thermal image of a growth experiment with resistance heater using bottom insulation (left, center) with IR camera (left) and TC measurements (center) indicated by arrows; optical image of the power supply (right).

The power supply temperature is evaluated in figure 3 (center) using the IR image in combination with fixed-emissivity stickers attached on the surrounding insulation. A difference of 53°C is observed, that in tendency fits to the crucible temperature asymmetry. This temperature difference is assumed to be caused by additional contact resistances at the screw connections shown in figure 3 (right). This is supported by a separate investigation in a table setup using a Sourcemeter device (Keithley, model 2450), where variation of the screw tightening at constant current of 1 A indicates large changes of the
supply resistance in the range 28–163 mΩ. Additional voltage measurement directly at the graphite heater (excluding power supplies) and outside of the furnace (including power supplies) at different currents is summarized in table 1. The resulting power loss at the supplies is found to be decreasing with increasing current thus increasing temperature and is hence assumed to be caused by a temperature dependency of the contact resistances; additional investigation is required here. For the case with bottom insulation (95 A) shown in figure 3, 13% of the power is lost in the supplies.

**Table 1.** Measured electrical parameter (rms) with the graphite resistive heater.

| Current [A] | U inside [V] | U outside [V] | P inside [W] | P outside [W] | Ratio [in/out] |
|-------------|--------------|---------------|--------------|---------------|----------------|
| 19.9        | 2.2          | 2.7           | 43.8         | 53.7          | 0.81           |
| 40.5        | 4.0          | 5.0           | 162.0        | 202.5         | 0.80           |
| 60.5        | 5.7          | 6.8           | 344.9        | 411.4         | 0.84           |
| 80.1        | 7.1          | 8.3           | 568.7        | 664.8         | 0.86           |
| 100.2       | 8.5          | 9.8           | 851.7        | 982.0         | 0.87           |

The influence of the insulation level on the required heater power under air atmosphere is summarized in table 2; voltage measurements outside of the furnace are used for power determination. A power reduction of 64% is achieved comparing the experiments without and with full insulation; the cap has an influence of 19%. In a separate growth experiment without insulation under vacuum condition (see table 3) a 72% lower heater power is required, indicating a strong influence of convective cooling. Investigation of the influence of the insulation shape on the cooling airflow using a systematic comparison to the vacuum experiment could give further insights.

**Table 2.** Heater powers with different insulations (B=bottom, S=side, T=top) under air atmosphere.

|              | without | B + ½ S | B + S | B + S + T |
|--------------|---------|---------|-------|-----------|
| Heater Power | 912.0 W | 743.8 W | 499.6 W | 324.5 W   |

A qualitative comparison of heater power consumption in model experiments is summarized in table 3. The results show that, without insulation, the power consumption of the induction heating is lower than that of resistance heater by approx. 80% under vacuum and 90% under air. Comparing the fully insulated resistance heating setup from table 2 to the induction heating setup without insulation under air, there is still a difference of approx. 70%. However, it should be noted that these are the effective heater powers and not the actual powers consumed by the supply units. The power consumption of the induction generator could be significantly higher than the effective power of 45 / 105 W, while power losses in the supply of the resistance heater are expected to be much smaller [19]. These aspects will be investigated later.

**Table 3.** Comparison of heater powers from growth experiments with a control temperature of 236°C (without insulation). For the induction heater, the values are estimated by numerical simulation.

| Heater     | Vacuum | Air  |
|------------|--------|------|
| Resistance | 254.4 W| 912.0 W|
| Induction  | 45.0 W | 105.0 W|

A general comparison between inductive and resistive heating is hardly possible: in crystal growth application there are various economic and process-specific requirements that are not considered in this study. For example, resistance heating generators work with DC or low frequency currents and are less complex and thus less expensive than induction heating generators at radio frequencies. However,
inductively heated furnaces are better suited for growth processes with high temperatures (e.g. oxide crystals) or high purity requirements, because the crucible / melt are the hottest part in the system and no additional impurities are introduced at the heater surface.

4. Application to the crystal growth - experimental results

Growth experiments with both heater types are performed to analyze the achievable crystal diameter for various process parameters, cf. [12]. In the induction heating setup, the focus is set on the influence of pull velocity and melt control temperature under air atmosphere, vacuum and air cooling with an additional fan. The resulting diameters of 23 crystal grown under different conditions, evaluated at a crystal length of 100 mm, are shown in figure 4 (left); a selection of crystal photographs is presented in figure 4 (right). Generally, we observed decreasing crystal diameters with increasing melt temperature (crystals B, C, D, E) or pull velocity. This is the expected behavior from theoretical considerations. Active cooling of the crystal using the fan almost doubles the obtained crystal diameter (crystal A). In contrast, under vacuum conditions the resulting diameters are very small (crystal F). This indicates a strong influence of convective cooling in the present setup. On the surface of all crystals except F, a spiral structure is visible. The pitch of the spiral changes with pull velocity and crystal rotation (which is held constant here). The spiral structure is probably caused by an asymmetric temperature distribution. The spiral structure is most prominent on crystal A grown with asymmetric active cooling from the side.

![Figure 4](image)

**Figure 4.** Experimental results from growth experiments with induction heating: resulting crystal diameters at length of 100 mm for different pull velocities, control temperatures and atmospheric conditions (left); crystals grown with a pull velocity of 4 mm/min (right).

In the resistance heating setup, the focus in the initial studies is set on the influence of the insulation. Air atmosphere and a melt control temperature of 236°C are used for investigation of the cases (a) without insulation, (b) bottom insulation, (c) half side insulation, and (d) full side insulation. In the cases (a) and (b) a growth velocity of 1 mm/min, in the cases (c) and (d) of 4 mm/min is used due to crystal size limitations. The resulting diameters and crystals are shown in figure 5. It is found that, in the present setup, increasing insulation increases the obtained crystal diameter. The assumed reason is that the heater temperature decreases with more insulation required to maintain the same melt temperature. In consequence, the radiative heating of the crystal is reduced. A spiral structure is visible on the surface of the crystals, similar to the inductively heated case.

In addition to the insights into the growth processes discussed above, the obtained results are highly valuable for validation of numerical simulations: broad parameter studies including comprehensive in-situ measurements are hardly possible outside the model experiment but are required for systematic examination of simplifications and assumptions made in modeling of crystal growth processes [7]. Phenomena observed in the present study, such as the convective cooling, asymmetries in temperature field, and asymmetric crystal shape are often neglected in literature but may have significant influence.
Further analysis with experiments and simulations optimized for scaling to real processes are planned for the future.

Figure 5. Experimental results from growth experiments with resistance heating: (a) without insulation, (b) bottom insulation, (c) half side insulation, and (d) full side insulation. Crystal diameter (left), obtained crystals (right).

5. Conclusions and outlook

A new model experiment setup for the CZ growth has been presented: extensive in-situ measurements are applied in a small-scale (melt diameter 50/100 mm) low-temperature process using the model material tin (melting point of 232°C) to get new insights into the characteristics of both induction and resistance heating during growth experiments.

For both induction and resistance heating, an asymmetric temperature distribution with deviations of approx. 1°C is observed in the crucible. In the induction heating setup, correlation with an asymmetric magnetic field of the inductor is assumed. In the resistance heating setup, heater temperature asymmetries are observed, particularly in the power supplies. At the power supplies of the resistance heater, temperature-dependent power losses of approx. 15% of the total heating power are measured, caused by contact resistances at screw connections. Despite nonexistent insulation, the induction heating setup outperforms the resistance heating in terms of effective heating power. However, the total power consumption of the supply units has not been investigated in detail so far. Further investigation is also planned regarding the asymmetric magnetic field of the inductor, the resistance heater temperature distribution, and the influence of the insulation shape on the convective cooling.

Growth experiments using the induction heating show an expected relation between melt temperature, pull velocity and crystal diameter; the obtained dataset gives valuable input for validation of numerical models [14,15]. In the resistance heating setup, the crystal diameter increases with increasing insulation level due to a reduction in heater temperature. Again, this behavior is as expected, but highlights the value of model experiments for investigation of design optimizations that is of much higher effort in real growth furnaces.

For future investigations, various new in-situ measurement techniques are currently under development, and a larger and more flexible experimental platform (MultiValidator) is under construction. Both will offer the basis for even more sophisticated experiments with in-situ measurements of the CZ process and other crystal growth techniques.
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