Float zone single crystals for testing rods, pulled under electron beam heating

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Abstract. The article is devoted to the description of mathematical modelling and attempts to grow silicon single crystals from a pedestal. The crystals are intended to be used for impurity composition testing in rods with a diameter of 300 mm grown with electron beam heating. The testing is being planned both by the method of FTIR spectroscopy and by functional testing of devices that might be manufactured using single crystals grown from pedestal. The article also describes the improvements of equipment, which were necessary for crystal growth attempts, and substantial difficulties that occurred in the process and hindered single crystal growth, allowing to obtain only a polycrystalline sample.

1. Introduction
KEPP EU developed new method of achieving polycrystalline silicon rods by electron beam heating method (EB rods), which are intended for future applications like float-zone (FZ) single crystal growth. The developed method allowed to obtain rods of 300 mm in diameter [1], which is remarkable, because electromagnetically heated FZ technology for 300 mm single crystals has not been developed yet.

Main requirements to such rods are purity, stable prescribed geometry and mechanical strength. The content of main impurities, such as boron and phosphorus, must be less than 10 ppta. Detection of such low level of contaminants in silicon is possible only by FTIR Spectroscopy at liquid He temperature. For these tests, EB rods should be melted and recrystallized as single crystals. To reduce additional melt contamination during this stage, crucible-free methods should be used. The present article is focused on a pedestal method due to its relative simplicity in comparison with FZ method.

In the pedestal method, electromagnetic (EM) inductor is used to melt upper surface of the polycrystalline rod, which is called pedestal, and single crystal is pulled upwards from the molten zone. Additional pedestal heating from the side may be used to increase the deflection of the melting interface and thus reduce the chance of melt freezing.

The work consists of three parts: modelling of crystal growth from pedestal, creation of a puller based on available laboratory equipment and the actual growth of single crystals. The modelling processes were carried out for the growth of control rods of 12-20 mm in diameter [2].

2. Modelling
The numerical model was quasi-stationary and included only cylindrical phase of crystal pulling. To reduce time consumption and allow for numerous calculation studies, axially symmetrical
approximation was used, and the melt flow was not considered. Inductor was optimized to increase melt height at the system axis and therefore decrease the risk of melt freezing. More details about the calculation procedure are described in [3].

Figure 1 shows the temperature distribution, calculated for different diameters of crystal and pedestal. The obtained optimal values of integral electromagnetic heat $Q_{EM}$ and additional pedestal side heating $Q_{add}$ are disproportionally increasing for larger pedestals, raising the ratio $Q_{EM}/Q_{add}$. The position of additional heater was optimized using heat transfer calculations with FEMM program in simplified axially symmetrical representation of a flat pedestal, see Figure 1, right top. Heat fluxes $q = \lambda \frac{\partial T}{\partial n}$ were imposed as boundary conditions on all pedestal surfaces except bottom surface, where fixed temperature was used to ensure calculation convergence. Here $\lambda$ is heat conductivity and $n$ – normal coordinate. Calculations predicted that the optimal position of heater, which creates maximal temperature difference $\Delta T$ between the pedestal centre and its rim, is dependent on pedestal radius. Thus optimal $z_Q$ (the distance from the additional heater to pedestal edge) increases from 30 mm for 37.5 mm pedestal to 40 mm for 50 mm pedestal. Moreover, $\Delta T$ becomes less sensitive on $z_Q$ when pedestal radius increases.

![Figure 1](image.png)

**Figure 1.** Left: temperature field in silicon ($T_0 = 1687$ K is melting temperature), for the system with 12 mm crystal grown from 75 mm pedestal. Middle: temperature field for the system with 44 mm crystal grown from 100 mm pedestal. Integral values of electromagnetic heating $Q_{EM}$ (black oval corresponds to the inductor) and additional pedestal side heating $Q_{add}$ (rectangle corresponds to the additional heater) are given. Right top: the scheme of simplified geometry and boundary conditions for additional heater position optimisation. Right bottom: temperature difference between the pedestal centre and its rim, over additional heater position, calculated for different pedestal radii $R_p$.

3. **Process development**

3.1. **Puller creation**

Vacuum furnace for laboratory electron beam melting (Figure 2, left) was chosen as a base machine for processes with induction heating. A standard high-frequency generator FTEC 30 (Figure 2, middle) was used as a power source. The single-circuit design of the FTEC 30 was replaced by a two-circuit design, which is energetically more efficient [4]. In order to increase the efficiency of the system and reduce the influence of uncontrolled induced currents, we have changed constructively the HF tube, primary
(anode) circuit, the circuit of separating capacitors, and inter-loop air transformer. Then we have added a tank of capacitors and have put all of that in a separate unit (Figure 2, right).

The power required for crystal growth processes was obtained from the results of numerical modelling and based on the literature data [5]. According to the model (see Figure 1), an upper estimate of total power required for 44 mm crystal growth is approximately 7 kW. On the other hand, the comparison of the power consumed by power suppliers with the capacity to grow single crystals in Steremat FZ1510 and Leybold-Hereus 1450/35 shows that the efficiency of such systems is 5-8%. It suggests that if the main coil power consumption (for single crystal diameter up to 44 mm) is around 3 kW, the power of the FTEC 30 generator is sufficient for the process.

3.2. Growth experiments
The rod with diameter of 75 mm, pulled under electron beam heating, was used as a pedestal. It had been obtained using the equipment described in [1]. The purity of silicon had not been measured, as the rod was intended only to be used in pedestal process development. However, the potential purity of EB rods was shown to be comparable with that of the high-purity reference sample of FZ silicon [1].

Despite the long work to harmonize the HF tube with the electrical load, it was possible to melt the pedestal upper surface only partially (see Figure 3, left). The power supplied by the bottom heater was changed from 2.6 kW (predicted by the model) to the value that was sufficient to melt pedestal side surface (see figure 3, right). However, it did not help to fully melt the upper surface of the pedestal.
The power supply was increased by replacing the transformer with a more powerful (+20%), regular HF tube FU 307S at ITK 30-2 (+300%). We have appropriately matched the HF tube with the electrical load, and it allowed to melt the upper surface of pedestal completely (Figure 4, left). We successfully connected the seed crystal to the melt (Figure 4, right). However, it was not possible to pull a single crystal due to significant risk of spontaneous crystallization.

The described result was obtained using a coil that was manufactured according to the model [2]. After trying to conduct a process using a coil with the prolonged bottom part to concentrate EM field closer to the pedestal centre (similar to the one used in [6]) we have not obtained a positive result. The photographs of both coils and the pedestal with a non-molten centre are shown in Figure 5, left.

These difficulties were overcome by tuning of system parameters and improving heat flux distribution in the system. The first growth process was performed and irregular-shaped polycrystalline rod with diameter of 11-16 mm and length of 159 mm was obtained (see Figure 5, right). The shape of it demonstrates that the growth process is highly unstable, and its control is very challenging.

An important discrepancy between the mathematical model and the experiment should be addressed. The former demonstrated the existence of a steady-state solution for different inductor parameters and crystal diameters, thus predicting good stability of the process, at least for relatively small crystals. The experimental growth process, however, was highly unstable. Some of the possible explanations are T gradient oscillations induced by non-stationary melt flow, 3D features of one-turn EM inductor, insufficiently sensitive power control in the experiment and different ambient conditions for thermal radiation and EM field. Although some of the mentioned effects, such as melt flow, were investigated previously [2], additional numerical studies are needed to improve the validity of the mathematical model by detecting which simplifications induce discrepancies between the model and the experiment.

Figure 4. Pedestal upper surface during the process and after (left) and seed crystal during the process and after (right).
Figure 5. Top left: photograph of the used coils; the coil manufactured in accordance with the model (lower coil) and the coil with the modified middle part, which is closer to pedestal centre (upper coil). Bottom left: fused pedestal with a non-molten centre. Top right: the growth process with improved thermal conditions. Bottom right: irregular-shaped polycrystalline sample that was obtained from the most successful growth process.

4. Conclusions
It was shown that the laboratory furnace for electron beam melting can be used as a base for creating equipment for pedestal method of single crystal growth. The inductor, proposed by numerical modelling, was capable to create the molten zone and the simulated necessary power of bottom heater was close to the experimental one.

The process with pedestal diameter 75 mm and seed crystal with diameter 12 mm was performed and polycrystalline sample was pulled. Its irregular shape demonstrates that the process is unstable, therefore the used quasi-stationary model might be insufficient for process development, and time-dependent models could be more useful.

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