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Growth of large scale silicon crystals by the rf-heated Float Zone technique

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Abstract

Improvements of the radio-frequency (rf) heating for Float Zone growth of silicon single crystals are presented. Details with respect to the shape and design of the rf-inductor are described. The specific growth parameters for 4" and 6" ingots are given and the required power consumption is discussed. The formation of spikes during the melting of the feed rod is set in relation to the frequency and temperature dependence of the skin-depth. The doping via the gas phase is explained and results for the absorption efficiency are presented.

Introduction

For silicon mono-crystals, the Float Zone technique provides material of maximum quality [1-4]. Compared to the Czochralski method, the only alternative for silicon single crystal growth, the Float Zone material is 2 to 3 order of magnitude lower in oxygen [4], shows lower levels of metallic impurities, and allows more uniform axial resistivity profiles. Float Zone is a truly crucible-free technique, the silicon is molten by an radio-frequency inductor placed near the feed rod [5, 6]. Float Zone silicon is the material of choice for power electronics like thyristors, IGBT´s or any application where oxygen would lower the performance. Today, the standard diameter is 4" to 6", but 2" or 3" are also still on the market. A few companies world-wide are capable to grow 8" ingots, which also marks more or less the physical limitations of the process.

In the following, new approaches for the improvement of the Float Zone technique will be shown, we will discuss in detail the heating and melting of the material, arrangement of the inductor and the feed rod, the power consumption and the doping from the gas phase. Special emphasis is put on the melting of the feed rod and the discussion of the process parameters.

Experimental set-up and experimental results

The experimental arrangement itself looks relatively simple, a schematic drawing is shown in Fig.1, but the requirements for the mechanical stiffness, the quality and stability of the rf-generator and the accuracy of the pulling spindles are very high. In addition, the specification for the feed rod material is significantly more vigorous than for any other growth techniques.

For the experiments, two different Float Zone machines had been used, a smaller one (FZ-14, PVA TePla) for ingots up to 4" and a large one (FZ-35, PVA TePla) for ingots up to 6" (and capable for 8"). For diameters larger than 4" the argon inert gas pressure is increased, the FZ-35 can handle an overpressure of 5 bar. According to the Paschen law, the overpressure reduces the risk of argon ionization caused by the high voltage / high frequency. Argon ionization is a serious problem and is one of the limitations for larger diameters.

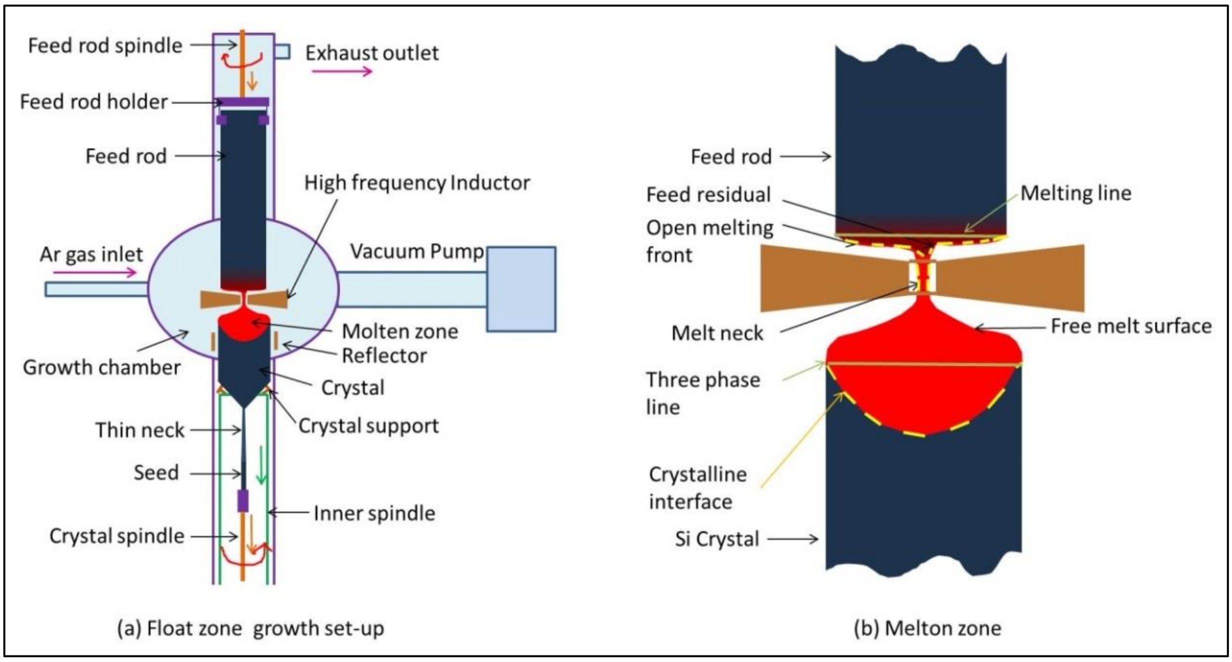

Fig. 1: Schematic arrangement of the silicon Float Zone process. The mono crystal and the feed rod are rotating in opposite directions. The silicon melt flows as a thin film along the bottom side of the feed rod and forms a liquid bridge, connecting the feed with the melt lake on top of the growing ingot.

A picture of a 4" Float Zone is given in Fig. 2. The feed rod (above the inductor) is connected to the growth section (below the inductor) by a small liquid bridge only (therefore, the name "needle-eye-technique" is often used for this method), the liquid silicon flows along the melting feed rod towards the center and into the melt lake on top of the growing crystal.

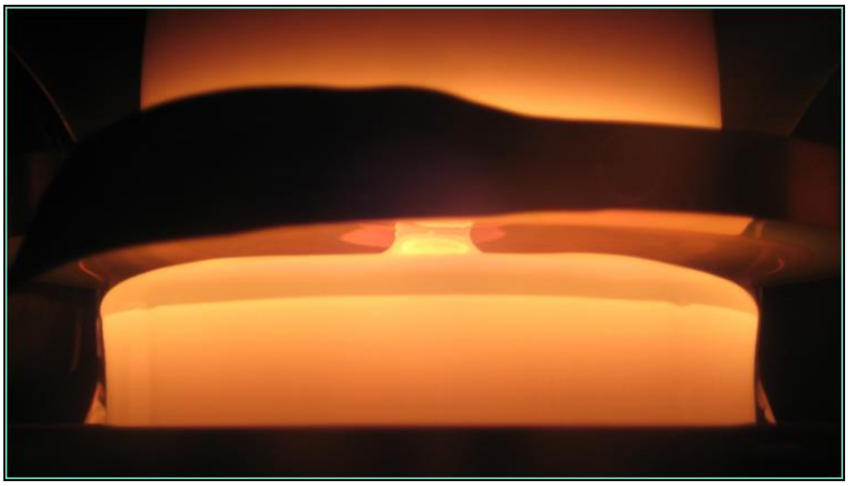

Fig. 2: Picture of a 4" silicon Float Zone. The dark ring in the center of the image is the rf-inductor. Liquid silicon appears darker than the solid one because of the lower emissivity of the melt compared to the crystal.

The inductor (or induction coil) itself, shown in Fig. 3, is a single-winding copper plate, water cooled and some 30 to 50% larger in diameter than the anticipated ingot. It might be round or square shaped, that is of lower importance, but it needs well defined steps on the upper side, i.e. the side which is responsible for the melting of the feed rod. These steps should correspond to the diameter of the feed rod and should just be slightly larger than the feed diameter. Essential for the uniformity of the electromagnetic field are the side slits, length and width are related to the process parameters and have to be optimized for each diameter. Normally, 3 side slits and one main slit are used. Without side slits or with slits too short, the radial temperature profile becomes non-uniform, which results in back-melting / accelerated growth with each crystal revolution. Growth rates above a certain limit initiate crystal defects, which might lead to structure loss. It turned out that towards the growing interface, a smooth, slightly convex-shaped inductor geometry is most helpful and results in stable growth.

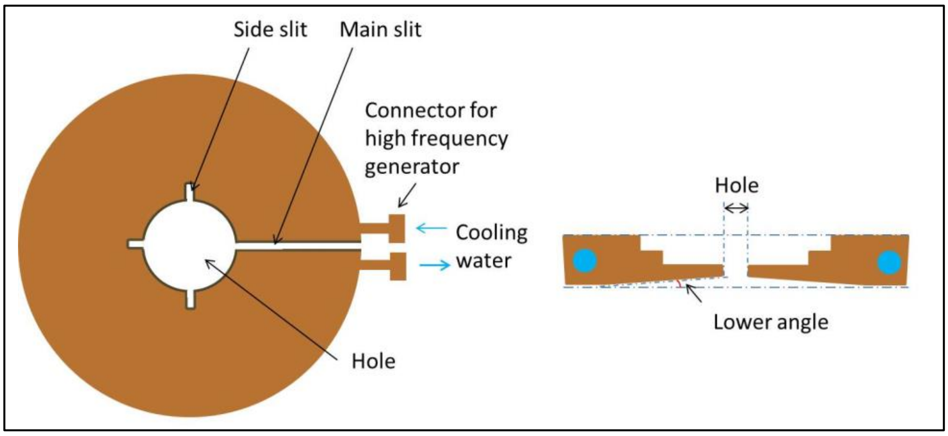

Fig. 3: Drawing of a typical Float Zone inductor. The side slits generate a more uniform electromagnetic field. The steps on the upper side support the melting of the feed rod.

A significant challenge for melting and growing silicon by the radio-frequency technique is the wide range of the electrical conductivity of silicon. Silicon is nearly insulating at room temperature, depending on the dopant level, the resistivity is in the range of 0.05 Ω·cm (heavily doped) to >1,000 Ω·cm for undoped material. Near the melting point (T=1350), the resistivity is lowered to $3 \cdot 10^{-3}$ Ω·cm (doped or undoped) and drops by more than one and a half orders of magnitude beyond the melting point $7 \cdot 10^{-5}$ Ω·cm (T_liquid=1420°C) [7]. This has a significant impact on the electromagnetic coupling and finally, the heat transfer. In particular, two problems have to be handled by the operator: (I) starting the process: warming the silicon to a temperature, where it couples with the rf-field and (II) control the interface of the melting feed rod. In order to establish a stable Float Zone, frequencies in the range of 2 to 3 MHz are used. Lower frequencies result in pulsating melt heating and difficult to control zone heights [4]. Looking at the skin depth of silicon as a function of the temperature (Fig. 4), we observe a strong variation of the penetration depth.

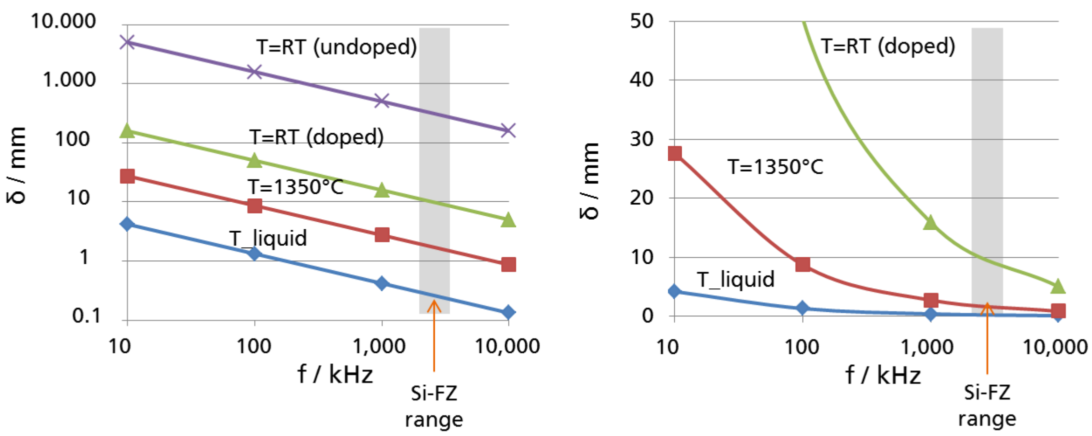

Fig. 4: Skin depth as a function of the frequency of the rf-inductor and the temperature of silicon. Liquid silicon behaves like a metal, but in the solid state, the electrical conductivity drops drastically and the skin depth becomes very large. As a consequence, the electromagnetic coupling is weak and the heating effect is low.

In the liquid state, the skin depth and thus the heat transfer is localized to the surface-near area of just a few hundred micrometers. Liquid silicon absorbs rf-heating very eagerly. Near the melting point, small variations of the temperature in the feed rod result in strong changes in the heat transfer. In the solid state, skin depth might reach from around 1 mm (near the melting temperature) to several hundred millimeters, the colder the silicon gets. As a consequence, if the feed rod is not melting smoothly but forms some hillocks or spikes, they will stick out the feed rod surface and will come closer to the water cooled inductor. At that point, it is a self-amplifying process: the closer the spike comes to the inductor, more heat is extracted by the water-cooling and the rf-heating becomes less and less effective. Melting will not be possible anymore without moving the feed rod to a larger distance to the rf-inductor, but in that case, the liquid bridge would be interrupted. The process has to be stopped or the inductor will be damaged. In order to prevent that a scenario, a proper selection of the feed material is required, the velocity of the feed rod should not exceed some 4 mm/min. In addition, a second inductor (active or passive) might be installed near the melting interface with an inner diameter somewhat larger than the feed rod. If some spikes are observed, the melting / solidifying process has to be slowed down to give the system more time for melting.

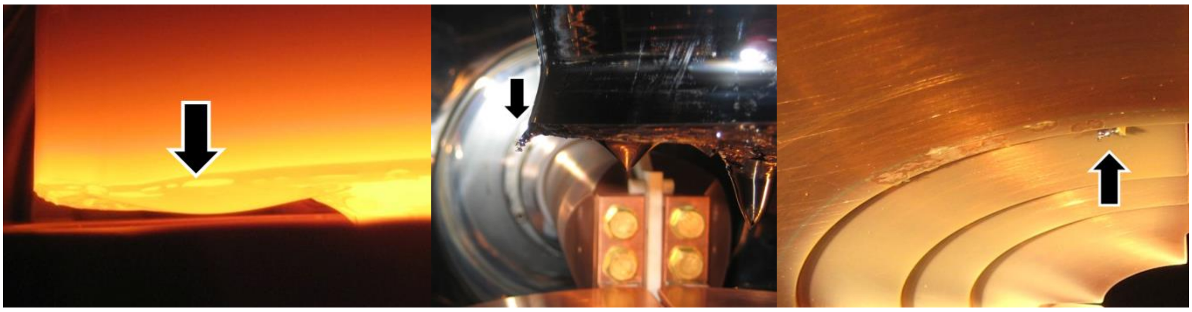

Fig. 4: Formation of spikes during the melting process of the feed rod. Once a spike becomes too large, it will be cooled too strongly by the water-cooled inductor and is not coupling with the rf anymore and will finally touch the inductor. Left: irregularities on the surface of the melting feed rod. Middle: Feed rod after the process was stopped: spikes had been formed. Right: A silicon spike touched the inductor and resulted in a shortcut.

To overcome the problem of the insulating behavior of undoped silicon at room temperature during the starting phase, we place a SiC-coated graphite ring between inductor and silicon feed rod: the graphite couples easily and heats the silicon by radiation to temperatures where the skin depth is small enough that the feed rod is further heated up by the rf-coil directly.

Compared to the Czochralski method, where the silicon feed stock is completely molten right at the beginning of the process, we have a different scenario in case of the Float Zone technique, where the feed material is continuously melted during the process and only a small amount of melt is formed at a time. In such a way, using undoped feed material and adding the dopend material in-situ during the crystallization, we are able to add exactly the amount of dopant as we consume by crystallization. Therefore, very uniform axial resistivity profiles are reached. The dopants, boron or phosphorous, are blown as a gaseous species (PH_3 or B_2H_6) directly onto the silicon melt. Liquid silicon absorbs them quite well, they decompose and will be incorporated into the growing ingot. The dopant gases are mixed with argon in a ratio of 1:10.000. This allows easy to control flow volumes and the concentration of phosphine or diborane are so low that they are not critical anymore and no special off-gas treatment is required. Our nozzle is placed below the rf-inductor in close vicinity to the silicon melt. For 4" ingots, we reach dopant levels of >3 $\Omega\cdot$cm (n-type) and >5 $\Omega\cdot$cm (p-type) respectively. For 6" ingots, they are slightly higher. Of course, higher dopant levels can be achieved by using a more concentrated mixture of dopant gas versus argon carrier gas. For the given arrangement, we determined an absorption rate of 16-18% for PH_3 and 8% for B_2H_6.

As mentioned above, the feed rod diameter should be slightly smaller than the growing ingot. In case of 4" crystals, we use 90 +0/-5 mm feed rods, for 6" ingots some 140 +0/-5 mm feed rods. An important issue is the power consumption, in particular in relation to pulling from the melt by the Czochralski technique. The 4" ingots, we grow with 3.2 mm/min and a total power consumption[1] of 44 kW/h has been determined. In case of 6", growth rate is reduced to 2.8 mm/min and a total power of 100 kW/h is needed. Of interest is the power consumption per kilogram solidified silicon, typical values are given in table 1. For the calculation, we use the total power consumption of the machine, including subsystems and periphery, not only the power of the rf-inductor (or the graphite heater in case of the Czochralski puller).

Ingot diameter	Growth rate [mm/min] (left: FZ, right: Cz)	Total power consumption [kW/h] (FZ / Cz)	Power consumption [kW per kg silicon] (FZ / Cz)
4"	3.2 / 1.2	44 / 90	12.5 / 68.3
6"	2.8 / 1.1	100 / 90	14.5 / 33.1
8"	2.2* / 1.0	120* / 90	12.3* / 20.5

*extrapolated values from the 6" process; our 8" process is still under development.

In particular for smaller diameters, the Float Zone technique provides much more favorable energy consumption data compared to the Czochralski method, simply because in case of pulling from the melt, we have to keep the whole melt volume liquid all the time. The much easier release of the latent heat in case of the Float Zone arrangement results in significantly faster growth rates and therefore shorter process times and strongly reduced power requirements. The shown values are of course also a function of the corresponding machine and might be somewhat different for other types of growth pullers. The increase of the power consumption from 4" FZ to 6" FZ results from the switch from the smaller FZ-14 machine to the larger FZ-35, capable to grow 8" ingots. Thus, the 6" ingots are grown with lower power efficiency. The values for the Czochralski-case are based on an EKZ-2700 from PVA TePla, which can handle some 60 kg of silicon. In any case, Float Zone growth needs significantly less energy per kilogram crystallized silicon. Of course, the requirements for the quality of the equipment and the feed material are higher, as well as the required skills of the operator.

Summary

The Float Zone technique produces silicon of highest quality. The contact-free, crucible-less process results in oxygen concentrations some 2-3 orders of magnitude lower compared to standard Czochralski material. The 4" ingots, we are able to grow with a pulling rate of 3.2 mm/min and a total power consumption of 44 kW/h, resulting in a power consumption of 12.5 kW/kg_Si. For 6" ingots, the growth rate has to be lowered to 2.8 mm/min, resulting in a slightly higher power consumption of 14.5 kW/kg_Si. Compared to standard Czochralski silicon, these values are considerably lower, because of the smaller melt volume of the Float Zone and the much higher growth rates. Blowing PH_3 or B_2H_6 directly onto the melt surface we determined absorption rates of 16-18% for phosphine and 8% for diborane. For p-type as well as for n-type, steady, uniform axial resistivity profiles can be achieved by the doping from the gas phase.

[1] The total power consumption takes into account not only the power at the rf-inductor, but the power pulled from the grid to operate the machine.

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