Large Silicon production at IKZ

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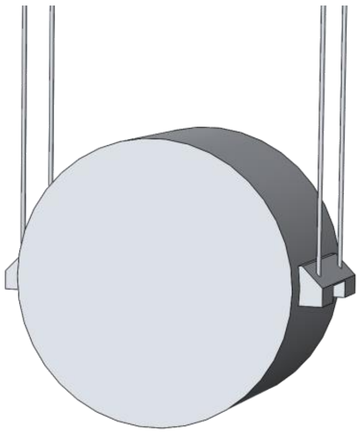
Crystalline Silicon Substrates for ET-LF cryogenic mirrors

Mirror Requirements & Crystal Properties

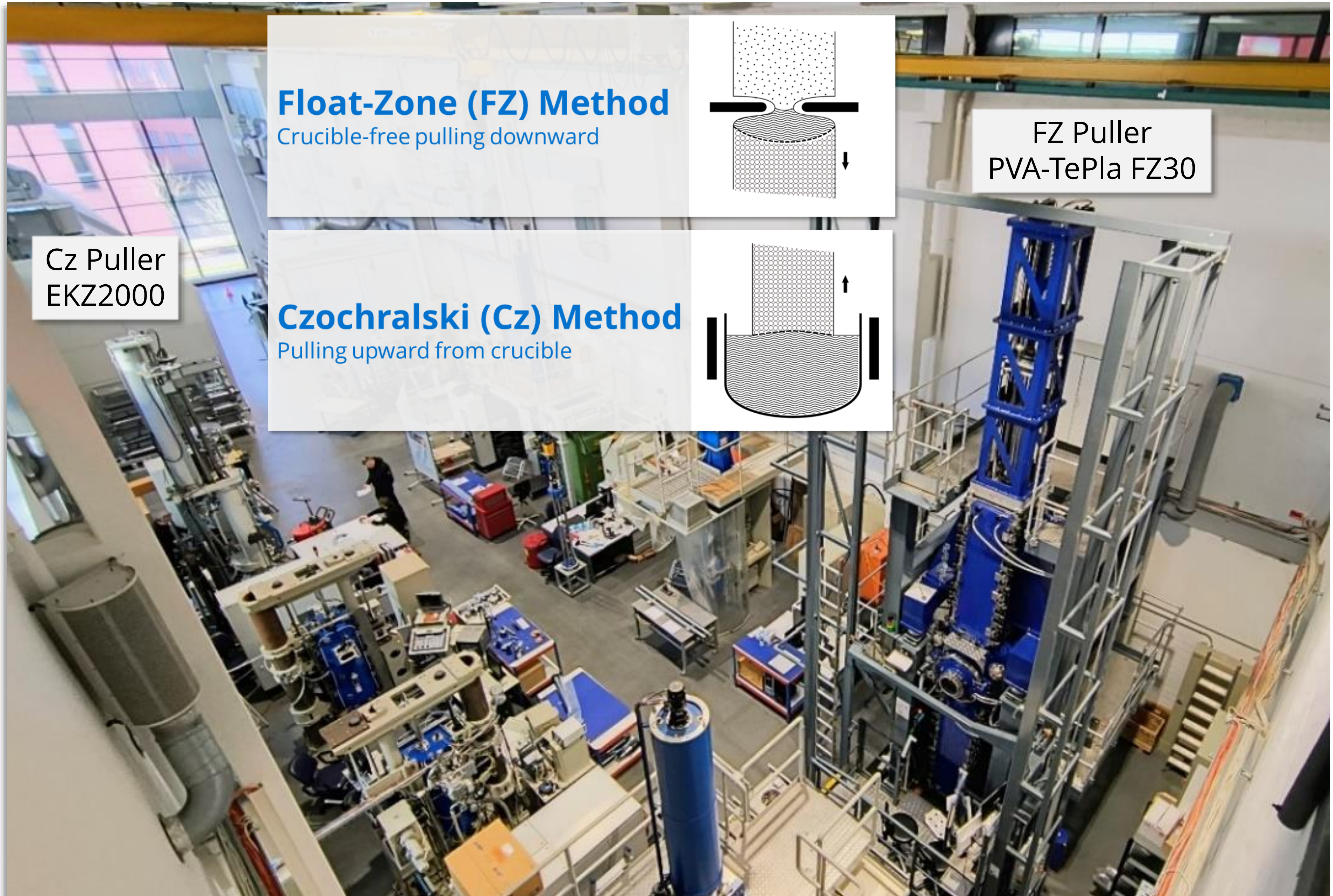
- Large-diameter > 45 cm
- Large Mass > 200 kg, thickness > 45 cm
- Optical transparency at 1550 nm
- low absorption (few 10^{-6}/cm for ITMs, 10^{-4}/cm for ETMs)
- Ultrahigh crystal purity (O,P,B, transition metals)
- No sources for optical scattering (dislocation-free single crystal)
- low mechanical loss at low temperatures



No established process can produce crystals that meet all these requirements

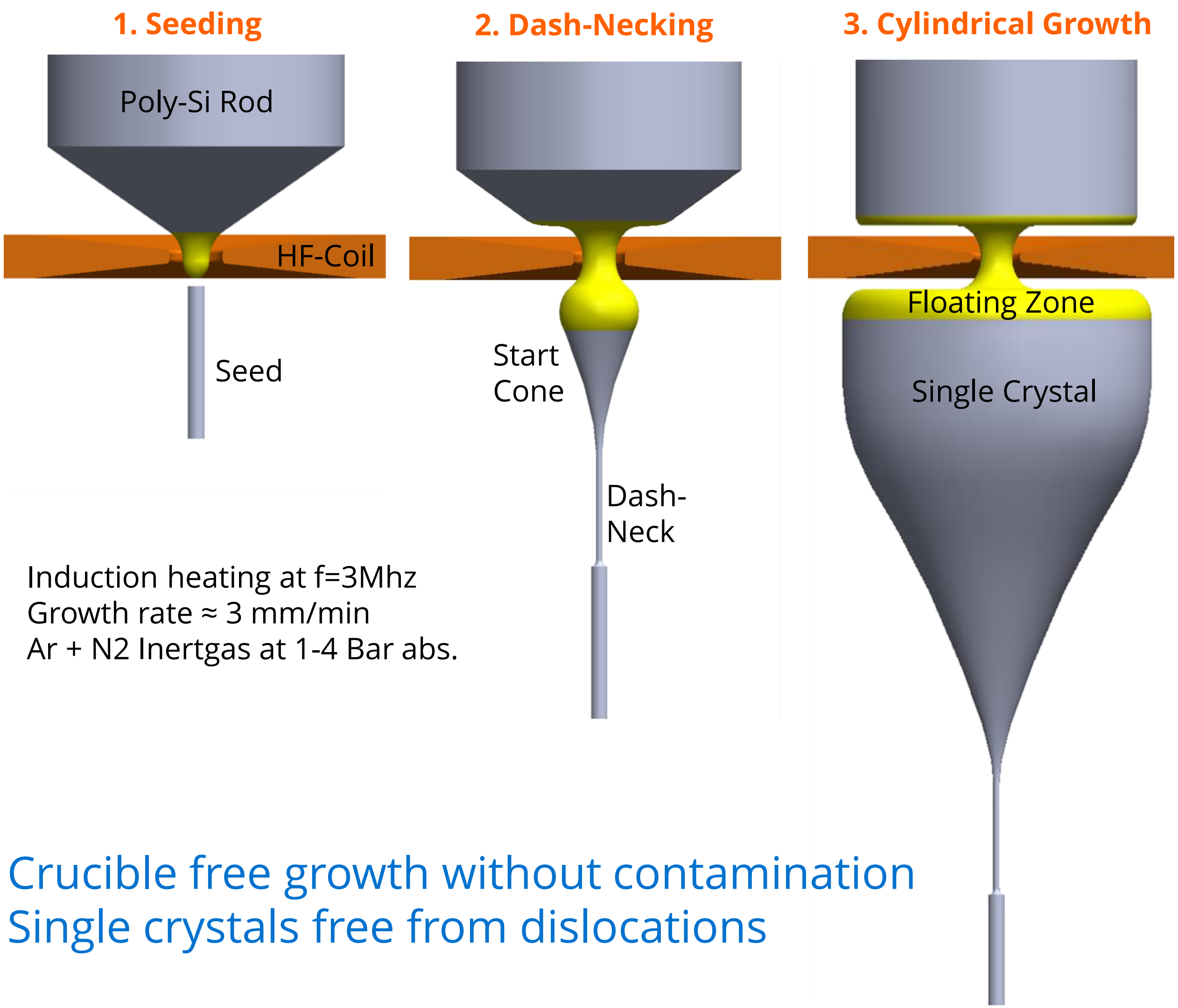
➡ **exploration of the development potential in crystal growth methods**

Silicon Crystal Growth Lab at IKZ



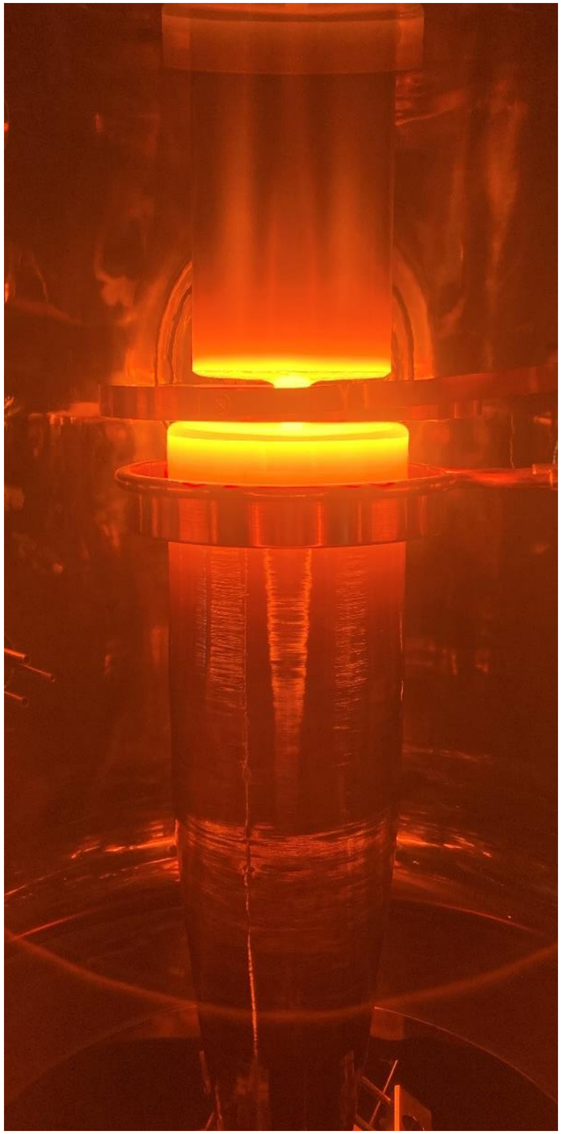
Growth of large-diameter FZ-Si crystals using the needle-eye technique

Process phases:



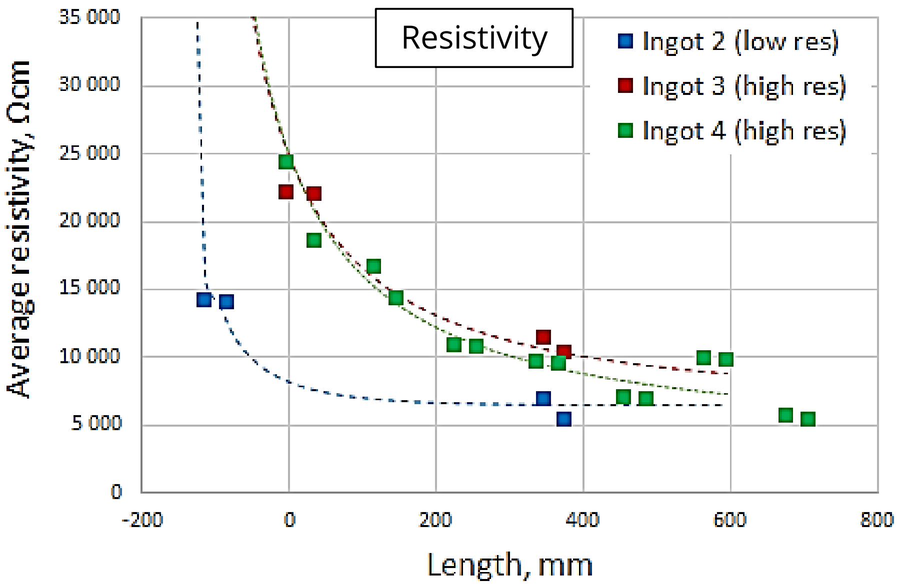
Induction heating at f=3Mhz
Growth rate ≈ 3 mm/min
Ar + N2 Inertgas at 1-4 Bar abs.

Crucible free growth without contamination
Single crystals free from dislocations



6 inch FZ crystal during growth

FZ is the only established method for large ultrahigh purity Si crystals



Specific resistivity along crystal length, measured on 6 inch crystals grown from different feed rod purity level at IKZ

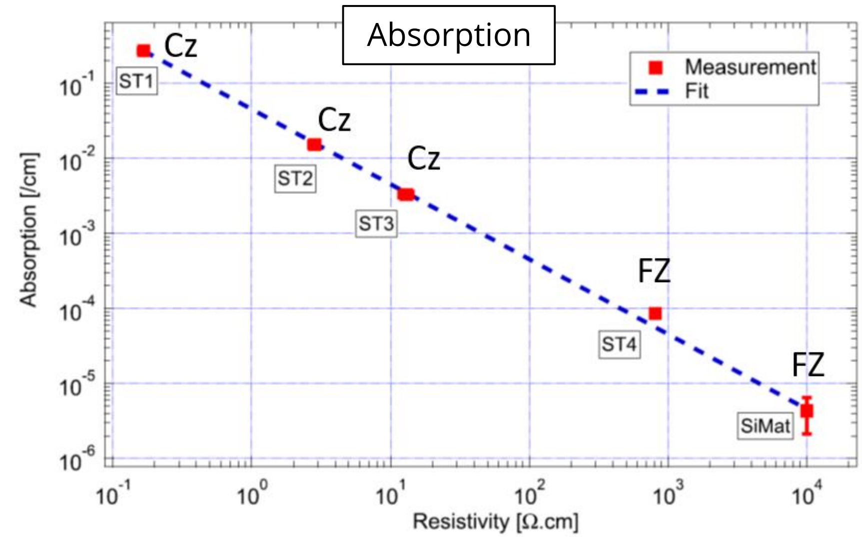


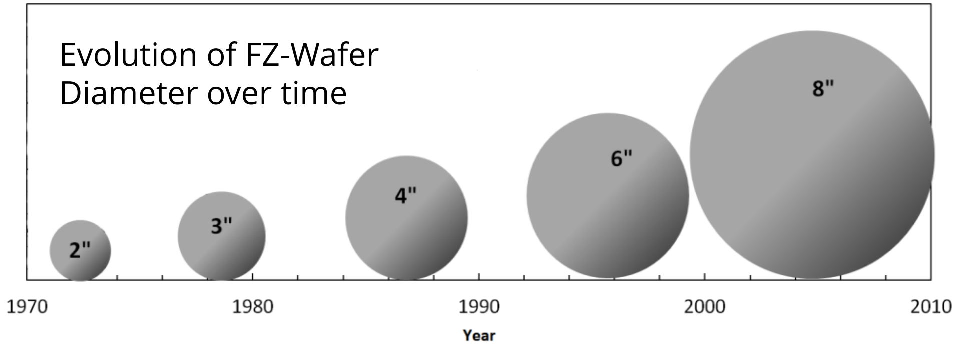
Fig. 3. Bulk optical absorption as a function of the resistivity for silicon material at 1550 nm. For the samples ST1 to ST4, the error bars are within the square data points.

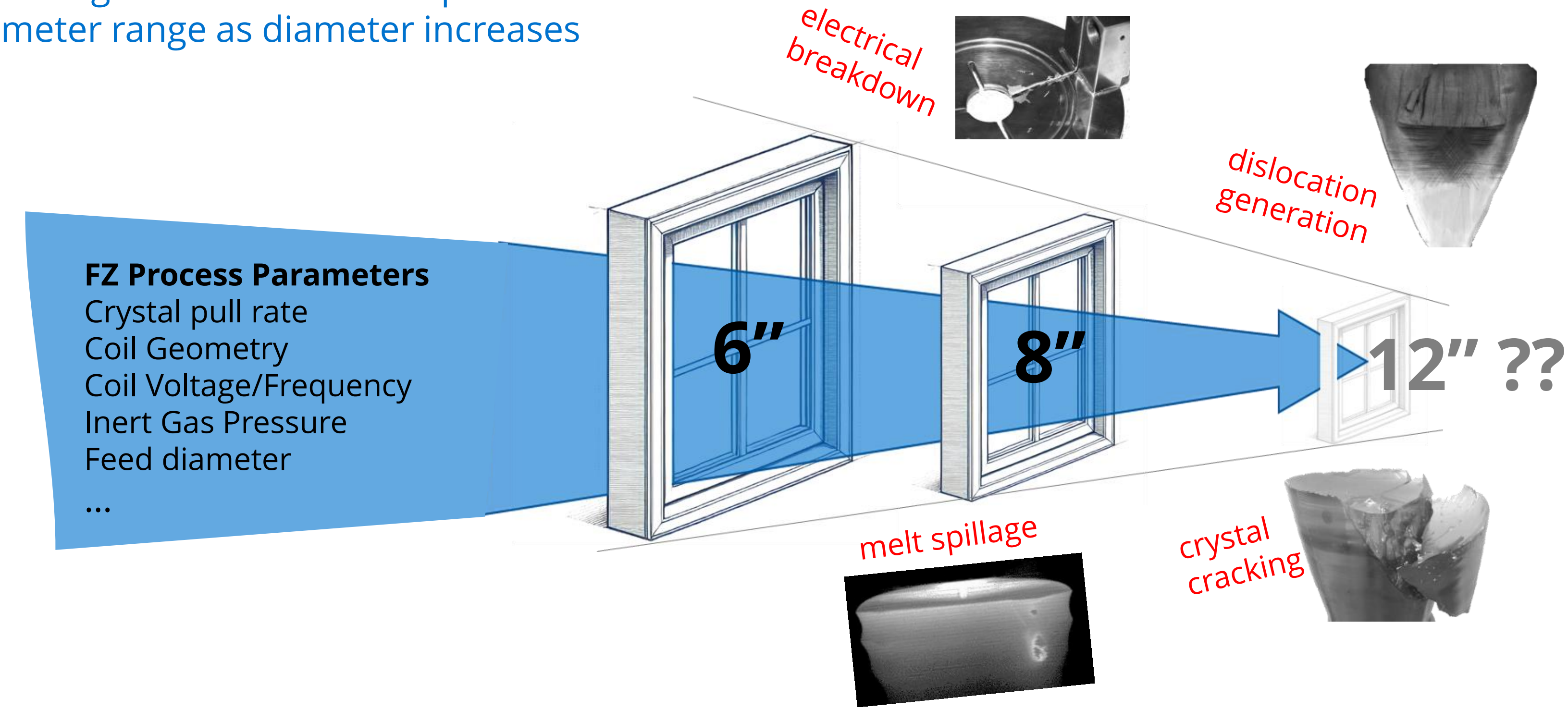
J. Degallaix et al, Opt. Lett. ***38****, 2047 (2013).*

➡ even higher purity in FZ-Si crystals can be achieved by using purer raw materials or by performing multiple FZ runs (impurity segregation effect)

FZ-Si Crystal Diameter Limitations

- 200 mm (8”) is the largest crystal diameter that can be achieved today
- Demand for FZ-Si crystals with larger diameter also in the Semiconductor industry
- Huge technological limitations in the industrial established FZ Process
- Decreasing window of feasible process parameter range as diameter increases





Float-Zone Method

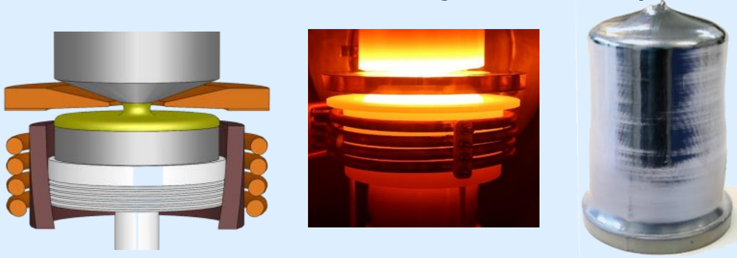
Novel crucible-free growth approaches

Silicon Granulate Crucible (Si-GC)

- Less diameter limitations (as in Cz)
- Dislocation-free growth
- No contamination during growth
- No high-purity raw material availabe

Dadzis et al. JEM 49 (2020)

Large-area seeding
with FZ Needle-eye technique



- no growth of start cone needed
- High purity achievable
- High dislocation density

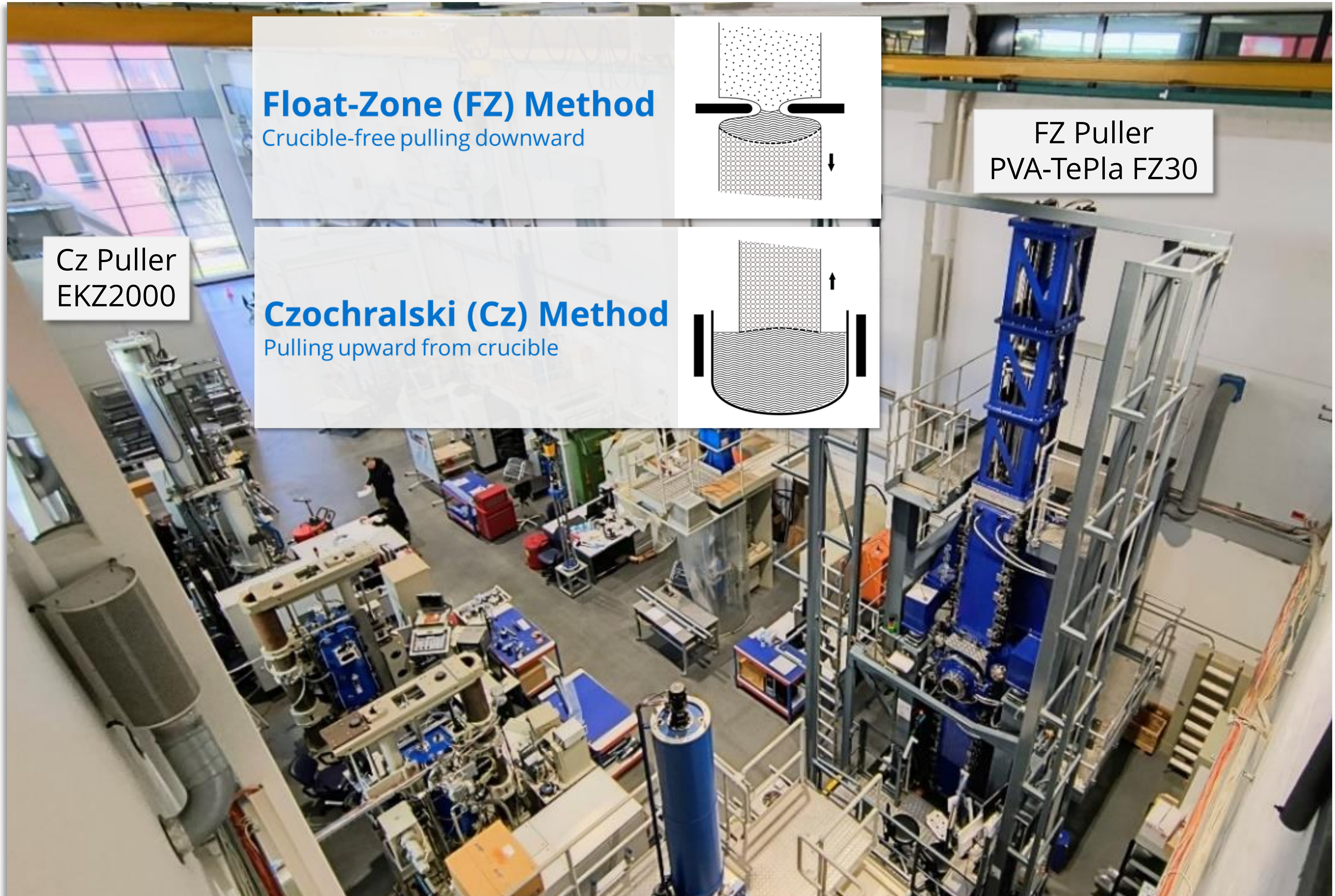
Menzel et al. JCG 515 (2019)

NeoGrowth
Large area seeding, radiation heating

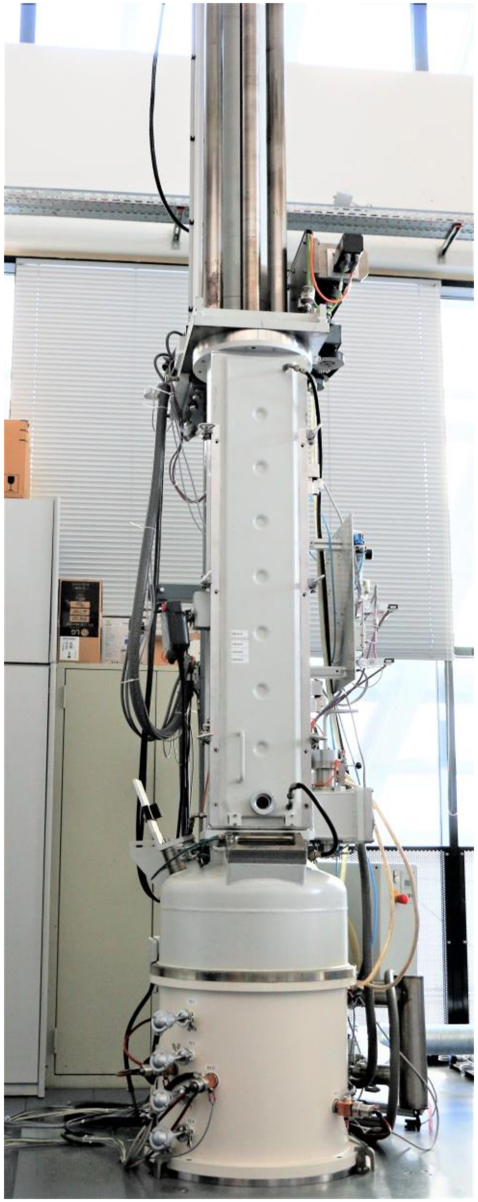
- Large-diameter 450 mm demonstrated
- Single crystal with low dislocation density
- not optimised for high purity

Stoddard et al. Prog. Photovolt. 26 (2018)

Silicon Crystal Growth Lab at IKZ

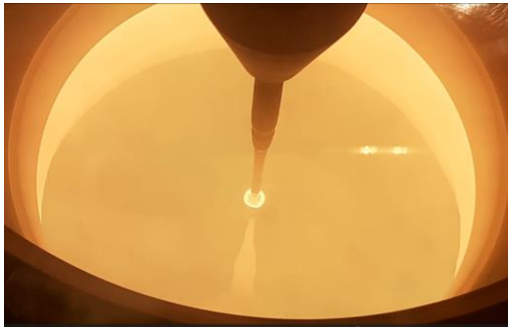


Czochralski Method

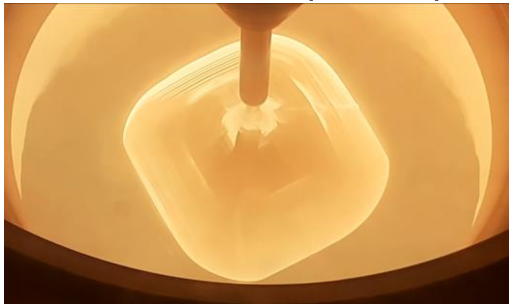


EKZ2000 at IKZ

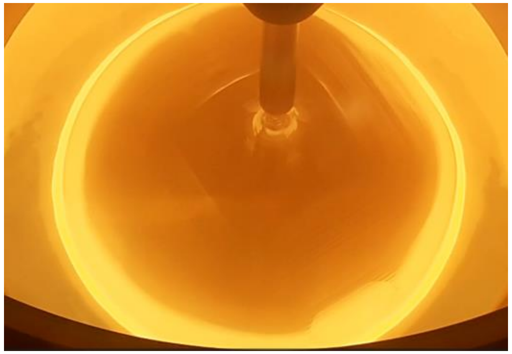
Seeding



Start cone (4 inch)



6 inch



Cz process phases

Typical Cz growth parameters at IKZ

- Crucible rotation: 8 rpm
- Crystal Rotation: -5
- Pulling rate : 1 mm/min
- Full melt level compensation
- Gas flow: 20 l/min
- Chamber pressure: 20 mbar Ar

Czochralski Method

- Less diameter limitations in Cz growth compared to FZ

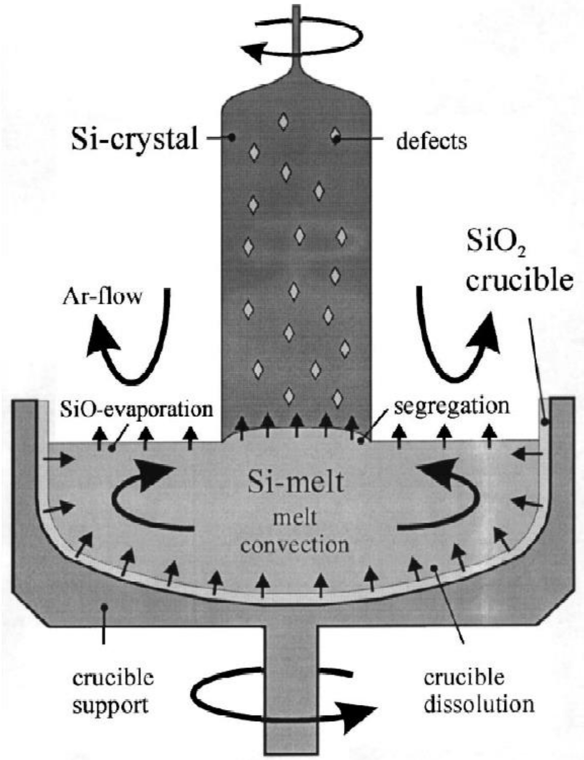
- 450 mm crystal diameter was already demonstrated in industry

- but Si crystal is contaminated during growth

- Impurities: O,C,P, B, transition metals...
- origin of impurities are the quartz crucible, crucible support, graphite heaters..

	FZ (6 inch)	Cz (6inch)
resistivity [Ωcm]	10.000	3.5
O_i-concentration [cm^{-3}]	< 1 x 10^{16}	< 7 x 10^{17}
C_s-concentration [cm^{-3}]	< 1 x 10^{16}	9 x 10^{16}
lifetime [µs] (unpassivated surface)	> 1000	ca. 43

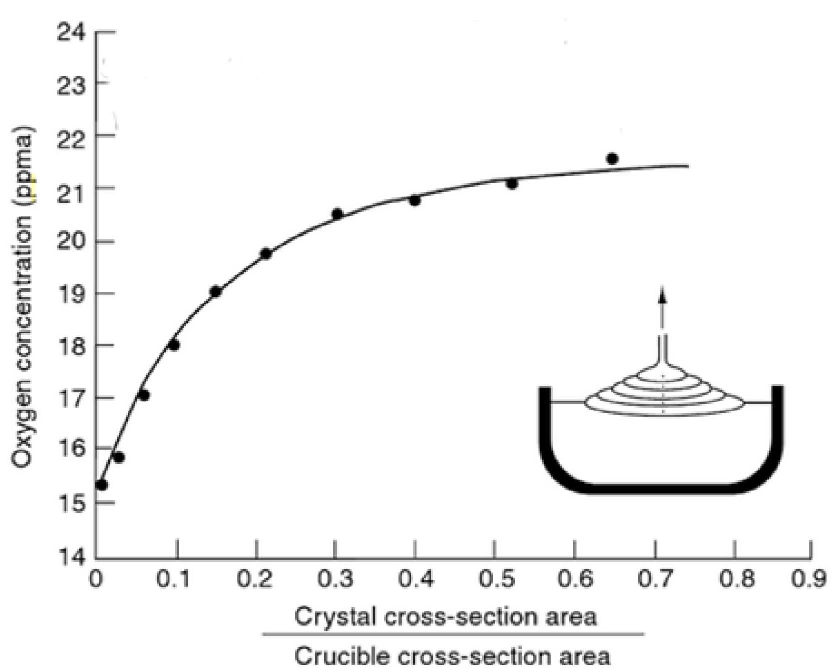
Typical properties of Si crystals grown at IKZ



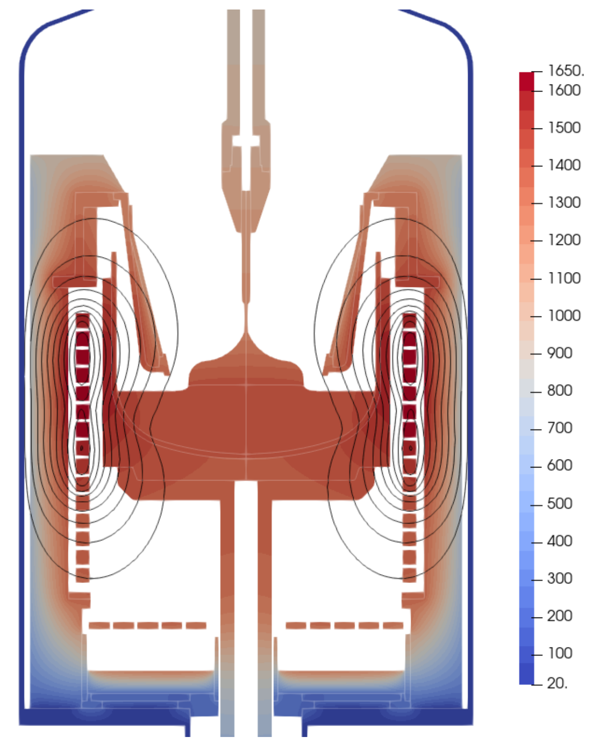
picture: G. Müller, Microelectronic Engineering 45 (1999) 135

Reducing impurity concentration in Cz-Si crystals

- Optimize crucible material
- Crucible coating (e.g. Si_3N_4)
- Crystal and crucible rotation
- Magnetic fields (e.g. KRISTMAG)
- Ratio free melt surface/crystal diameter
- Inert gas pressure and gas flow



Wang et al. JCG 576 (2021)



KRISTMAG®
heater magnet module
installed at IKZ

Model by Arved Wintzer (IKZ)

Conclusions

- **FZ offers highest purity but faces strong limitations in crystal diameter**
- **New crucible-free approaches could not meet FZ-Si quality yet**
- **Less diameter limitations in Cz but contamination during growth**
- **Some potential to improve purity in Cz-Si crystals**

⇨ Prioritization of relevant crystal properties for best mirror performance within an iterative development loop including test measurements