

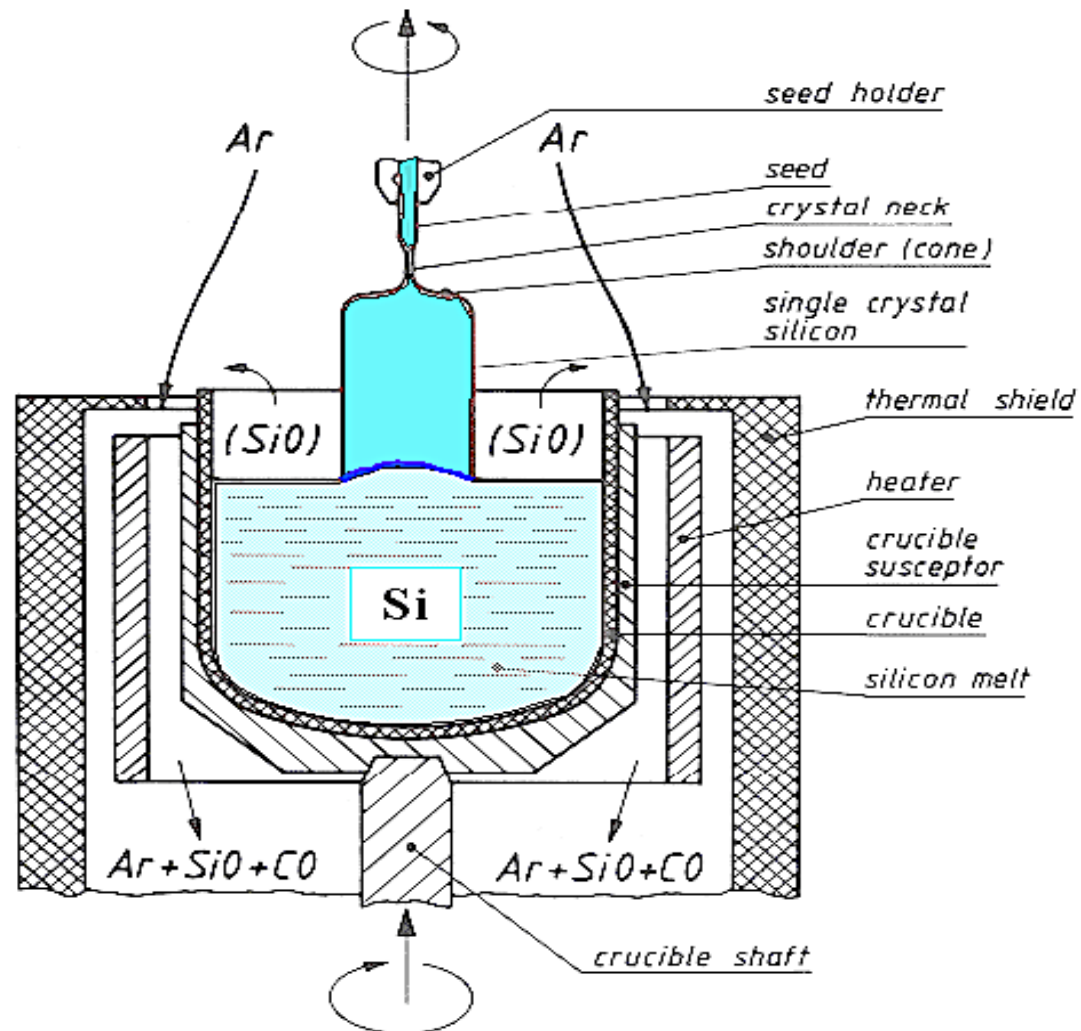
# CZOCHEWSKI METHOD

Growth of silicon Ingot(\*silicon wafer)

## Czochralski Method ...

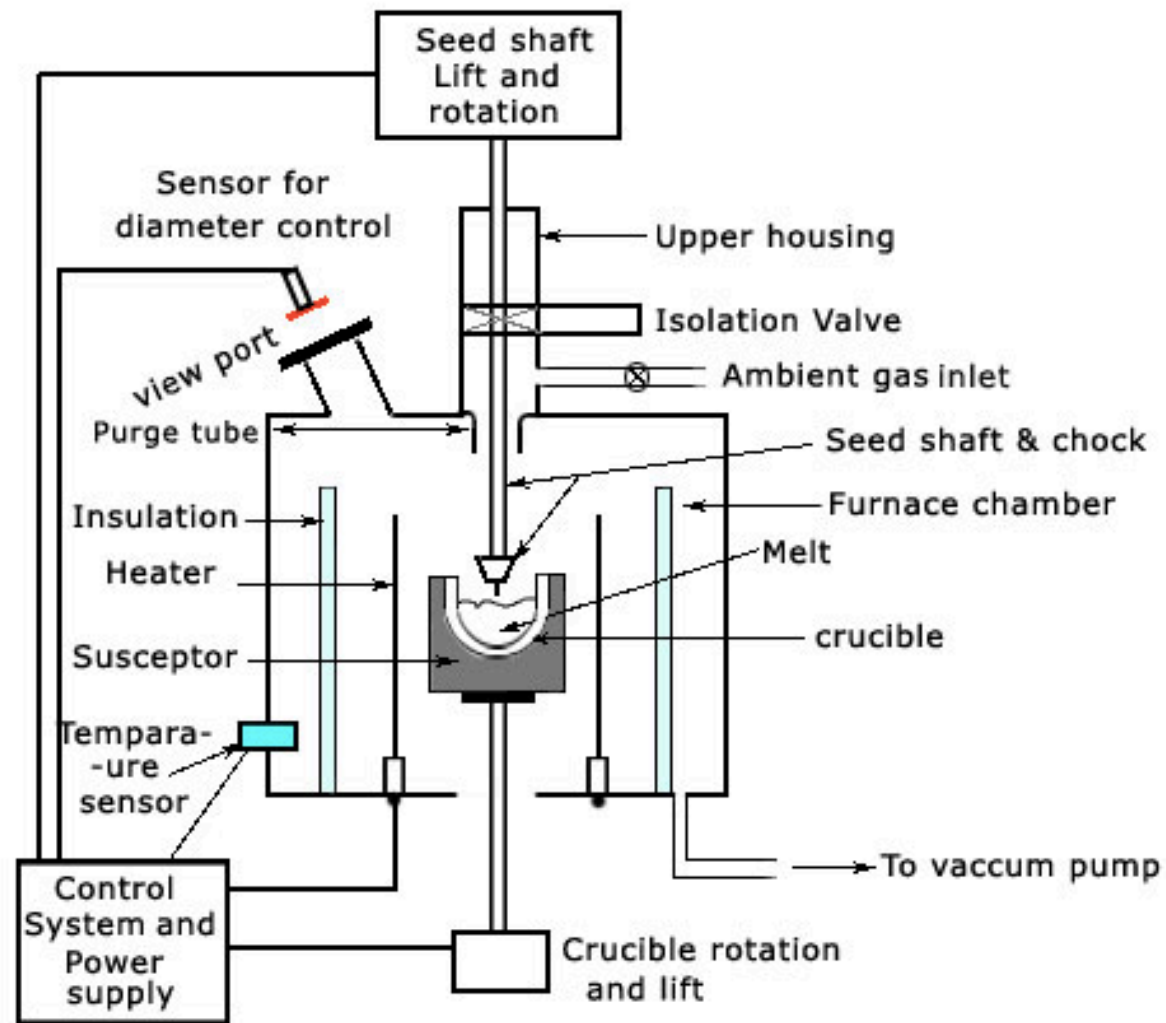
- This process uses Czochralski Crystal Grower.
- Poly-EGS is melted in a quartz-lined crucible at about  $1412^{\circ}\text{C}$ .
- Seed crystal is introduced into the molten poly-EGS to begin crystallization.
- Seed pulled slowly, allowing the crystal to grow on to it. Simultaneously, the crystal is rotated slowly to give slight stirring of the melt to prevent inhomogeneous solidification.
- Then Ingot is cooled at a very slower rate.

## Beginning of crystal growth





## A practical silicon crystal growing apparatus



- Czochralski system is also known as Liquid-Solid monocomponent System
- Four subsystem of CZ process

**1. Furnace-** It consist of quartz envelope. This quartz envelope may be water cooled. Crucible is a cup in which EGS is going to place and usually it is made up of quartz (Quartz is nothing but Silicon dioxide). So there is less chance of contamination.

After the crystal growth when you are cooling down the system, then because of the thermal mismatch usually the quartz crucible is going to crack. So, we cannot reuse the crucible and this quartz crucible is usually placed inside a graphite susceptor.

A **susceptor** is, we can view it as an outer jacket. That is, I have a bigger cup of graphite in which quartz cup is placed. The quartz cup is called the **crucible**, the graphite cup is called the susceptor and the graphite susceptor is placed on a heater.

**2. Crystal Pulling mechanism:** A pull rod is used and at the end of the pull rod, a small seed crystal is fixed in a chuck. The pull rod is pulled up during the crystal growth.

- The two parameter for growth process: i) The Pull rate  
ii) Crystal rotation



## PULL RATE AND GROWTH RATE

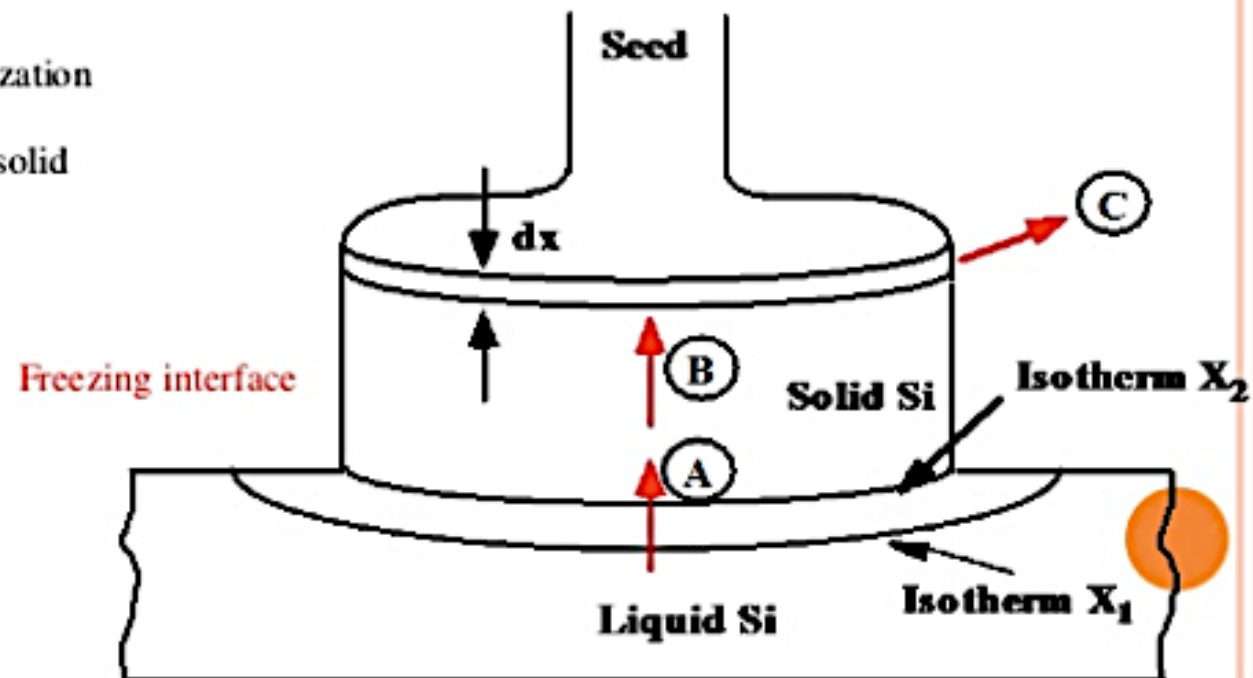
- Pull rate varies inversely with diameter.
- Growth rate depends upon the no. of sites on the face of crystal and heat transfer at the interface and can be greater than pull rate and even be negative.

A=Heat of crystallization

B=conduction in solid

C=Radiation

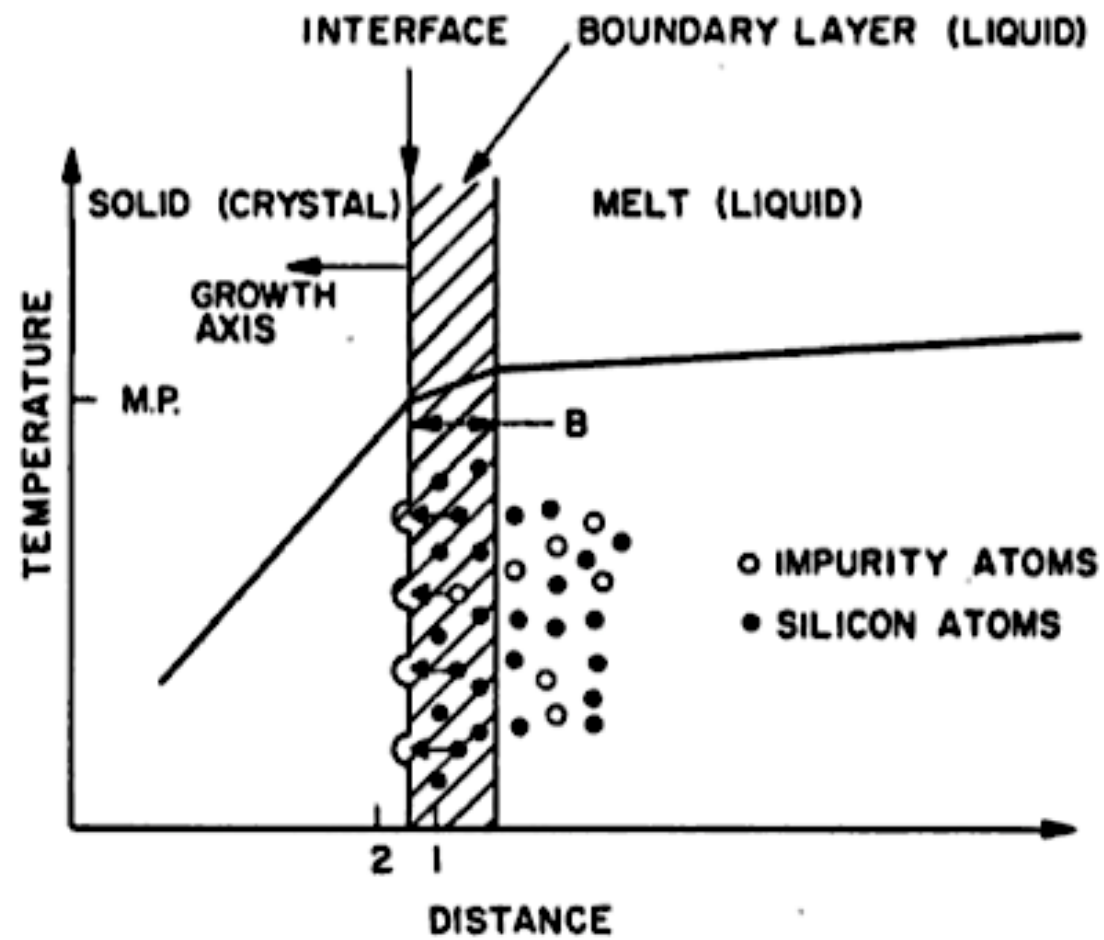
Liquid to solid  
→ HEAT ↑



$$L \frac{dm}{dt} + K_L \frac{dT}{dx_1} A_1 = K_S \frac{dT}{dx_2} A_2$$

- where  $L$  is the latent heat of solidification or latent heat of fusion
- $\frac{dm}{dt}$  is mass solidification rate
- $K_L$  is the thermal conductivity of the liquid
- $K_S$  is the thermal conductivity of the solid
- $\frac{dT}{dx_1}$  is the thermal gradient in the melt
- $\frac{dT}{dx_2}$  is the thermal gradient in the solid





- $L \frac{dm}{dt} = L \cdot A \cdot \rho \frac{dx}{dt}$

- $\frac{dm}{dt}$  rate of change in volume times the density.

- Consider thermal gradient in melt is zero, then

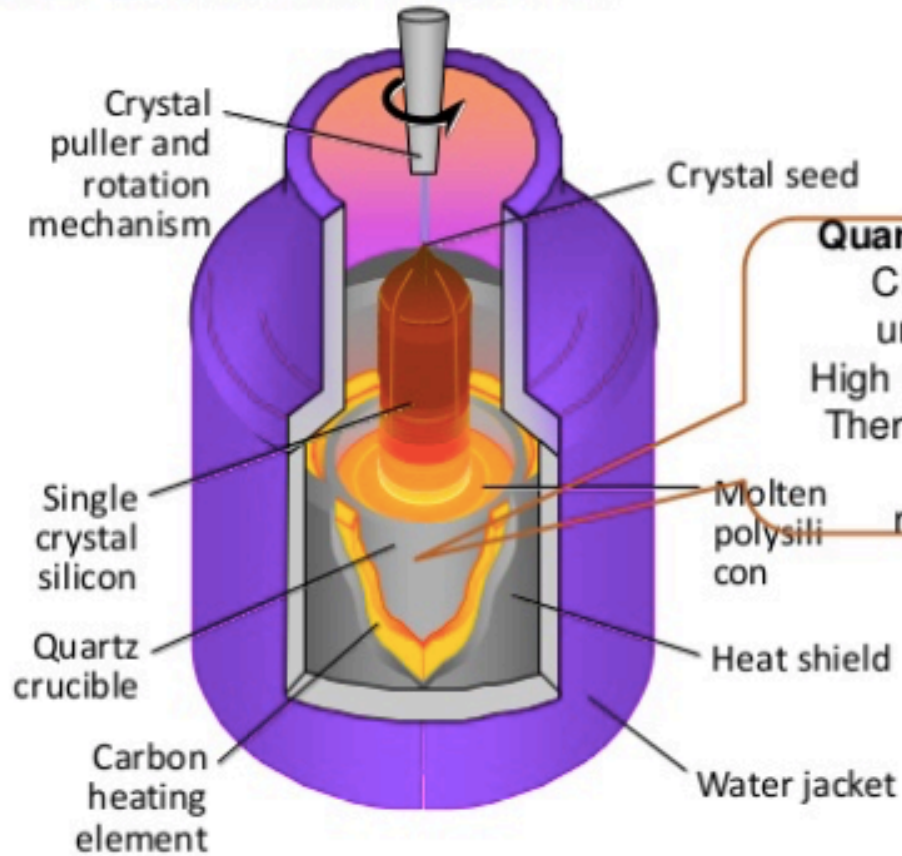
- $L \frac{dm}{dt} = K_S \frac{dt}{dx} A_2$

- $\frac{dx}{dt}$  is the pull rate. This is the rate at which we are pulling the crystal.

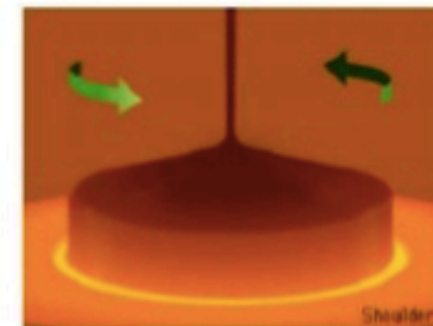
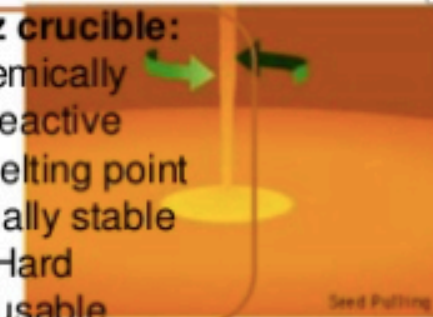
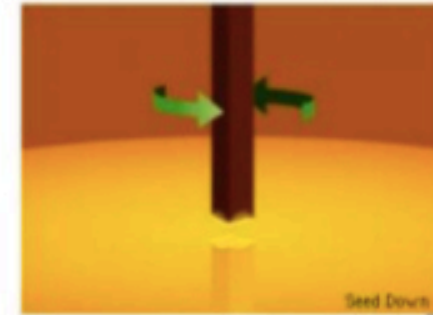
- $\frac{dx}{dt} = \frac{K_S}{L \cdot \rho} \frac{dt/dx}{2}$  This is maximum attainable pull rate.

- Practically the pull rate is less than the maximum pull rate

# CZ CRYSTAL PULLER



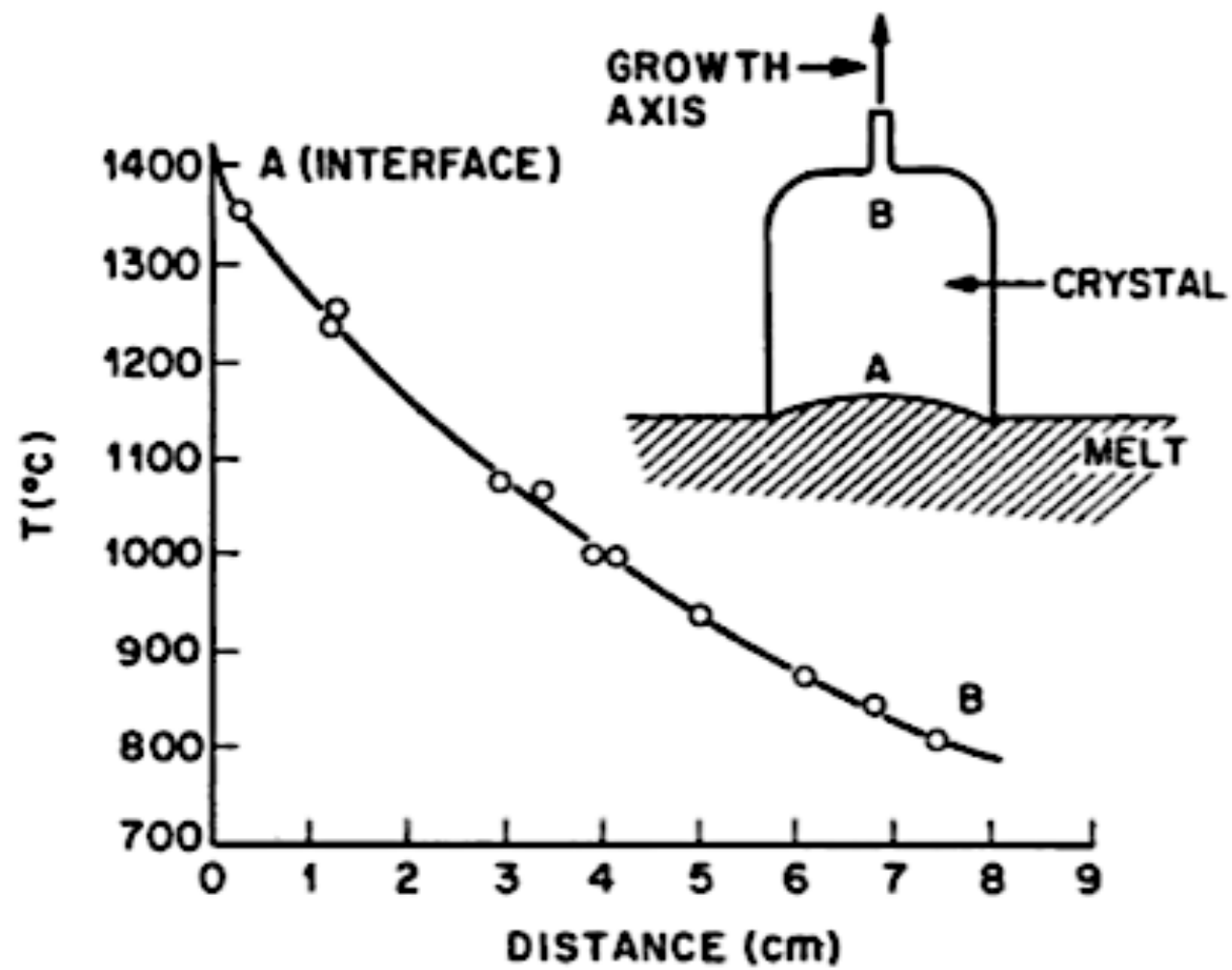
**Quartz crucible:**  
Chemically unreactive  
High melting point  
Thermally stable  
Hard  
reusable





- What happens if there will be a small change in the temperature of the melt. Because we are pulling out the crystal, the thermal conditions, they are continuously changing. So, in a practical system actually, a feedback control is used. That feedback control is used to supply the heat to the melt, so that the temperature is maintained.
- this heat loss, the heat needed to be supplied in the system, heat input minus heat output, the heat loss is almost entirely taken off by the latent heat of solidification

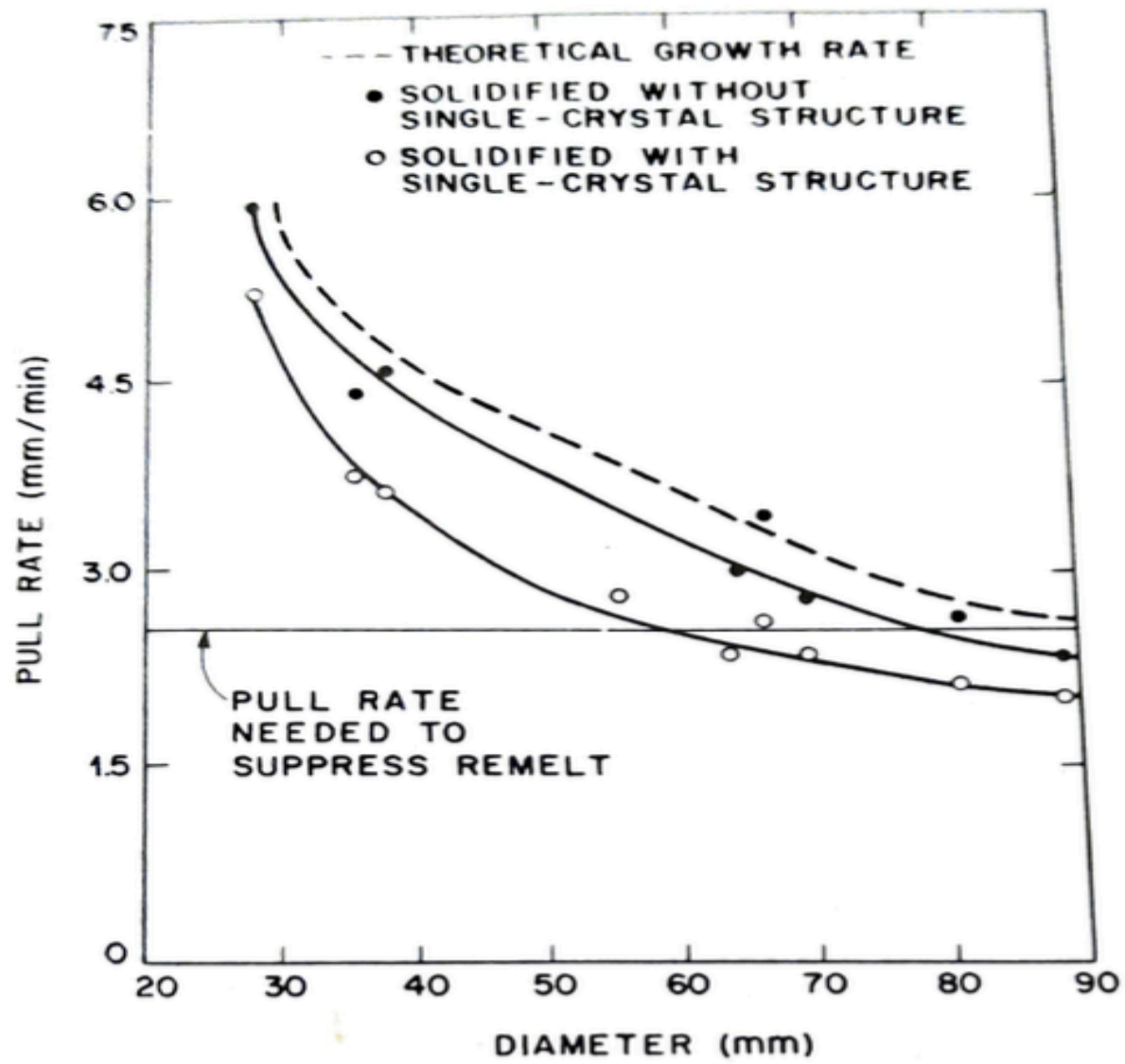
$$\begin{aligned}
 \text{○ } H_i - H_o &= L \frac{dm}{dt} & H_i - \text{Heat in,} \quad H_o - \text{Heat out} \\
 &= L \cdot A \cdot \rho \frac{dx}{dt}
 \end{aligned}$$



- Latent heat and the density, these are the material properties. So, the only variables are the area and the pull rate. Therefore the pull rate is going to vary inversely with the area for a given  $H_i - H_o$
- In other words, the larger your crystal, the smaller should be the pull rate.
- The defect finally in the crystal, it is going to depend quite significantly on the pull rate.
- The thermal point defects will try to come together into dislocation loops. This happens around  $950^{\circ}\text{C}$ .



- If we increase the pull rate we can avoid this thermal defects
- For a smaller diameter crystal, we can afford to use a large pull rate and when we use a large pull rate, the problem of this dislocation forming is less severe. That is the reason why 10 years back, the silicon crystals that were grown were almost exclusively 2 inch or 3 inch diameter;
- At the beginning of the integrated circuit revolution, the silicon wafers available were only of 2 inch or 3 inch diameter. As the system became perfect, more perfect, technologically more advance was made, it was, it became possible to grow large diameter crystal with low defect density.



## The dopant incorporation during crystal growth

- There is a different solubility in the crystal and in the melt.
- this difference is given by the segregation coefficient,
- Segregation coefficient  $K_0 = \frac{C_S}{C_L}$
- $C_S$  = Solid solubility in solid silicon
- $C_L$  = Equilibrium solubility in the melt.



- For most of the impurities  $K_0 < 1$ . It means solid solubility is less than equilibrium solubility in melt.
- Means as we grow the crystal, these impurities are preferentially left inside the melt, they do not come into the solid. Impurities are rejected by solid, they prefer to be in melt.
- As we are drawing the crystal, the melt is becoming progressively richer in the impurity.
- So, when you grow a crystal, we will find that the doping concentration will vary from one end to the other.
- The top end will have relatively less dopant concentration and the bottom end will be relatively more doped.

- Above case is not true for oxygen impurity. For oxygen  $K_0 > 1$ .
- Means it will try to come preferentially into the crystal.
- We are using quartz crucible, we are using quartz envelopes. Although the envelope is water cooled, but the crucible is heated. So, there is a possibility of oxygen incorporation during the crystal growth.  $O_2$  act as interstitial impurity and act as donor impurity in silicon crystal
- One possible remedy to reduce this is, we cut the grown ingot into wafers that is thin slices and then we heat these slices to temperature above  $600^{\circ}\text{C}$  and then, so when we heat it above  $600^{\circ}\text{C}$ , these complexes are dissolved, because the formation temperature is only around  $450$  to  $500^{\circ}\text{C}$ .



**3. Ambient control:** The ambient is very important in a crystal growth system. We are using a graphite susceptor and graphite heaters. Therefore, there must not be any oxygen inside the system.

- The whole thing will just go up in smoke. It will just form carbon dioxide and there will be no susceptor, no heater, nothing left.
- We required high purity single crystal silicon. If oxygen is present then it is going to react with silicon to form silicon dioxide. We cannot allow that, it will increase level of impurity in silicon. Therefore, we need to take care of oxygen.



- **Growth** must be conducted in an inert gas or vacuum. So that the hot graphite parts must be protected from oxygen and the gas around process should not react with the molten silicon
- First of all evacuate the quartz chamber and then fill it up with an inert ambient - argon or helium or nitrogen. Maintain the atmospheric pressure or sometimes even reduced pressure.

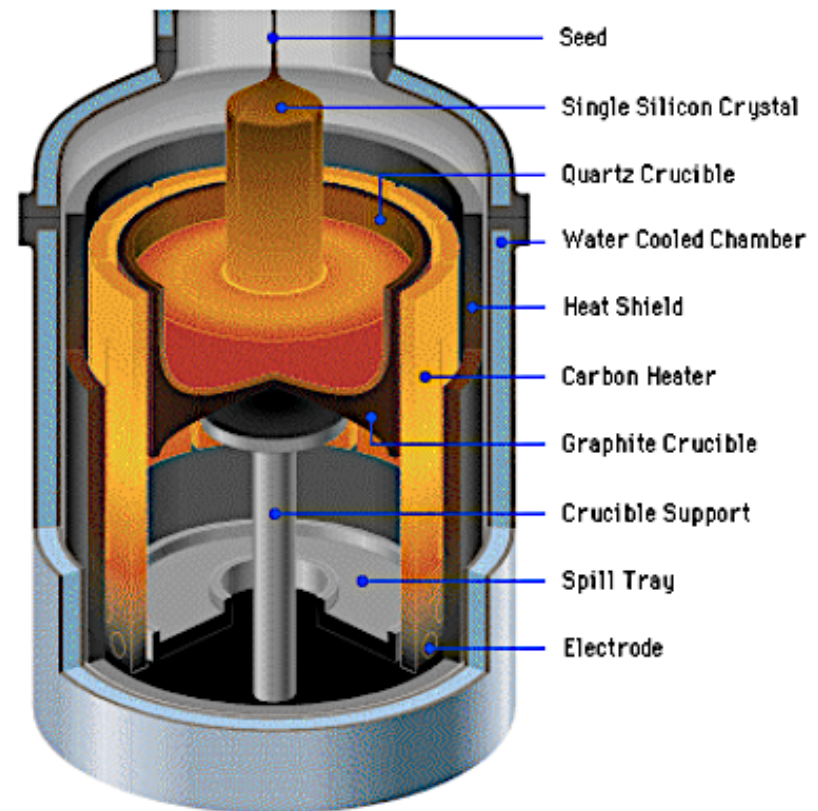
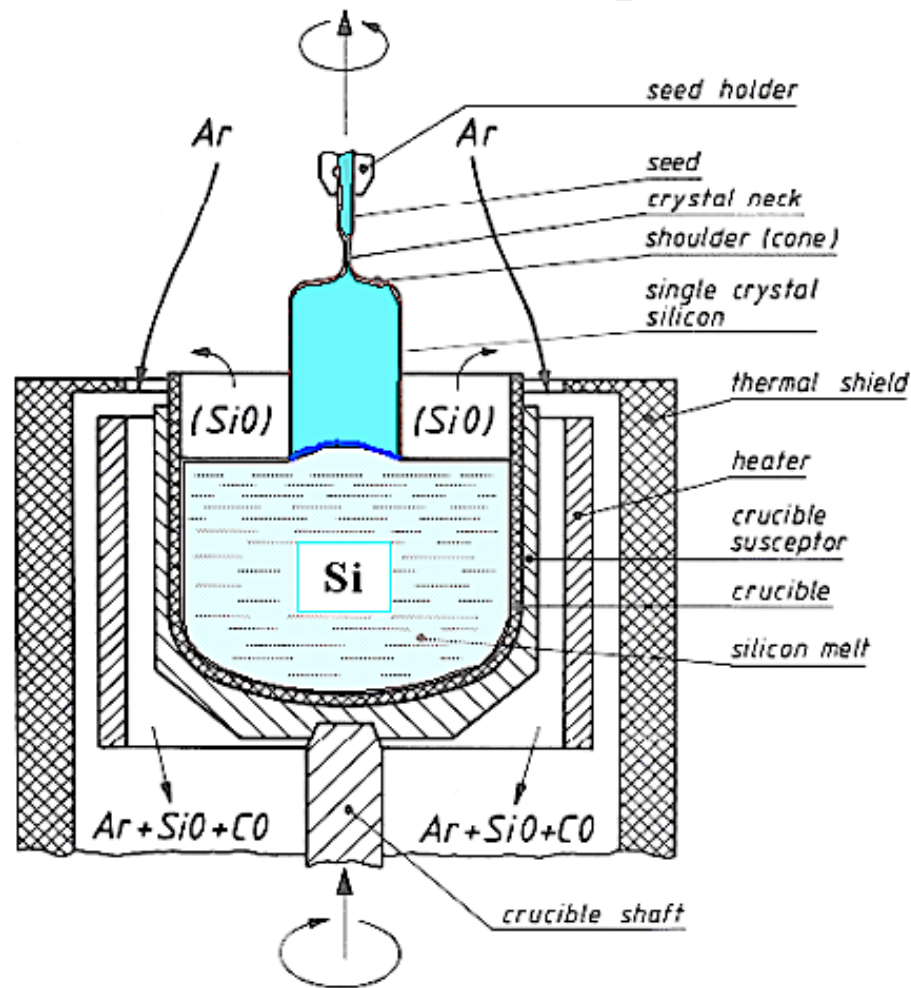
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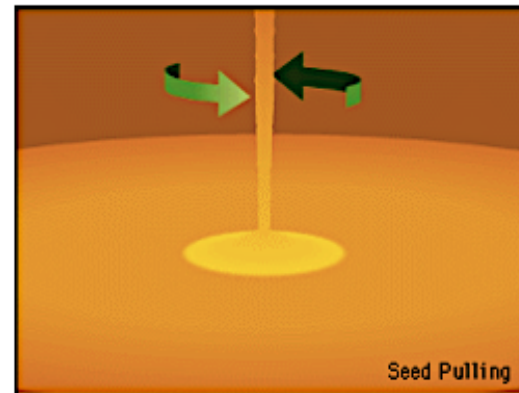
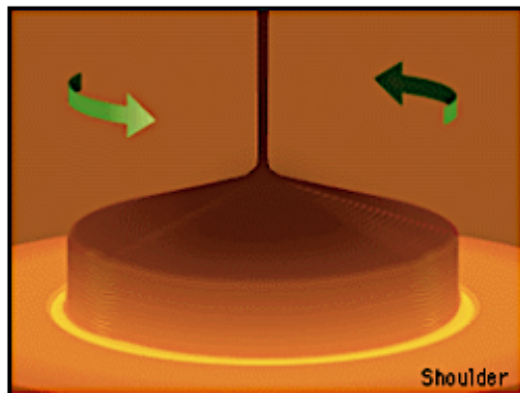
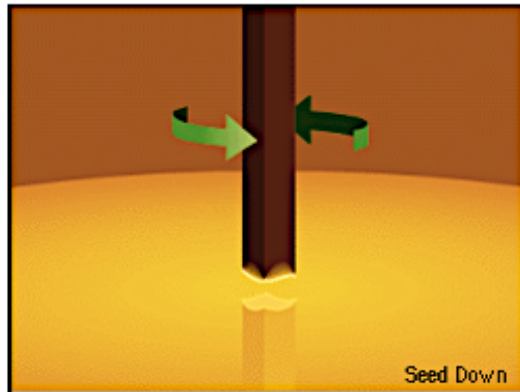
## 4. Control system

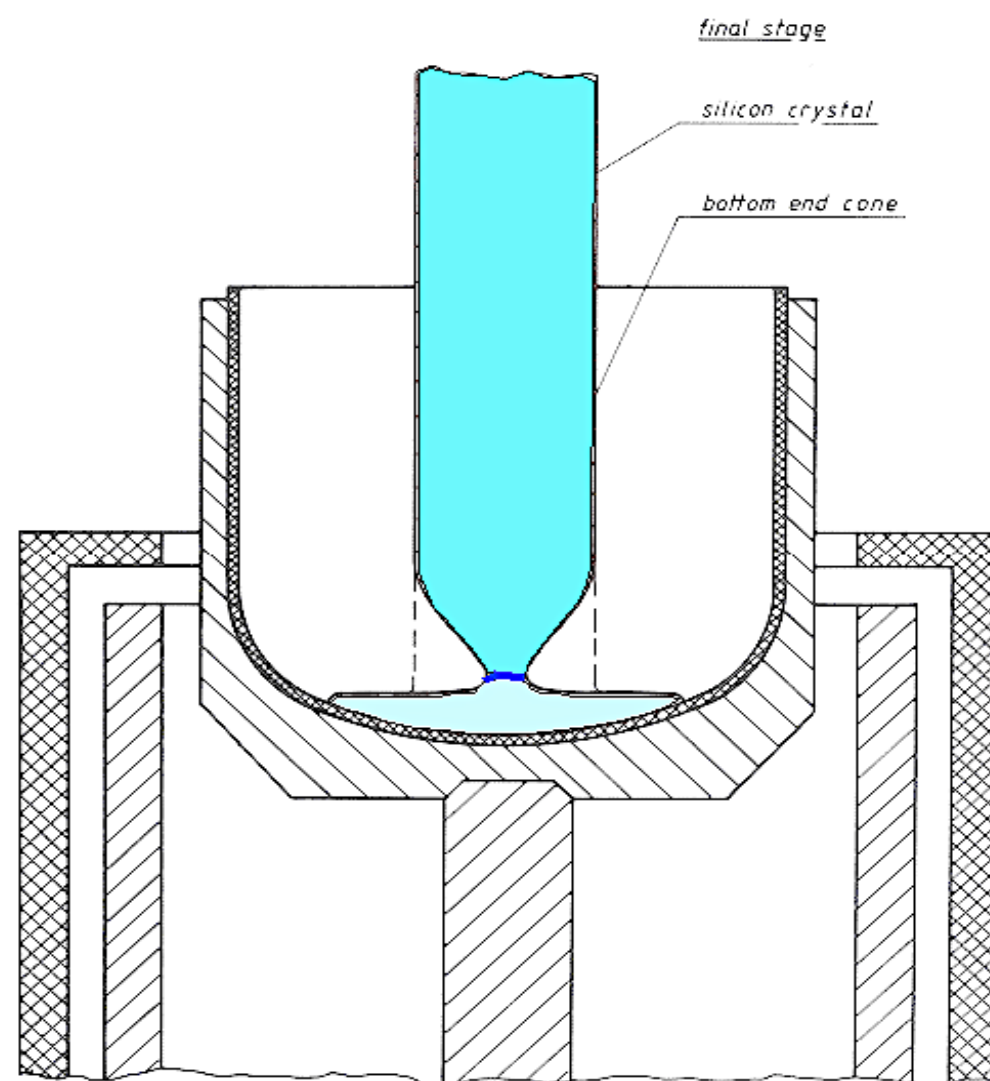


# Czochralski Crystal Grower....



# The Process...







- Take care to keep impurities out - do it in a clean room - and use hyperpure silica for your crucible.
- Make sure that the inside of the machine is very clean too and that the gas flow
- the gas you introduce but also the SiO coming from the molten Si because parts of the crucible dissolve
- Dissolve the Si in the crucible and keep its temperature close to the melting point.

- Since you cannot avoid temperature gradients in the crucible, there will be some *convection* in the liquid **Si**. You may want to suppress this by big magnetic fields.
- Insert your *seed crystal*, adjust the temperature to "just right", and start withdrawing the seed crystal.
- For homogeneity, *rotate* the seed crystal and the crucible.
- First pull *rather fast* - the diameter of the growing crystal will decrease to a few mm.
- Now decrease the growth rate - the crystal diameter will increase