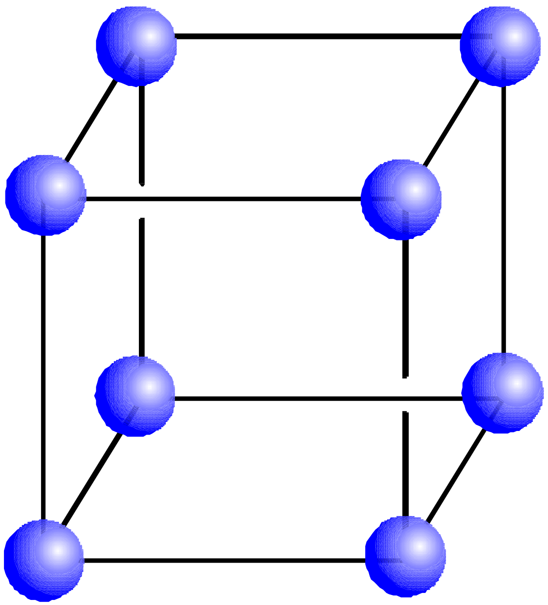
Hartmut S. Leipner

1. Crystal growth and defects



1.5 Defect formation

Misfit dislocations

Lattice constants

$a + \Delta a$

a

Misfit dislocations and growth

- Misfit between regions of different compositions (impurities, dopants) resulting in a change in the lattice constant
- Misfit at boundaries of growth pyramids or facets during non-planar growth

- Simple case of constant concentration gradient gives two perpendicular sets of edge dislocations with a density

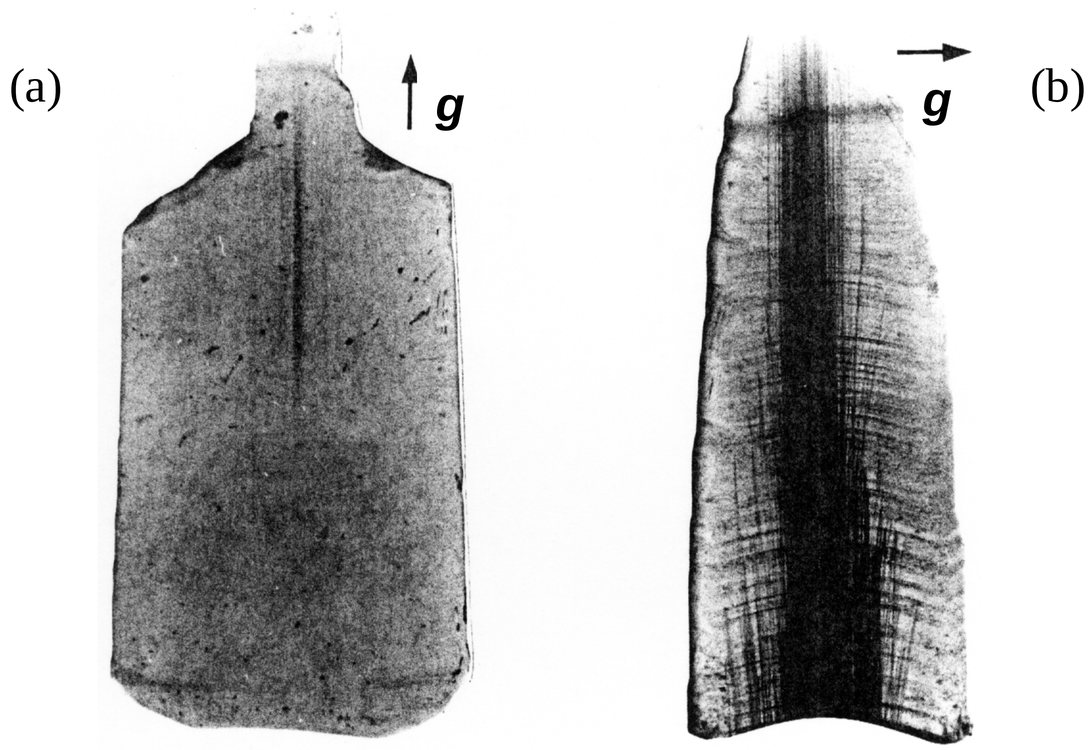
$$\rho = \frac{\alpha}{b} \operatorname{grad} c$$

(α linear expansion coefficient related to the concentration *c*,
b magnitude of the Burgers vector)

In-grown dislocations

- Propagation of dislocation during growth: a dislocation ending on the growth face will proceed with the growing crystal
- Spiral growth connected with screw dislocations
- Sources of dislocations: Inclusions, gas bubbles, precipitates
- Grown-in dislocations frequently found in non-crystallographic directions, which depend on the growth direction
- Post-growth movement of dislocations leads to irregular dislocation networks, cell structure

Propagation of dislocations



X-ray topography of grown-in dislocations. *a*) Propagation of dislocations from the seed in a Czochralski benzophenone crystal. *b*) Czochralski salol containing tiny gas bubbles and numerous dislocations starting from them. The length of the imaged crystals is about 46 mm, the thickness 1.2 mm. ***g*** is the diffraction vector; CuKα radiation.

[Klapper 1996]

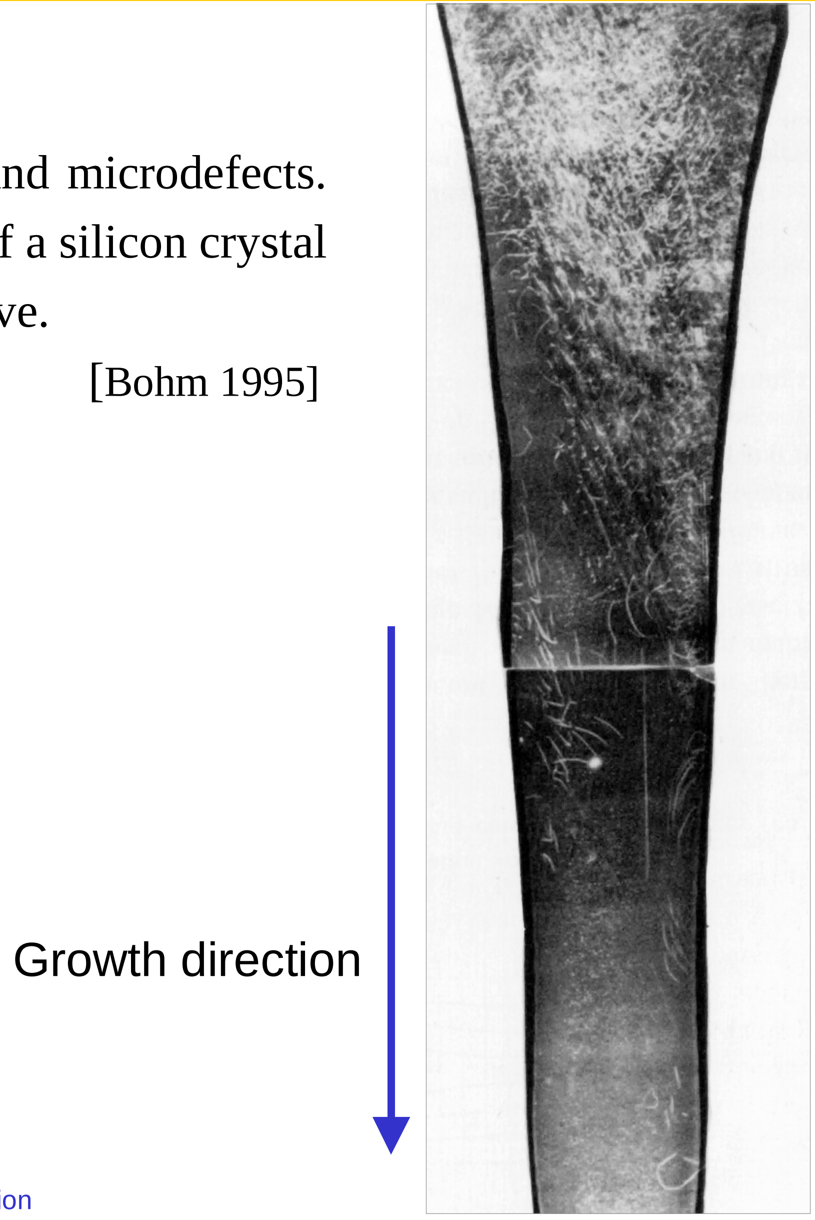
Crystals with a low dislocation density

- Start of the growth at the seed very critical and a high density of extended defects (dislocations, small-angle grain boundaries) is formed
- "Neck-and-shoulder" technique
- Remaining dislocation density usually 10^2 to 10^3 cm^{-2}
- Dislocation-free Si as a routine technology (avoid new formation of dislocations – no foreign phases, stress-free growth!)
- Si not defect-free, in addition to point defects it contains microdefects (*e.g.* swirls)

Extension of dislocations from the seed

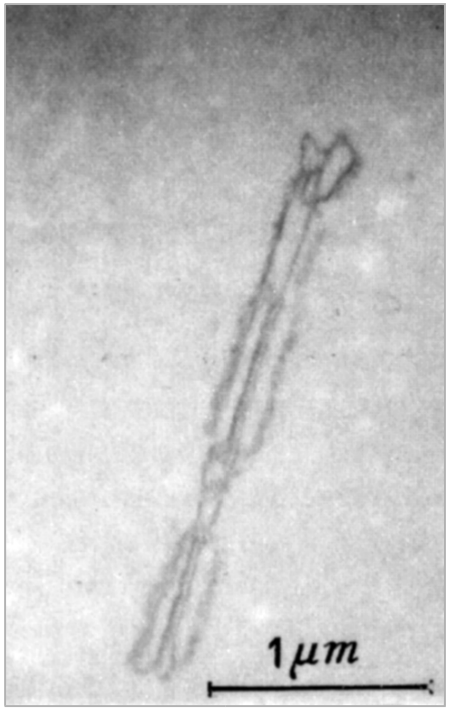
X-ray topography of dislocations and microdefects. A cross section of the neck region of a silicon crystal is shown. The seed was located above.

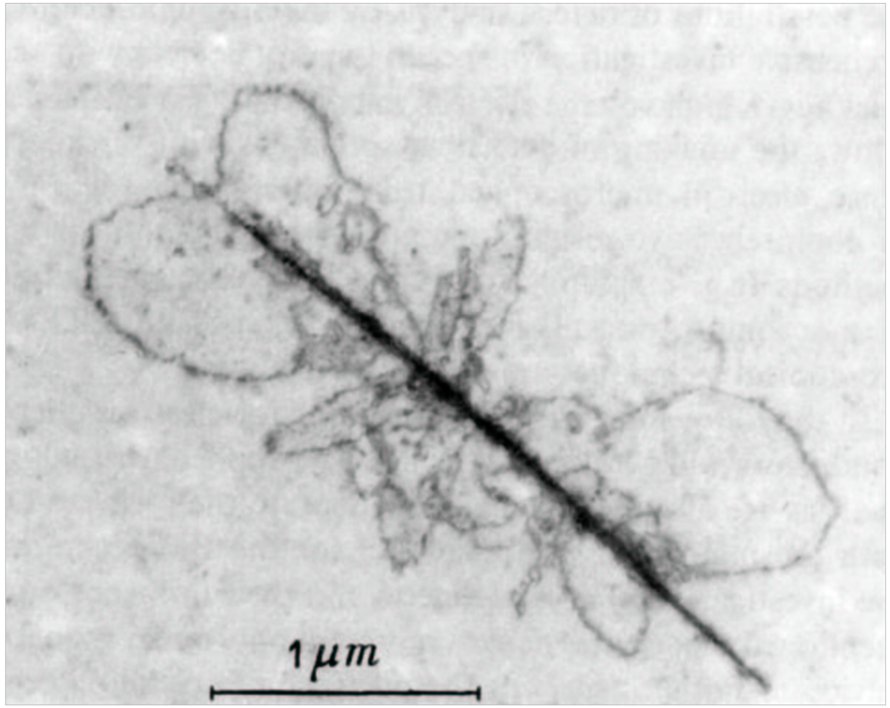
[Bohm 1995]



Microdefects in dislocation-free silicon

a)





b)

a) A swirl in floating-zone material, *b*) microdefect in Czochralski silicon.
TEM bright field, 1 MV.

[Gleichmann *et al.* 1987]

Condensation of point defects

Dislocation formation by condensation of vacancies

Vacancy loop [Wilke:88]

Density of loops

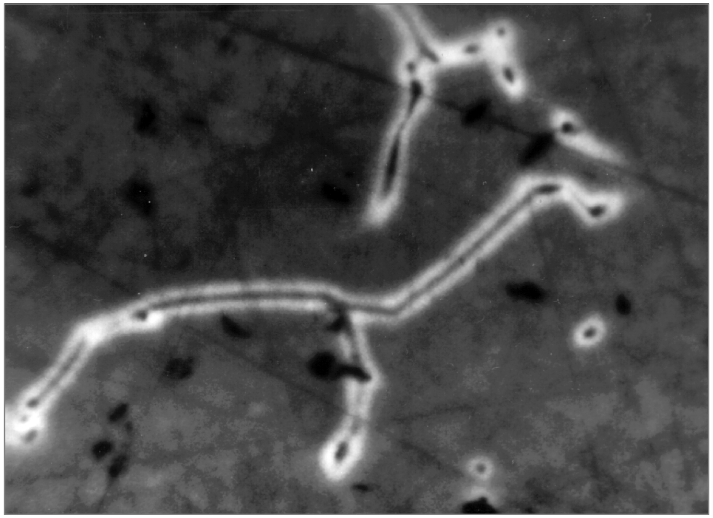
- Parameters: supersaturation of point defects, diffusion coefficient, cooling time
- Maximum density of dislocations according to Schoeck, Tiller (1960)

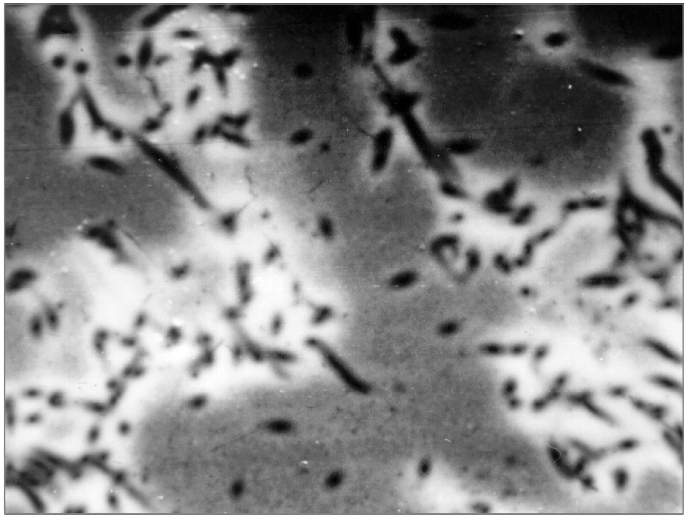
$$\rho = \frac{c_0 - c}{2rb}$$

  (c_0, c point defect concentration at melting point and at the given temperature, r loop radius)

- Quantitative estimations however difficult
- Not only agglomeration of point defects – such loops are sinks for other supersaturated defects
- Further growth or shrinking; gettering of impurities

Gettering





Dot–halo cathodoluminescence contrast as manifestation of the immobilization of in-grown dislocations by gettering of point defects

Halo, SEM 9897 n-GaAs

Two-dimensional defects

- ***Grain boundaries***
  polycrystalline growth; parasitic nucleation and merging of crystal parts
- ***Small-angle boundaries***
  grown-in; misfit; plastic deformation*
- ***Twins and stacking faults***
  grown-in; plastic deformation* and dissociation of perfect dislocations; phase transitions
- ***Antiphase boundaries and domains***
  phase transitions; spontaneous change in the orientation; non-stoichiometry

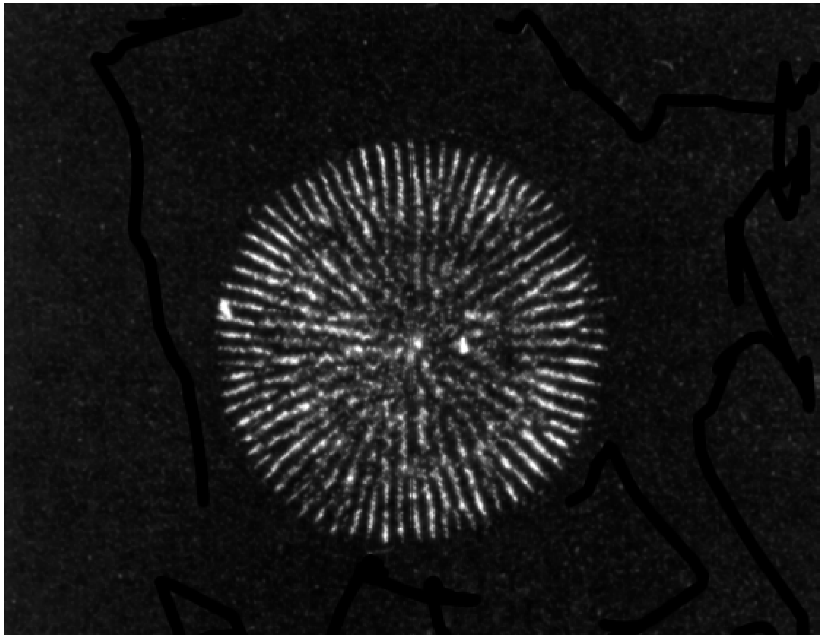
* induced by strain during growth/cooling

Inclusion of a foreign phase

- Coarse perturbation of homogeneity of a growing crystal
- Precipitation of a foreign phase; interaction with the defect structure of the crystal
- Microdefects only in dislocation-free (parts of) crystals, otherwise segregation at dislocations
- Temperature regime after crystallization important; quenching would be favorable to avoid precipitation, but thermal strain induces dislocations
- Capture of foreign phases: gas bubbles, inclusion of the solvent, impurities from the crucible

Interaction of precipitation and dislocations

0.1 mm



Precipitates in GGG (gandolinium gallium granate $Gd_3Ga_5O_{12}$)

[Wilke 1988/Bohm 1977]

1.6 Strain in the cooling crystal

Sources of strain

- ***Gravity***

  axial stress up to 0.05 MPa for a 500 mm long crystal

- ***Interaction with containers***

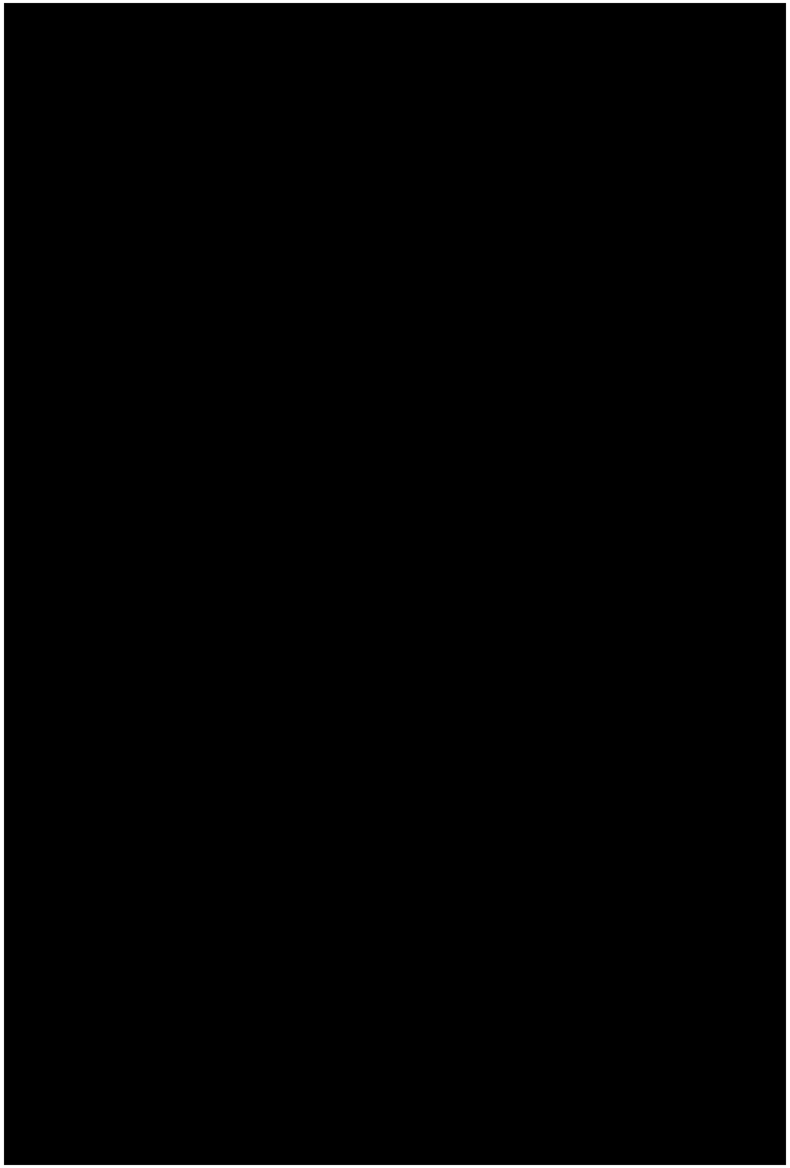
  good condition if wetting can be avoided;

  if the crystal sticks to the container, different thermal expansion coefficients may give rise to enormous forces (brittle fracture)

- ***Stress induced by inhomogeneities***

  striations, inclusions

Heat transfer during growth



Schematic visualization of heat exchange processes during LEC growth

Growth heat transfer [Völkl:94]

Concept of critical resolved shear stress

- ***CRSS*** – threshold value for plastic deformation of the crystal
- Wherever the elastic stress, obtained from the temperature field calculated, exceeds this critical value, plastic deformation occurs
- No differentiation between dislocation nucleation and multiplication, only qualitative estimations
- Main criticism of Alexander: not the existence of an unlocking stress for dislocation mobilization, but neglecting the time dependence: dislocation density is a function of the whole thermal and stress history of the crystal

Model of dislocation multiplication

Alexander-Haasen model of plastic deformation

- Concept of effective stress, which is needed for a dislocation to overcome the lattice resistance

$$\tau = \tau_{\mathrm{eff}} = \tau_{\mathrm{a}} - \tau_{\mathrm{i}}$$

- Stress due to the interaction of moving dislocations

$$\tau_{\mathrm{i}} = Gb \frac{\sqrt{\rho_{\mathrm{m}}}}{\beta} = D_0 \sqrt{\rho_{\mathrm{m}}}$$

(ρ_{m} density of *mobile* dislocations, D_0 strain hardening factor, β interaction factor)

- Dislocation velocity v

$$v = v_0 \left(\frac{\tau_{\mathrm{a}} - D_0 \sqrt{\rho_{\mathrm{m}}}}{\tau_0} \right)^m \exp\left(-\frac{Q}{k_{\mathrm{B}} T}\right)$$

Alexander–Haasen model

- Dislocation multiplication law

$$d\rho_m = K\,\tau_{\mathrm{eff}}^{\lambda}\,\rho_{\mathrm{m}}\,v\,dt = \rho_{\mathrm{m}}\,K\,v_0\left(\frac{\tau_{\mathrm{a}} - D_0\sqrt{\rho_{\mathrm{m}}}}{\tau_0}\right)^{m+\lambda}\exp\left(-\frac{Q}{k_{\mathrm{B}}T}\right)dt$$

- Orowan equation

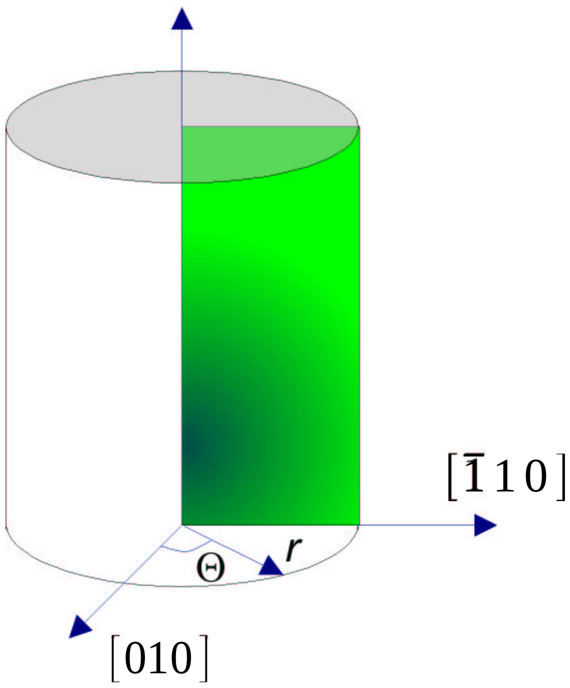
$$\dot{a} = \rho_{\mathrm{m}}\,b\,v = \rho_{\mathrm{m}}\,b\,v_0\left(\frac{\tau_{\mathrm{a}} - D_0\sqrt{\rho_{\mathrm{m}}}}{\tau_0}\right)^{m+\lambda}\exp\left(-\frac{Q}{k_{\mathrm{B}}T}\right)$$

- Slip *a* related via Schmid factor to (plastic) strain ε

$$\dot{a} \propto \dot{\varepsilon} = \dot{\varepsilon}_{\mathrm{tot}} - \dot{\varepsilon}_{\mathrm{elast}}$$

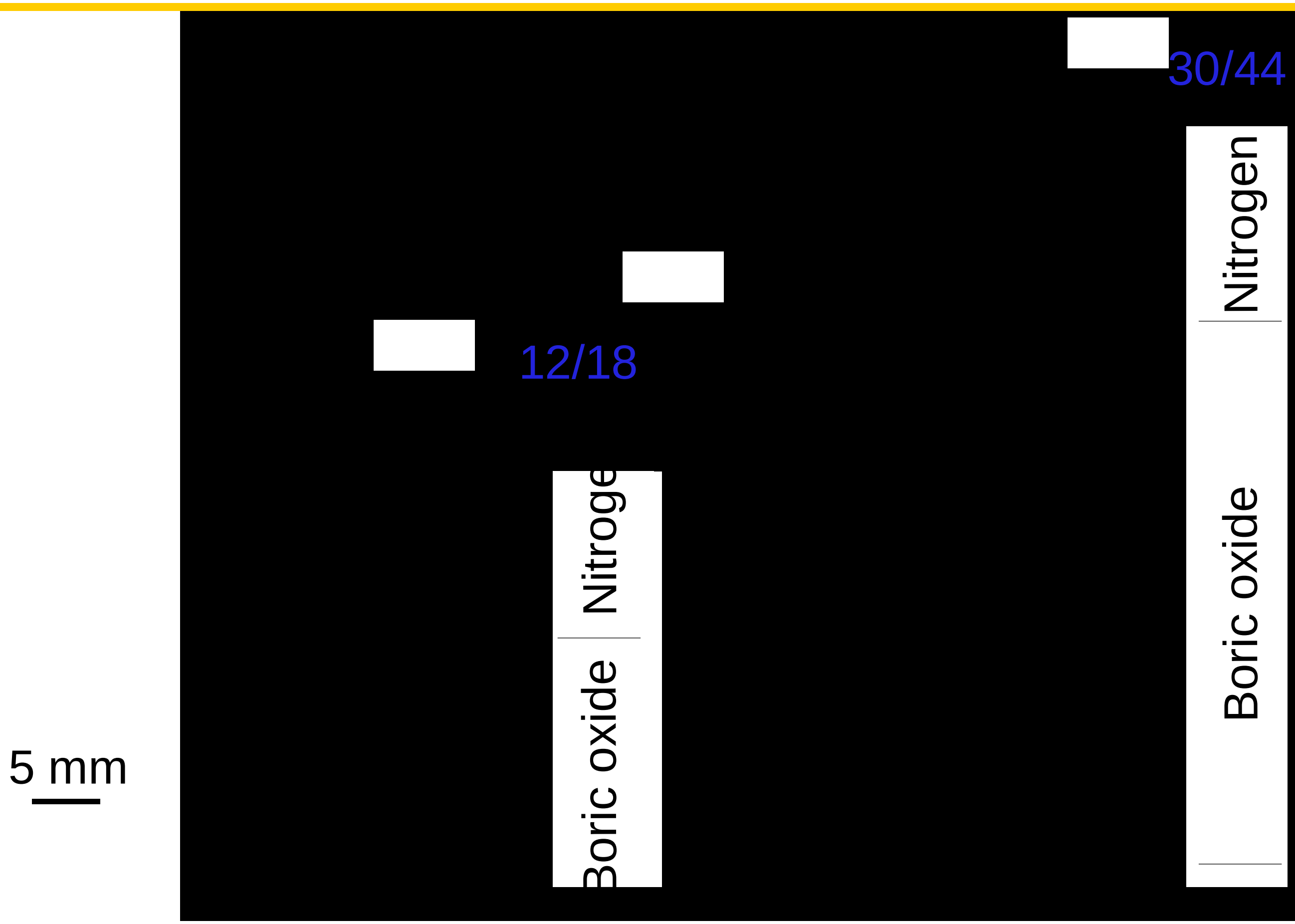
- The constants for GaAs: $m = 1.7$, $\lambda = 1$, $Q = 1.4$ eV, $v_0 = 1.8\times10^8$ m/s, $\tau_0 = 1$ MPa, $K = 7\times10^{-3}$ m/N, $D_0 = 3.12$ N/m
  [Tsai *et al.* 1993]

Thermal elasticity model



- Thermal stress calculations in cylindrical coordinates
- Assuming isotropic material and axisymmetric crystal
- Two-dimensional thermal elasticity model
- Thermal elastic stress components σ_{rr}, $\sigma_{\Theta\Theta}$, σ_{zz}, σ_{rz} $\rightarrow$ transformation to each slip system to simulate dislocation motion/multiplication

Isotherms in a growing crystal



Isotherms in InP crystals grown under 12/18 and 30/44 conditions denoting the initial/final boric oxide heights in mm. An additional afterheater was used to increase the temperature in the gas atmosphere in the 30/44 crystal.

[Völkl 1994]

Thermoelastic stress

- Inhomogeneous temperature distribution coupled with locally different expansion/contraction
- Resulting strain converted into stresses via Hooke's law
- Assuming completely elastic behavior over the whole temperature range *!*
- Theory of thermoelasticity
  see *e.g.* Timoshenko: Theory of elasticity (1951)
- Simple rules:
  i. Temperature field which is linear in all coordinates results in a stress-free state (free temperature bending)
  ii. Scaling of dimensions and temperature differences
  iii. Incompatibilities of the temperature field are sources of stress

Von Mises stress

Contour plots of the von Mises stress (in MPa) for the growth conditions of 12/18 and 30/44 InP crystals

[Völkl 1994]

$$\sigma_{vM} = \sqrt{\frac{(\sigma_{rr} - \sigma_{zz})^2 + (\sigma_{zz} - \sigma_{\Theta\Theta})^2 + (\sigma_{\Theta\Theta} - \sigma_{rr})^2 + 6\sigma_{rz}^2}{2}}$$

Resolved shear stress

- Glide forces on dislocations has to be calculated for in each slip system
- fcc structure: 12 systems of the type $\langle 1\,\bar{1}\,0 \rangle \{ 111 \}$
- ***Peach–Koehler equation*** for the force per unit dislocation length

$$\boldsymbol{F} = \boldsymbol{b} \cdot \boldsymbol{\sigma} \times \boldsymbol{l}$$

- The glide force is the projection of ***F*** in the slip plane, perpendicular to the dislocation

$$\boldsymbol{F}_{\mathrm{g}} = \boldsymbol{F} \cdot \boldsymbol{n} \times \boldsymbol{l}$$

- Resolved shear stress $\tau = F_g/b$

Dislocation multiplication during growth

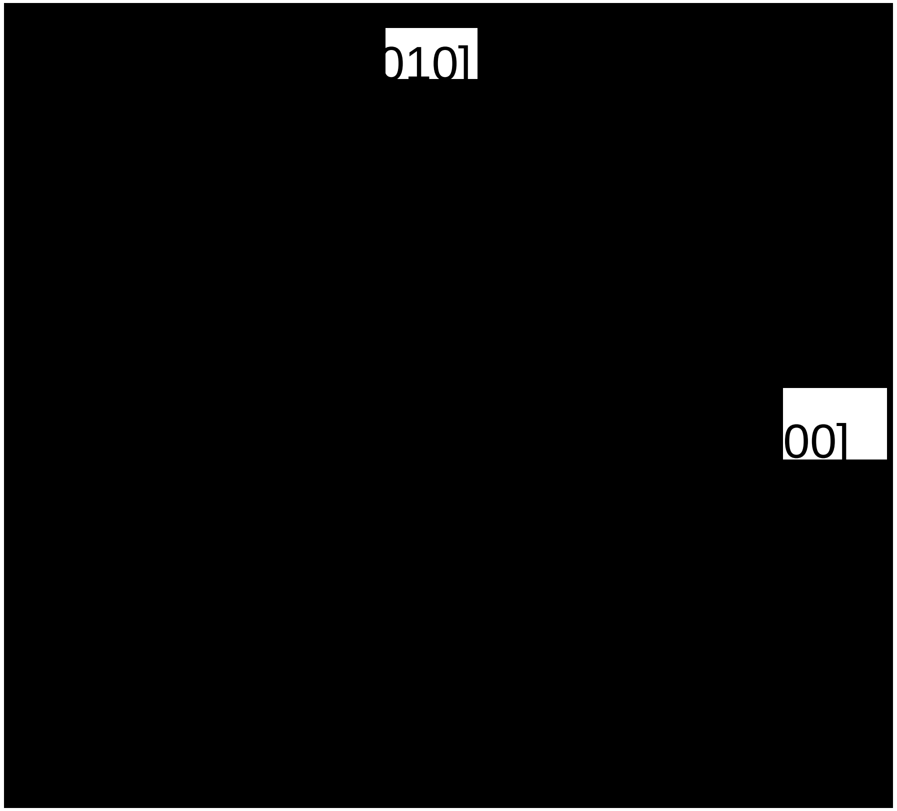
Rate of dislocation multiplication related to the growth velocity v_{gr}

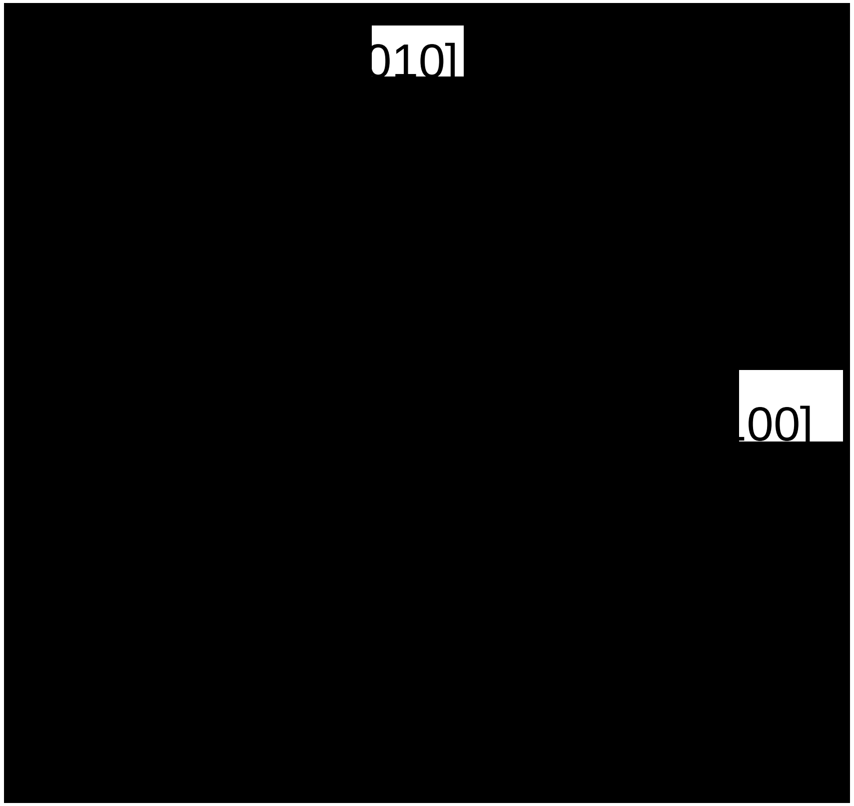
$$\dot{\rho}_{\mathrm{m}} = \frac{\partial \rho_{\mathrm{m}}}{\partial z}\frac{\partial z}{\partial t} = \frac{\partial \rho_{\mathrm{m}}}{\partial z} v_{\mathrm{gr}}$$

Result of numerical calculations on a $(1\bar{1}0)$ plane for an $[011]\{\bar{1}1\bar{1}\}$ slip system in GaAs.
m middle of the crystal, e edge,
c halfway position.

[Tsai *et al.* 1993]

Dislocation distribution in the slip systems

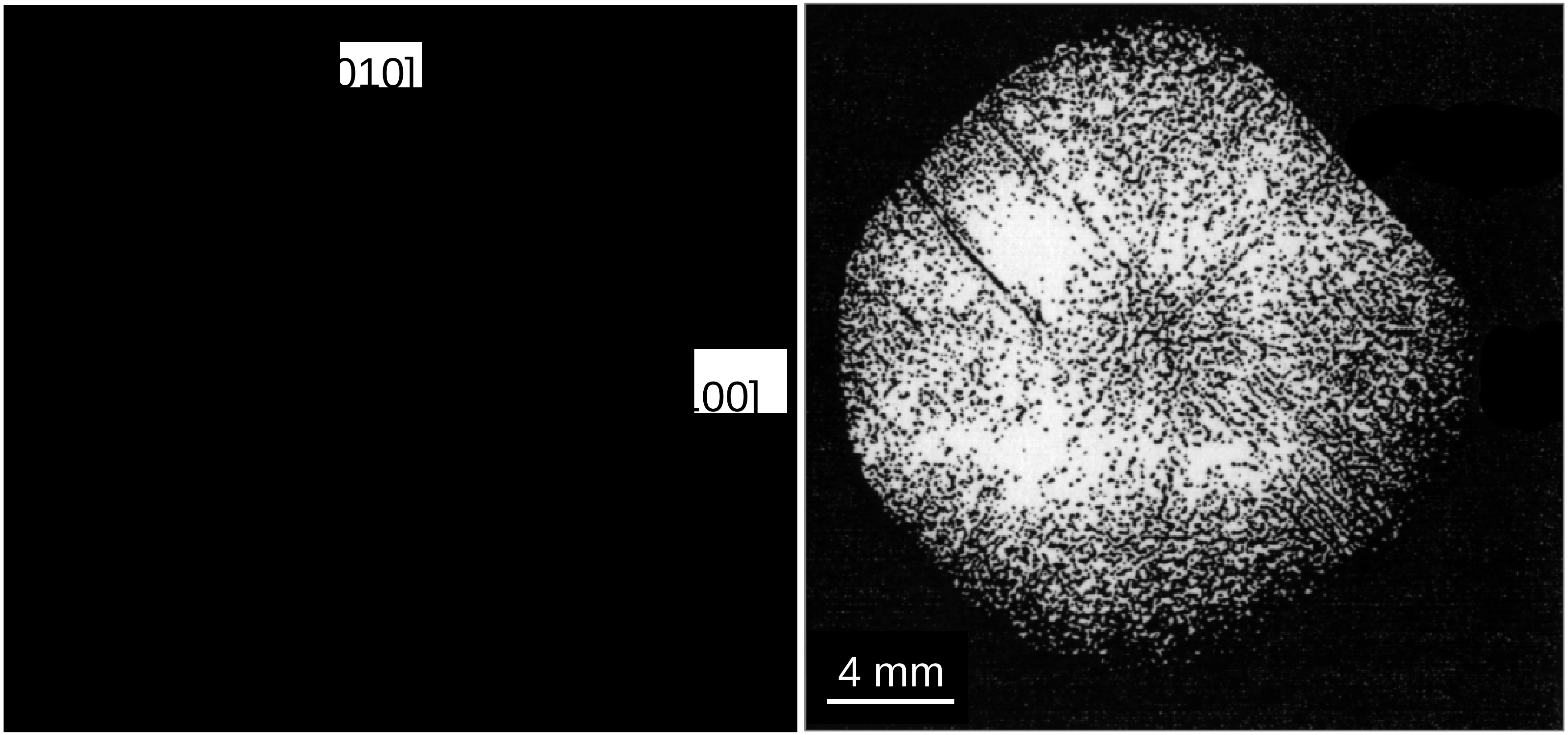




Dislocation density (in cm^{-2}) distribution on (001) GaAs wafers near the top end of the boule.
Left: $[\bar{1}10]\{\bar{1}\bar{1}1\}$, $[110]\{\bar{1}1\bar{1}\}$, $[110]\{1\bar{1}\bar{1}\}$, $[1\bar{1}0]\{111\}$,
right: $[011]\{\bar{1}1\bar{1}\}$, $[0\bar{1}1]\{1\bar{1}\bar{1}\}$ slip systems.

[Tsai *et al.* 1993]

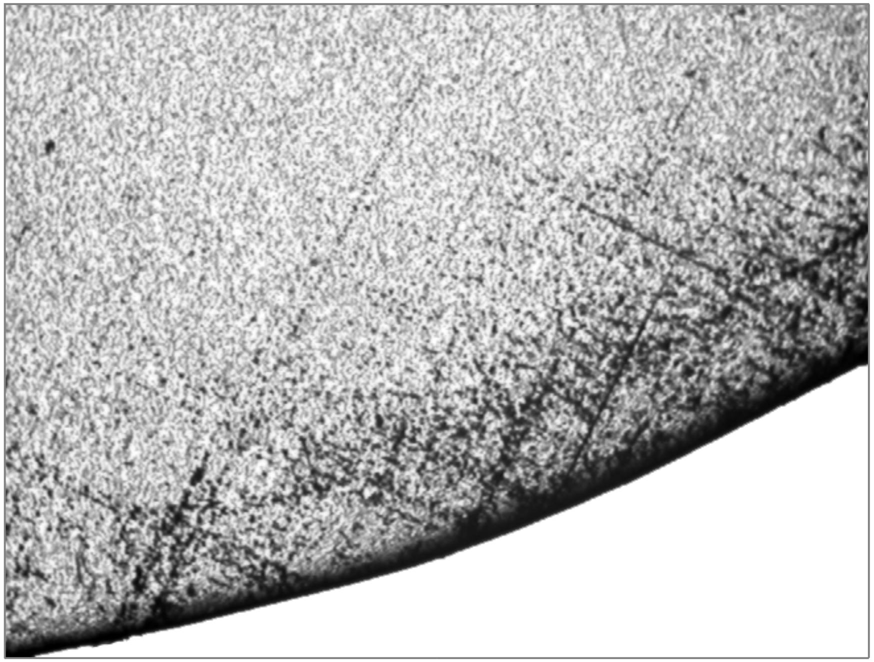
Dislocation pattern on a (001) GaAs wafer

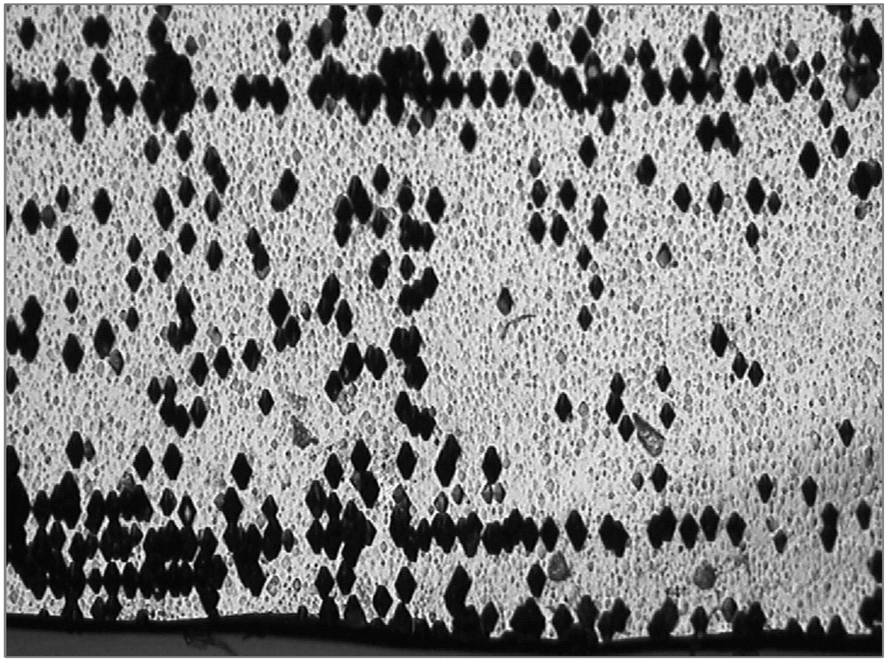


Sum of dislocation density (in cm^{-2}) distribution on a (001) GaAs wafer near the top end of the boule for all 12 slip systems. The macrophotograph shows a KOH-etched wafer.

[Tsai *et al.* 1993/Jordan *et al.* 1980]

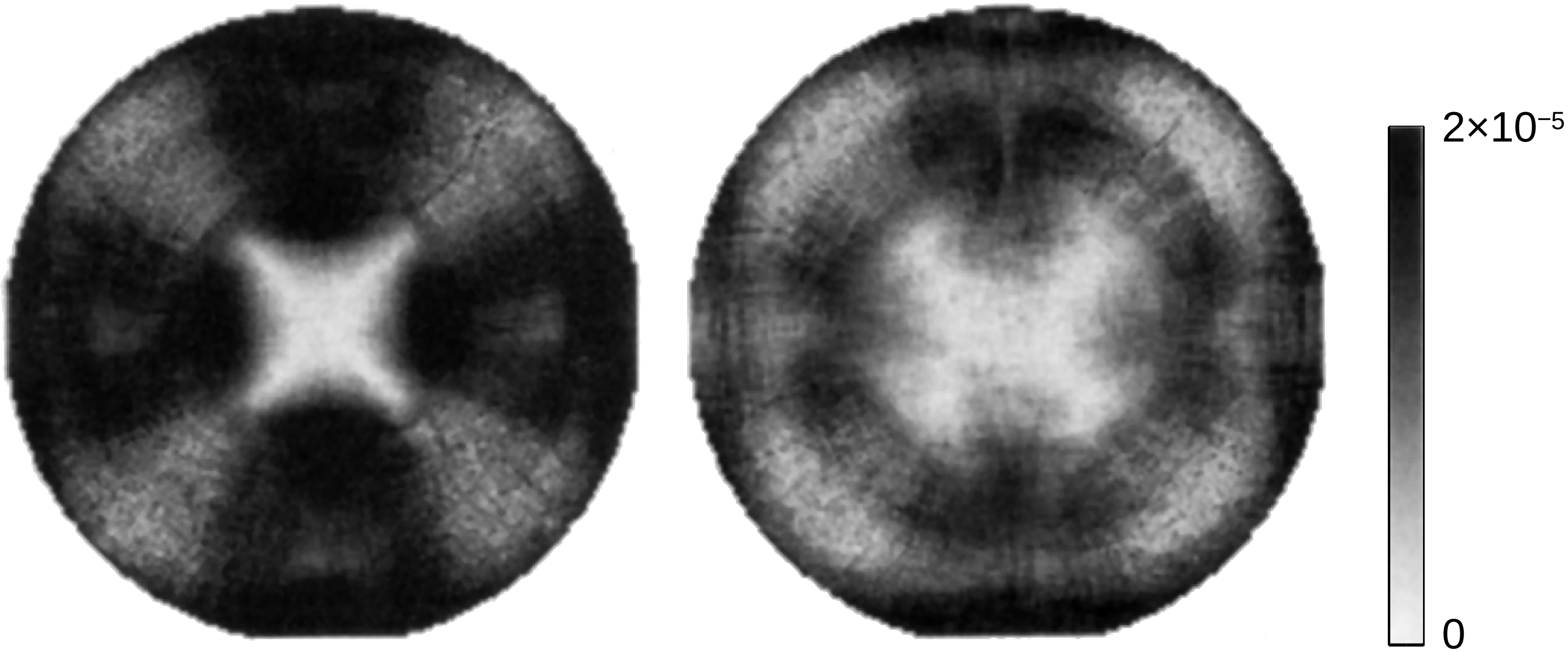
Dislocations near the edge of a GaAs wafer





Dislocation arrangement in slip lines near the wafer edge.
(001) GaAs etched with molten KOH.

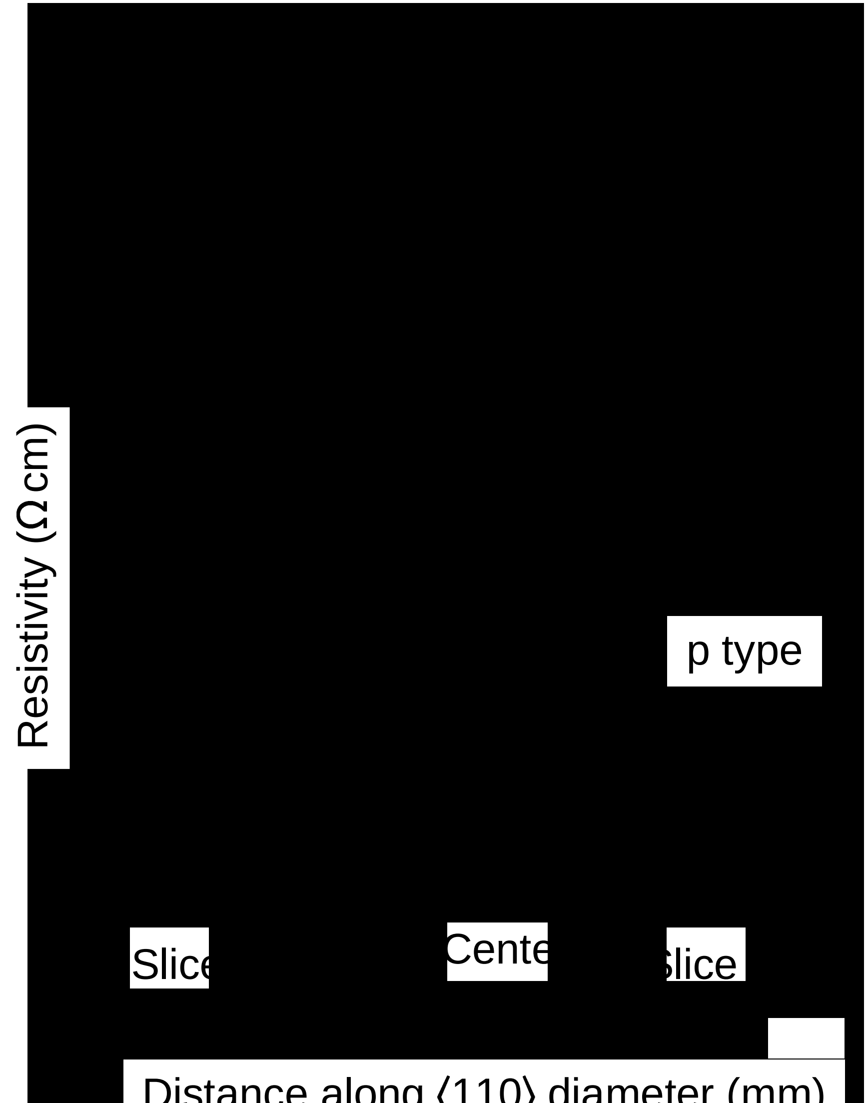
Residual strain



Residual strain profiles of $|\varepsilon_y - \varepsilon_z|$ measured in two different commercial 3” GaAs wafers with an infrared polarization microscope. $|\varepsilon_y - \varepsilon_z|$ is the difference in tensile strains along the radial and growth directions, *i. e.* a figure of the residual in-plane strain.

[Yamada *et al.* 1996]

Electrical inhomogeneity of GaAs wafers



Typical W-shaped dependence of the radial resistivity profile in GaAs for different ([As]/([As] + [Ga]) melt ratios

[Young *et al.* 1990]