Characterization of micro-/nano-structures

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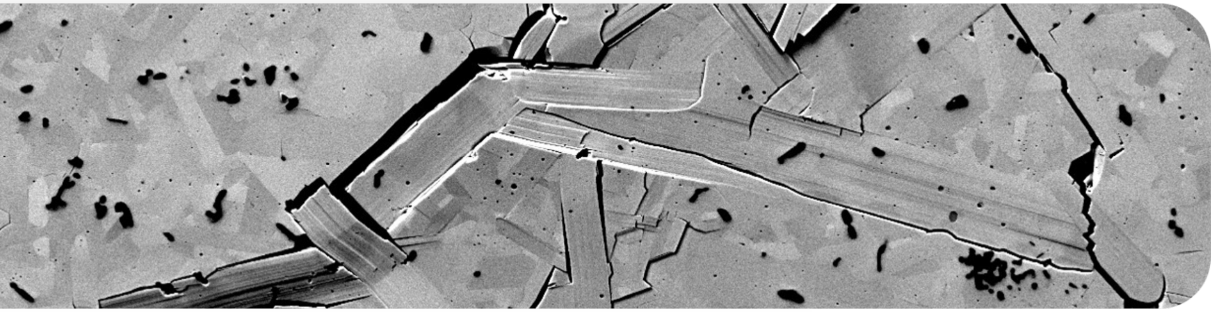
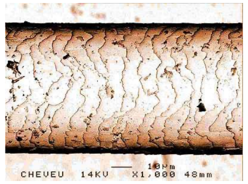
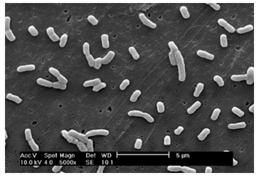


Some micro-/nano-structural features

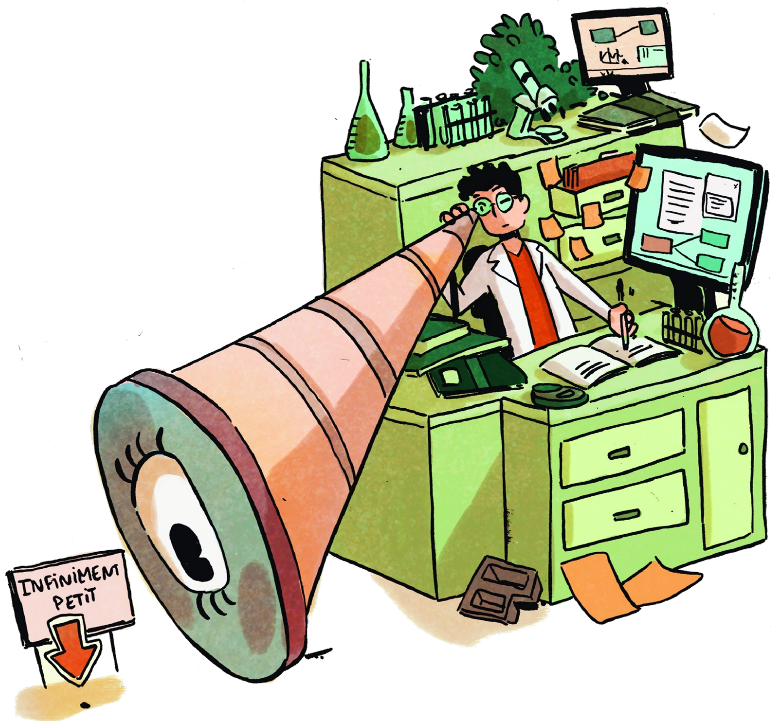
Some orders of magnitude

❖ Micrometer (1 µm = 0.000001 m = 10^{-6} m)





- Hair diameter = 50-100 µm
- Size of a bacteria = 0.1-10 µm



A. Guitton, Science en 4 Saisons – Hiver, *EDP Sciences (2023)*

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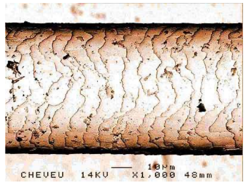

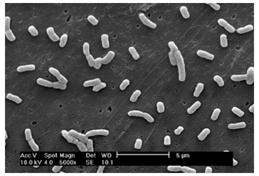

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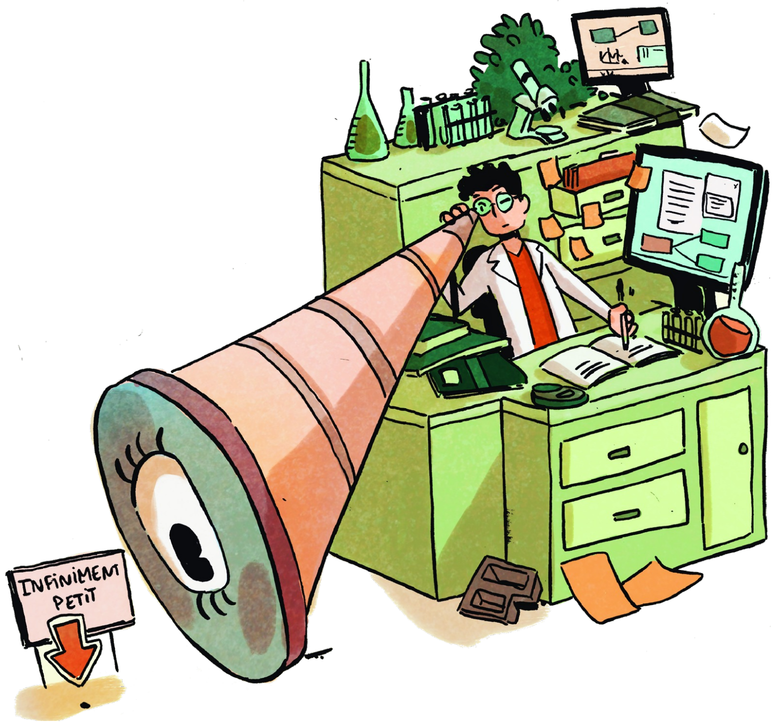

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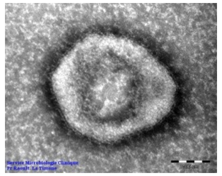

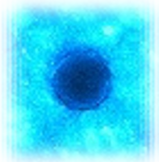

- Size of a virus = 20-450 nm

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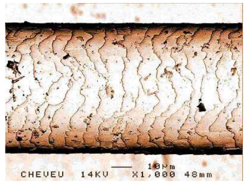
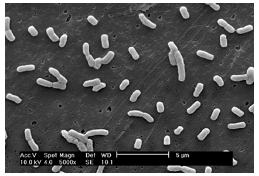

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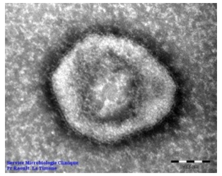
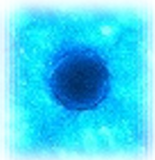

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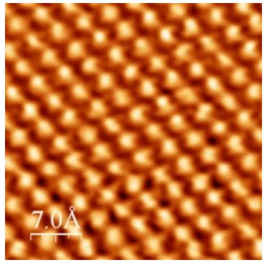
❖Atomic scale (1 Å = 0.0000000001 m = 10^{-10} m)



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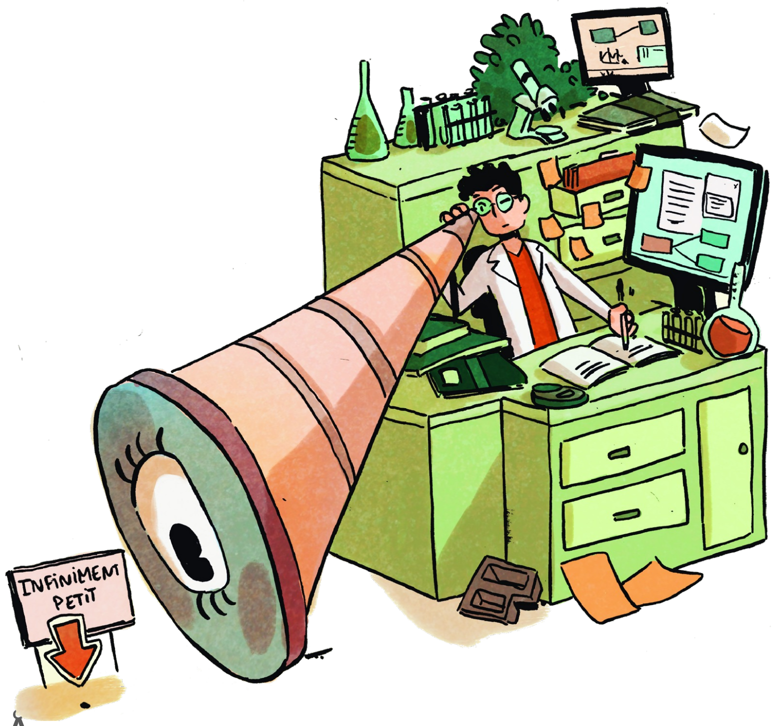

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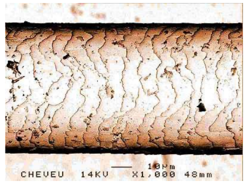
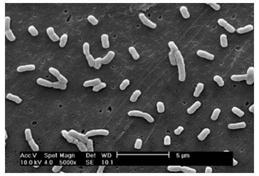

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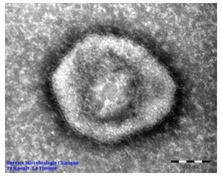
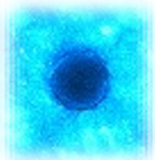

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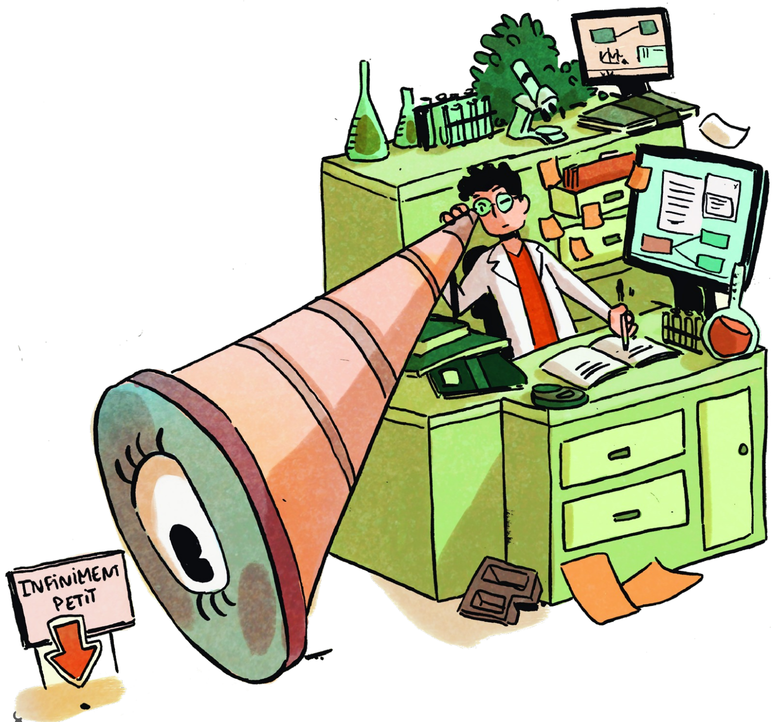

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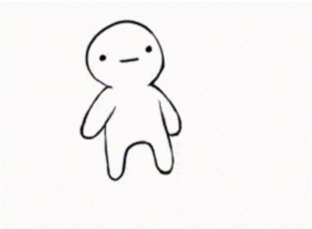
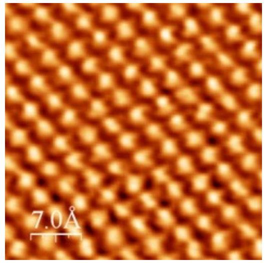

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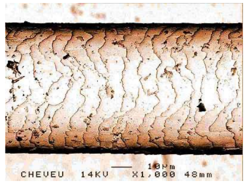

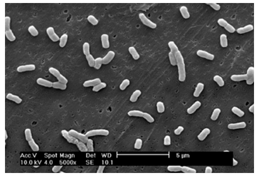

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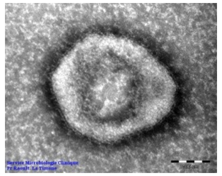

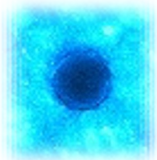

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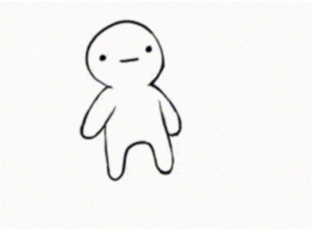

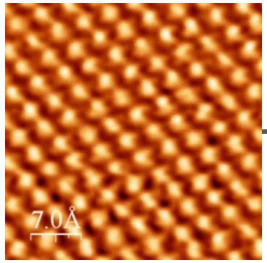

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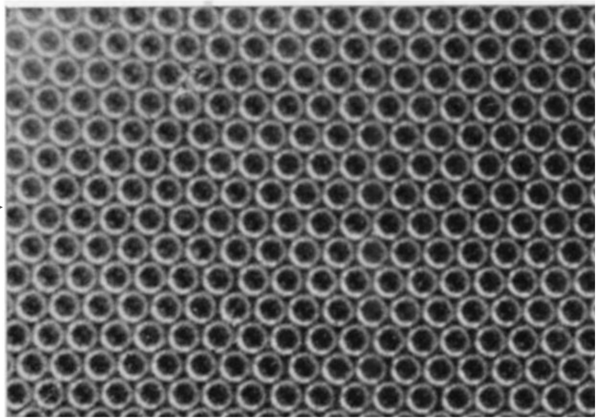
Stacking of spheres

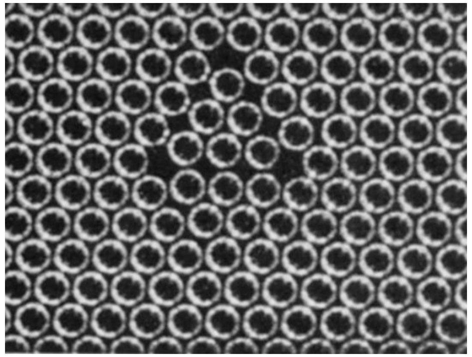
L. Bragg, J.F. Nye, *Proc. R. Soc. Lond.*, 1947



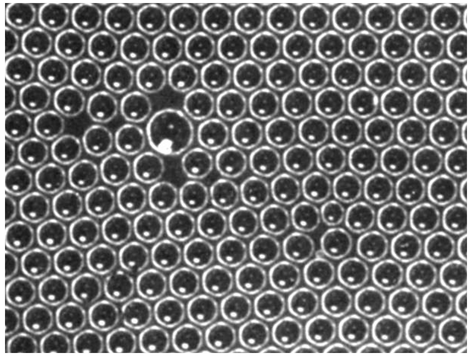
Defects in crystalline materials

0 dimension

Vacancy



Interstitial



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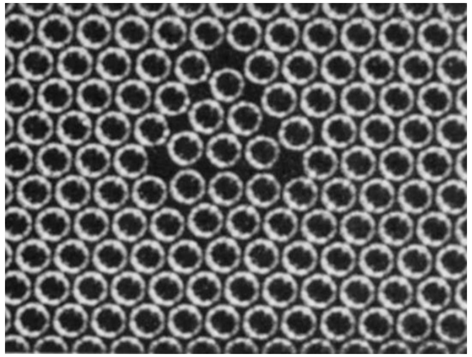

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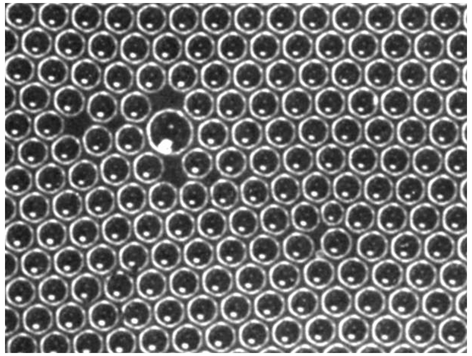
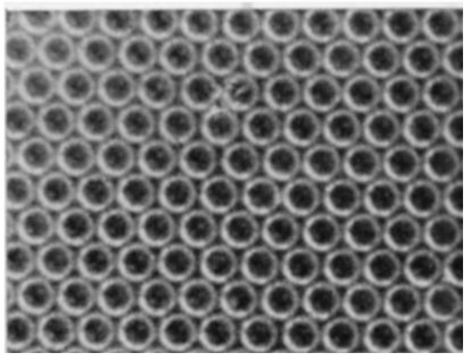


1 dimension

Dislocation



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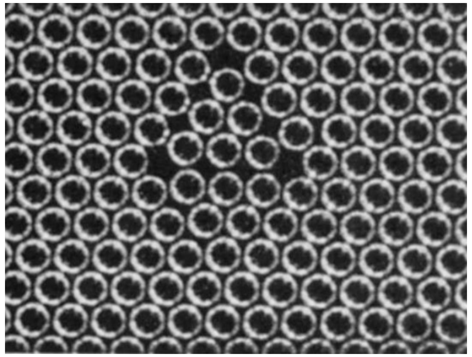

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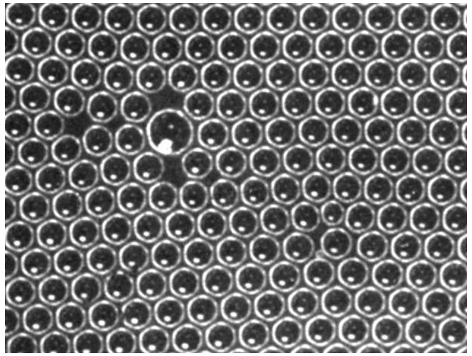

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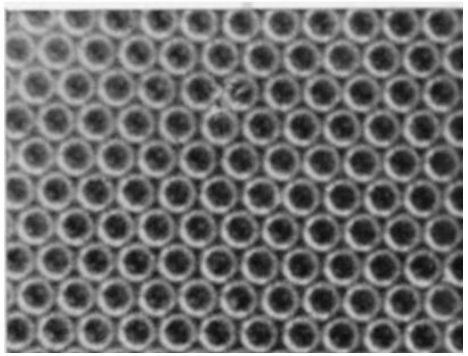
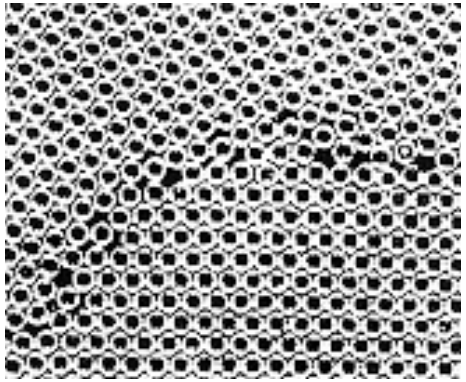


2 dimensions

Stacking fault

Grain boundaries



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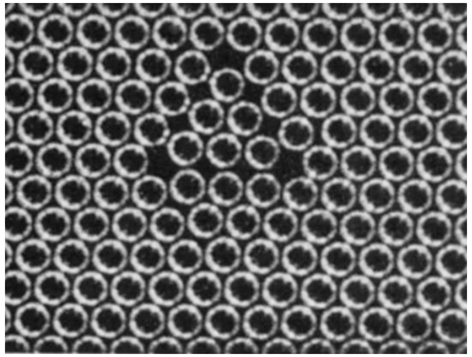

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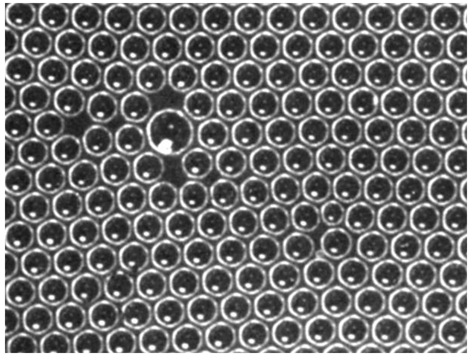

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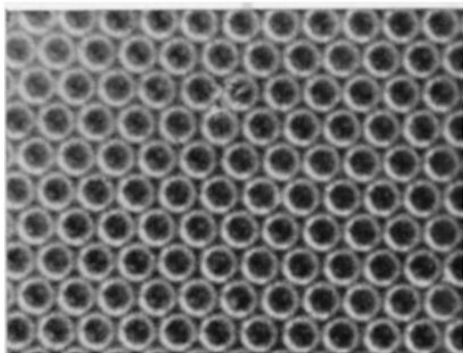


3 dimensions

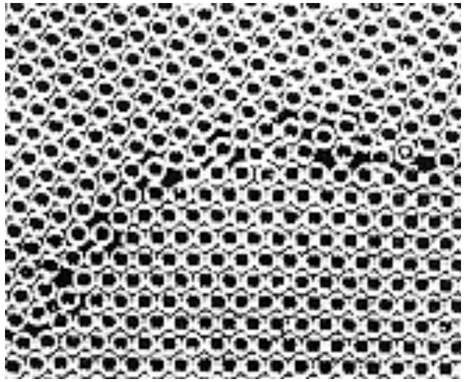
Inclusions, precipitates…

"Crystals are like people; it is the defects in them which tend to make them interesting!"
(Colin J. Humphreys)

2 dimensions

Stacking fault

Grain boundaries



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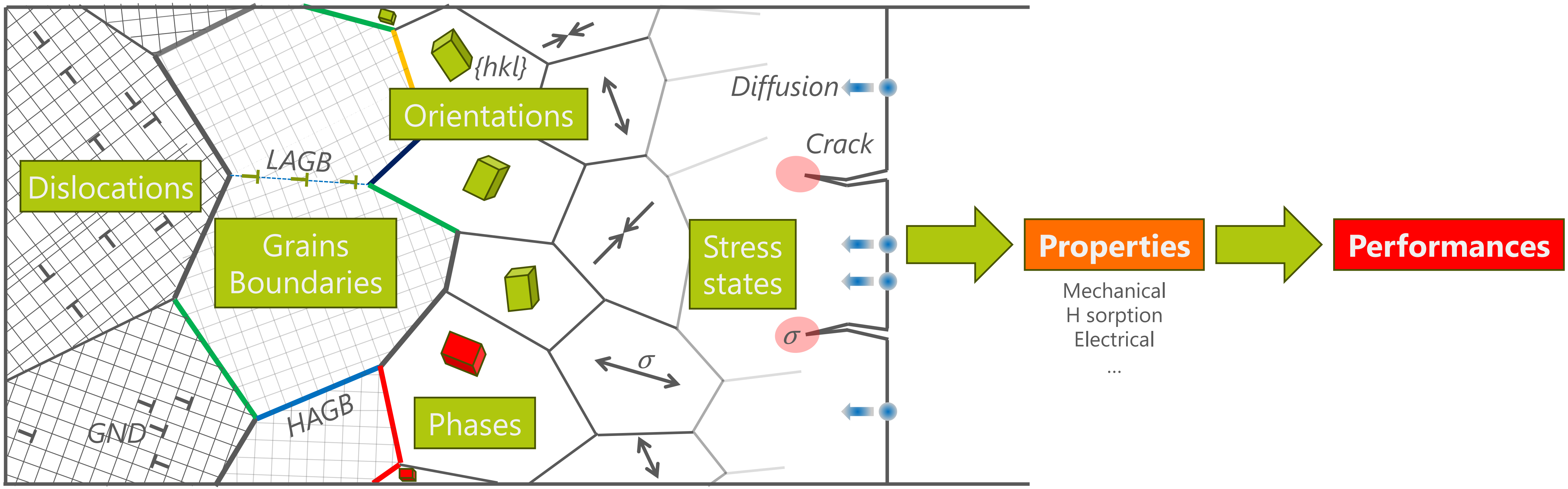
Micro-/nano-structures

Microstructure (~1 µm →1 mm) and nanostructure (~1 nm →100 nm) refer to the internal features of a material, at the micrometer and nanometer scales respectively, influencing its properties



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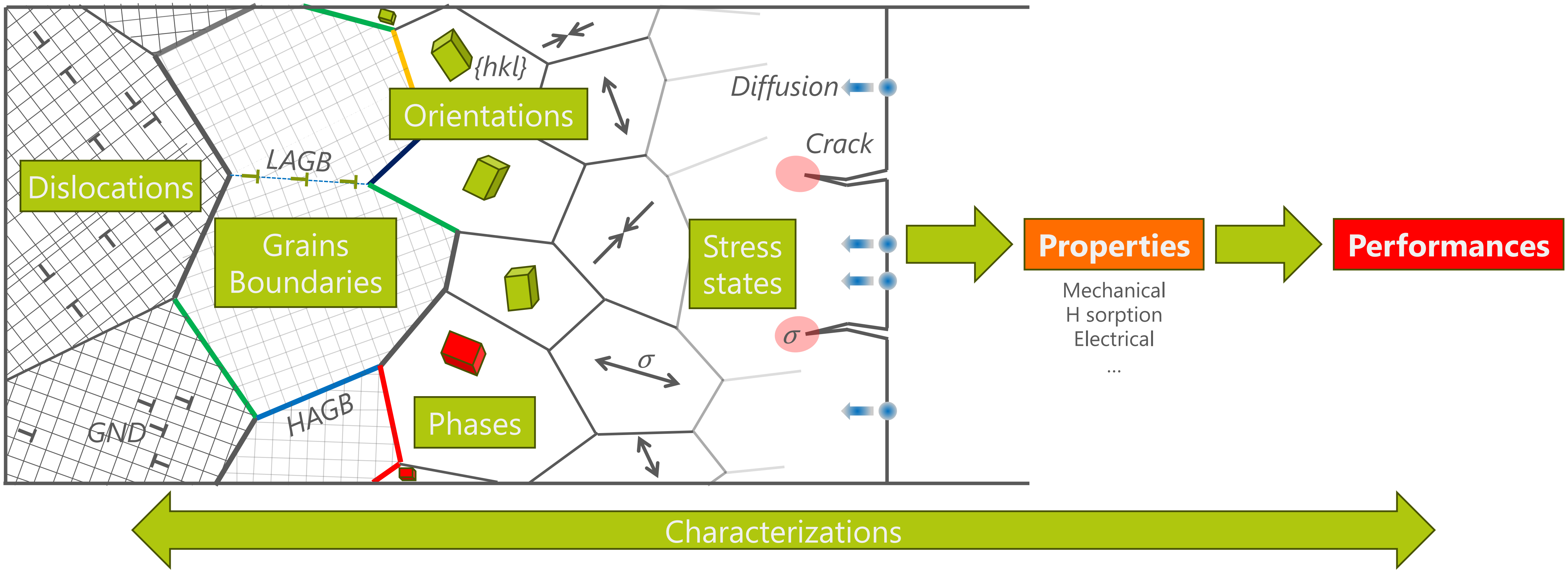
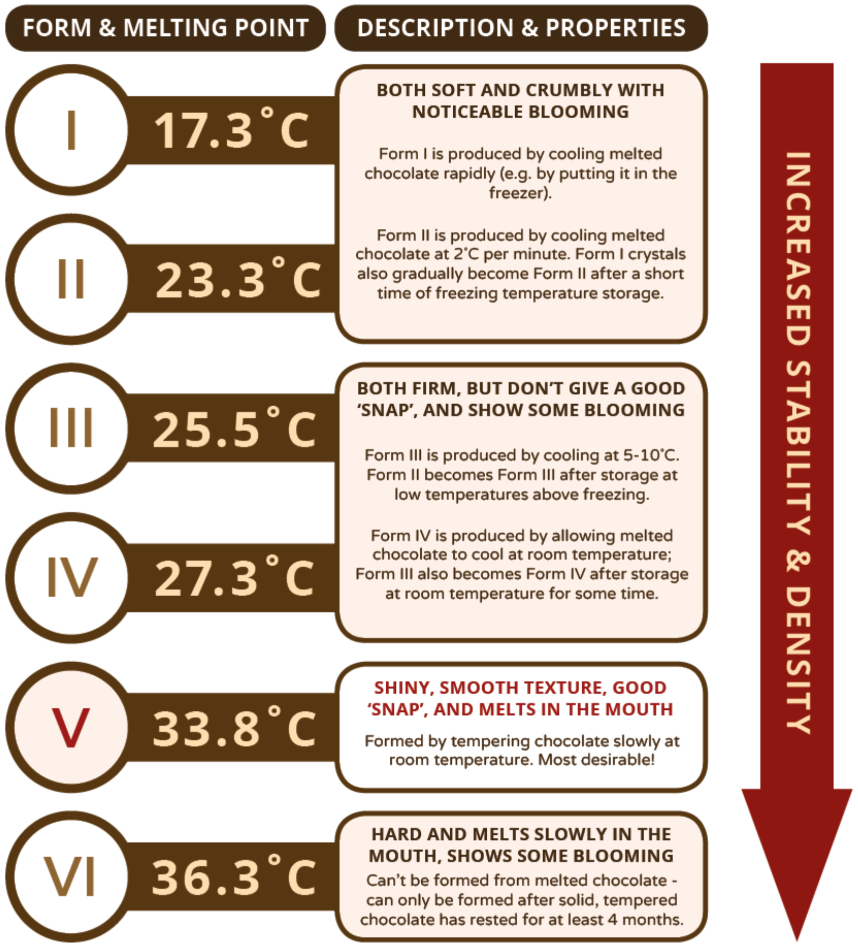


Phases



- **Definition:**
  - Distinct physical states of a material with uniform composition and structure.
  - Example:
    - ✓ cocoa butter polymorphism: exists in six polymorphic forms (I to VI), each with different melting points and stability.
    - ✓ Most desirable phase: Form V (β') – gives chocolate its smooth texture, glossy finish, and ideal snap.
- **Phase transitions:**
  - Heating and cooling control polymorphic transformation

P. Fryer et al., *MRS Bulletin.* (2000)

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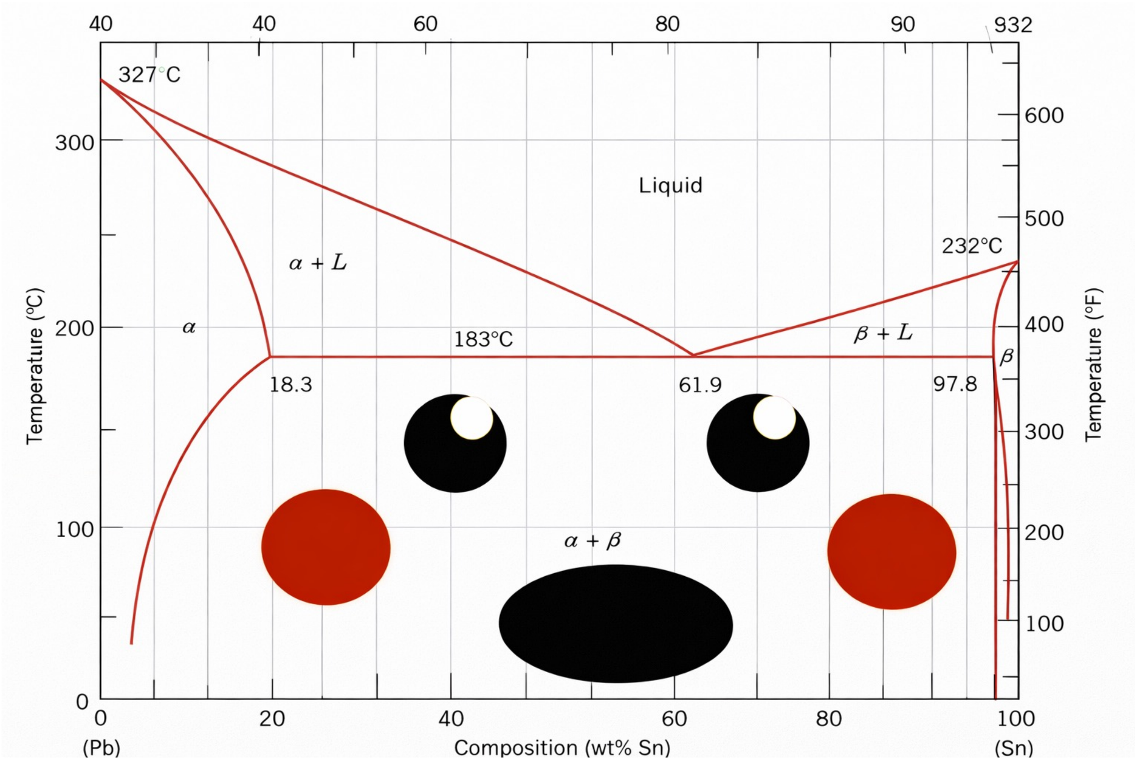

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- **Phase transitions:**
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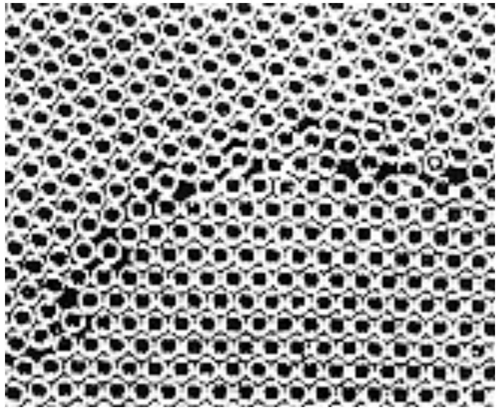
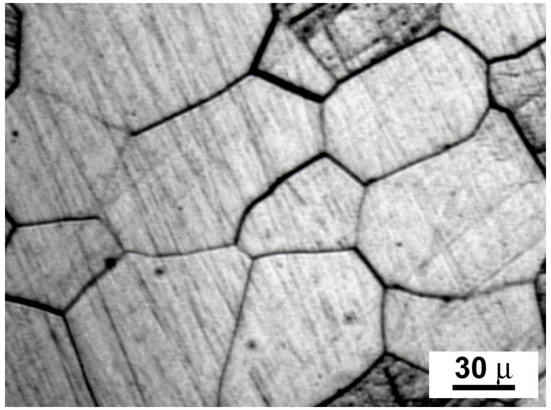
- **Phase diagram:**
  - Graphically represents phase stability as a function of temperature and composition.
- **Tie line (isothermal line):**
  - A horizontal line drawn in a two-phase region to determine the composition of coexisting phases.
- **Lever rule:**
  - Used in two-phase regions to determine phase fractions; applies to both metals and chocolate crystallization.
- **Importance in processing:**
  - Helps control microstructure, texture, and mechanical properties in both food and engineering materials.

P. Fryer et al., *MRS Bulletin.* (2000)

Grains

L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)





❖ Definition:

- Grains are individual crystallites in a polycrystalline material, each with a distinct crystallographic orientation.
- Can range from nanometers (nanocrystalline materials) to millimeters (coarse-grained materials).

❖ Description:

- Shape
- Size

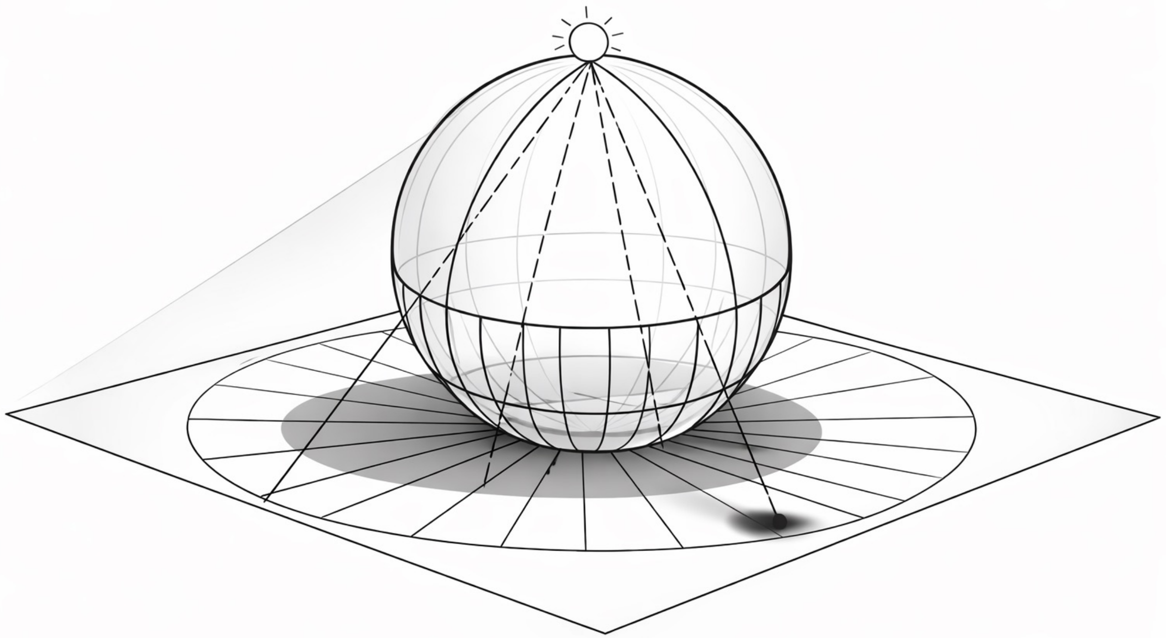
❖ Texture:

- Texture refers to a preferred orientation of grains in a polycrystalline material.

Basics

- **Light and shadow analogy:**
  - A sphere is illuminated by a point light source
  - The light source is placed at the north pole of the sphere
  - Each point on the sphere is connected to the light by a straight ray
  - The ray intersects a flat plane and creates a shadow point

↳ All shadow points form the stereographic projection



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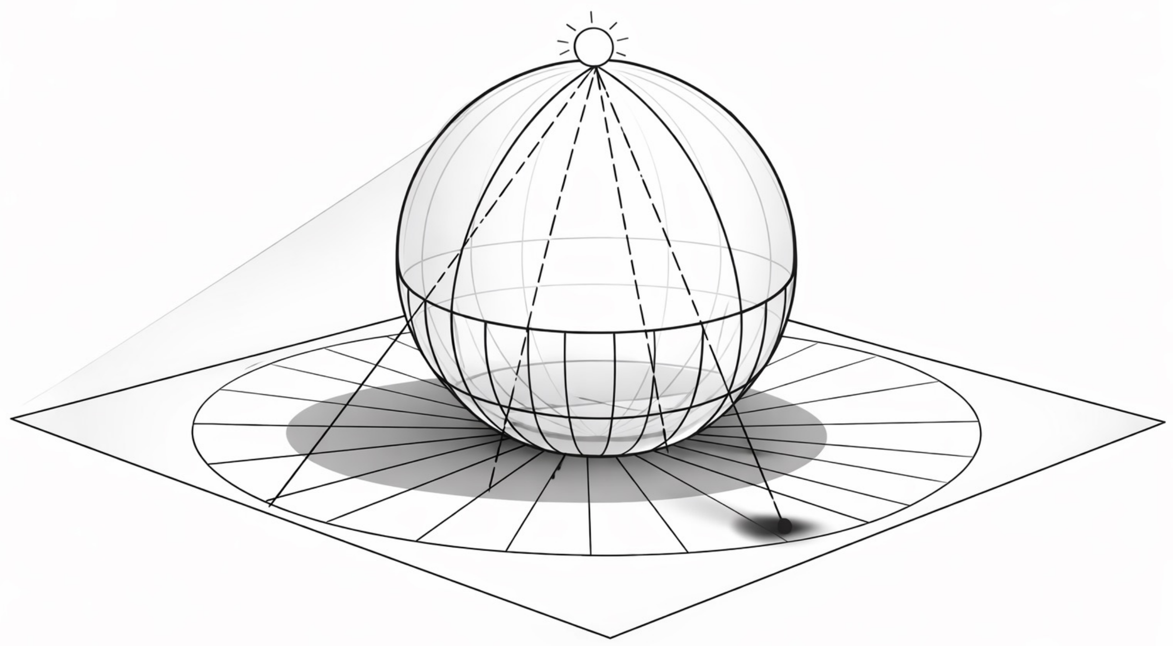
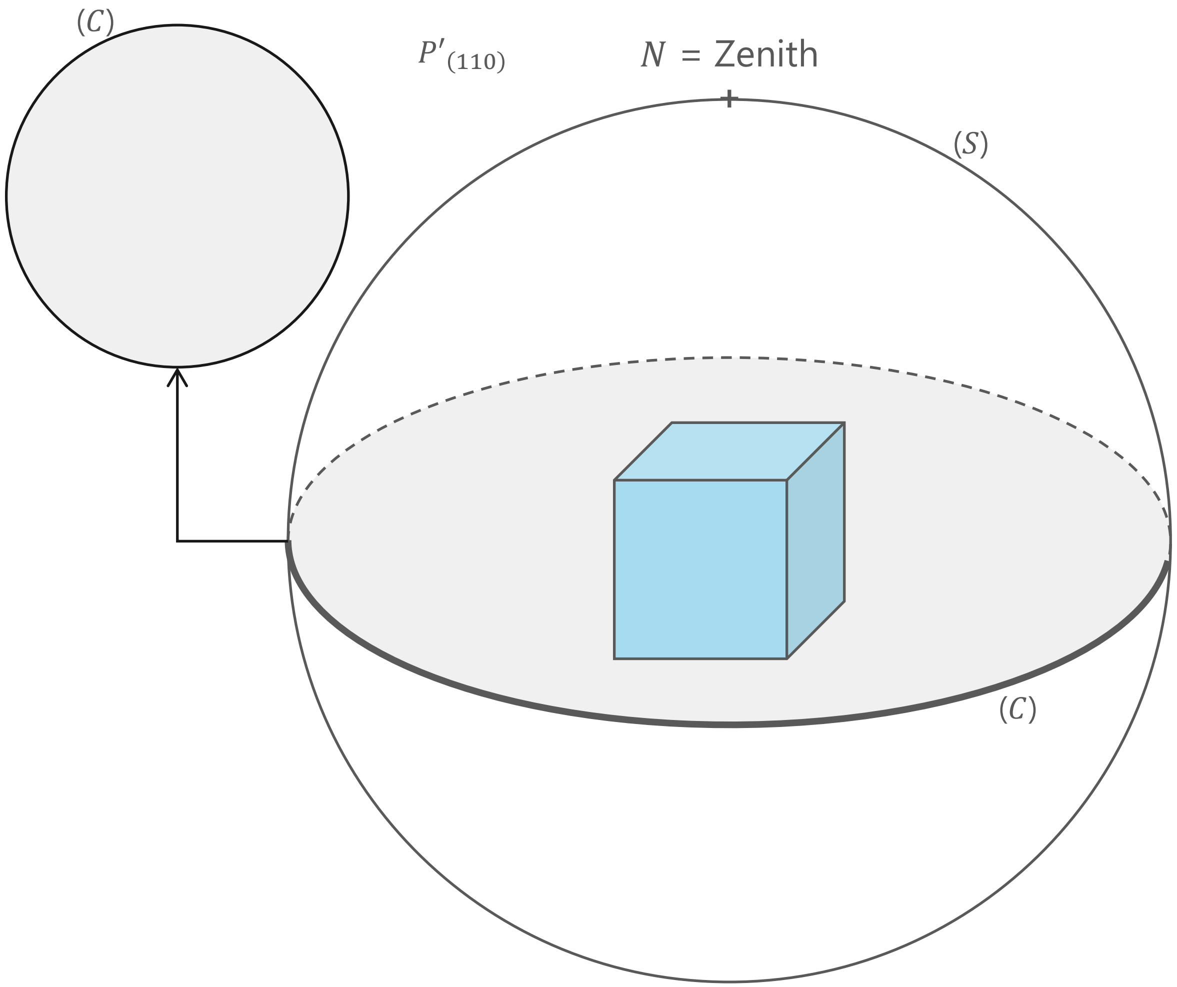


❖ **Key geometric property:**

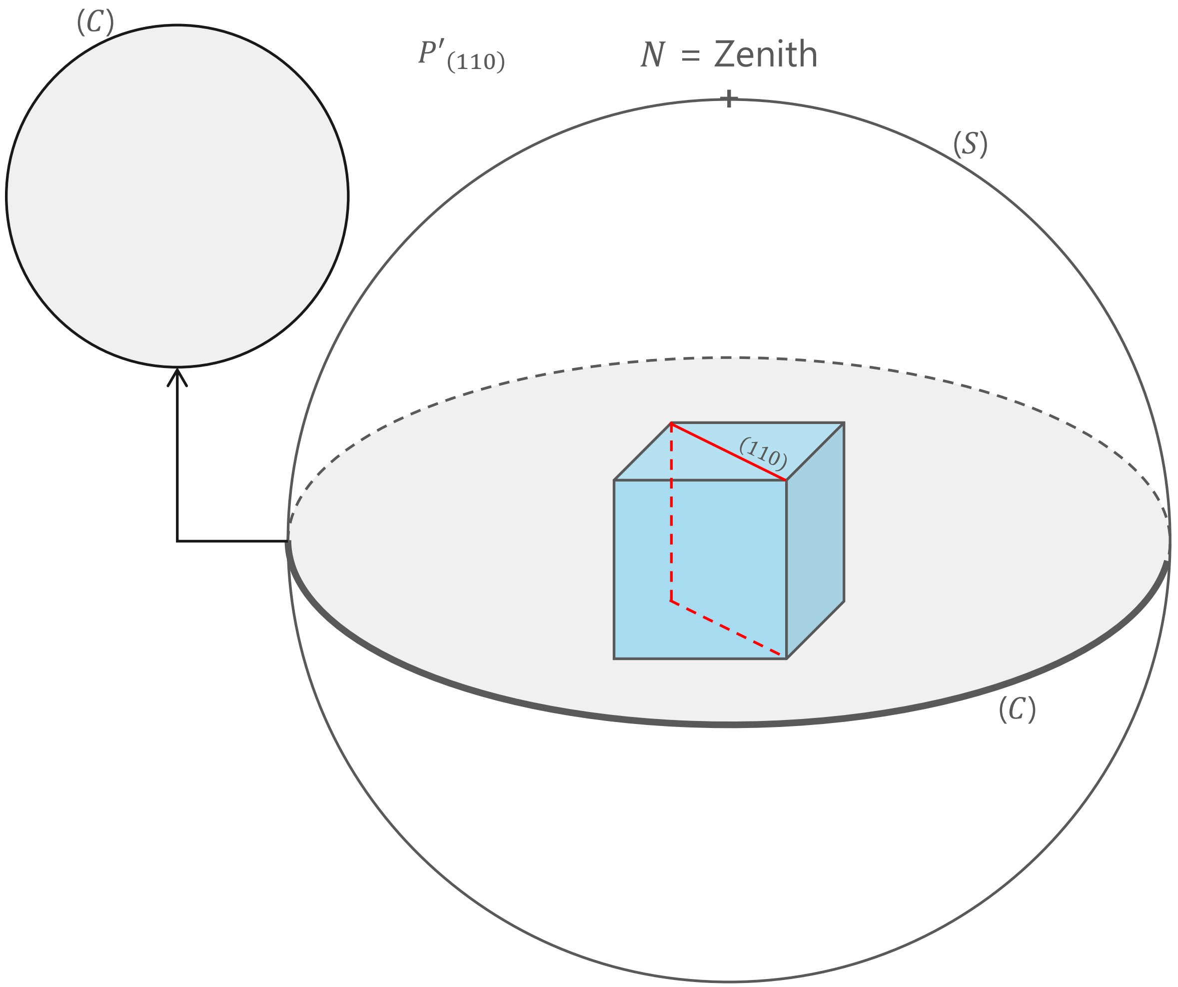
- Angles between directions are preserved
- Lengths and areas are not preserved

Stereographic projection of a plane via its normal



- Consider the crystal placed at the center of a reference sphere (S).

Stereographic projection of a plane via its normal



- Consider the crystal placed at the center of a reference sphere (S).
- Identify the crystallographic plane of interest, e.g., the (110) plane.

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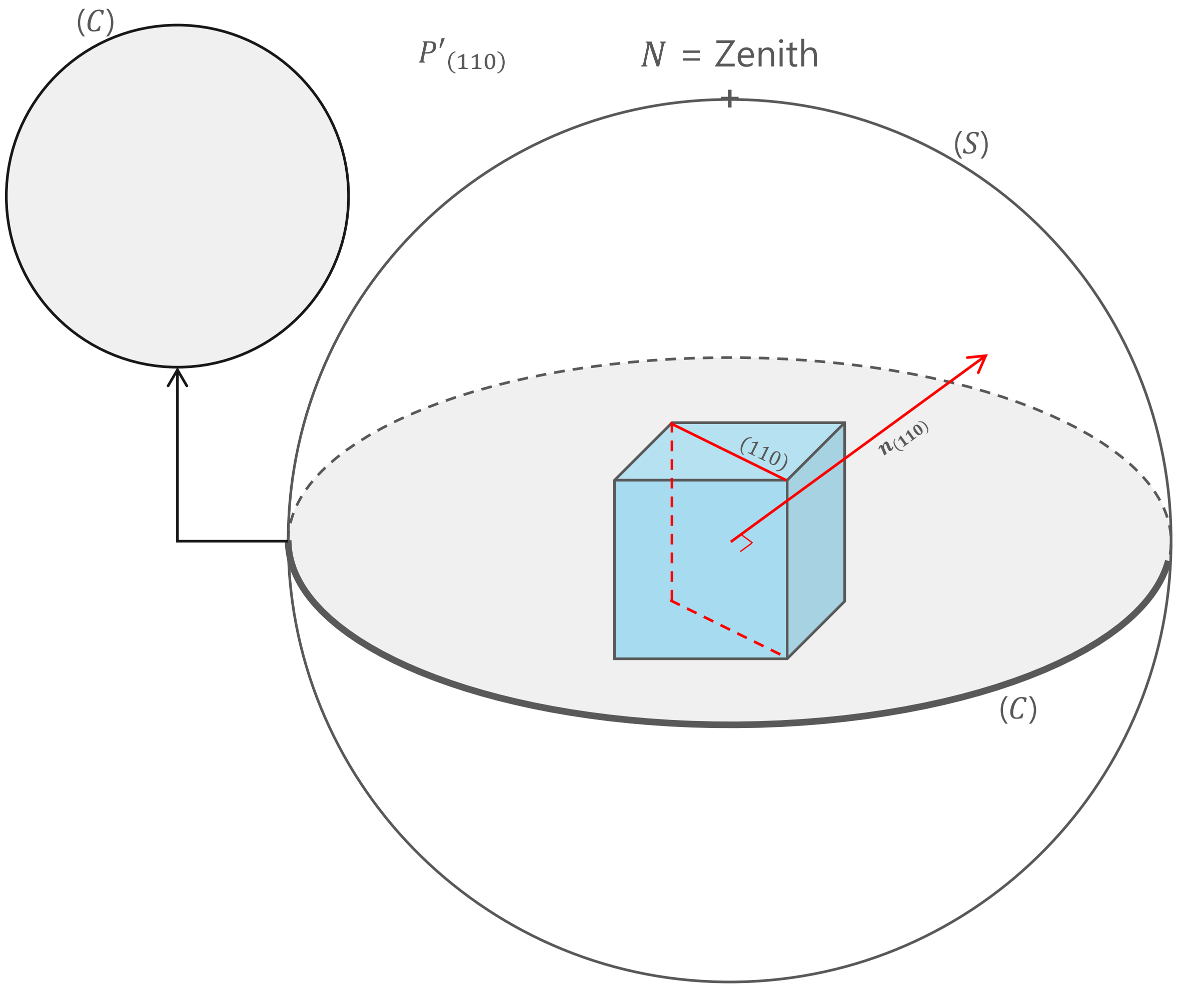

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- Draw the normal vector ($\boldsymbol{n}_{(\mathbf{110})}$) to this plane, starting from the center of the sphere.

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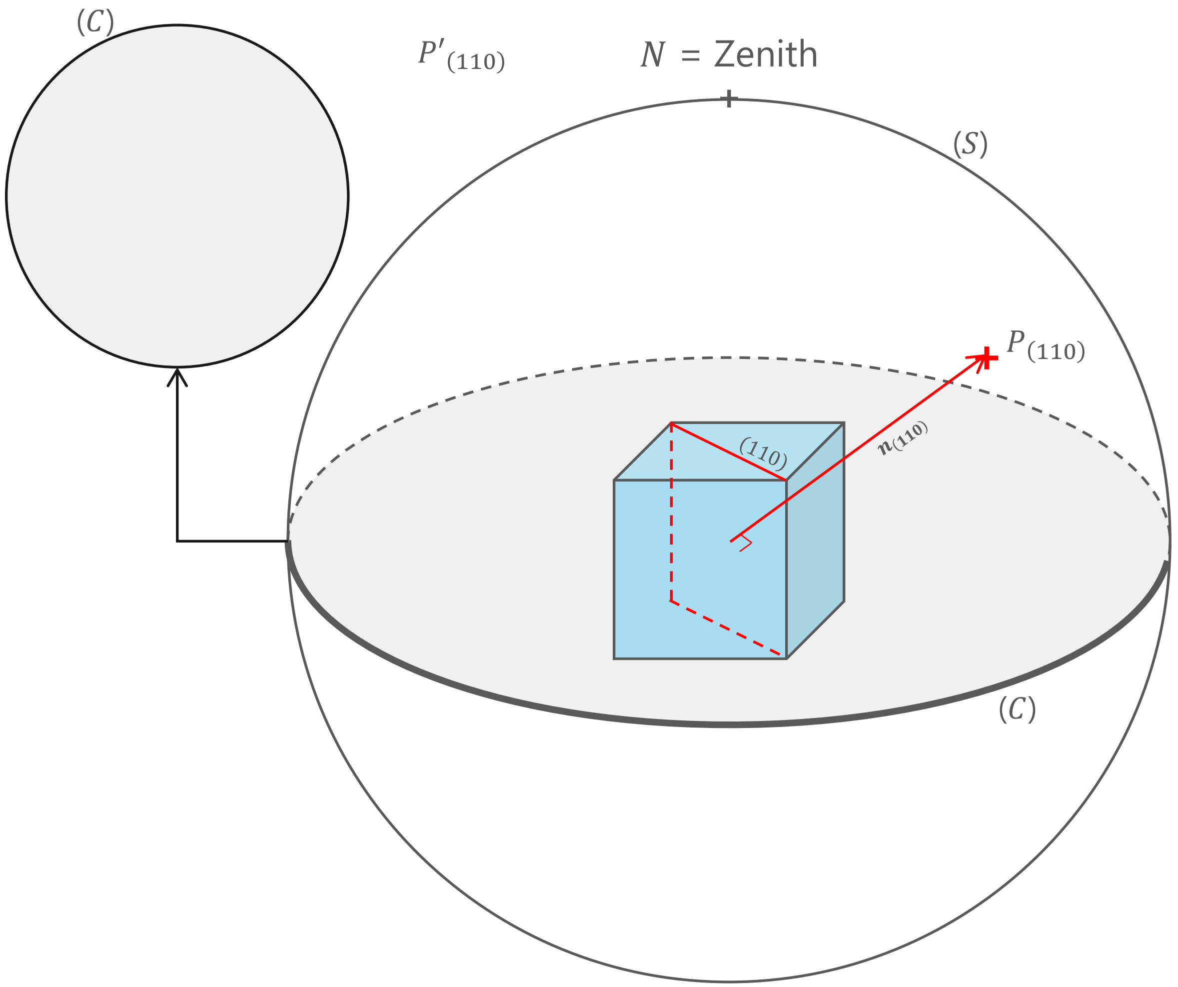

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- This normal intersects the sphere at a point $P_{(110)}$ on its surface.

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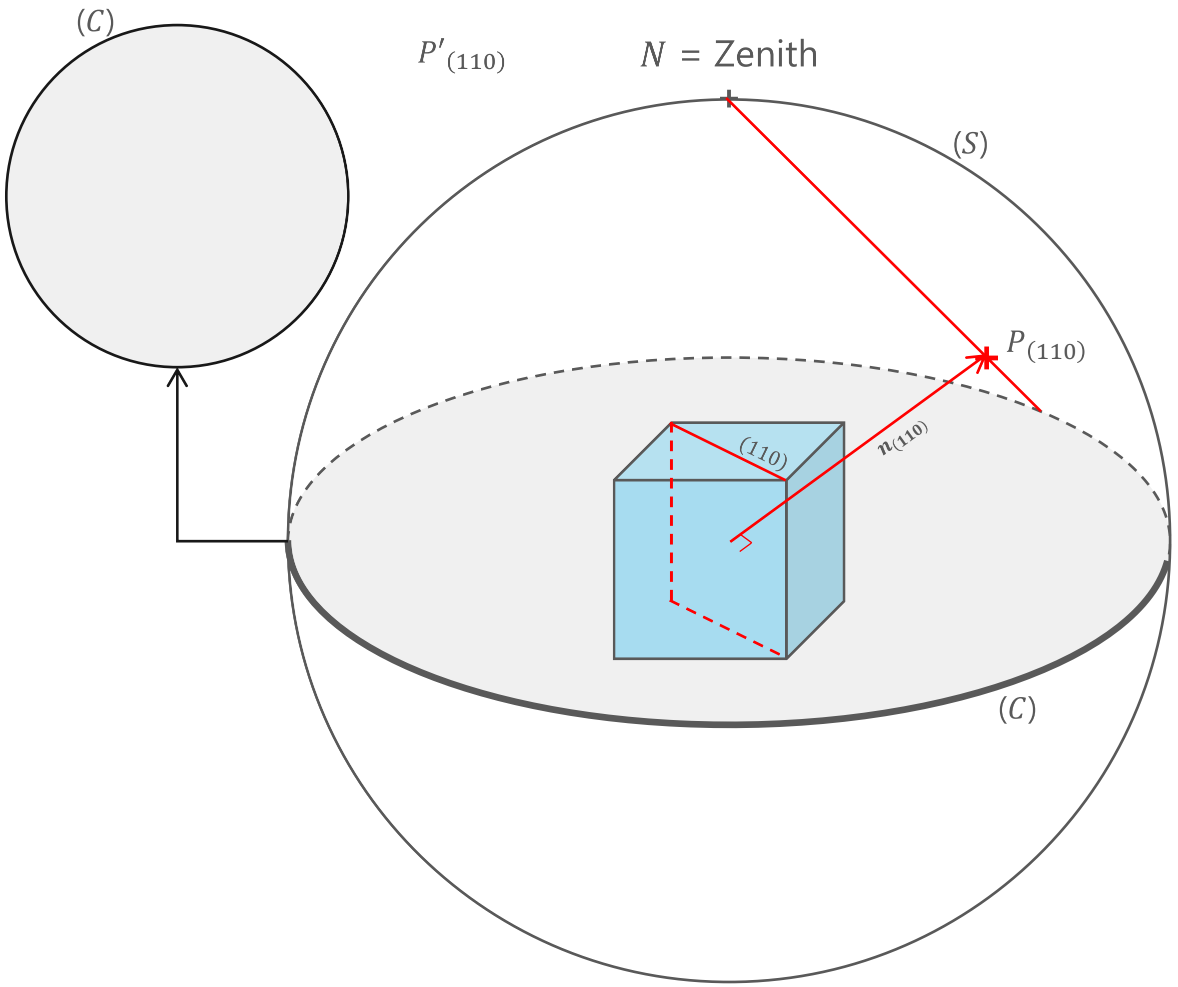

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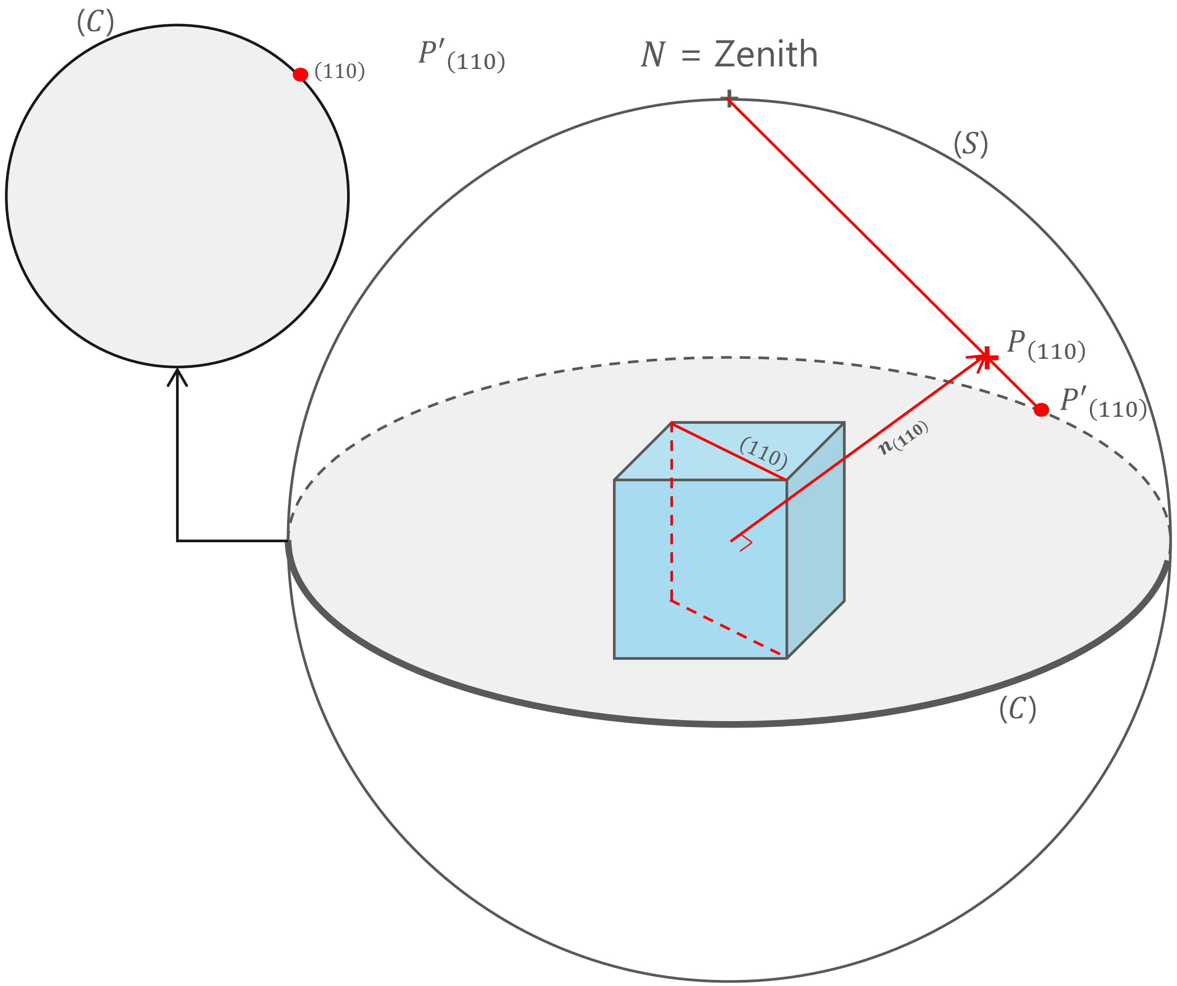
- Draw the normal vector $(\boldsymbol{n}_{(110)})$ to this plane, starting from the center of the sphere.
- This normal intersects the sphere at a point $P_{(110)}$ on its surface.
- Project point $P_{(110)}$ onto the equatorial plane (C) along a straight line from the North pole (N) of the sphere.

↳ The North pole is used as the projection point
↳ Any direction in the upper hemisphere is inverted ($\boldsymbol{n} \rightarrow -\boldsymbol{n}$) before projection.

Stereographic projection of a plane via its normal



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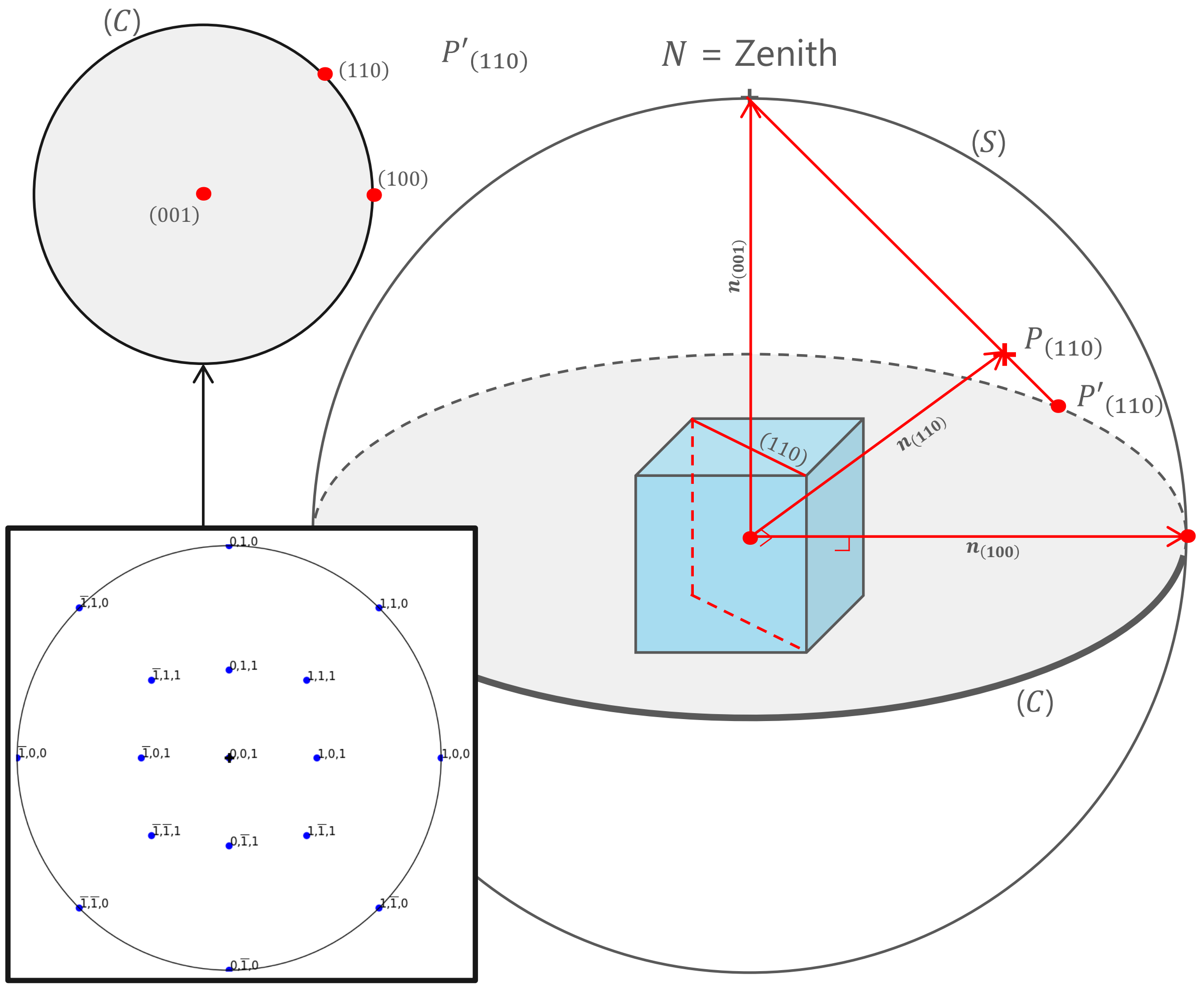
- Identify the crystallographic plane of interest, e.g., the (110) plane.
- Draw the normal vector ($\boldsymbol{n}_{(110)}$) to this plane, starting from the center of the sphere.
- This normal intersects the sphere at a point $P_{(110)}$ on its surface.
- Project point $P_{(110)}$ onto the equatorial plane (C) along a straight line from the North pole (N) of the sphere.
- The intersection point on the equatorial plane is the projection point $P'_{(110)}$.

Stereographic projection of a plane via its normal



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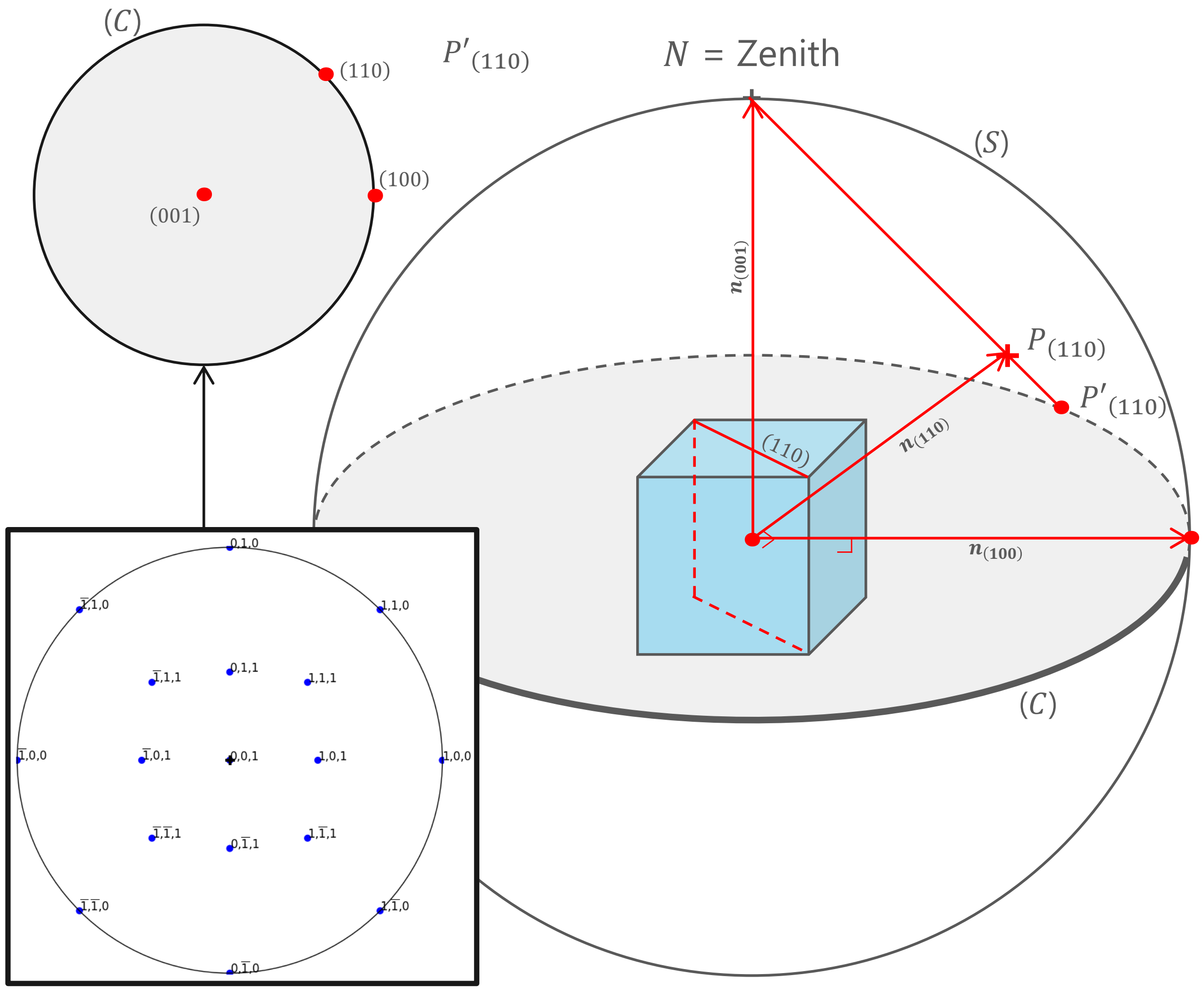

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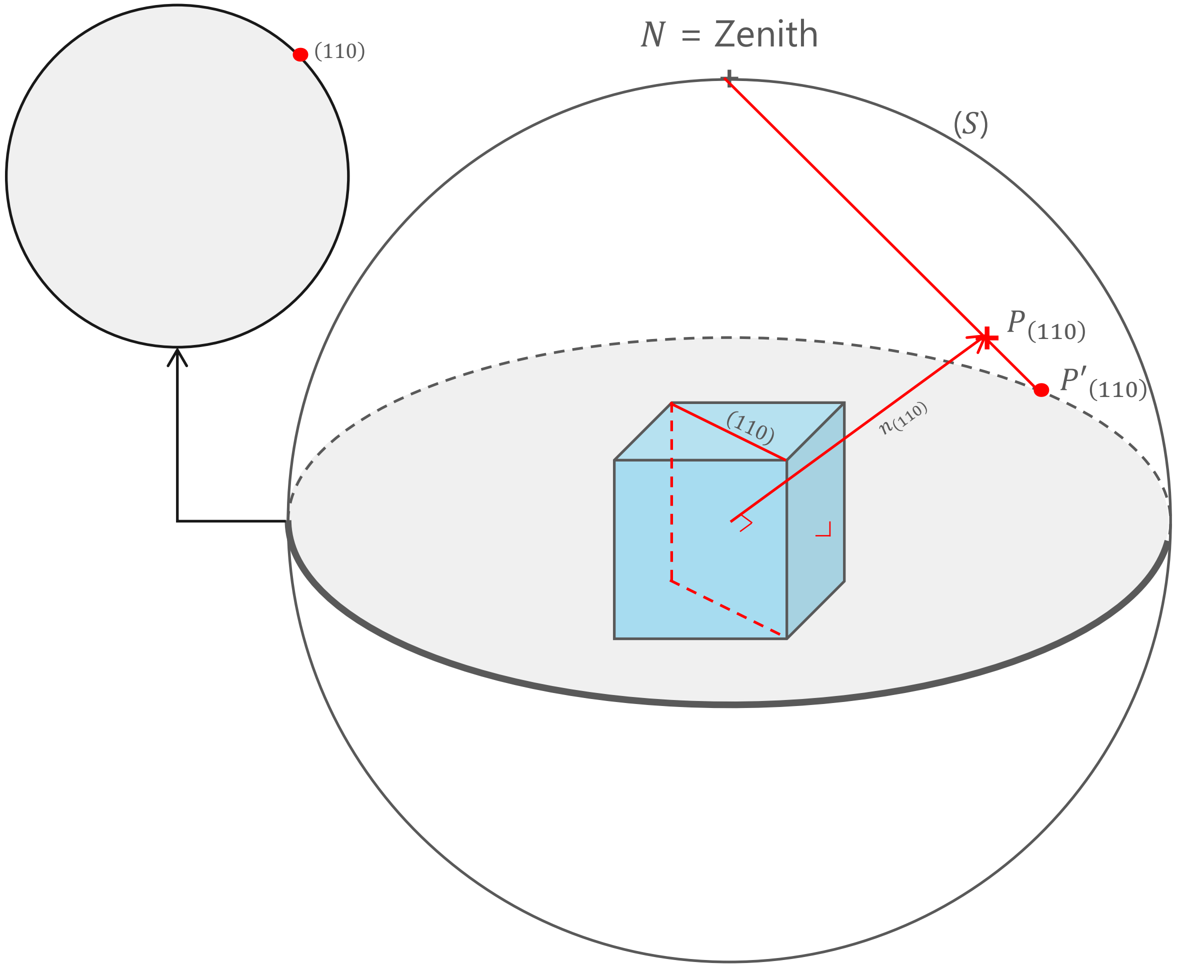
↳ Each crystallographic direction is represented by a single point on the stereographic projection

Stereographic projection of a plane via its trace



- Place the crystal at the center of the reference sphere (S).
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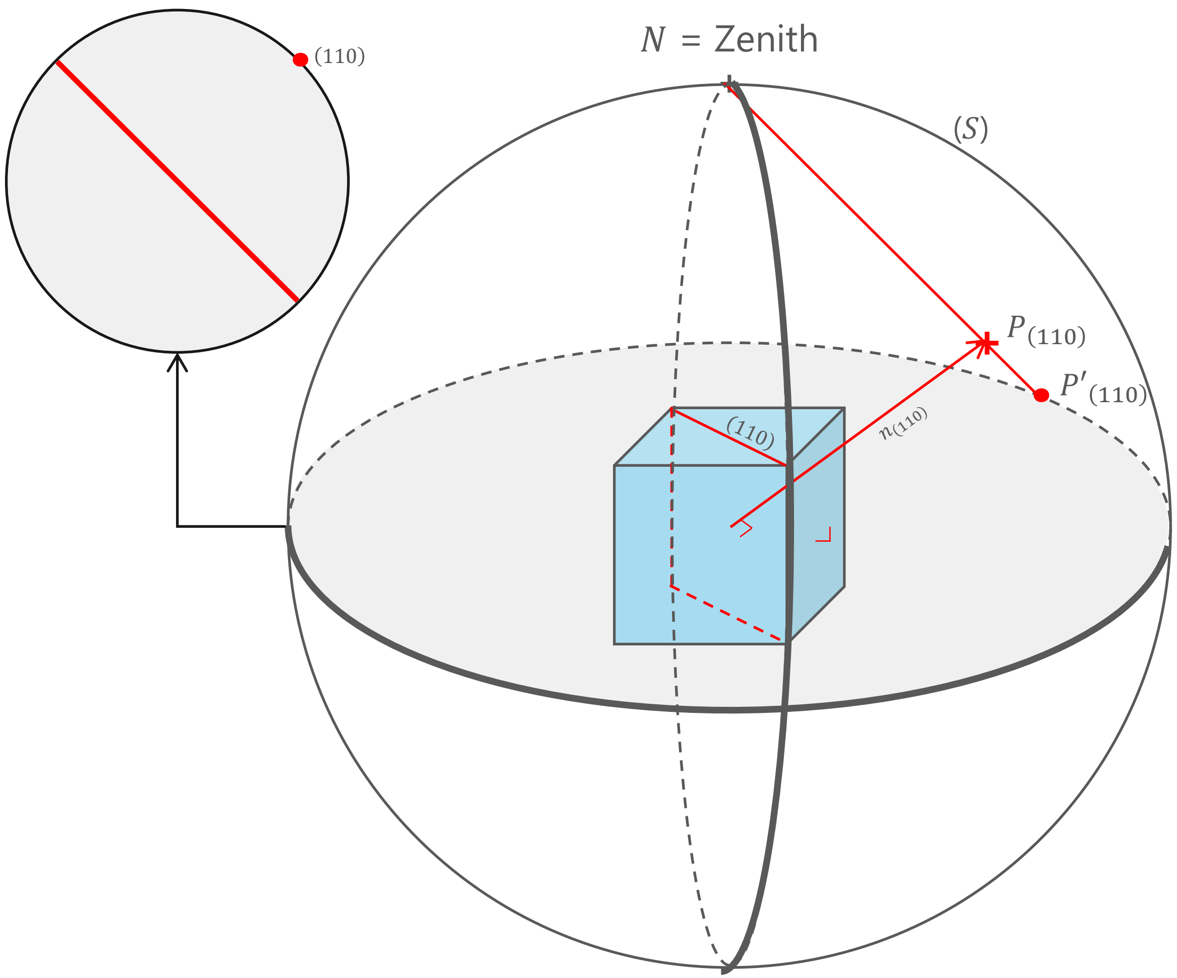
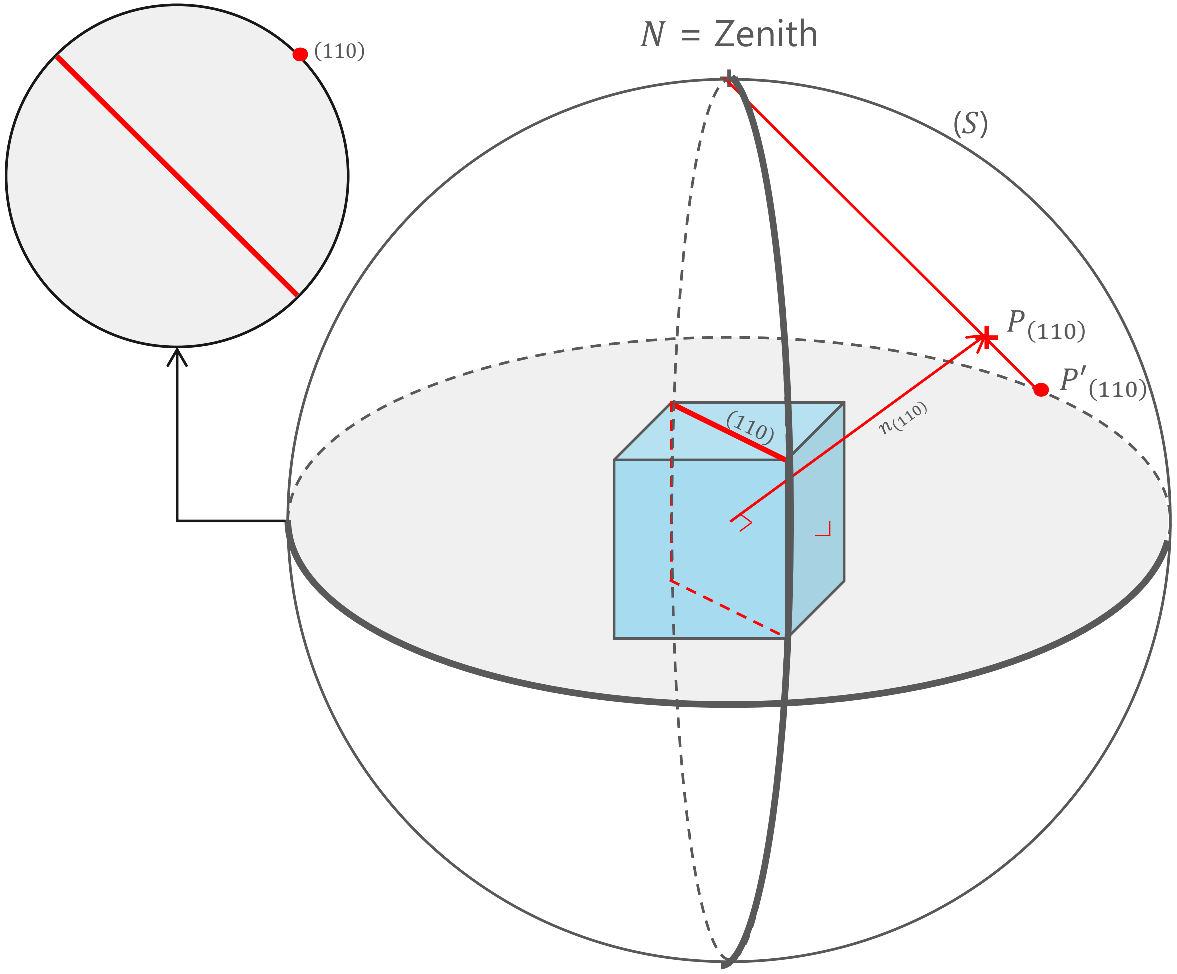
Stereographic projection of a plane via its trace



- Place the crystal at the center of the reference sphere (S).
- Identify the (110) crystallographic plane within the crystal.
- Imagine the intersection between this plane and the sphere: this defines a great circle on the sphere's surface.
- This great circle represents all directions lying within the (110) plane.

Stereographic projection of a plane via its trace



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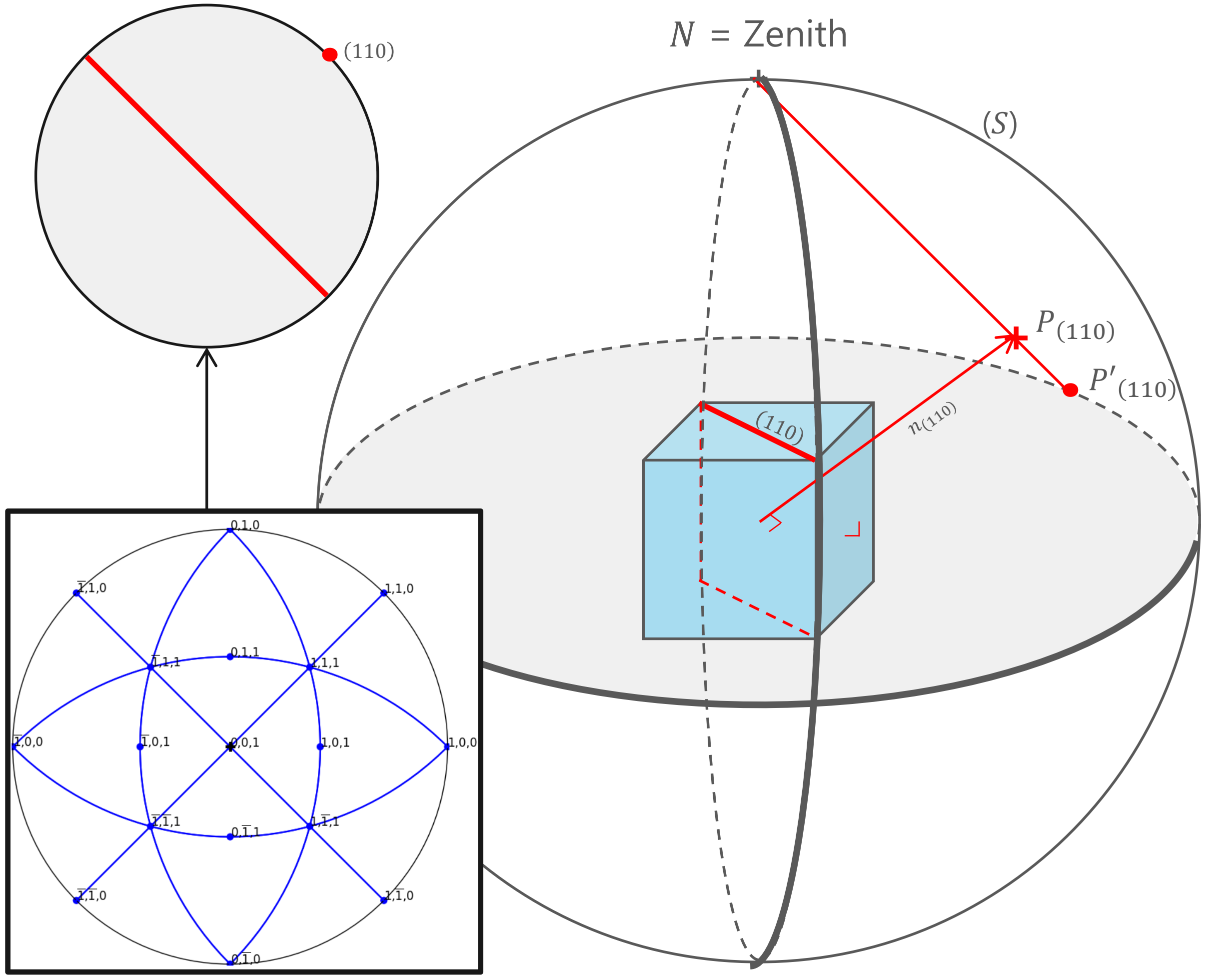

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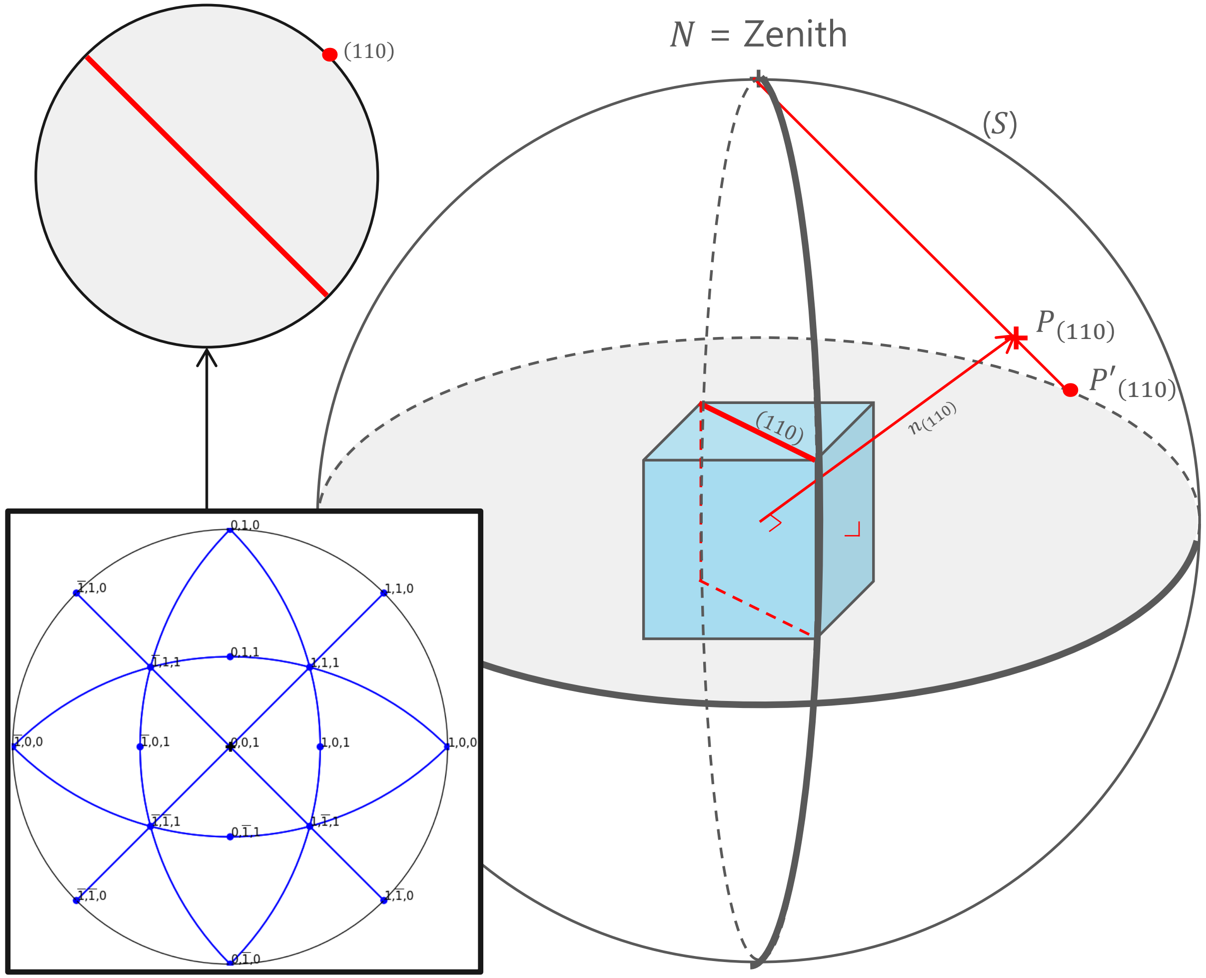

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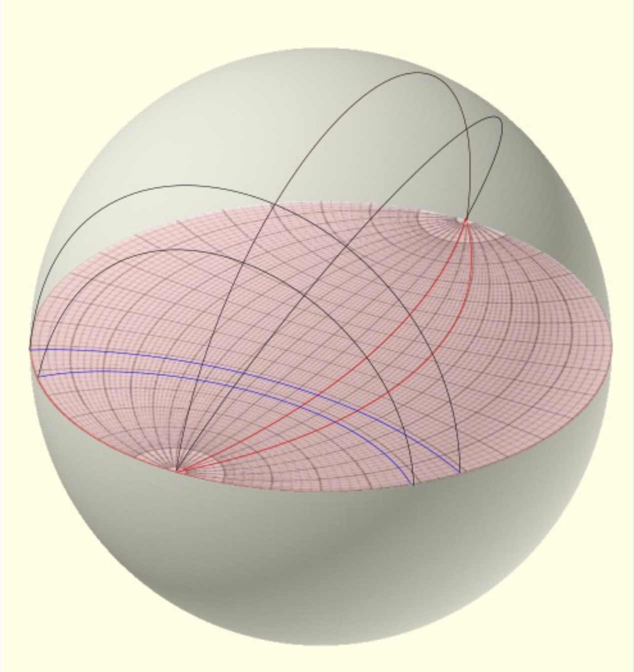
↳ The great circle trace shows all directions lying in the plane on the 2D projection.

The Wulff net



- The Wulff net is built using a stereographic projection of a reference sphere.

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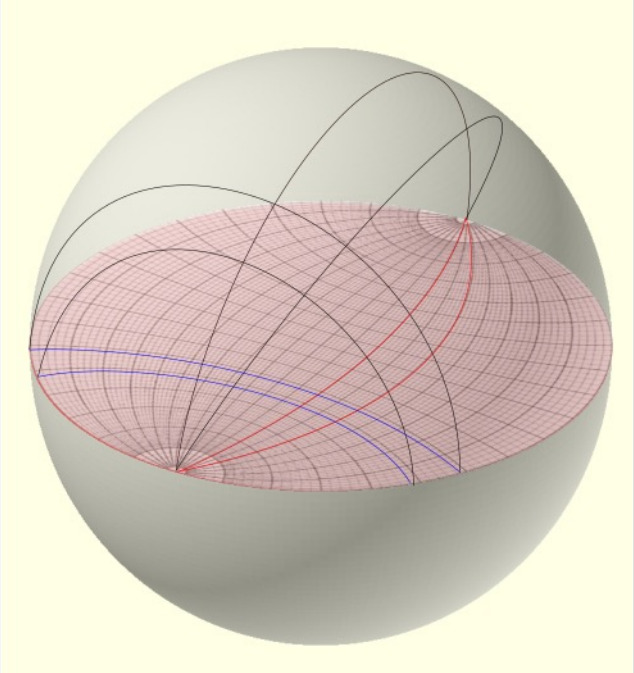

- The Wulff net is built using a stereographic projection of a reference sphere.
- It consists of a grid made up of two families of planes:
  - **Meridians:** represent planes with a constant azimuthal (rotation around the vertical axis) angle; they appear as straight lines radiating from the center.
  - **Parallels:** represent planes with a constant inclination angle from the vertical axis; they appear as concentric arcs or circles.

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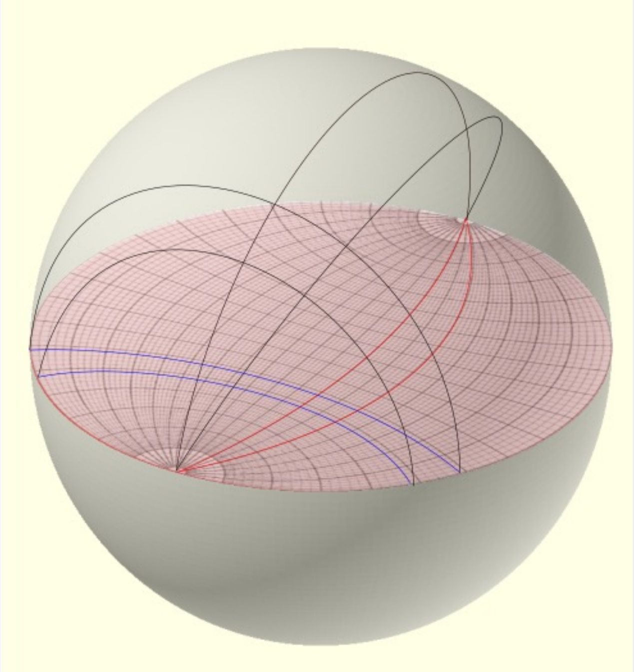

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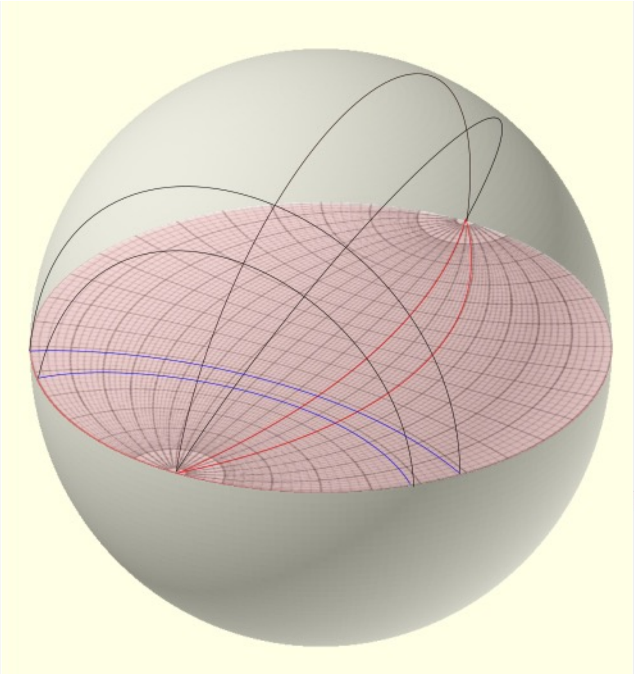

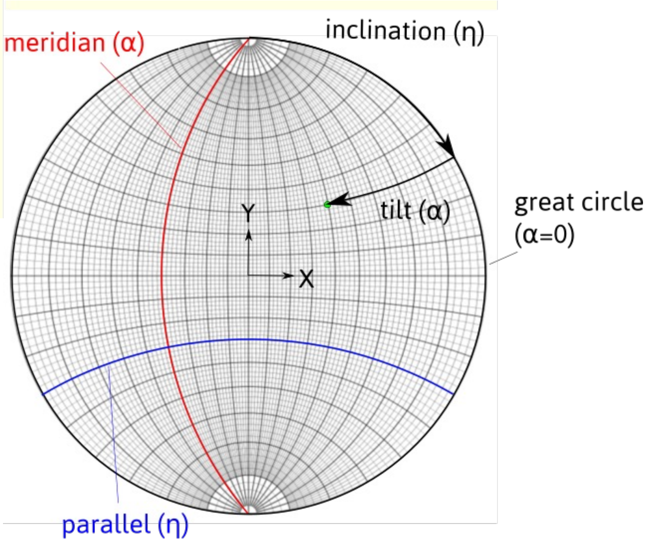

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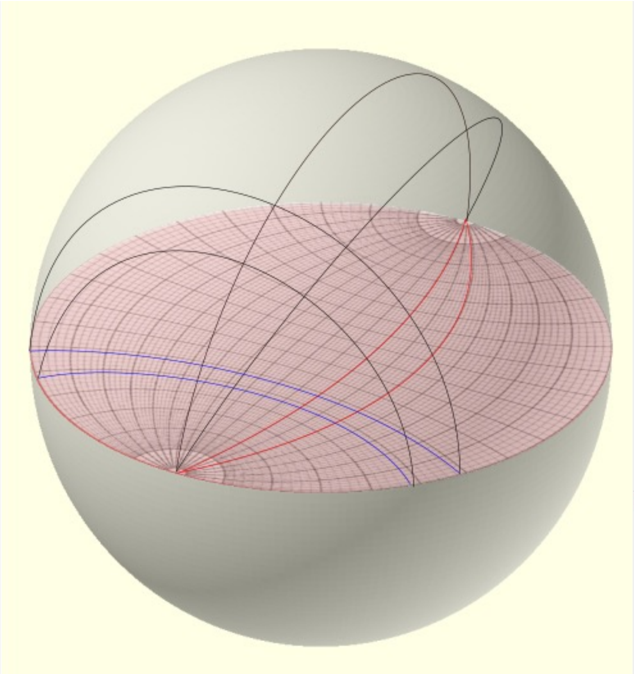

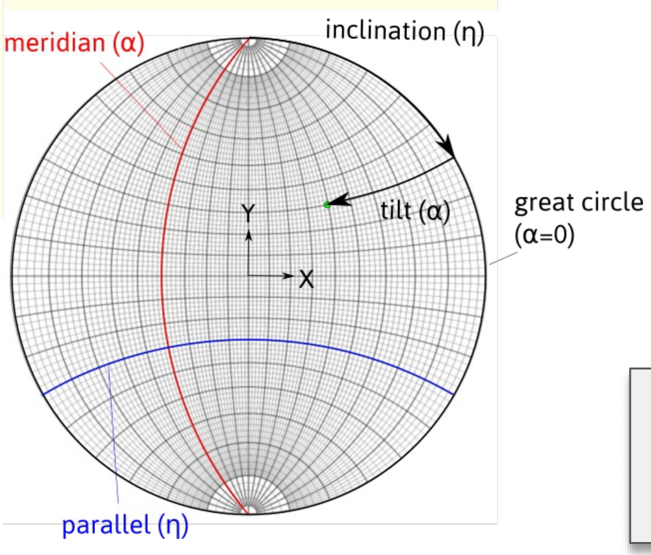

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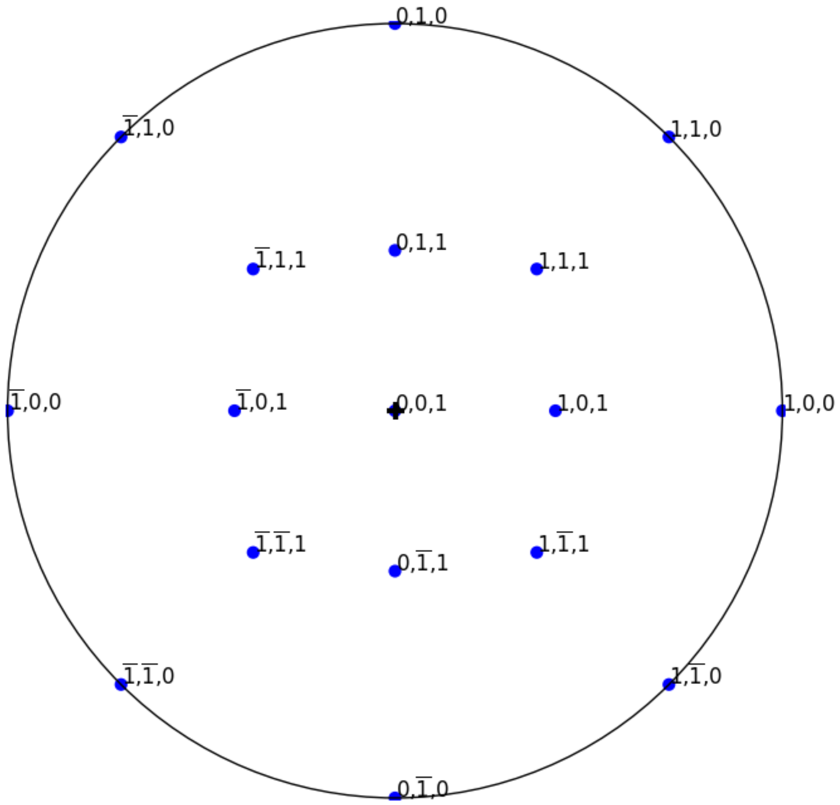
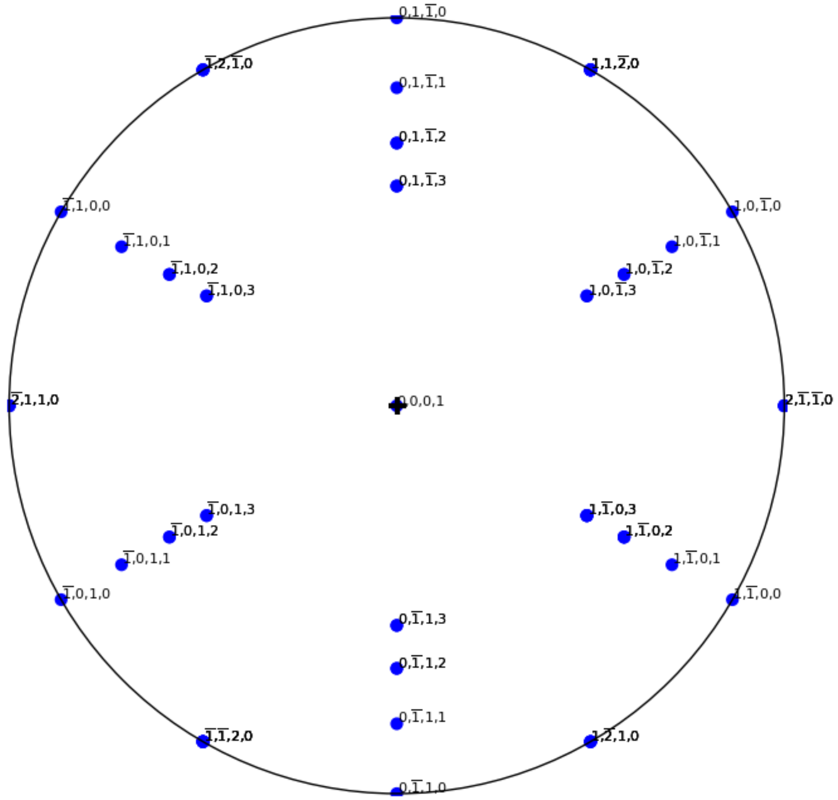
↳ The Wulff net serves as a reference tool to measure angles and construct stereographic projections accurately.

Some examples

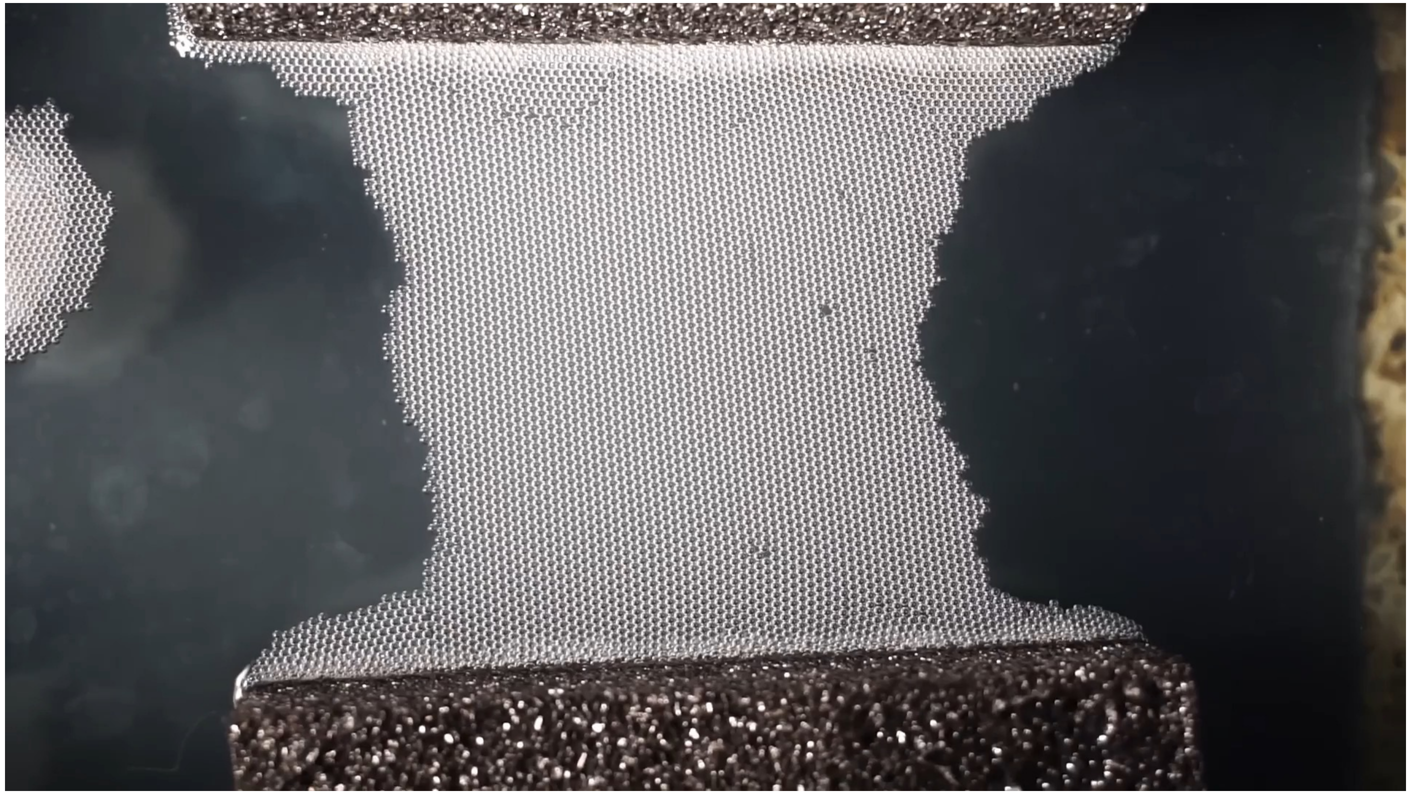
Cubic system

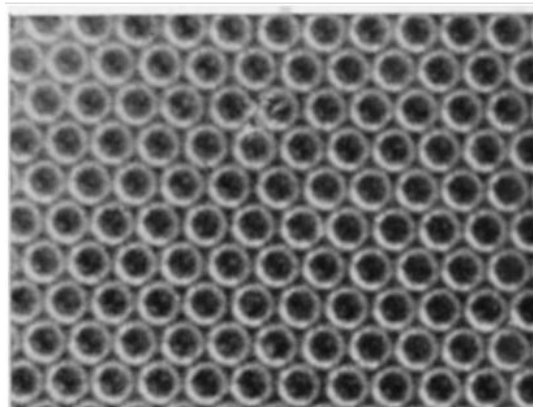


Hexagonal system



Dislocations





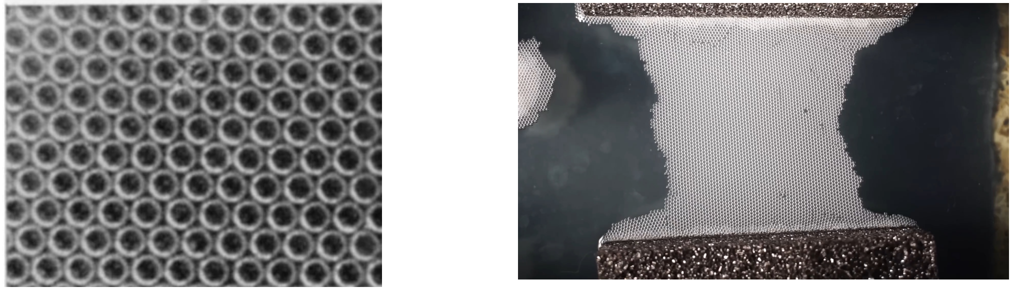
L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)

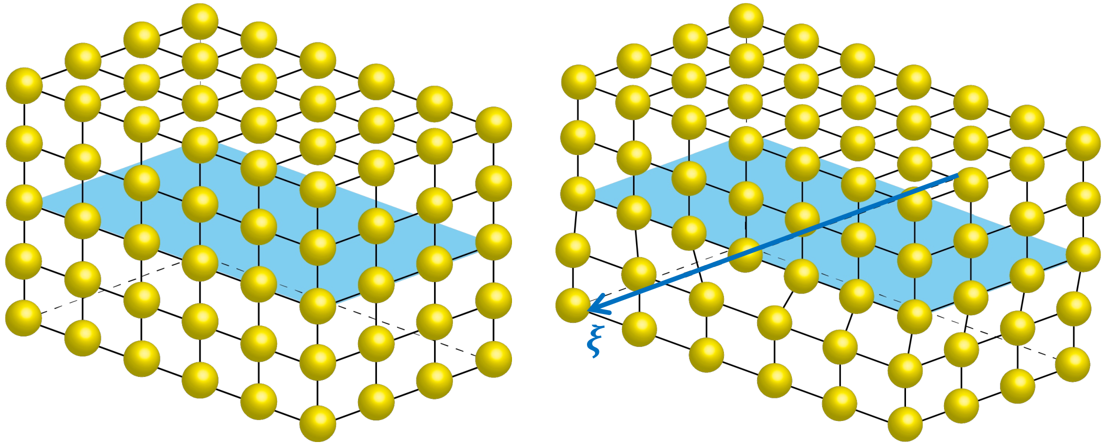
Dislocations

❖ Definition:

- Line defects in a crystalline material where atoms are misaligned.
- They represent a discontinuity in the crystal lattice that allows deformation to occur at lower stresses.
- Characterized by ***b*** and ξ.

L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)





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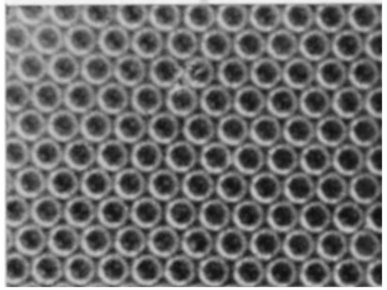

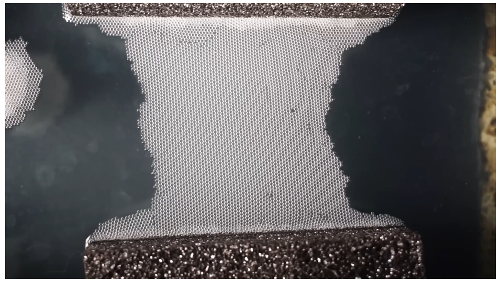

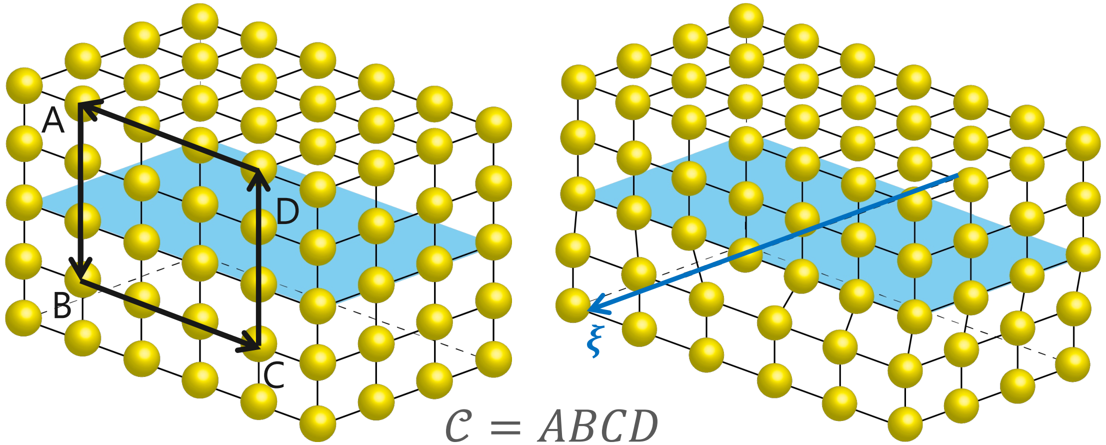

$\mathcal{C} = ABCD$

Dislocations

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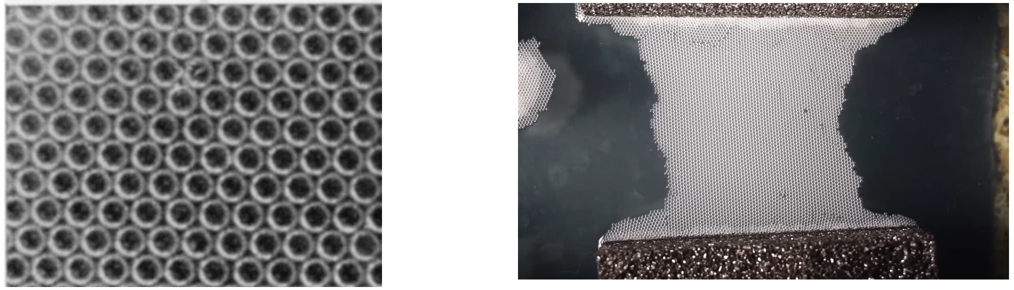
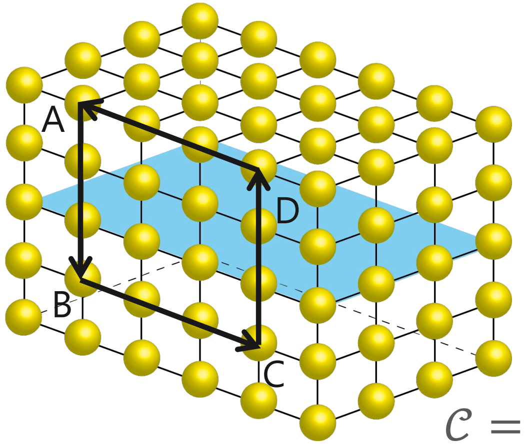
- Characterized by $\boldsymbol{b}$ and $\boldsymbol{\xi}$.

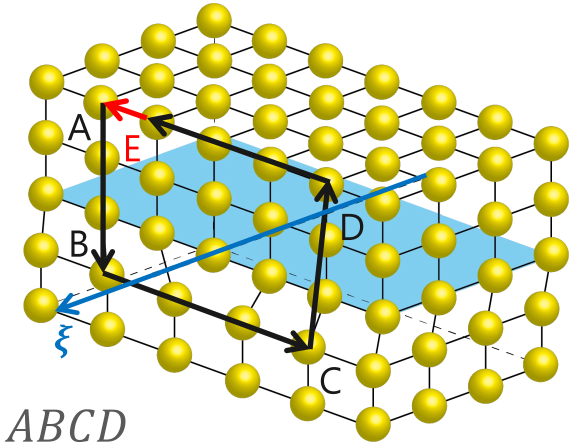
❖ Types:

- *Edge dislocation:* characterized by an extra half-plane of atoms inserted into the lattice ($\boldsymbol{b} \perp \boldsymbol{\xi}$).
- *Screw dislocation:* results from a shear distortion, forming a helical ramp in the crystal lattice ($\boldsymbol{b} \parallel \boldsymbol{\xi}$).
- *Mixed dislocation:* a combination of edge and screw dislocations ($\boldsymbol{b} \angle \boldsymbol{\xi}$).

L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)







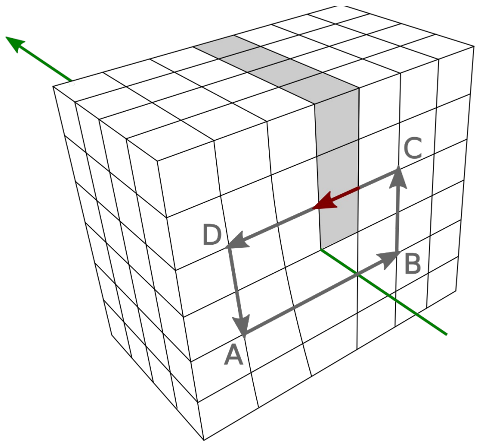
$\mathcal{C} = ABCD$

$$\boldsymbol{EA} = \boldsymbol{b} = \oint_{\mathcal{C}} \boldsymbol{U}^{e} \mathrm{d}\boldsymbol{r} \Rightarrow b_i = \oint_{\mathcal{C}} \frac{\partial u_i}{\partial x_j} dx_j$$

Dislocation types

Edge

Characterized by an extra half-plane of atoms inserted into the lattice ($\boldsymbol{b} \perp \boldsymbol{\xi}$).



Symbol: ⊥

F. C. Frank, *Proc. Phys. Soc. A*, 62, 131–134 (1949)

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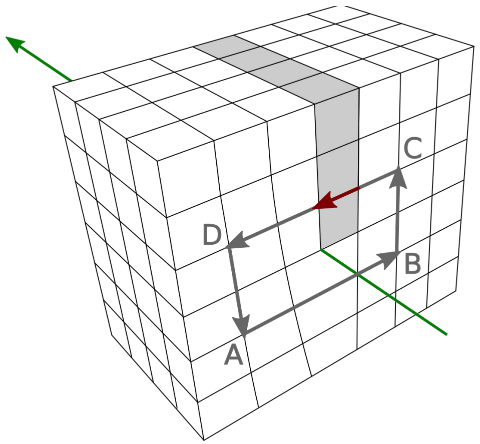

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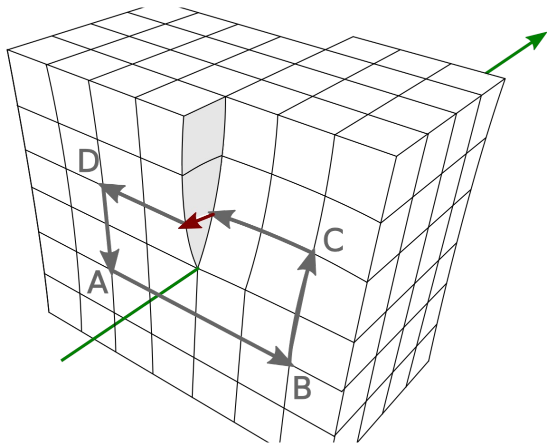
Screw

Results from a shear distortion, forming a helical ramp in the crystal lattice ($\boldsymbol{b} \parallel \xi$).



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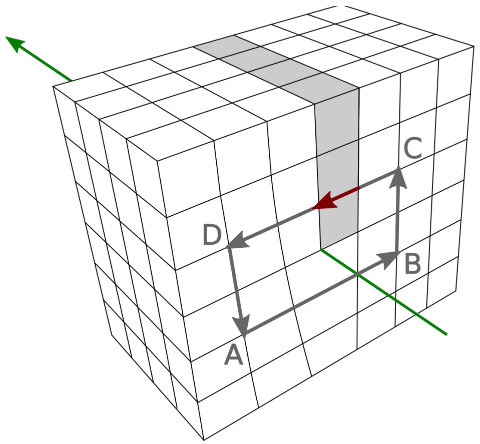

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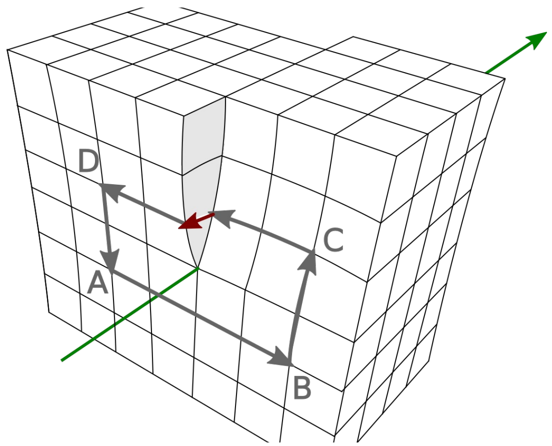
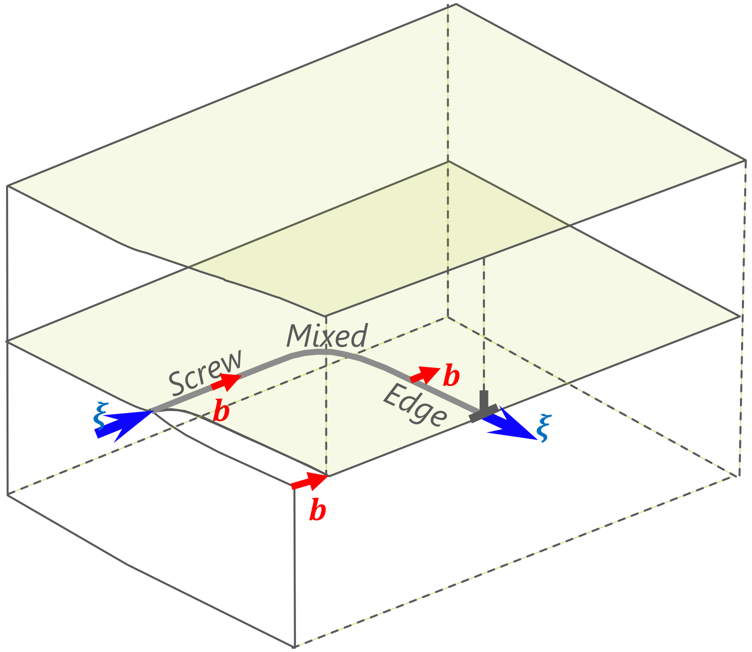


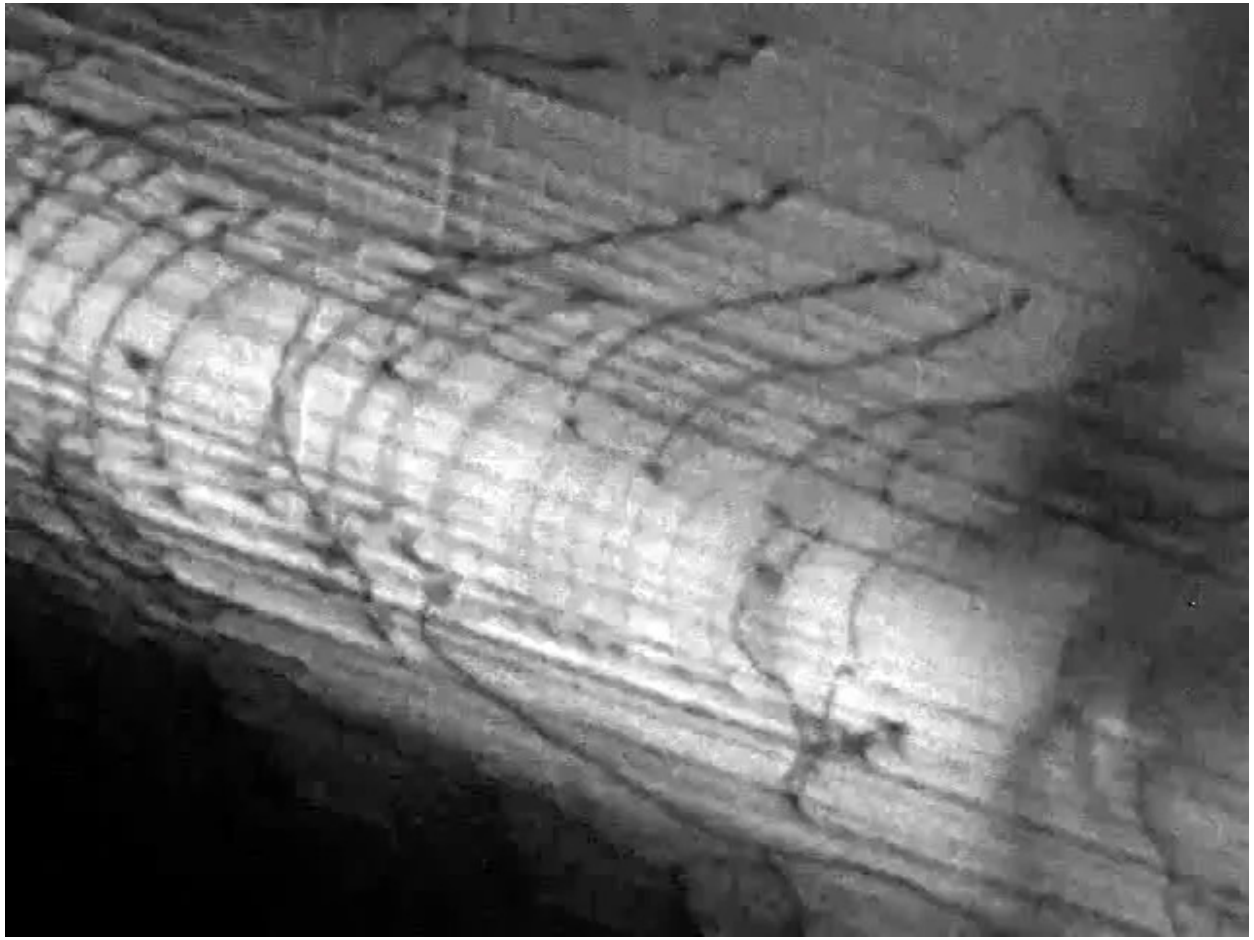
Mixed

A combination of edge and screw dislocations ($\boldsymbol{b} \angle \boldsymbol{\xi}$).



F. C. Frank, *Proc. Phys. Soc. A*, 62, 131–134 (1949)

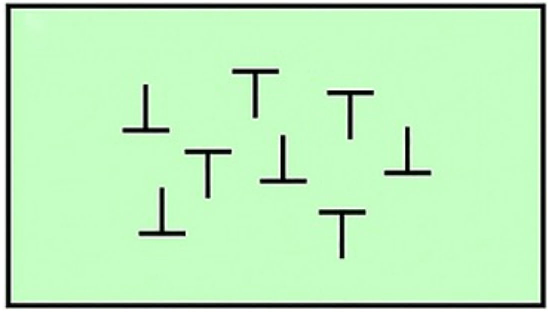
Dislocation glide -in 304 stainless steel



J. Kacher et al., *Proc. Acta Mater.*, 60, 6657–66724 (2012)

Different types of dislocation densities

Statistically stored dislocations (SSD)



⇒ No lattice curvature at long range

J.F. Nye, *Acta Metal.* (1953)

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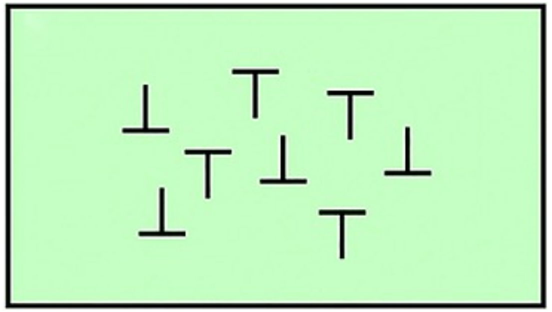

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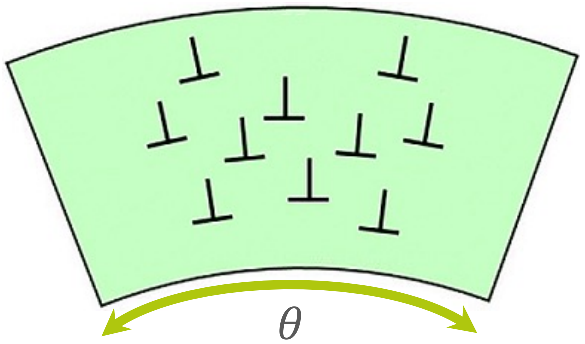
Geometrically necessary dislocations (GND)



⇒ Lattice curvature at long range

$$\rho_{GND} \propto \frac{\theta}{\|\boldsymbol{b}\|\delta x}$$

J.F. Nye, *Acta Metal.* (1953)

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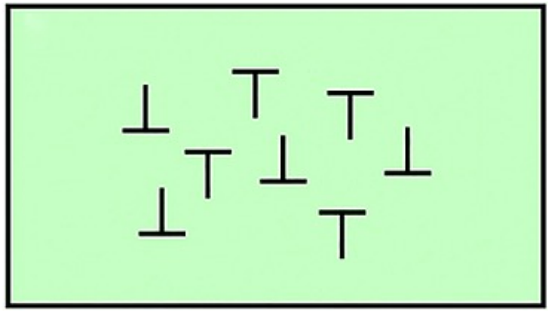

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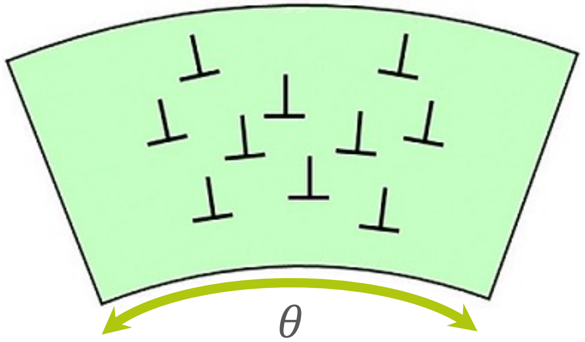


⇒ Lattice curvature at long range

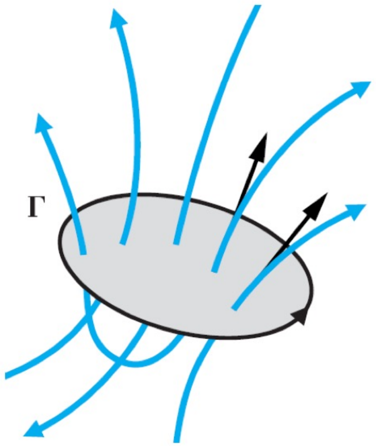
$$\rho_{\mathrm{GND}} \approx \frac{1}{\|\boldsymbol{b}\|}\|\boldsymbol{\alpha}\|;\ \|\boldsymbol{\alpha}\| = \sqrt{\alpha_{ij}.\alpha_{ij}}$$

$$\boldsymbol{\alpha} = curl\ \boldsymbol{\varepsilon} + \mathrm{tr}(\boldsymbol{\kappa_e}) \cdot \mathbb{I} - \boldsymbol{\kappa_e}^T$$

$$\kappa_{ij} \cong \frac{\Delta\omega_i}{\Delta x_j};\ \varepsilon_{ij} = \frac{1}{2}\left(\frac{\partial u_i}{\partial x_j} + \frac{\partial u_j}{\partial x_i}\right)$$

J.F. Nye, *Acta Metal.* (1953)

The Nye tensor



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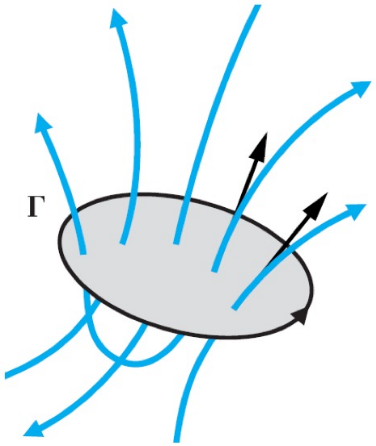

- The net Burgers vector on the closed circuit Γ limiting the surface S

$$\boldsymbol{B} = \left(\oint_{\Gamma} \boldsymbol{u}(\boldsymbol{x})\boldsymbol{n}\mathrm{d}S \right)\boldsymbol{b} = \oint_{\Gamma} \boldsymbol{\alpha}\boldsymbol{n}\mathrm{d}S$$

Where

$$\boldsymbol{\alpha} = \boldsymbol{b} \otimes \boldsymbol{u}(\boldsymbol{x})$$

J.F. Nye, *Acta Metal.* (1953)

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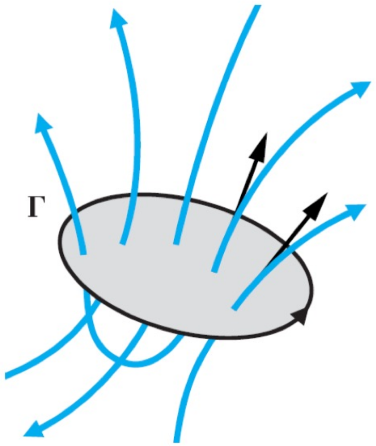

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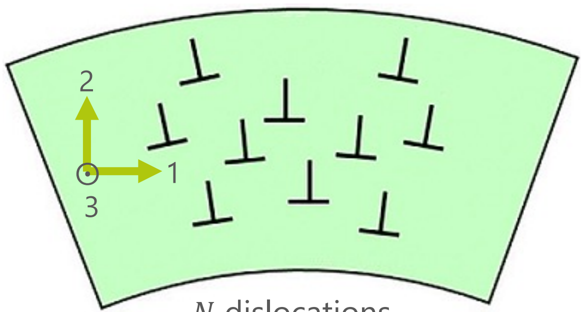


Edge dislocations (1 of 2 types)

$$\boldsymbol{B}_{edge} = Nb\boldsymbol{e_1} = \boldsymbol{\alpha}\boldsymbol{e_3}S$$

$$\boldsymbol{\alpha} = \frac{N}{S}\boldsymbol{b}\otimes\boldsymbol{u}(\boldsymbol{x}) = \frac{N}{S}b\boldsymbol{e_1}\otimes\boldsymbol{e_3} = \rho_{GND}\boldsymbol{e_1}\otimes\boldsymbol{e_3}$$

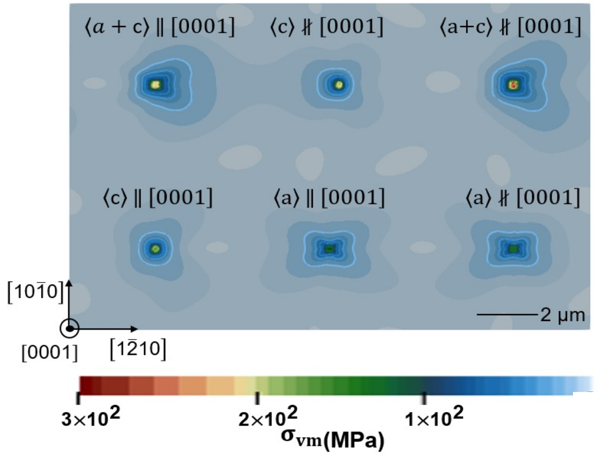
Screw dislocations

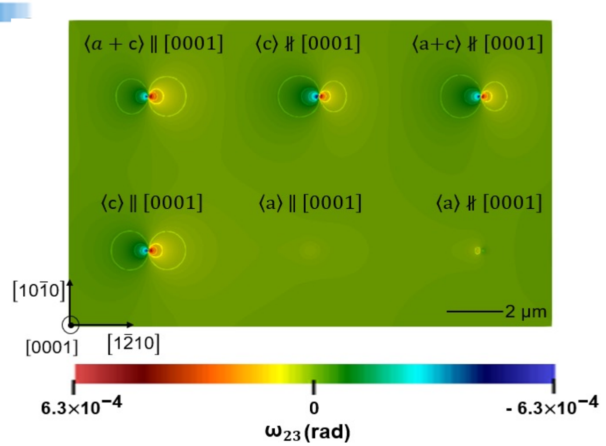
$$\boldsymbol{B}_{screw} = Nb\boldsymbol{e_3} = \boldsymbol{\alpha}\boldsymbol{e_3}S$$

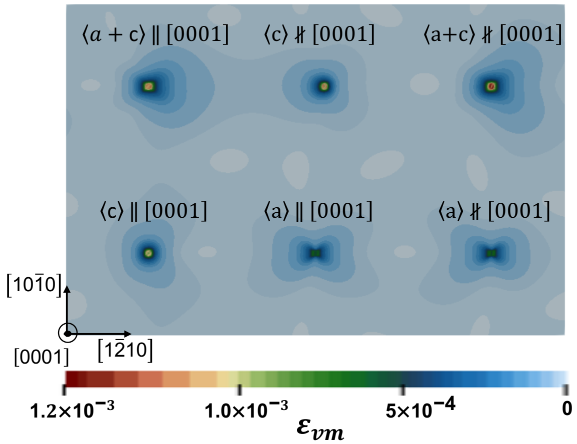
$$\boldsymbol{\alpha} = \frac{N}{S}\boldsymbol{b}\otimes\boldsymbol{u}(\boldsymbol{x}) = \frac{N}{S}b\boldsymbol{e_3}\otimes\boldsymbol{e_3} = \rho_{GND}\boldsymbol{e_3}\otimes\boldsymbol{e_3}$$

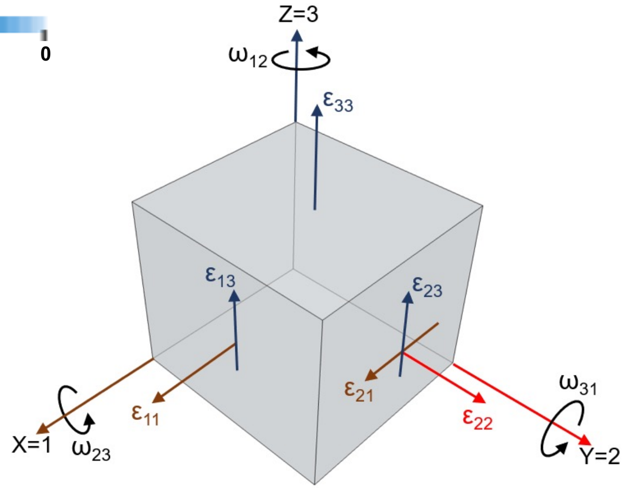
J.F. Nye, *Acta Metal.* (1953)

Elastic fields of dislocations





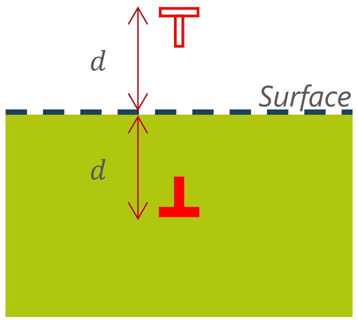




Dislocations induce elastic fields, which are key to their detection and analysis.

F. El Ajjouri et al., *Submitted.* (2026)

Effect of free surfaces on dislocations



❖ **Surface presence alters the elastic field of a dislocation loop.**

⇒ An image force appears to satisfy the zero-traction boundary condition at the surface.

⇒ Physically, it corresponds to the attraction between the real dislocation and a virtual dislocation of opposite Burgers vector ($-\boldsymbol{b}$) placed symmetrically on the other side of the surface.

P.P. Groves et al., *Philos. Mag.* (1970)

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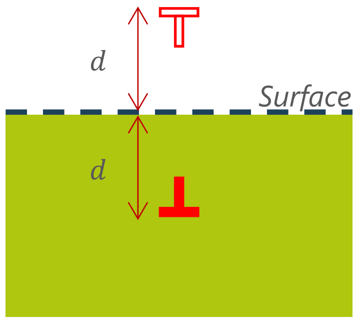

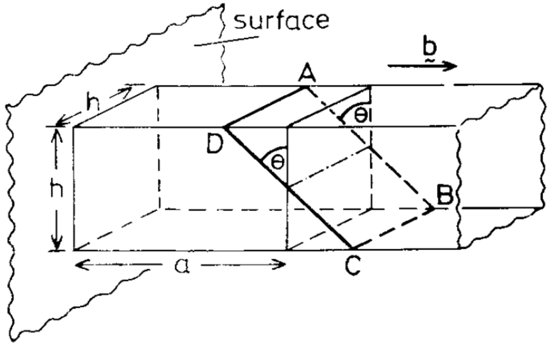

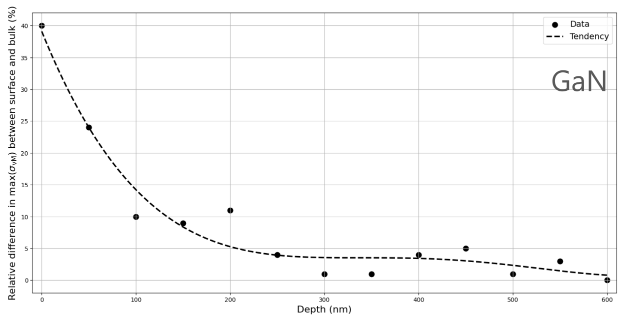

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  ⇒ Physically, it corresponds to the attraction between the real dislocation and a virtual dislocation of opposite Burgers vector ($-\boldsymbol{b}$) placed symmetrically on the other side of the surface.

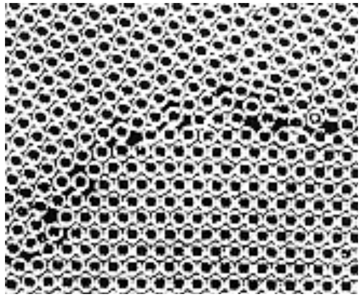
- **Surface effects are short-ranged:**
  - Significant influence when depth
    $$a \leq 2h - 3h$$
    a: distance from loop center to surface; h: loop size (length of one side of rectangular loop)
  - For $a > 3h$, the dislocation behaves as in an infinite medium.

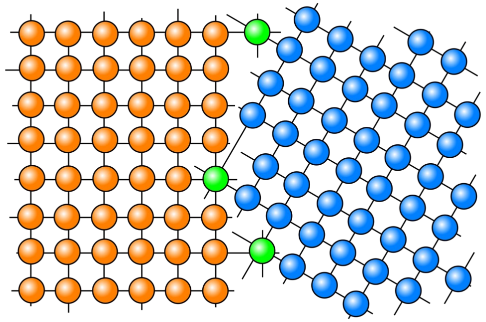
↳The behavior of dislocations is influenced by the presence of free surfaces

P.P. Groves et al., *Philos. Mag.* (1970)
F. El Ajjouri et al., *Submitted* (2026)

Grain boundaries

L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)





❖ **Definition:**

- GBs are interfaces where crystals of different orientations meet in a polycrystalline material.
- Five parameters to describe a GB
  - ✓ 1 disorientation angle (θ) → 1 parameter
  - ✓ Unitary rotation vector ($\boldsymbol{w}$) → 2 parameters
  - ✓ Unitary vector for the GB plane normal ($\boldsymbol{n}$) → 2 parameters

L. Priester, *Springer Science & Business Media.* (2012)

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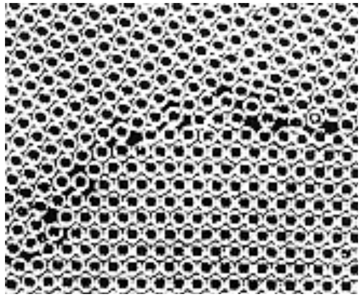
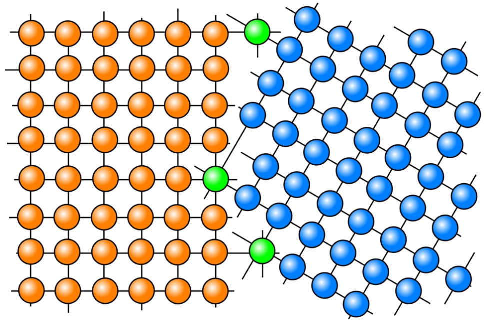
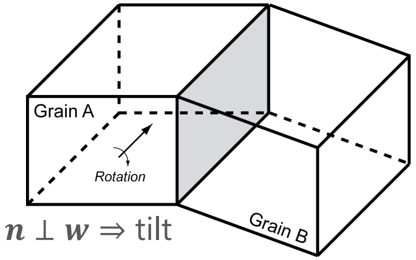
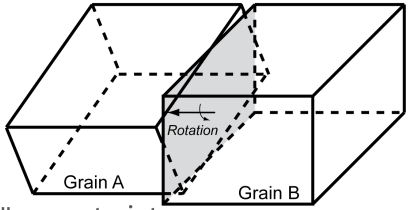






$n \perp w \Rightarrow$ tilt



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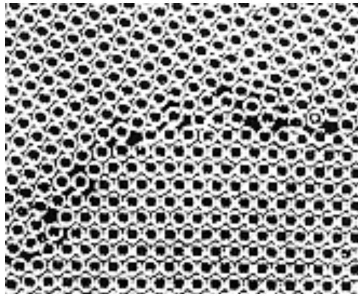

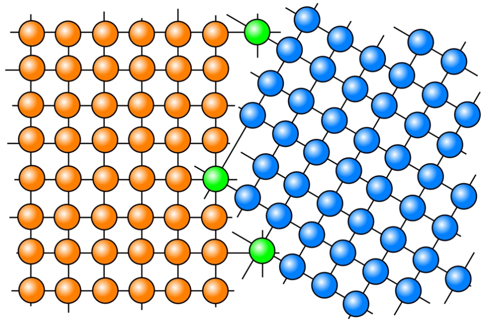

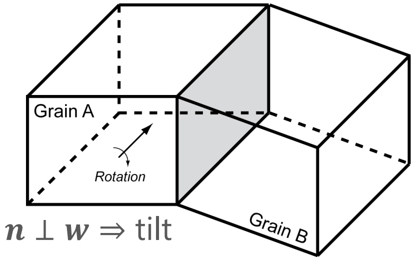

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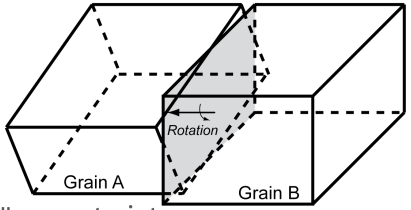

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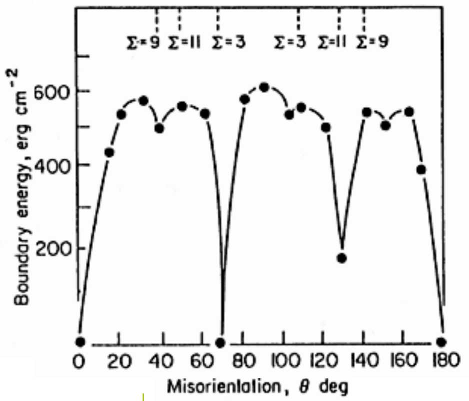
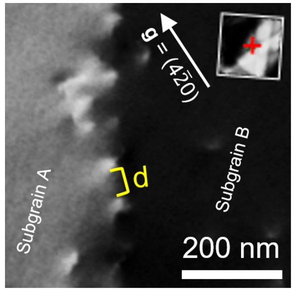
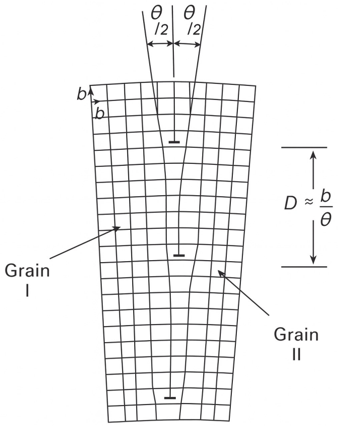




- $D = 60 \pm 5.52$ nm
- $\boldsymbol{b} = \frac{a}{2}\langle 110 \rangle$

$\Rightarrow \theta = 0.24 \pm 0.02°$



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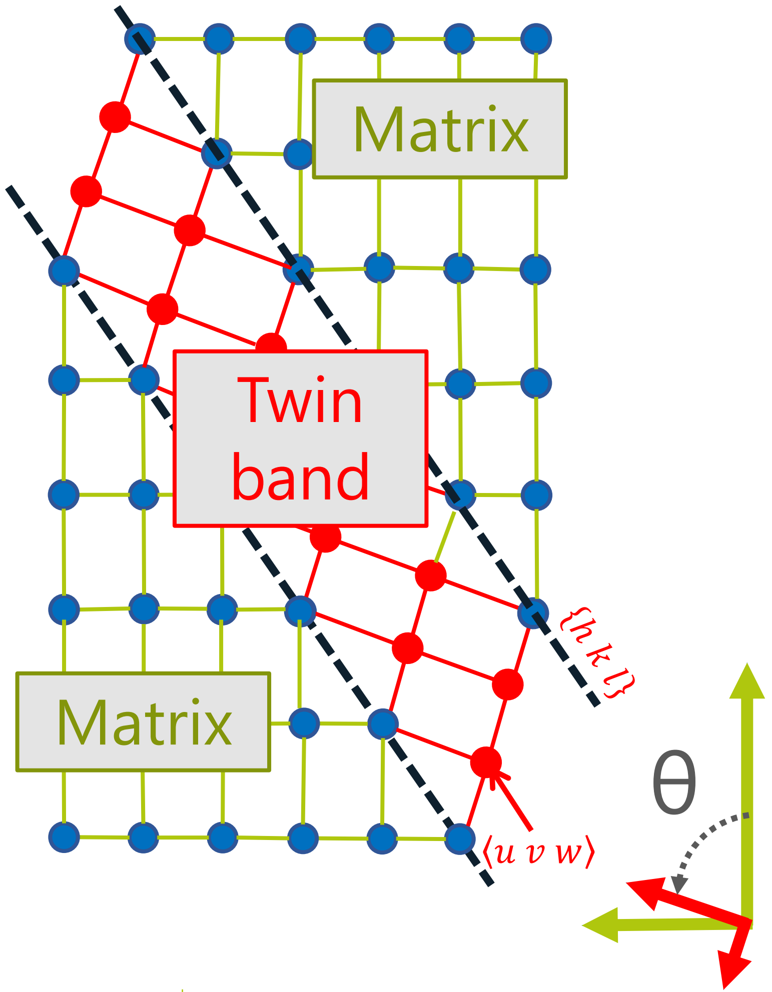
- Low-Angle GB (LAGB): $\theta \lessapprox 15°$

$$\theta = 2\sin^{-1}\left(\frac{|\boldsymbol{b}|}{2D}\right)$$

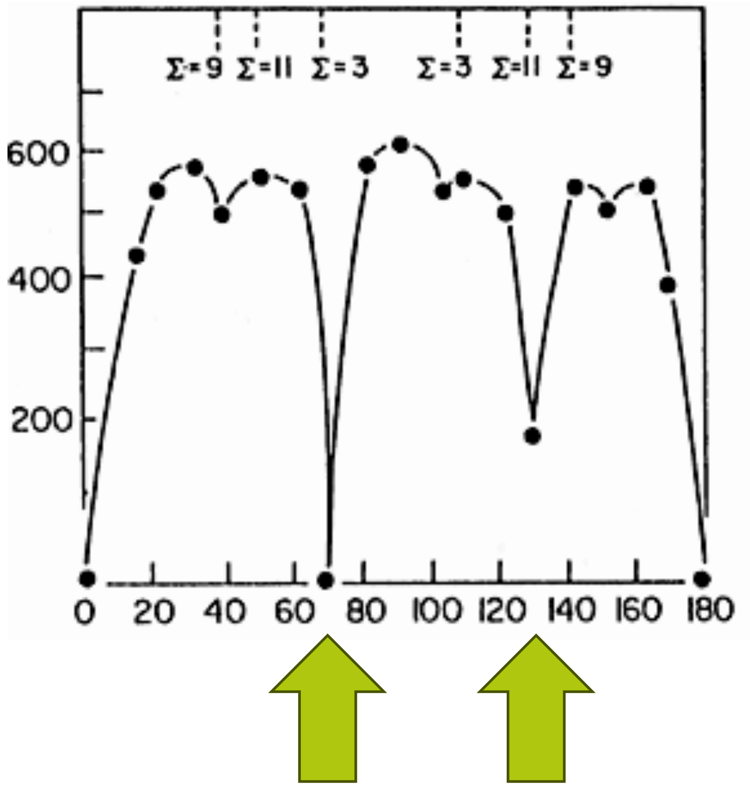
- High-Angle GB (HAGB): $\theta \gtrapprox 15°$

L. Priester, *Springer Science & Business Media.* (2012)
F. Habiyaremye *et al.*, MSEA (2021)

Focus on the twinning



- Identical crystalline structure
- Mirror reflection
- System: $\langle u\ v\ w\rangle\{h\ k\ l\}$
- Low-energy HAGB
- Tilted by a specific angle (θ)



L. Priester, *Springer Science & Business Media.* (2012)
R. Genin, *PhD Defense* (2025)

Punctual defects

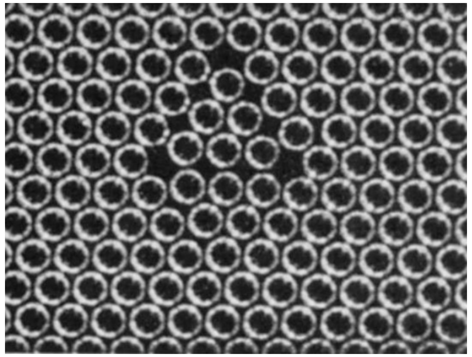
❖ Definition:

- Point defects are atomic-scale imperfections localized at or around a single lattice site in a crystalline material.

❖ Types:

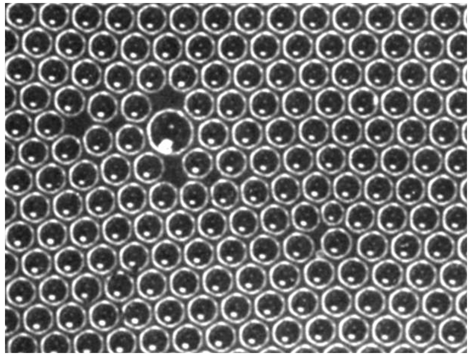
- *Vacancies:* Missing atoms in the lattice.
- *Interstitials:* Extra atoms positioned in the interstitial sites between the regular lattice points.
- *Substitutional defects:* Foreign atoms replacing host atoms in the lattice.

Vacancy



L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)

Interstitial



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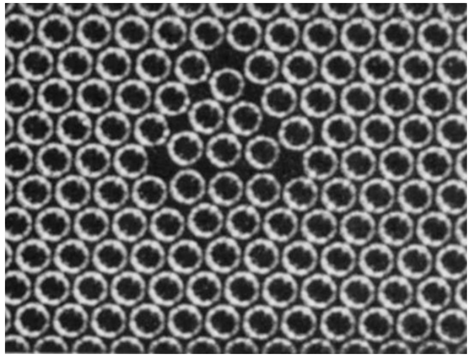
❖ Diffusion:

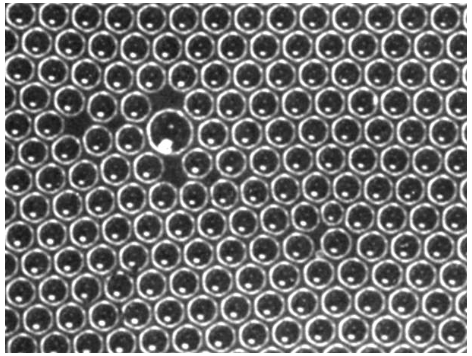
- They enable atomic diffusion, which is governed by Fick's Laws…

Vacancy



L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)

Interstitial



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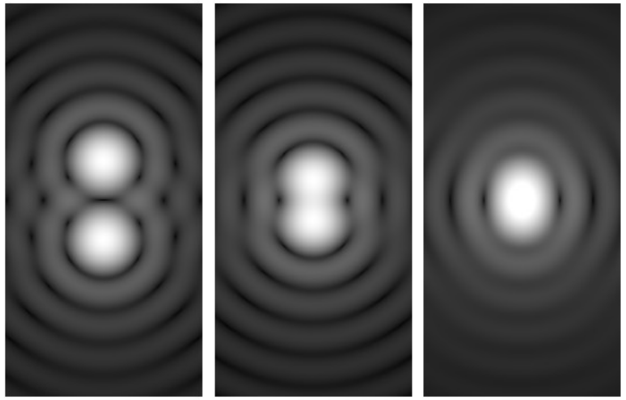
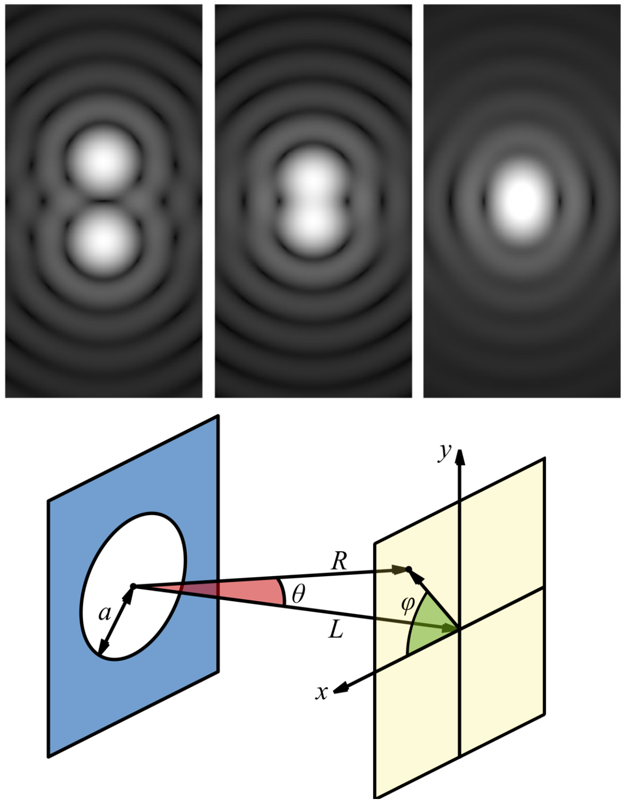
Limit of resolution



❖ **Airy disk and Rayleigh criterion**
- It refers to the ability to distinguish between very close details.

Limit of resolution



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$$I(\theta) = I_0 \left(\frac{J_1(ka \sin \theta)}{ka \sin \theta} \right); k = \frac{2\pi}{\lambda}$$

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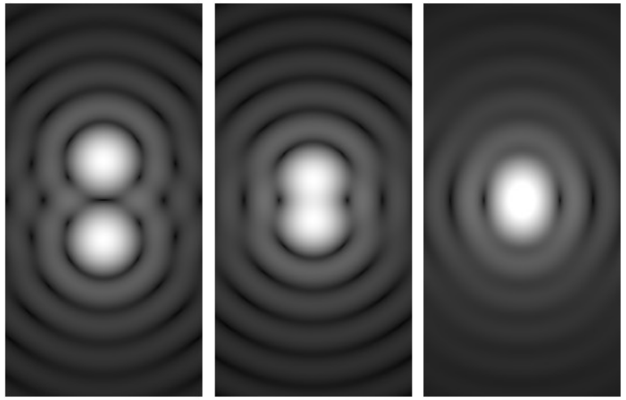

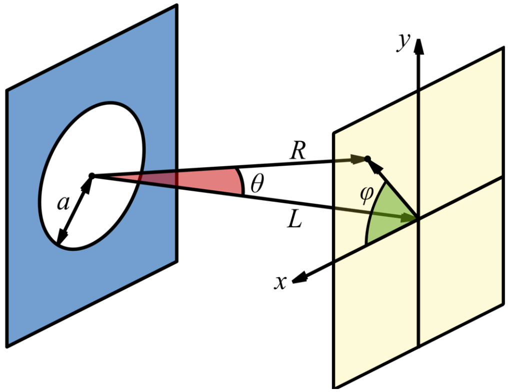

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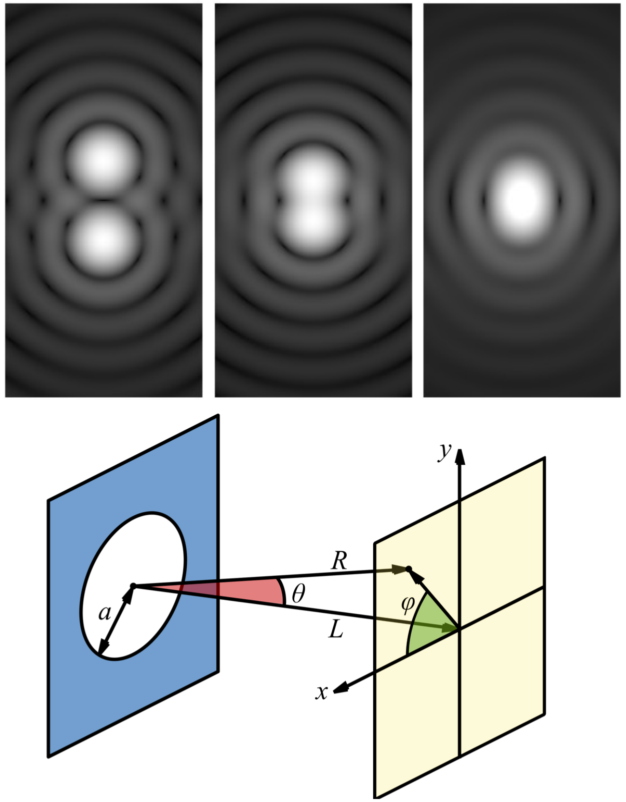
$$\sin\theta = 0.61\frac{\lambda}{a} \sim \theta$$

Limit of resolution



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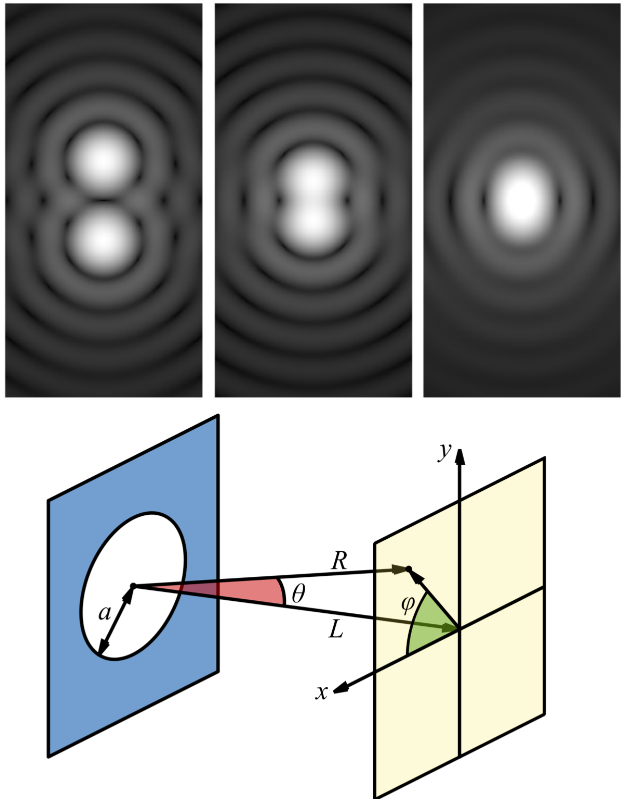

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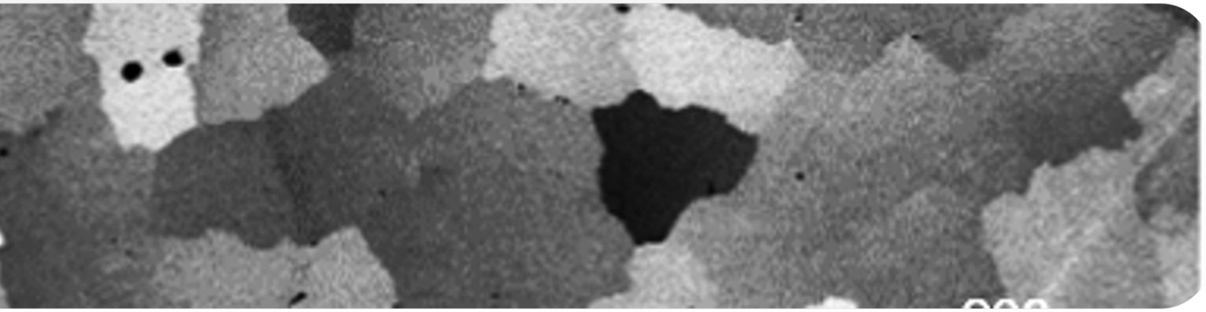
➯ Two objects cannot be resolved if they are closer than one wavelength λ.



Characterization by X-rays diffraction

Meso- Micro-meter scale

"Radiations studied by M. Röntgen"

Wilhelm RÖNTGEN
(1845 – 1923)
1 Nobel Prize (1901)

- **On the evening of November 8, 1895, in Würzburg, Germany:**
  - Röntgen made a screen fluoresce near a Crookes tube inside a black box.
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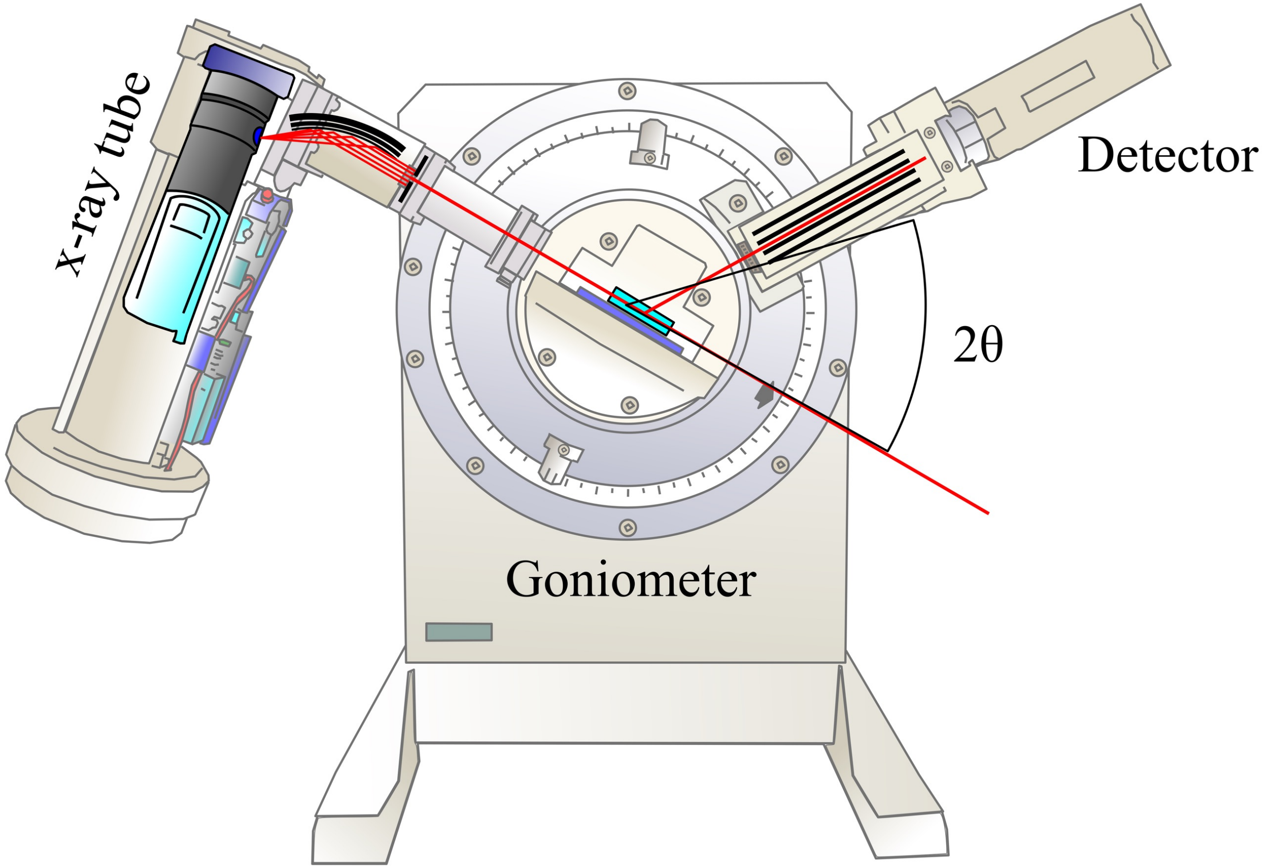
Since Röntgen did not understand their nature, he called these rays, X-rays!

W.C. Röntgen, *Ann. Phys.* (1898)

The experimental setup: the diffractometer

The diffractometer (Bragg-Brentano configuration)



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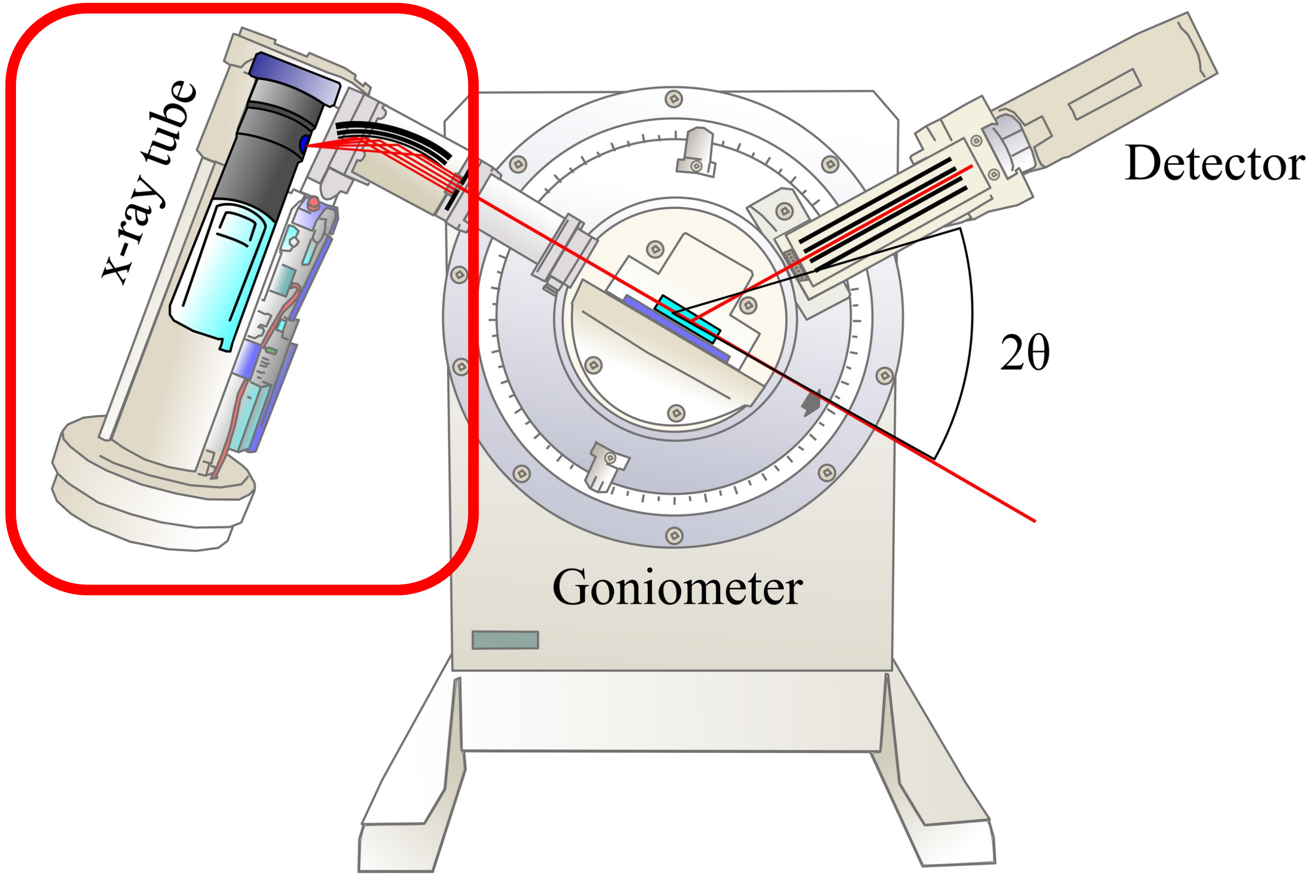
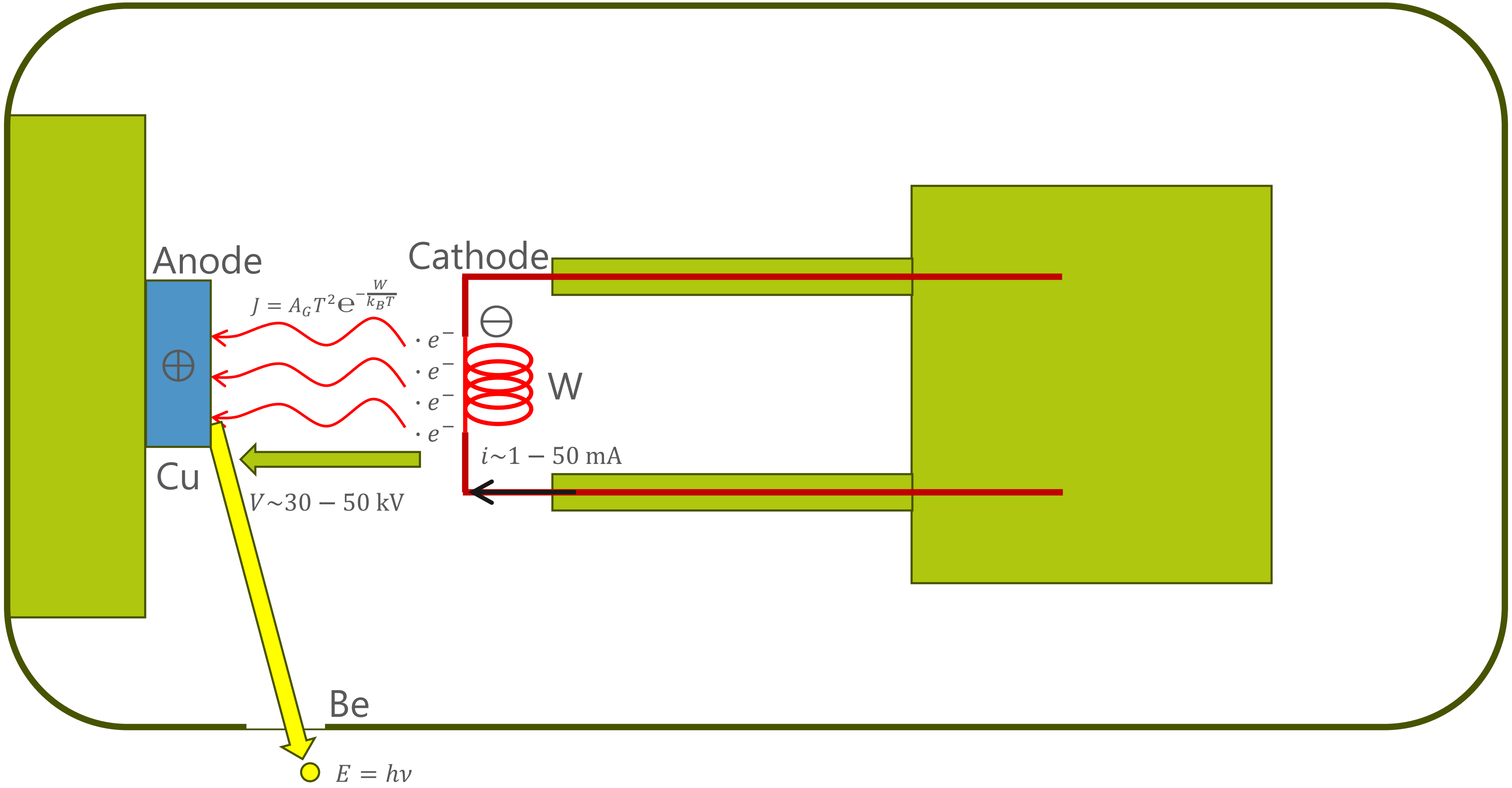


X-ray production



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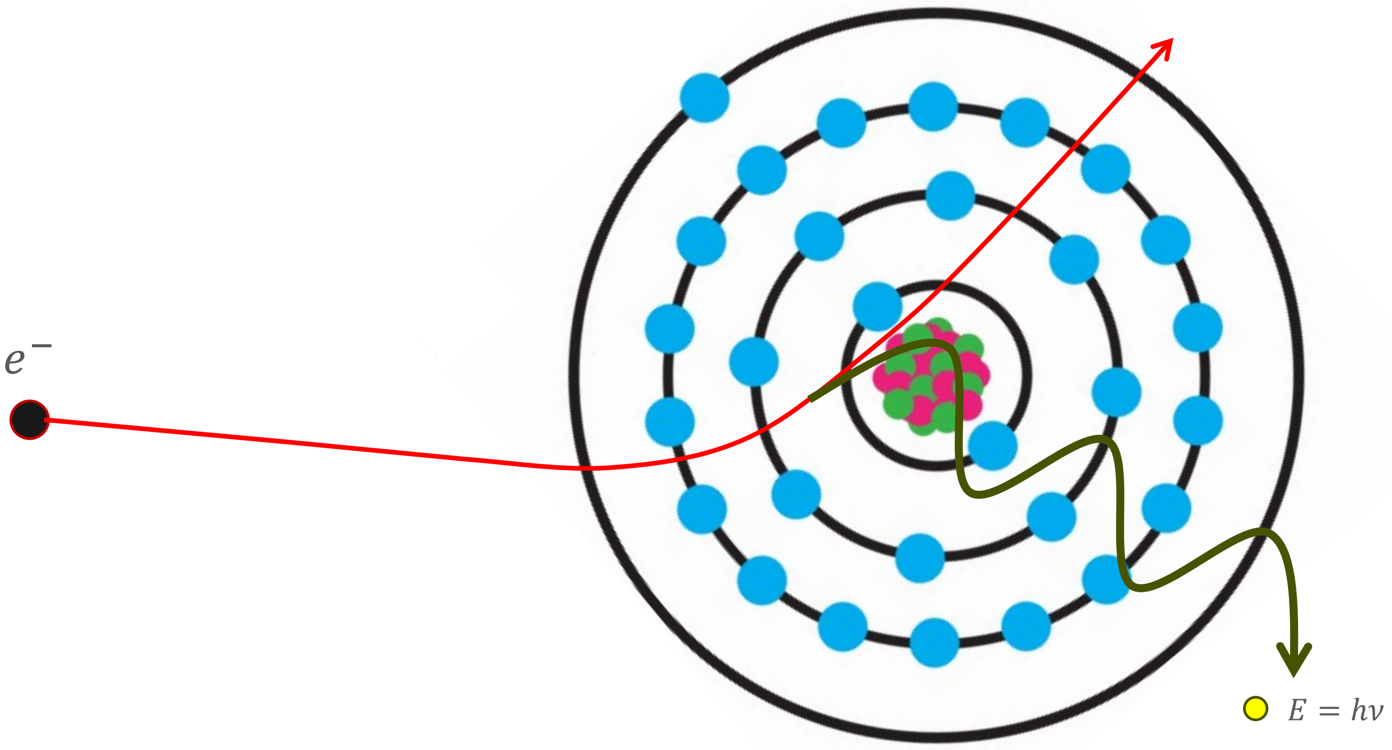
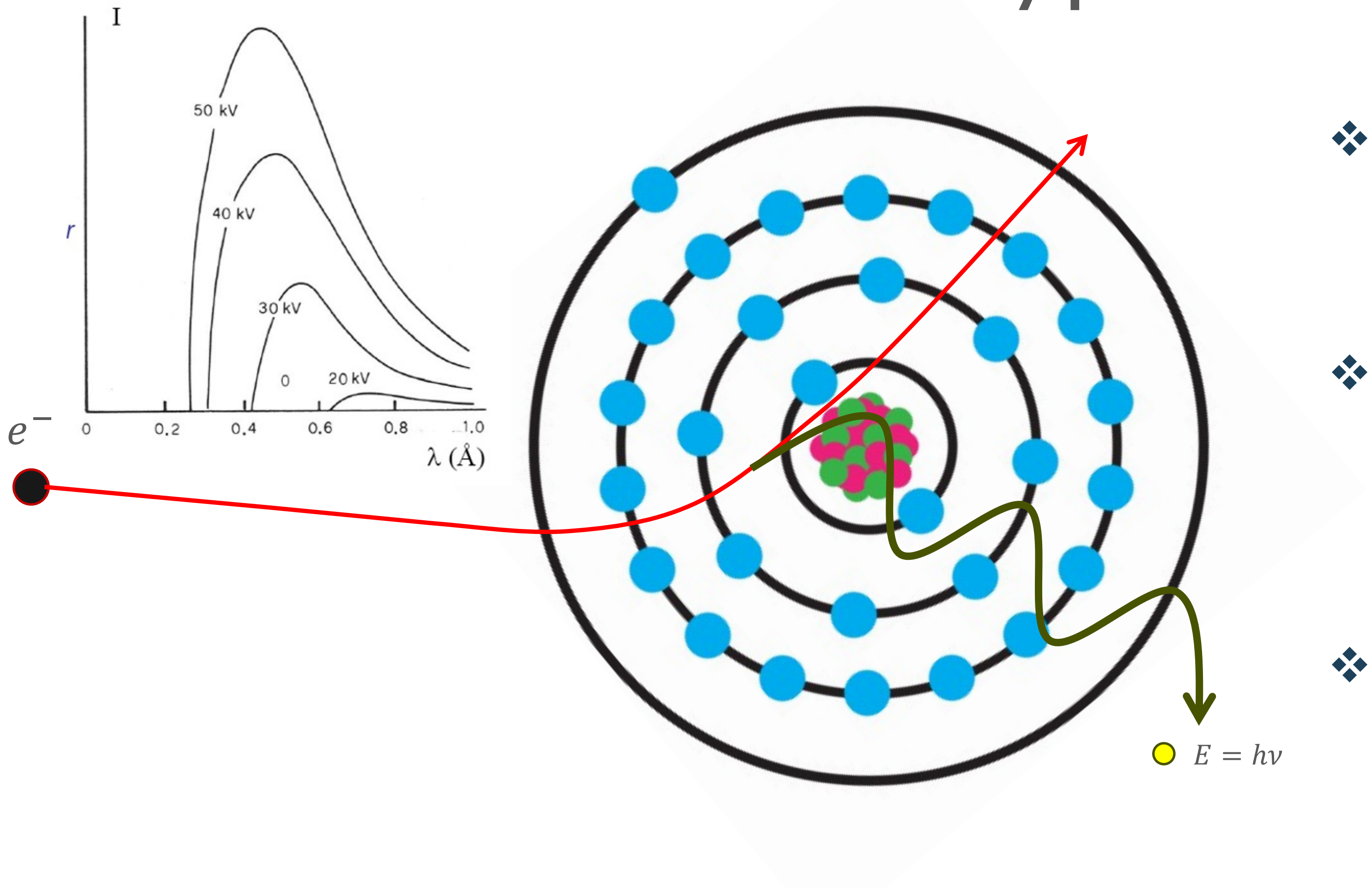


Bremsstrahlung radiation

❖ **Definition:**
- X-ray radiation produced when an electron is decelerated or deflected by the nucleus.

❖ **Mechanism:**
- Electrons lose kinetic energy when interacting with atomic nuclei.
- Lost energy is emitted as X-ray photons.

X-ray production



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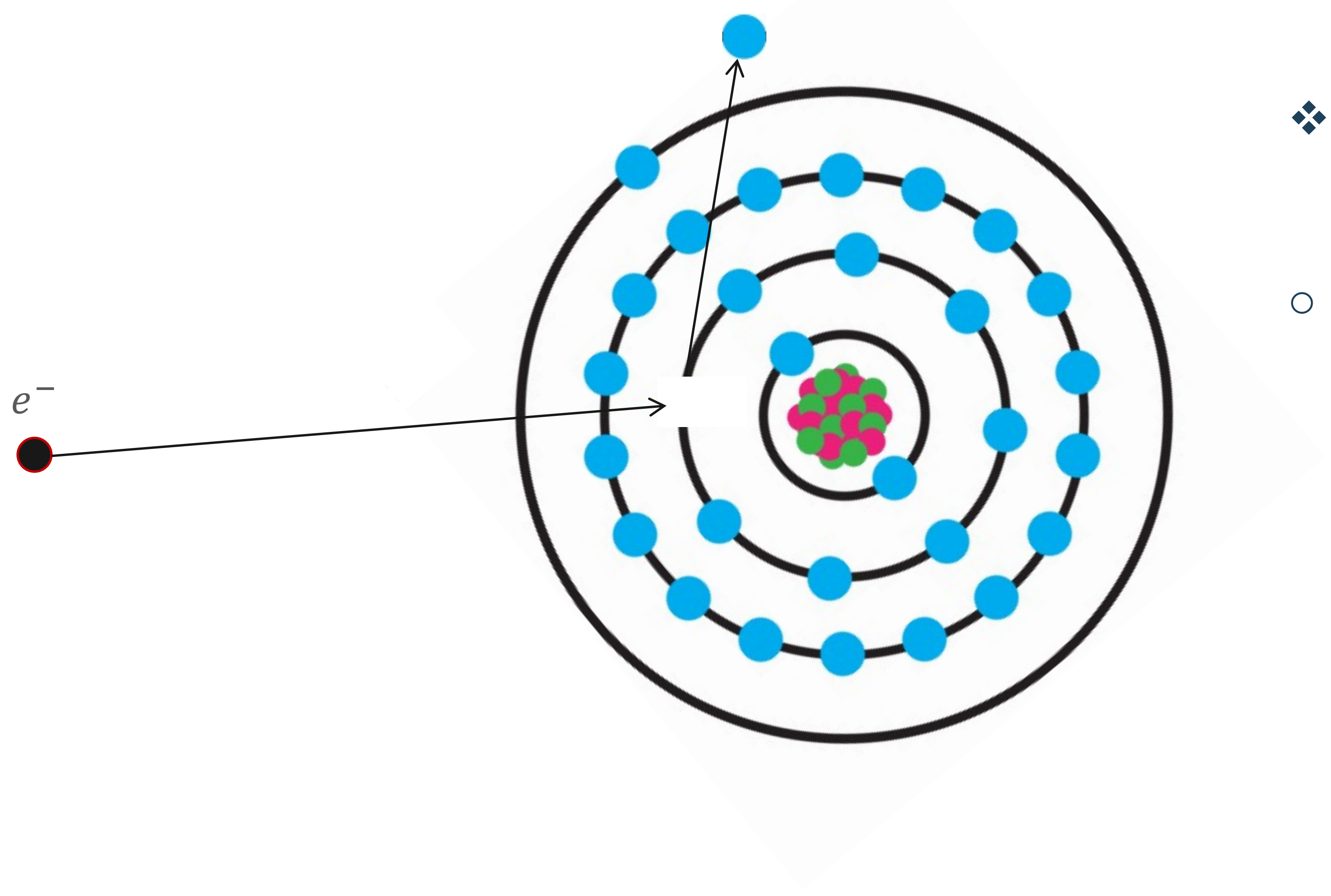
  - Lost energy is emitted as X-ray photons.
- **Characteristic:**
  - *Continuous:* due to gradual deceleration of electrons.
  - Higher for heavier nuclei ($\propto Z^2$).

↳ Bremsstrahlung radiation is the primary contributor to the background in X-ray emission spectra.

X-ray production



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- **Mechanism:**
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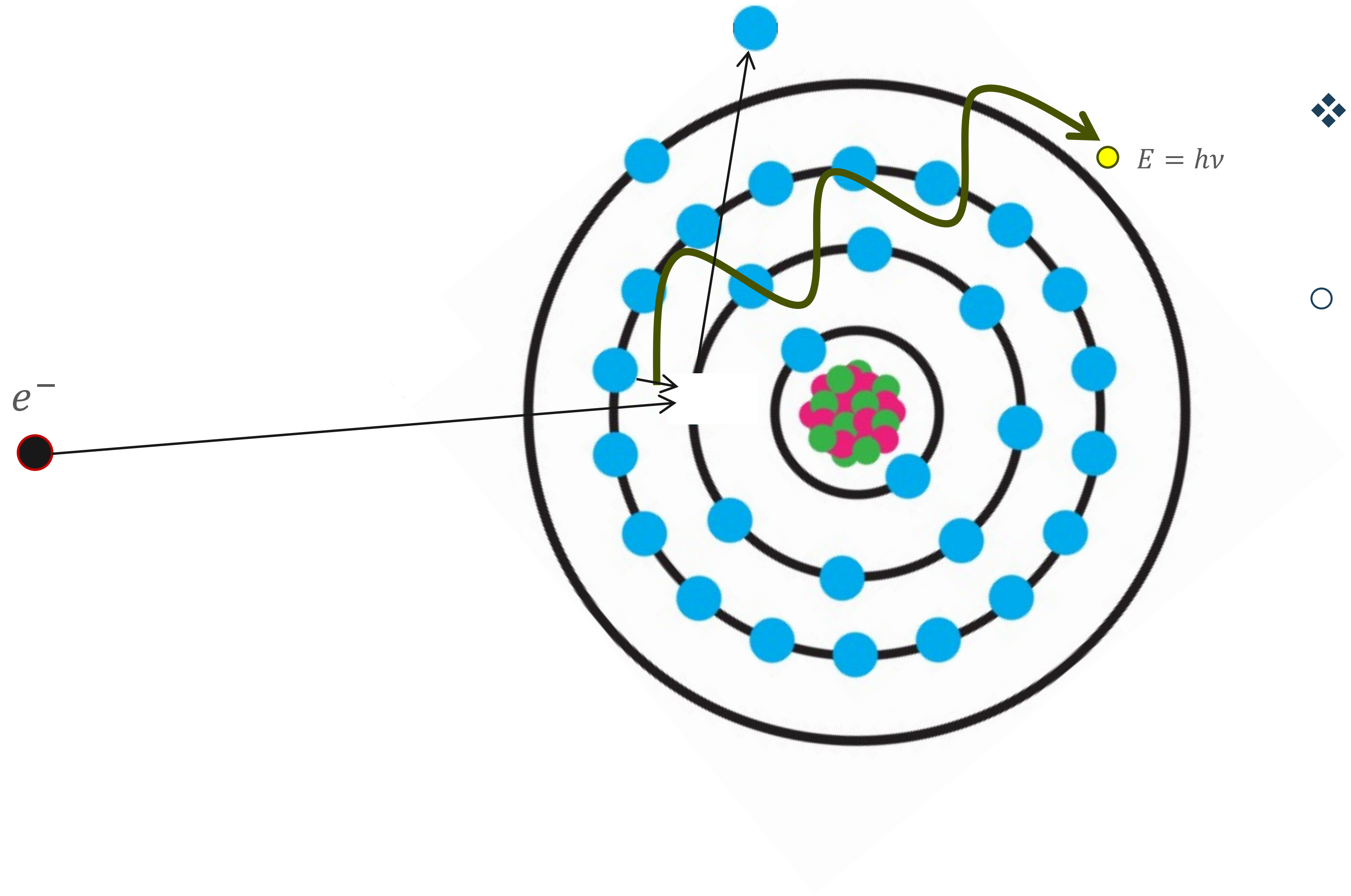
X-ray production



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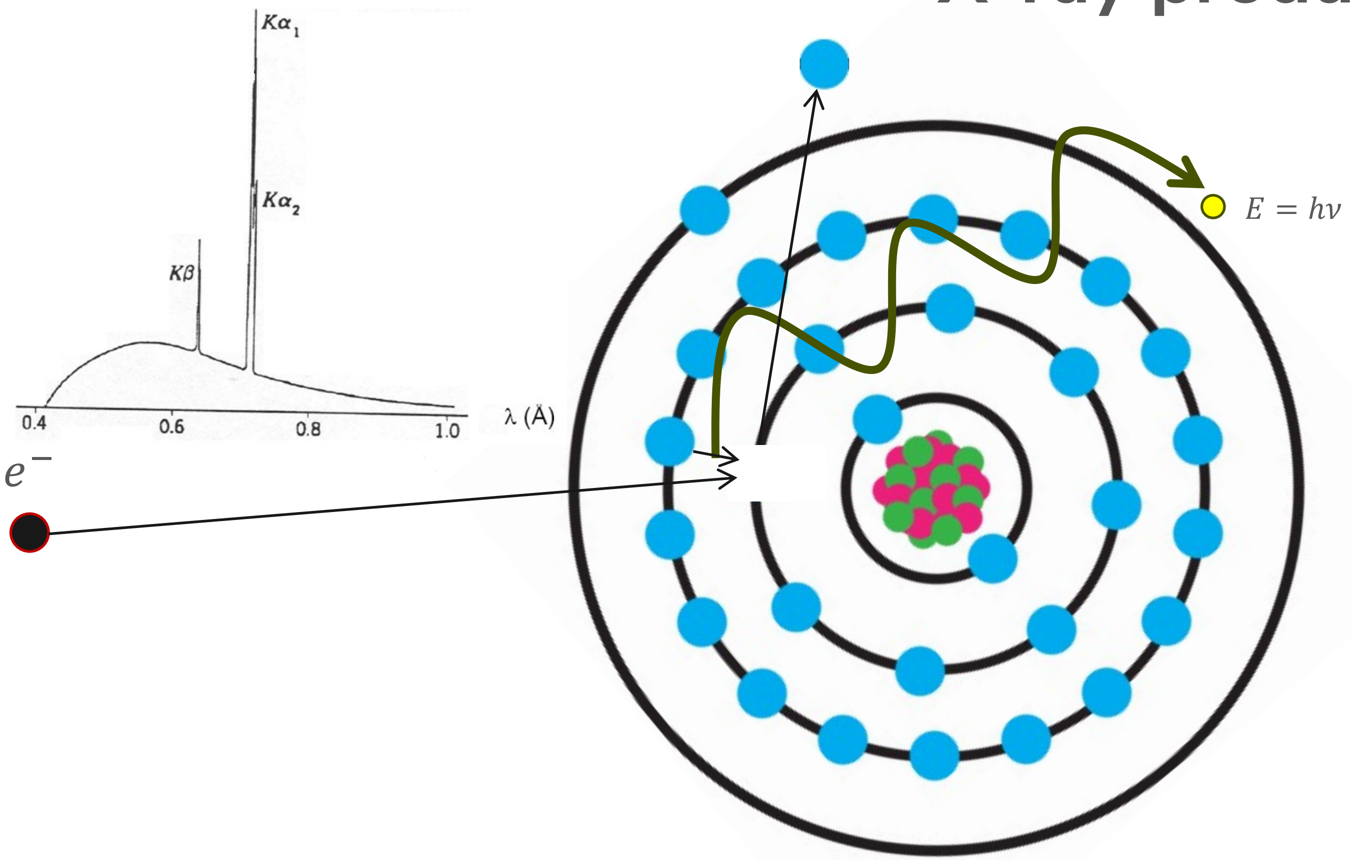

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❖ **Characteristic:**

- Distinct peaks
- Element-specific

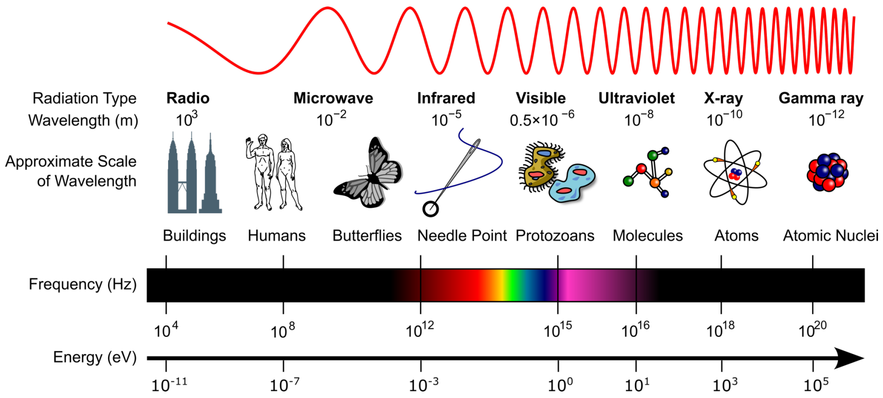
⇘Characteristic X-rays enable elemental analysis, aiding in materials characterization.

What are x-rays?

Electromagnetic waves

- X-rays are electromagnetic waves. If they have enough energy (E_{rad}~ 10 keV), some electrons can be removed from atoms or molecules, thereby ionizing them (E_{ion}~ 10 eV).

$$1\ eV = 1.60\times10^{-19}\ \mathrm{J}$$



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where h: Planck's constant; ν:frequency; ω: pulsation

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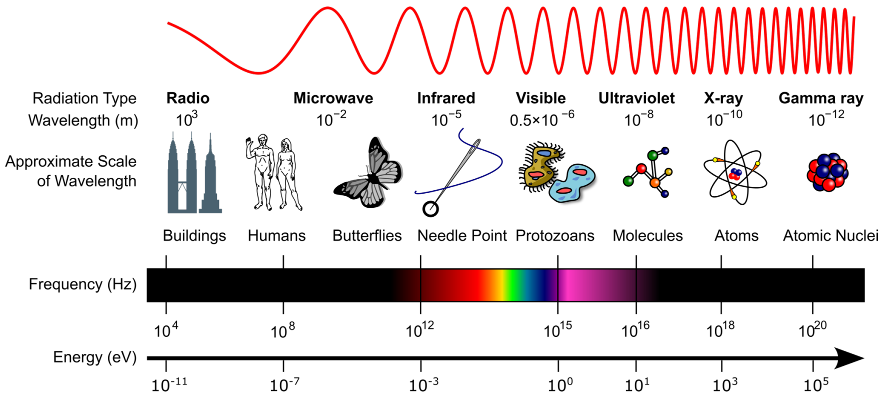

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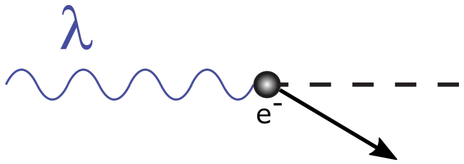
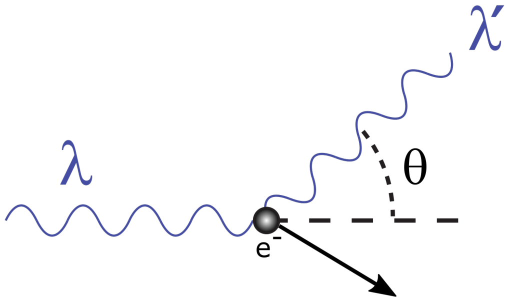
Photon-matter interaction

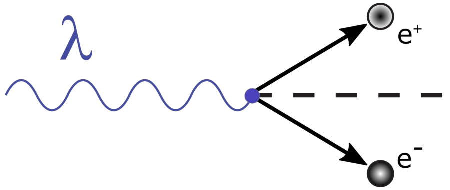
❖ **Photoabsorption effect (10 eV – 100 keV):** A photon transfers all its energy to an electron, ejecting it if the energy exceeds the electron's binding energy.



❖ **Compton scattering (10 keV – 1 MeV):** An incident photon collides with a free or loosely bound electron, losing energy and changing direction. The electron gains this energy and is ejected with a new direction and velocity.



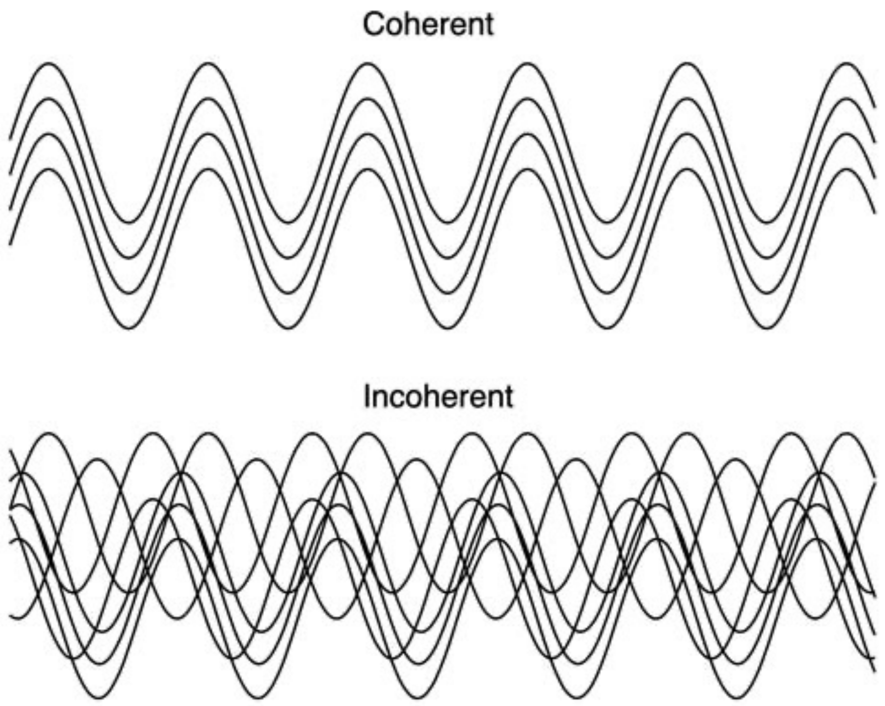
❖ **Pair production ($>$1 MeV):** A high-energy photon interacts with an atom's nucleus and converts into an electron-positron pair.



Coherency

❖ **Definition:**

- Coherence describes the stability of an X-ray wave's phase in time and space, crucial for producing clear interference patterns.



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- Temporal coherence (energy stability)
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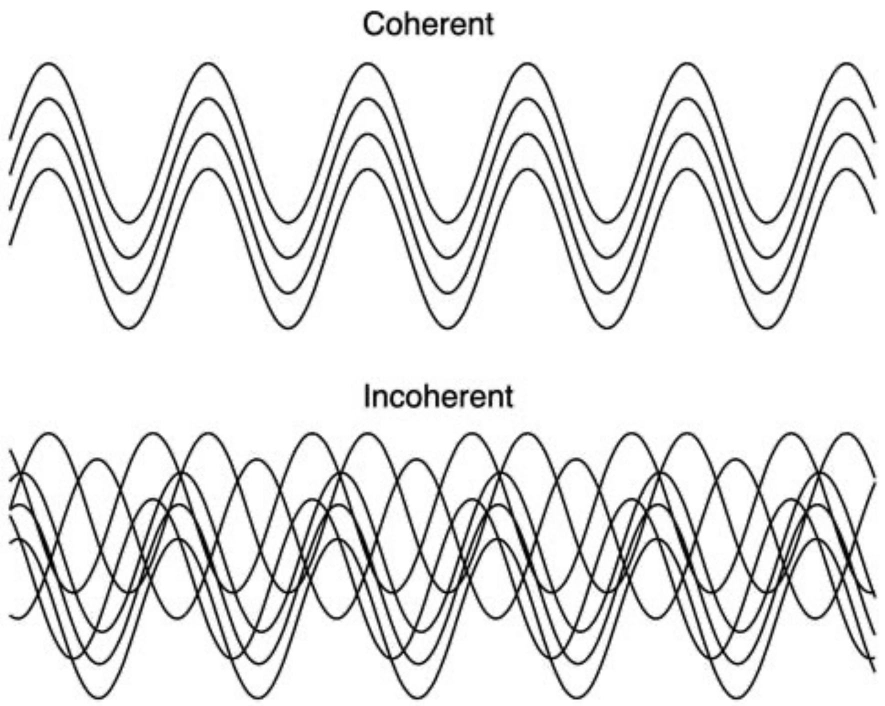

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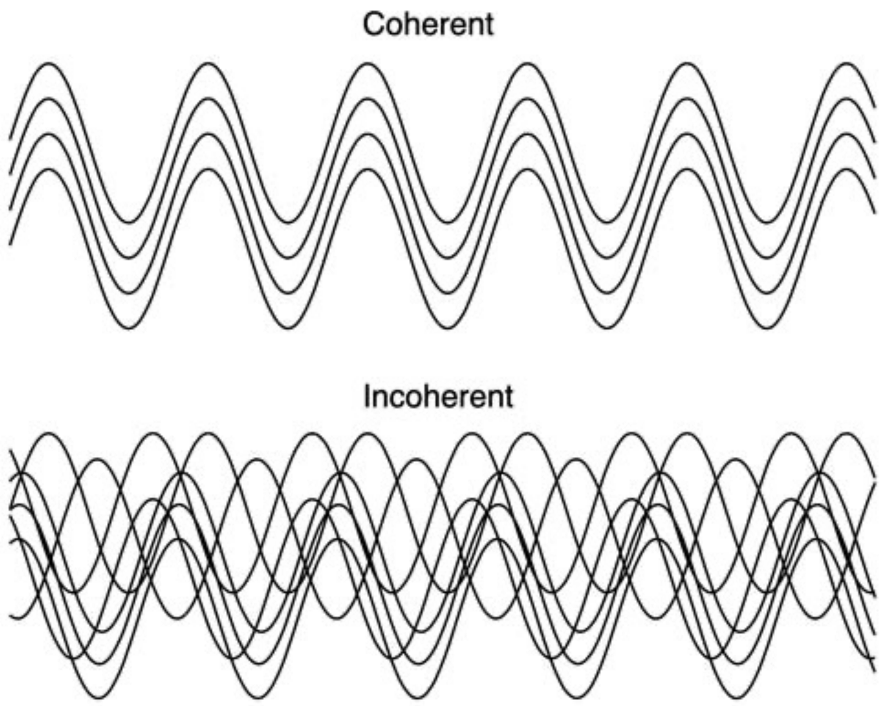
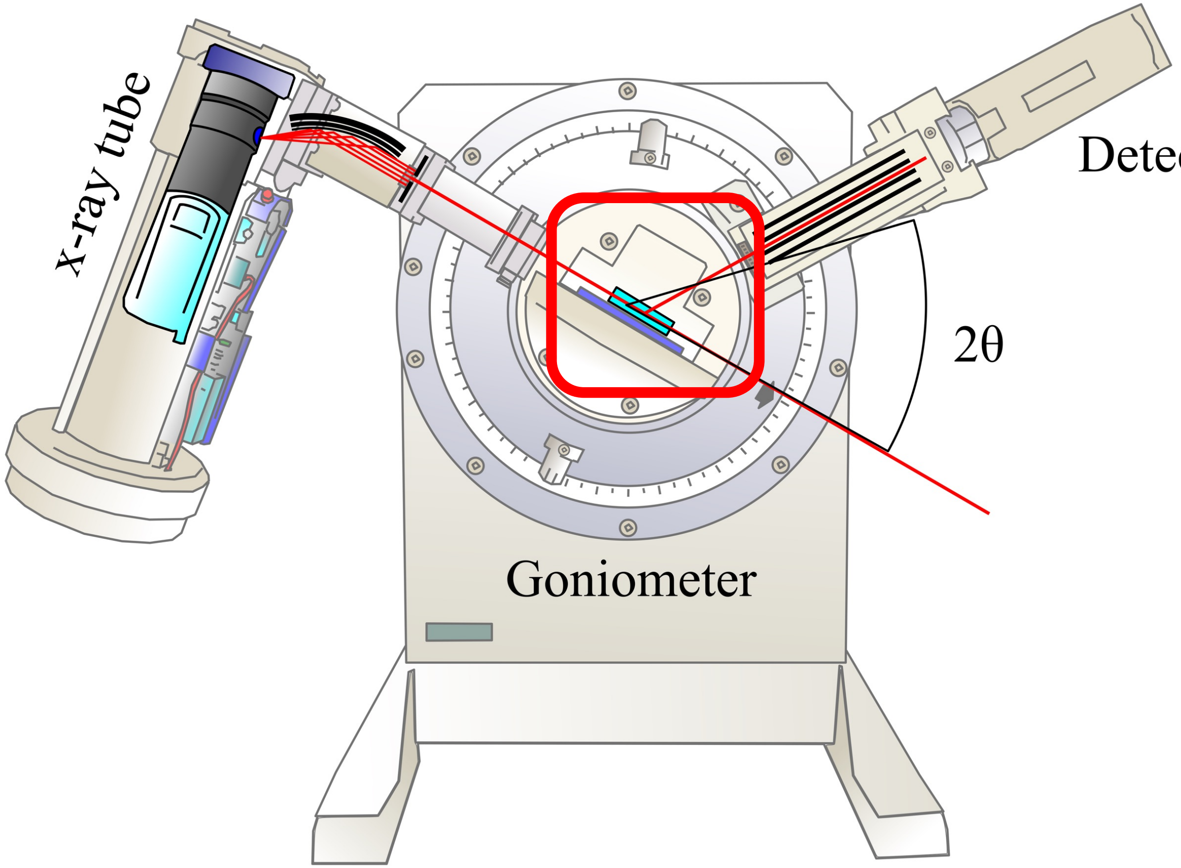
  - Spatial coherence (wavefront uniformity)
    - ✓ Measures phase correlation across different points in the X-ray beam.
    - ✓ Affects the sharpness of diffraction spots in XRD.
    - ✓ High spatial coherence is achieved with small, collimated sources (e.g., synchrotron X-rays).

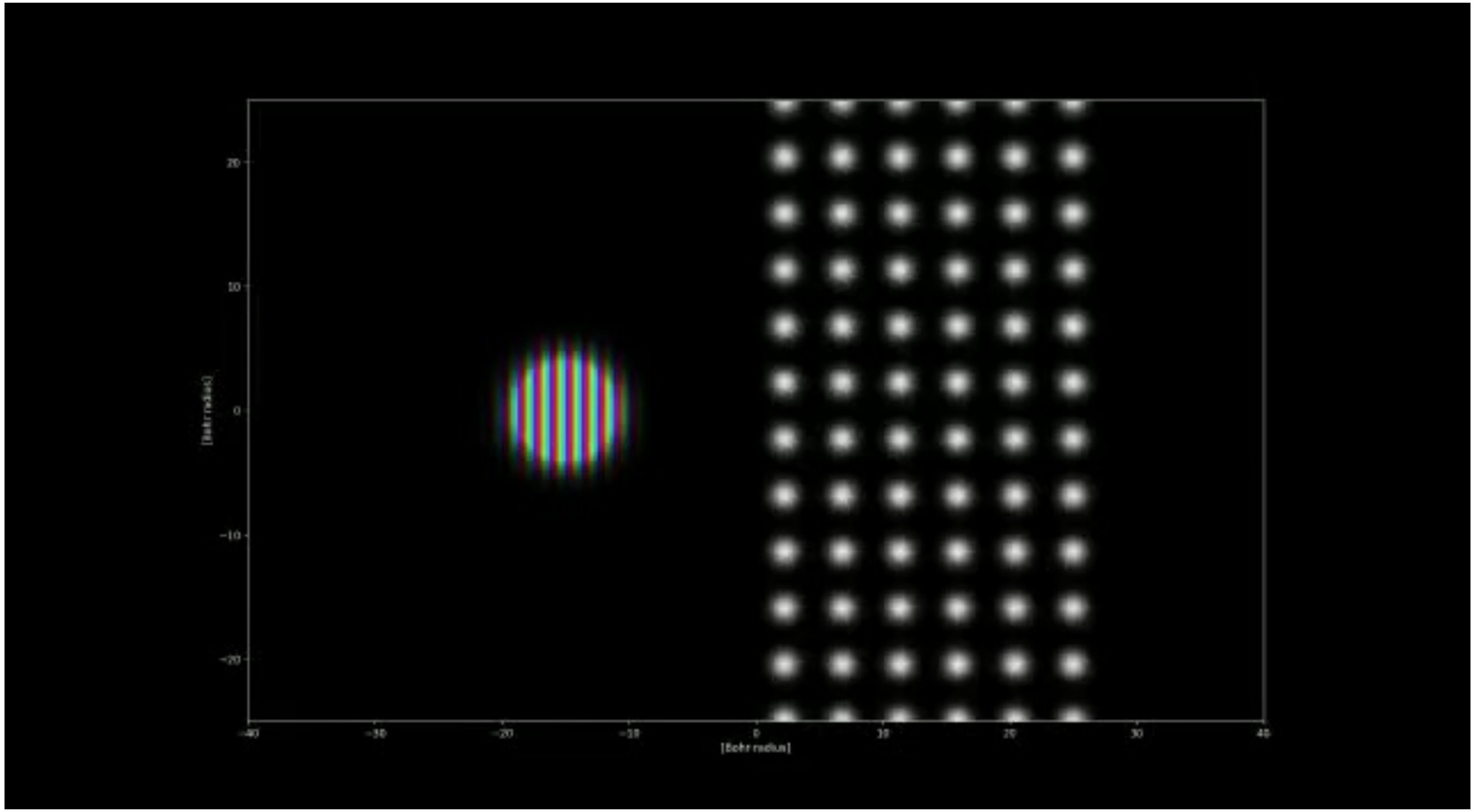


The experimental setup: the diffractometer

The diffractometer (Bragg-Brentano configuration)



The diffraction



Interaction X-ray matter

❖ **Liquids or amorphous materials:**

- X-rays are scattered in many directions, leading to broad, diffuse halos.

W.H. Bragg, W.L. Bragg, *Proc. R. Soc. Lond. A.* (1913)

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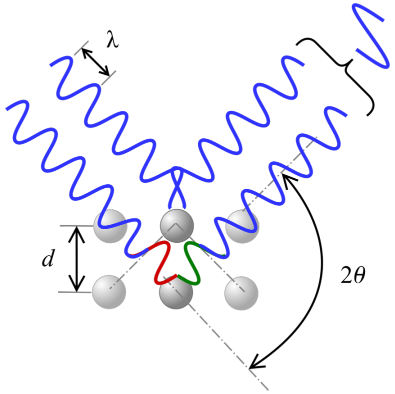

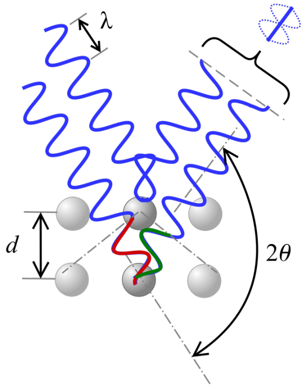

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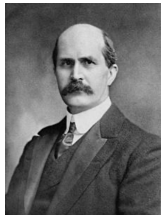
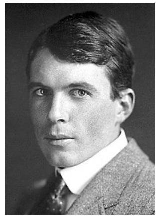
This suggests that X-rays interact in a structured manner with the internal atomic planes of the crystal.



William Henry BRAGG
(1862 – 1940)

William Lawrence BRAGG
(1890 – 1971)

2/2 Nobel Prize (1915)



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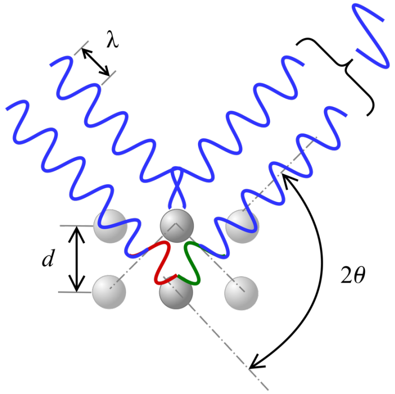

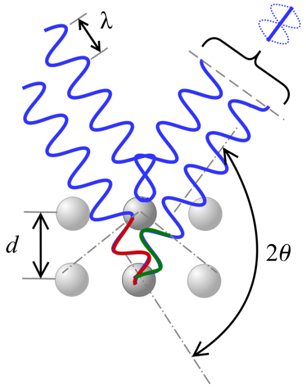

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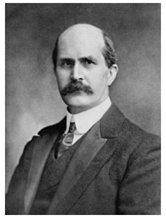

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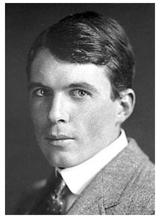

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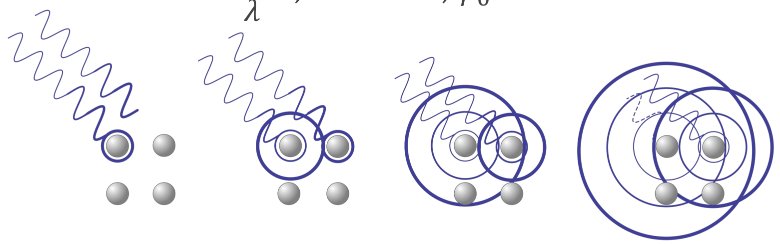
- **X-ray electromagnetic waves propagate in the direction of $\boldsymbol{u}$ and oscillate in time and space:**

$$A_0 \cos(2\pi \boldsymbol{k} \cdot \boldsymbol{r} - \omega t + \varphi_0) \longrightarrow \Re e\{\psi(\boldsymbol{r}, t) = A_0 \exp[-i\omega t] \exp[i(2\pi \boldsymbol{k} \cdot \boldsymbol{r} + \varphi_0)]\}$$

$$\boldsymbol{k} = \frac{1}{\lambda}\boldsymbol{u}\ ;\ \omega = 2\pi\nu\ ;\ \varphi_0 = 0$$



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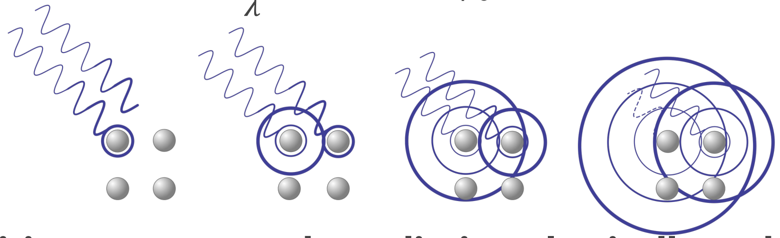

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$$\text{Elastic} \Rightarrow \|\boldsymbol{k}\| = \left\|\boldsymbol{k}' = \frac{1}{\lambda}\boldsymbol{u}'\right\|$$

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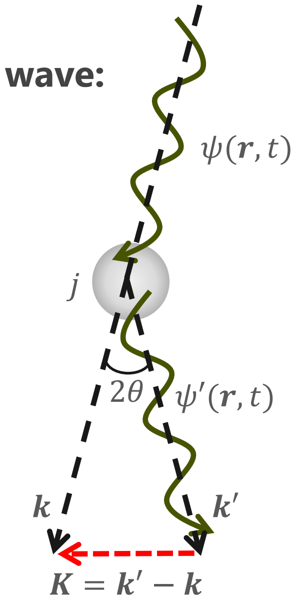

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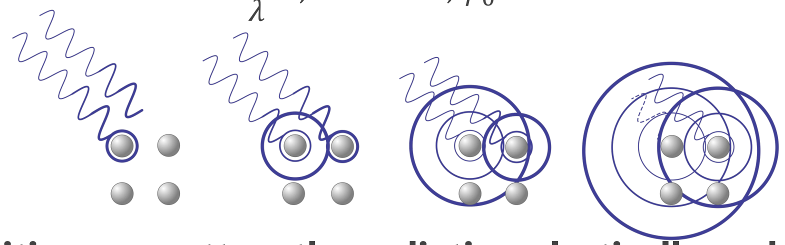

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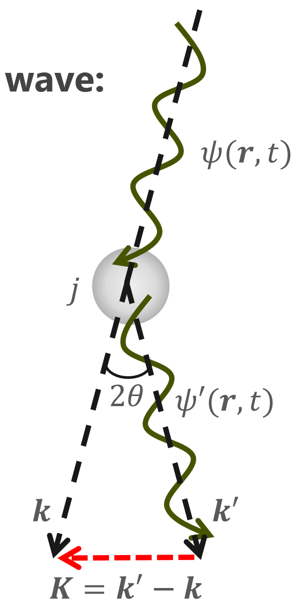

❖ **For a given unit cell of the crystal composed of n atoms:**

$$\psi'(\boldsymbol{r},t) = \sum_{j=1}^{n}\psi'_j(\boldsymbol{r},t) = A_0\exp[-i(\omega t + 2\pi\boldsymbol{k}'\cdot\boldsymbol{r})]F(\boldsymbol{K})$$

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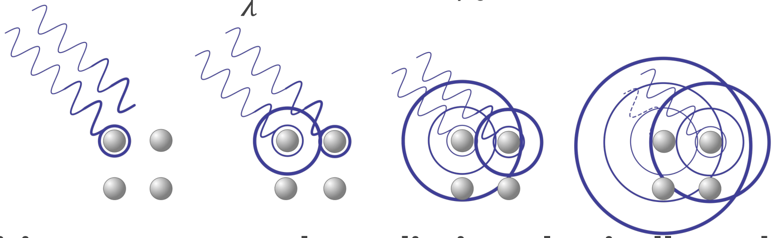

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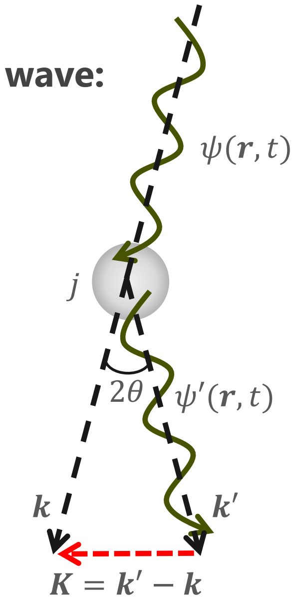

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Therefore, under the diffraction condition, $\boldsymbol{K}$ is identified with a vector $\boldsymbol{G}$ whose basis is defined as:

$$\boldsymbol{G} = h\boldsymbol{b_1} + k\boldsymbol{b_2} + l\boldsymbol{b_3}$$
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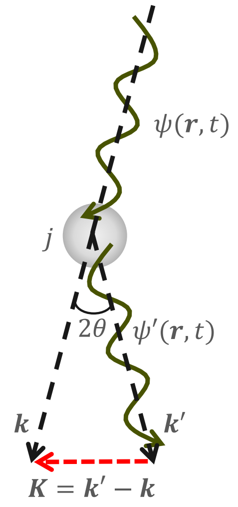
➪ Constructive diffraction occurs when $\boldsymbol{K}$ corresponds to a vector of the reciprocal lattice.

The Bragg’s law

- **From the Laue Conditions:**
  - Constructive diffraction occurs when $\boldsymbol{K}$ corresponds to a vector of the reciprocal lattice.
    $$\Rightarrow \boldsymbol{K} = \boldsymbol{G}$$
  - We can show that: $\|\boldsymbol{G}\| = \frac{2\pi}{d}$

$$\hookrightarrow n\lambda = 2d\sin\theta$$



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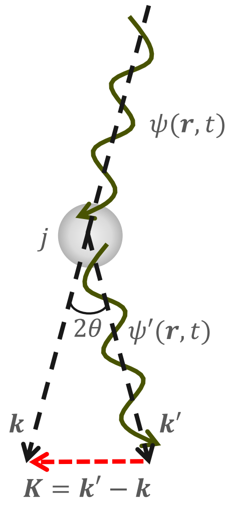

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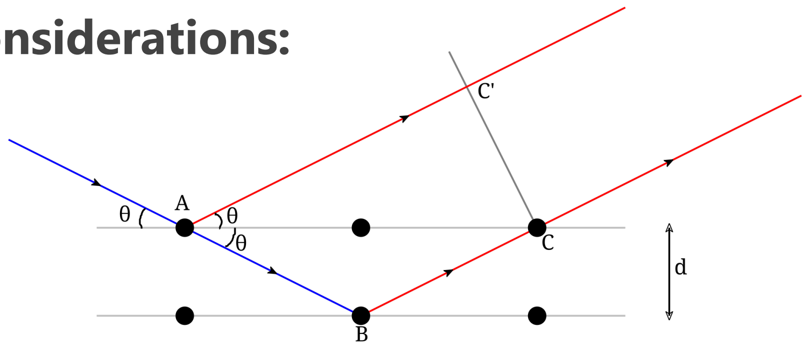

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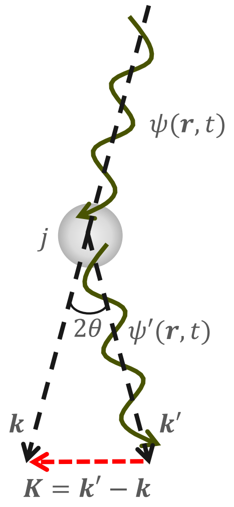

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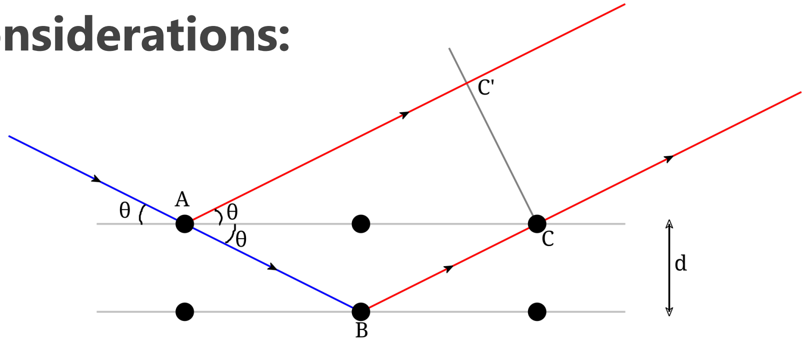


- The path difference $\Rightarrow (AB) - (BC) - (AC') = \frac{d}{\sin\theta} + \frac{d}{\sin\theta} - \frac{2d}{\tan\theta}\cos\theta$
- Constructive diffraction occurs when both waves are in phase $\Rightarrow n\lambda = (AB) - (BC) - (AC')$

$$n\lambda = 2d \sin\theta$$

The Bragg's law: extra

- $\|\boldsymbol{G}\|$:

$$\boldsymbol{G} = h\boldsymbol{b_1} + k\boldsymbol{b_2} + l\boldsymbol{b_3}$$

$$\boldsymbol{b_i} = 2\pi \frac{\boldsymbol{a_j}\times\boldsymbol{a_k}}{\boldsymbol{a_1}\cdot(\boldsymbol{a_2}\times\boldsymbol{a_3})} \Rightarrow \boldsymbol{b_i}\cdot\boldsymbol{a_i} = 2\pi\,\delta_{ij}$$

$$\|\boldsymbol{G}\|^2 = \boldsymbol{G}\cdot\boldsymbol{G} = (h\boldsymbol{b_1} + k\boldsymbol{b_2} + l\boldsymbol{b_3})\cdot(h\boldsymbol{b_1} + k\boldsymbol{b_2} + l\boldsymbol{b_3}) = h^2\boldsymbol{b_1}\cdot\boldsymbol{b_1} + k^2\boldsymbol{b_2}\boldsymbol{b_2} + l^2\boldsymbol{b_3}\boldsymbol{b_3} + 2hk\boldsymbol{b_1}\boldsymbol{b_2} + 2hl\boldsymbol{b_1}\boldsymbol{b_3} + 2kl\boldsymbol{b_2}\boldsymbol{b_3}$$

Let's suppose a orthogonal unit cell:

$$\boldsymbol{b_i} = 2\pi \frac{\boldsymbol{a_j}\times\boldsymbol{a_k}}{\boldsymbol{a_1}\cdot(\boldsymbol{a_2}\times\boldsymbol{a_3})} \Rightarrow \boldsymbol{b_1} = 2\pi\frac{bc}{abc}\boldsymbol{a_1} = \frac{2\pi}{a}\boldsymbol{a_1};\ \boldsymbol{b_2} = \frac{2\pi}{b}\boldsymbol{a_2};\ \boldsymbol{b_3} = \frac{2\pi}{c}\boldsymbol{a_3}$$

$$\Rightarrow \|\boldsymbol{G}\|^2 = \left(\frac{2\pi h}{a}\right)^2 + \left(\frac{2\pi k}{b}\right)^2 + \left(\frac{2\pi l}{c}\right)^2 = (2\pi)^2\left(\frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}\right)$$

$$\Rightarrow \|\boldsymbol{G}\| = \frac{2\pi}{d_{hkl}}$$

- **Bragg's law demonstration from Laue conditions:**

$$\Rightarrow \boldsymbol{K} = \boldsymbol{G} \Leftrightarrow \boldsymbol{k'} - \boldsymbol{k} = \boldsymbol{G} \Rightarrow (\boldsymbol{k'} - \boldsymbol{k})^2 = \boldsymbol{G}^2 \Leftrightarrow \boldsymbol{G}^2 = \boldsymbol{k'}^2 + \boldsymbol{k}^2 - 2\boldsymbol{k'}\cdot\boldsymbol{k}$$

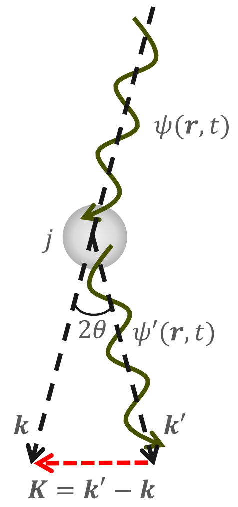
Elastic scattering $\Rightarrow \|\boldsymbol{k'}\| = \|\boldsymbol{k}\| = k$

$$\Rightarrow \boldsymbol{G}^2 = 2k^2 - 2k^2\cos 2\theta = 2k^2(1-\cos 2\theta) = 4k^2\sin^2\theta$$

But $\|\boldsymbol{G}\| = \frac{2\pi}{d_{hkl}}$

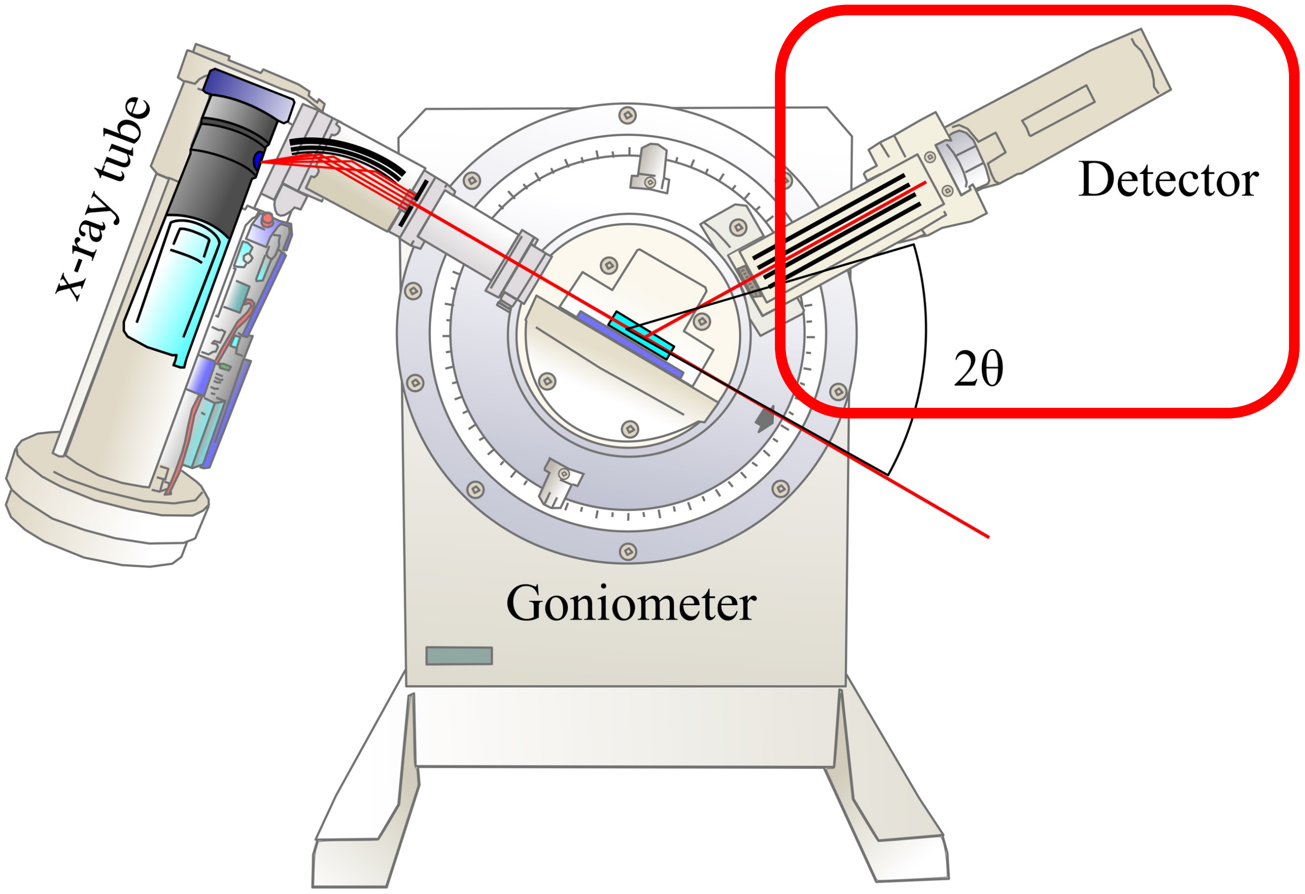
$$\Rightarrow \frac{2\pi}{d_{hkl}} = 4k^2\sin^2\theta = 4\left(\frac{2\pi}{\lambda}\right)^2\sin^2\theta$$

$$\Rightarrow 2d_{hkl}\sin\theta = \lambda$$



The experimental setup: the diffractometer

The diffractometer (Bragg-Brentano configuration)



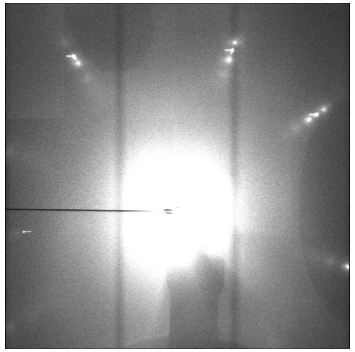
X-ray diffraction techniques

Polychromatic beam

$$n\lambda = 2d \sin\theta$$

- Several wave lengths $[\lambda_1; \lambda_2]$
- Sample: one monocrystal
- In the Bragg's law:
  - Fixed: d, θ
  - Varied: λ

⇒ **Isolated spots corresponding to the $\{\lambda, \theta, d\}$ combinations**



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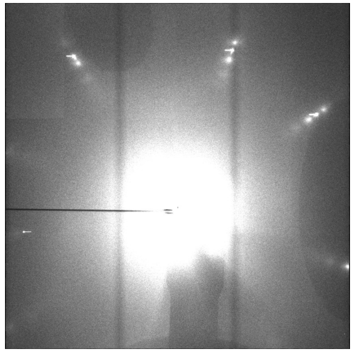

Monochromatic beam

- One unique waves length λ
- Sample: powder (random oriented crystals)
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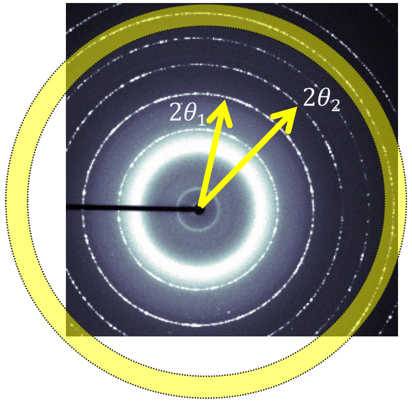
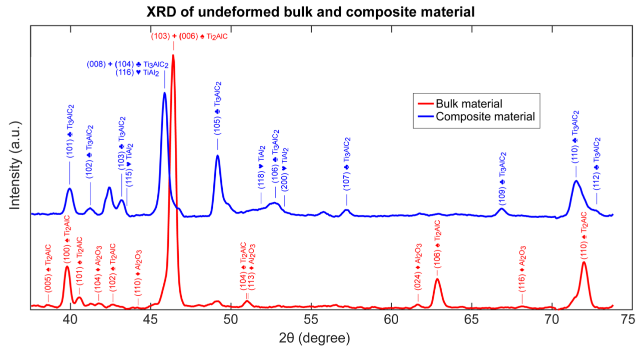
⇒ **Concentric rings or peak diffractograms**



$$\int_0^{2\pi} I(r, \varphi) \mathrm{d}\varphi$$



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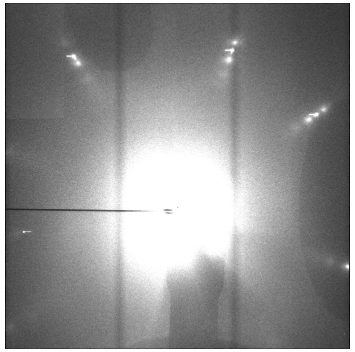

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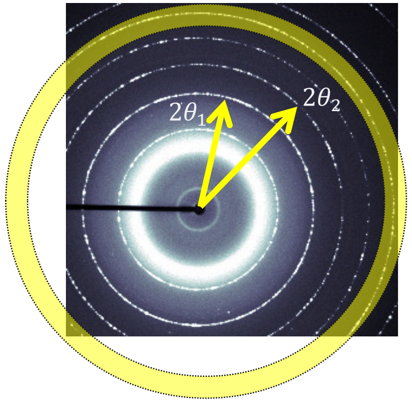

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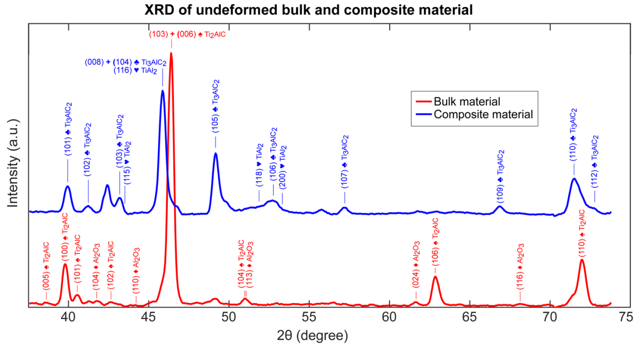
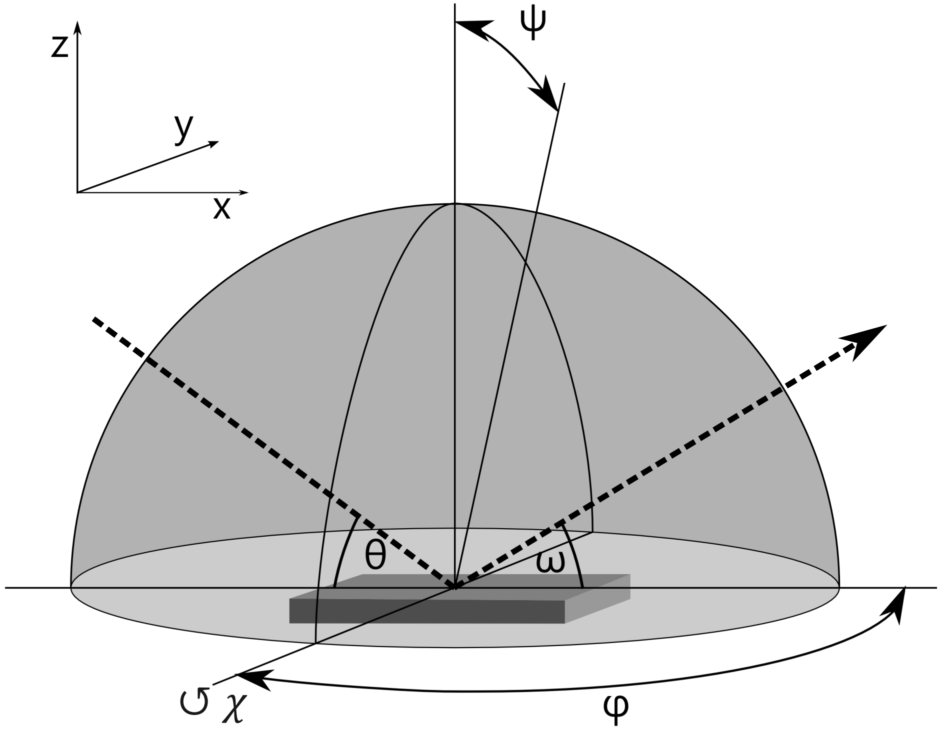


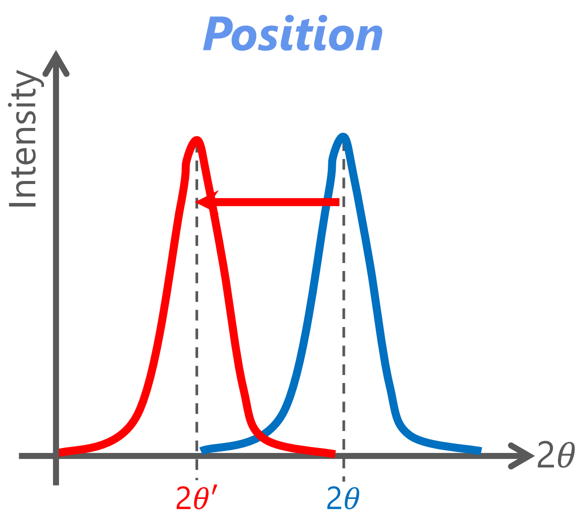
One crystal, many wavelengths – that's Laue.
Many crystals, one wavelength – that's powder.

Angle definitions for powder diffraction



- 2θ: angle between the incident beam and the diffracted beam
- ω: angle of incidence, i.e. the angle between the incident beam and the sample surface
- ψ: angle between the sample surface normal and the normal to the diffracting planes
- χ: tilt angle of the sample, measured from the vertical axis
- φ: in-plane rotation angle of the sample around the surface normal

Shape of the diffracted peaks



- **Peak shift:**
  - $n\lambda = 2d_{hkl} \sin\theta$
    - ✓ $2\theta \searrow \Rightarrow \sin\theta \searrow \Rightarrow d_{hkl} \nearrow \Rightarrow$ macrotraction
    - ✓ $2\theta \nearrow \Rightarrow \sin\theta \nearrow \Rightarrow d_{hkl} \searrow \Rightarrow$ macrocompression
- **Average elastic strain in the direction normal to the $(h\ k\ l)$ planes:**

$$\varepsilon_{hkl}^{e} = \frac{\Delta d_{hkl}}{d_{hkl}^{0}}$$

Shape of the diffracted peaks

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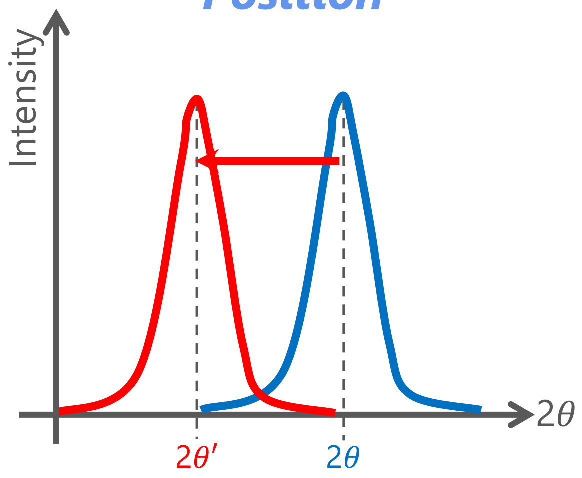

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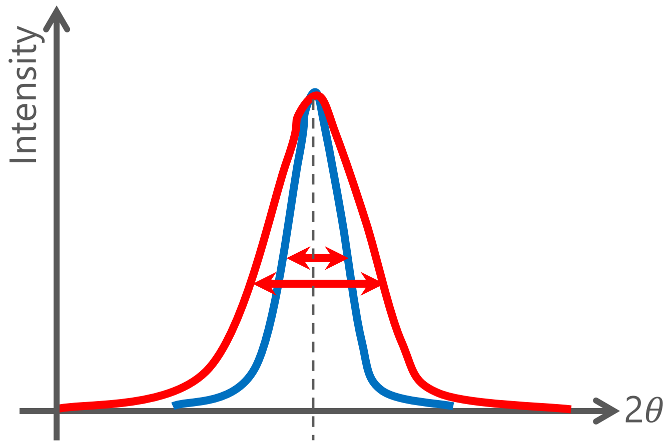

- **Peak width:**
  - Crystallite size (L)

$$FWHM \propto \frac{\lambda}{L\cos\theta}$$

  - Lattice strain heterogeneities
    - ✓ Dislocation density (ρ)

$$\rho \approx \frac{FWHM^2\cos^2\theta}{Kb^2}$$

    - ✓ Microstrain

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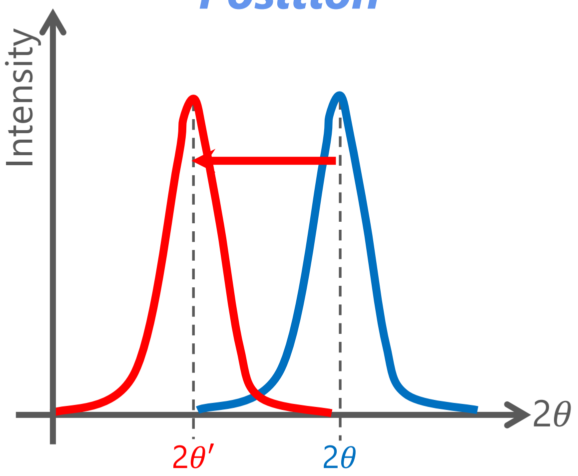

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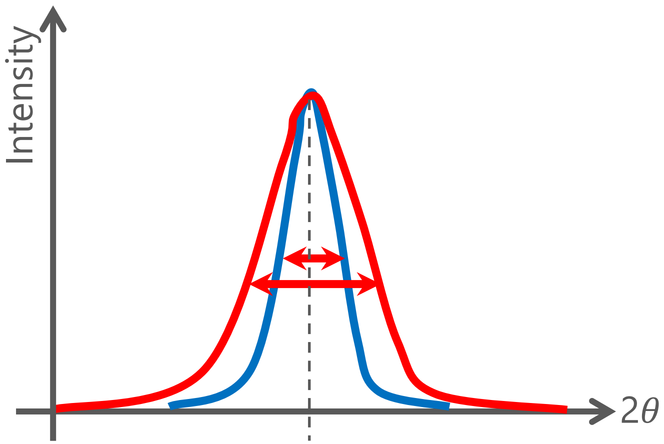

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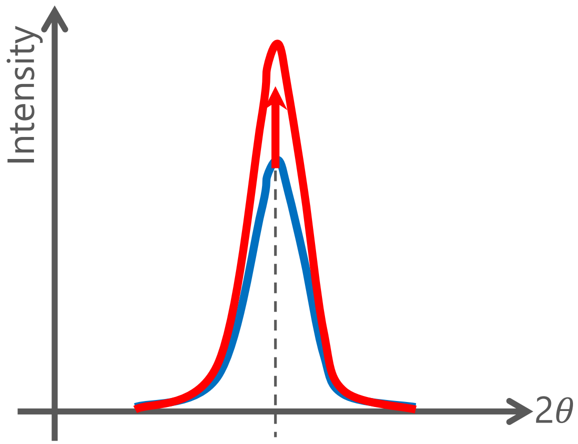

- **Peak height:**
  - Structure factor (F)
  - Phases
  - Texture

Example: Irradiation of Ti_2AlC with Ni^{3+} ions

Irradiation Ni^{3+} (@500°C)	
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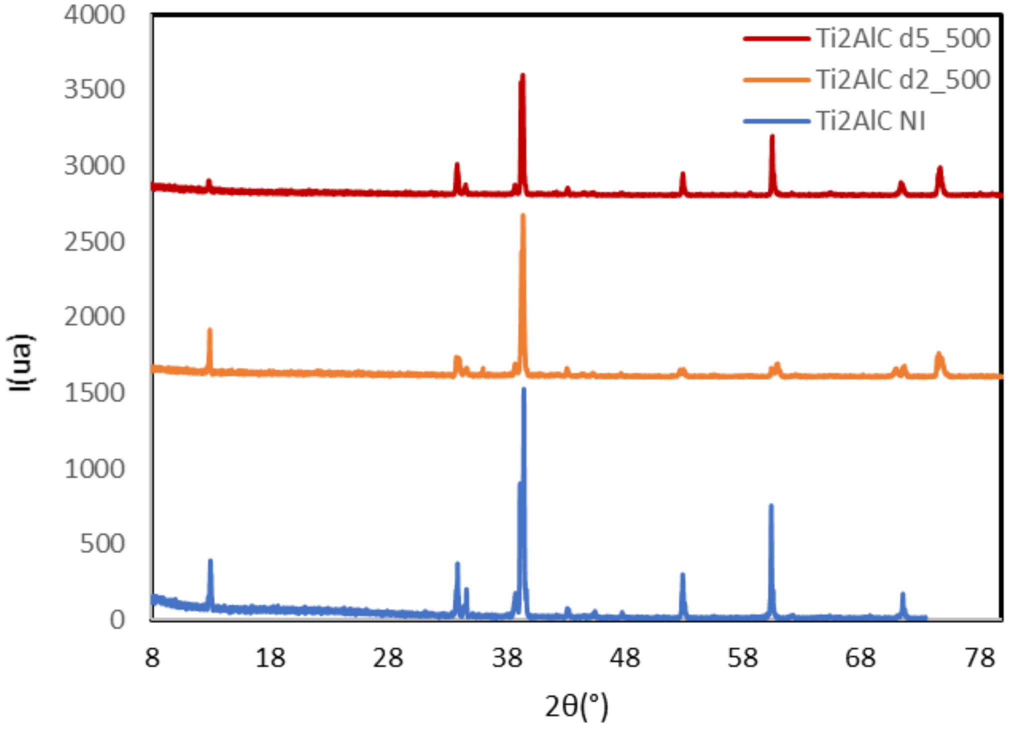

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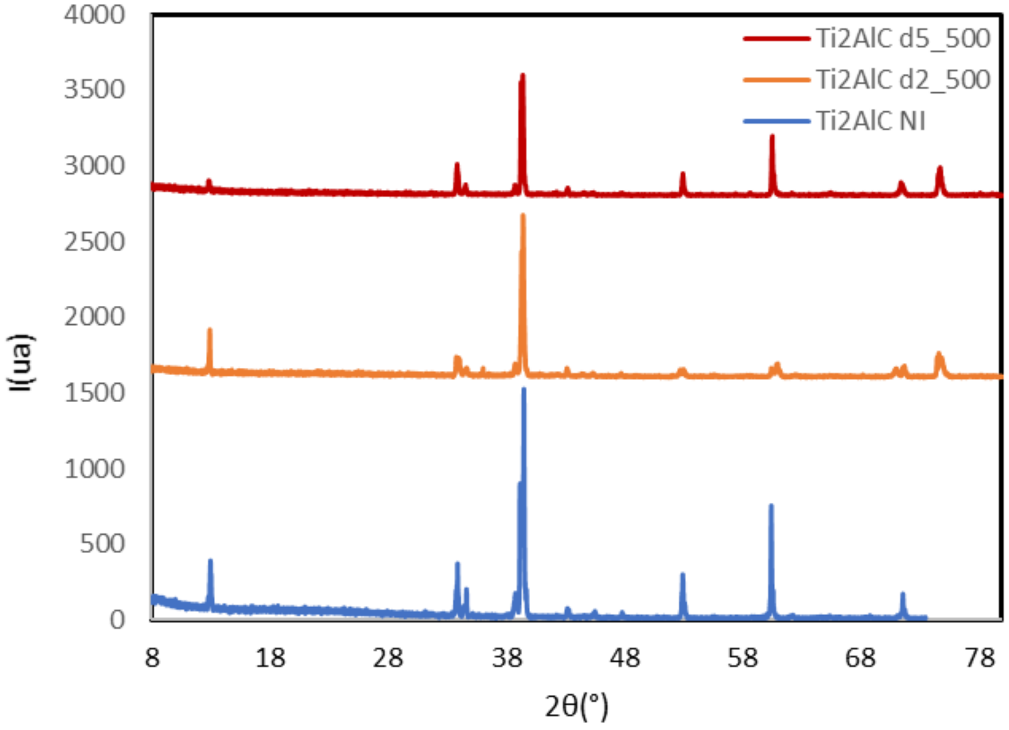

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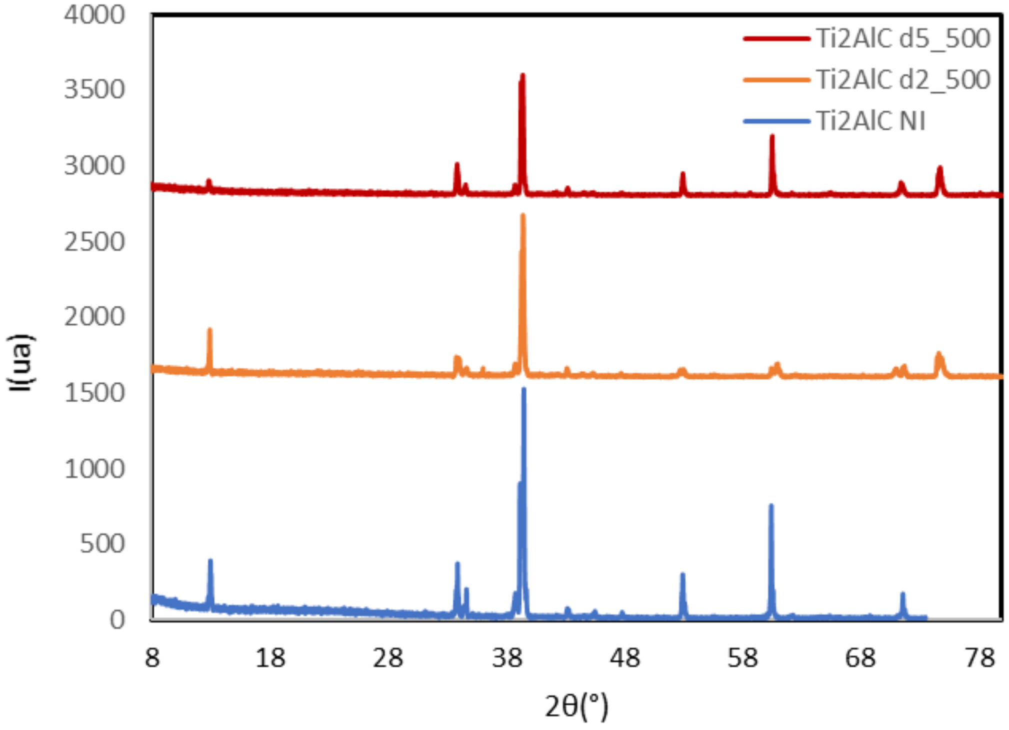

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M. Josse, *PhD Thesis* (2025)

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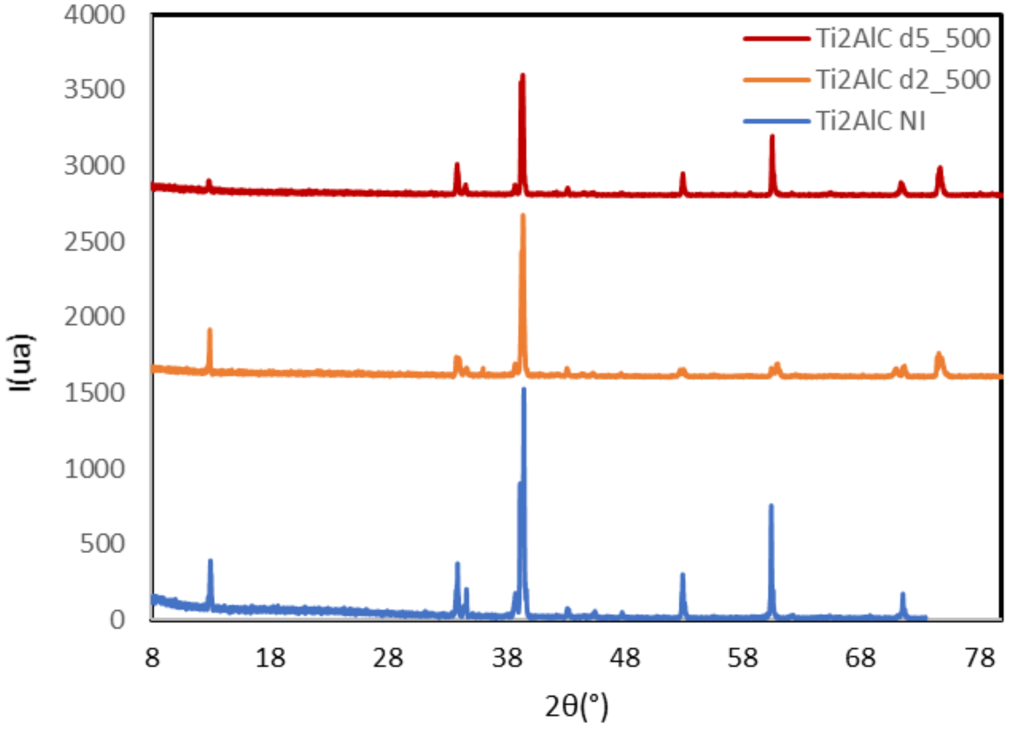

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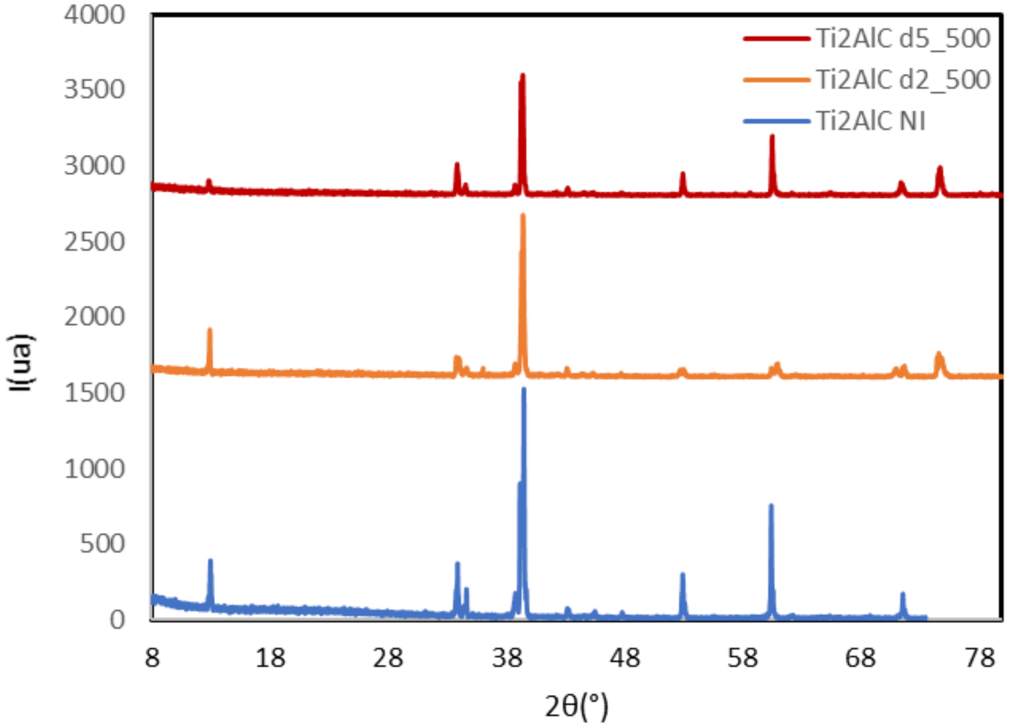

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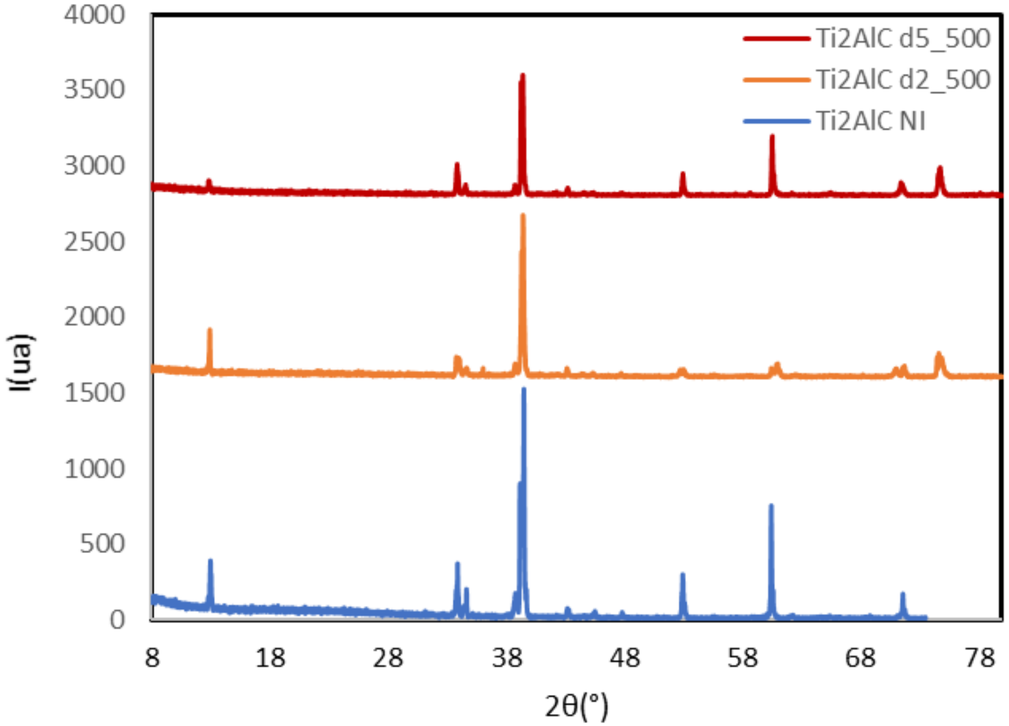

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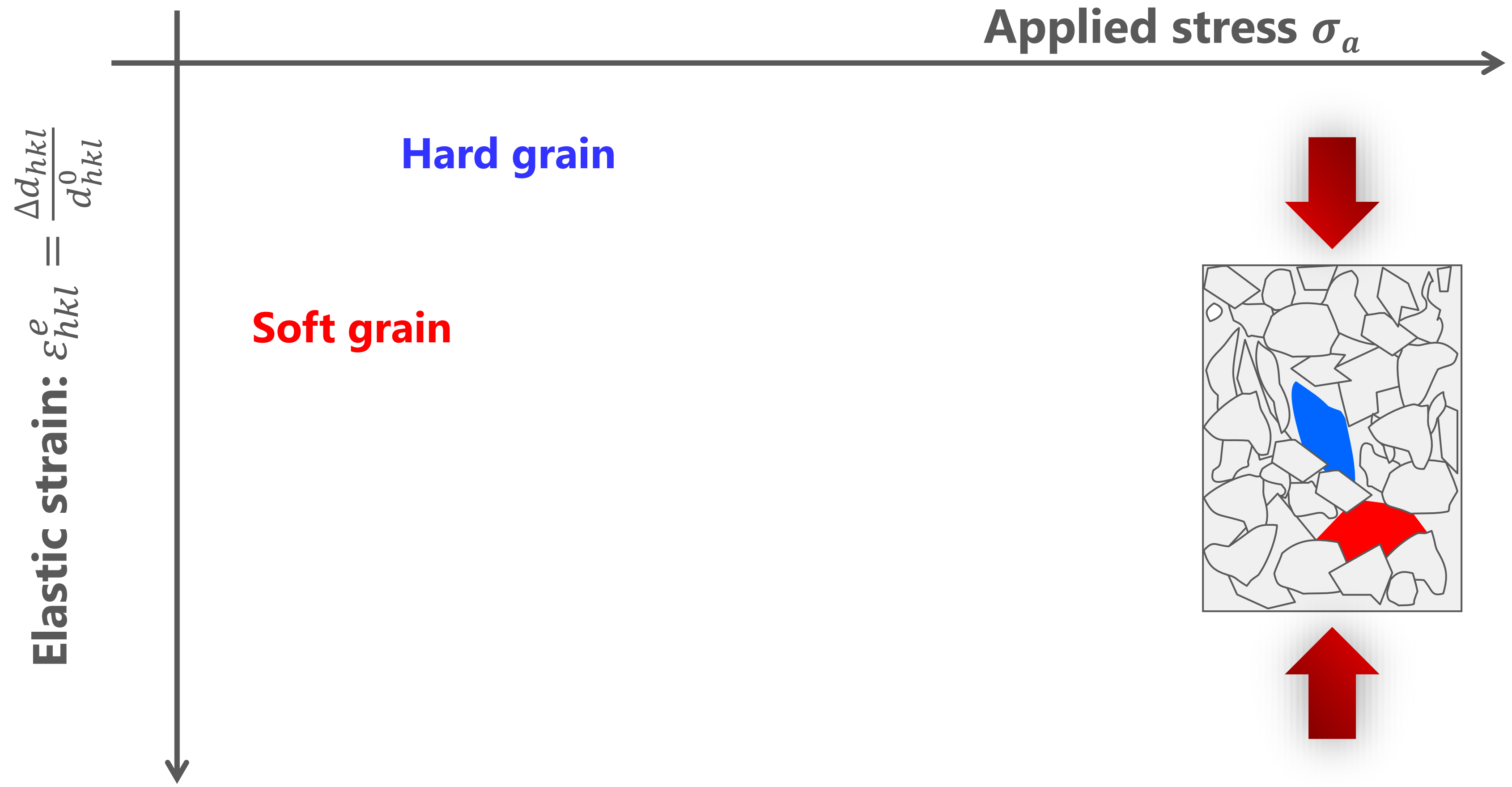
↳The structure of Ti_2AlC seems preserved after irradiation, which is significant for radiation tolerance.

M. Josse, *PhD Thesis* (2025)

Evolution of the elastic strain



A. Guitton et al., *App. Phys. Let.*(2014)

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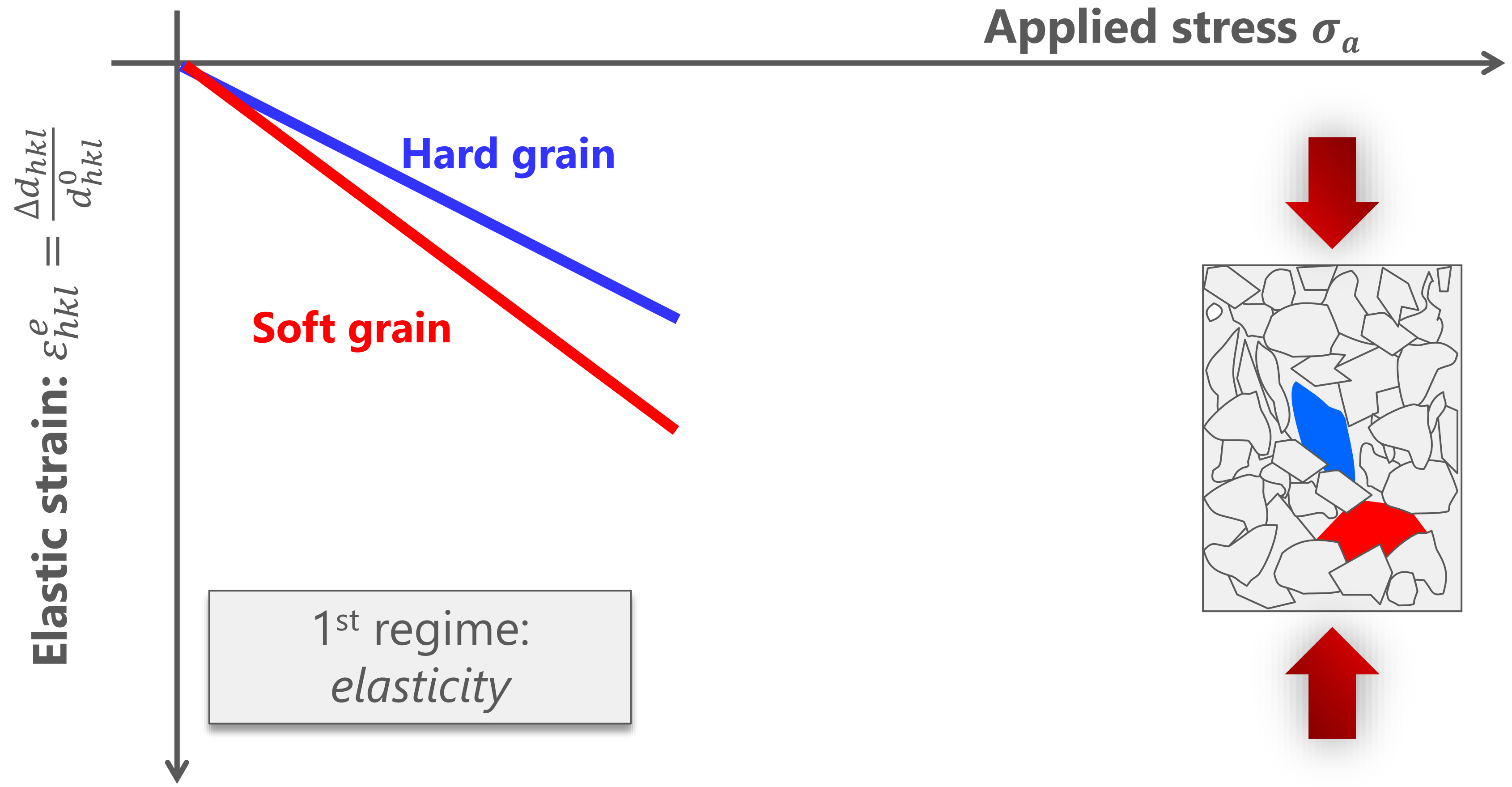

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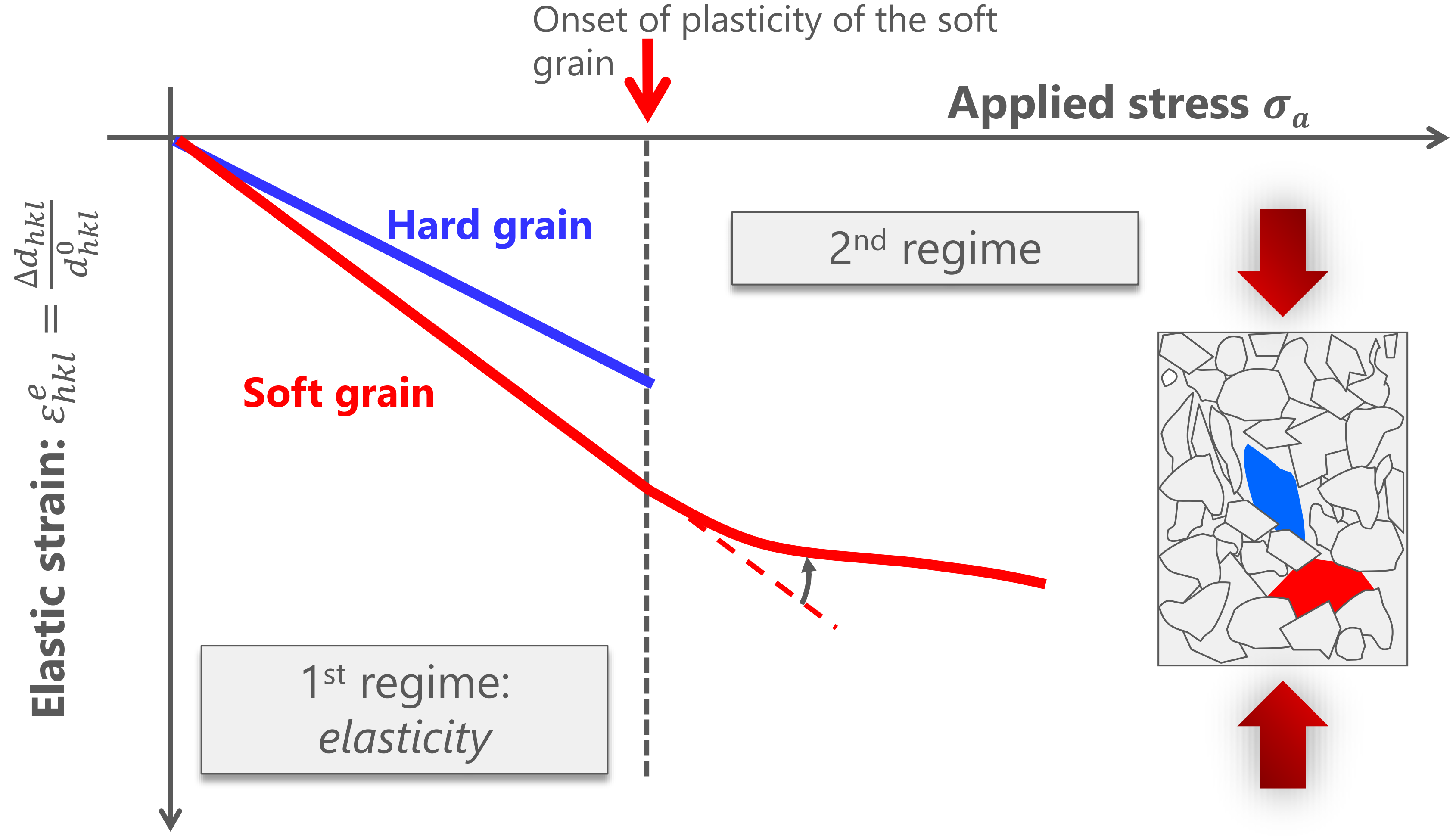

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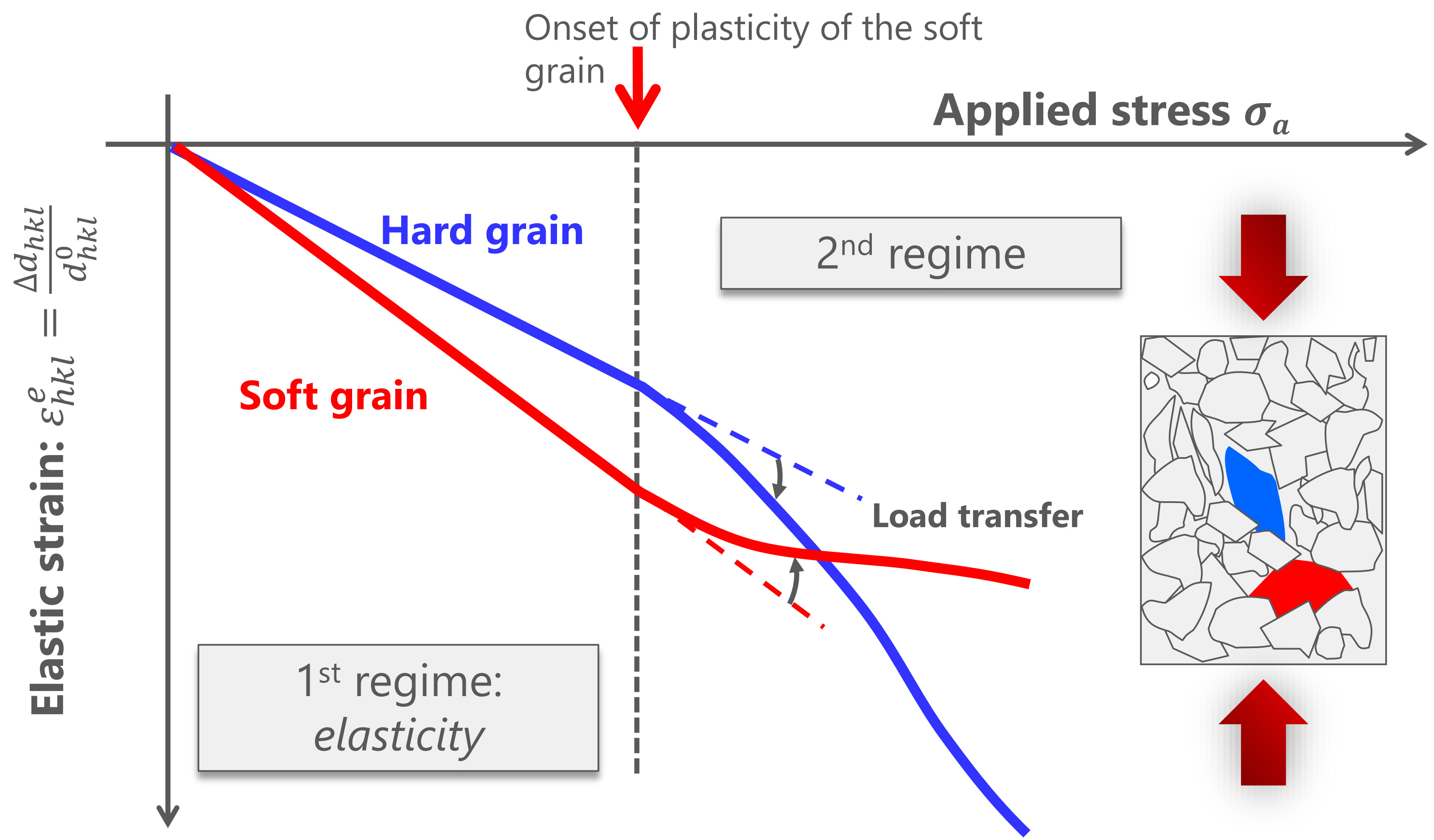

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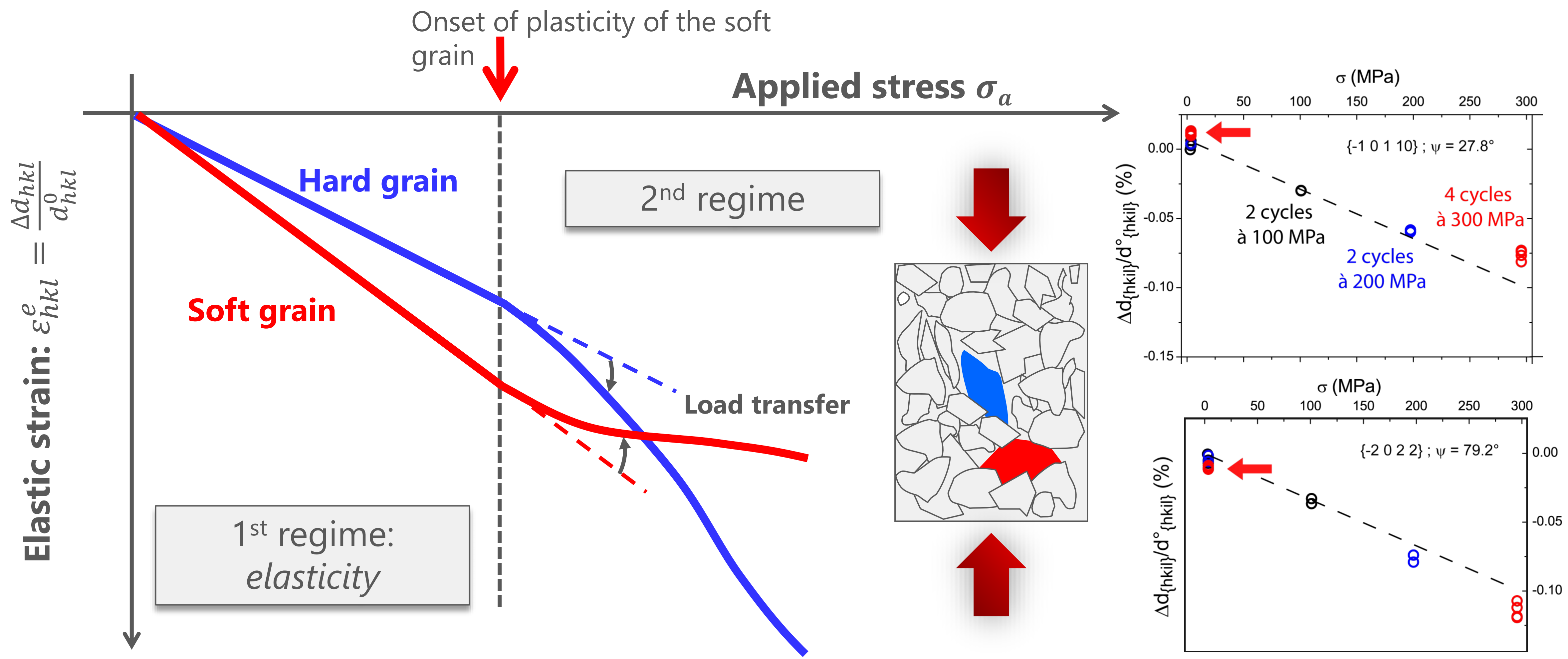

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Residual stresses are internal stresses that remain within a material after all external forces or thermal gradients have been removed.

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$$\varepsilon_{hkl}^{\psi\varphi} = \frac{1+\nu}{E}\sigma_\varphi\sin^2\psi - \frac{\nu}{E}(\sigma_1+\sigma_2) \Rightarrow \varepsilon_{hkl}^{\psi\varphi} = f(\sin^2\psi) = A\cdot\sin^2\psi + B$$

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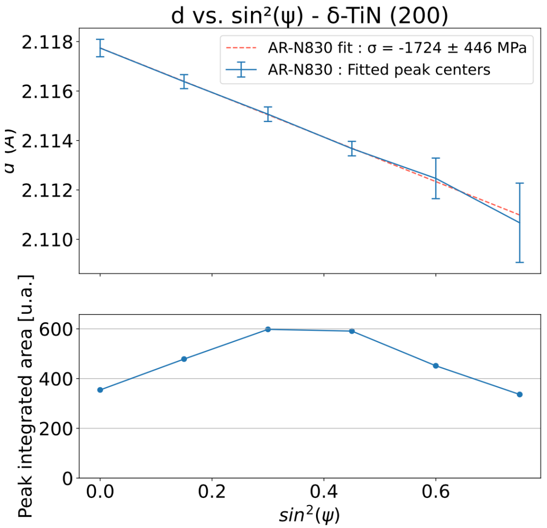

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Plotting $\varepsilon_{hkl}^{\psi\varphi} = f(\sin^2\psi)$ yields a straight line; making residual stress measurable through its slope.

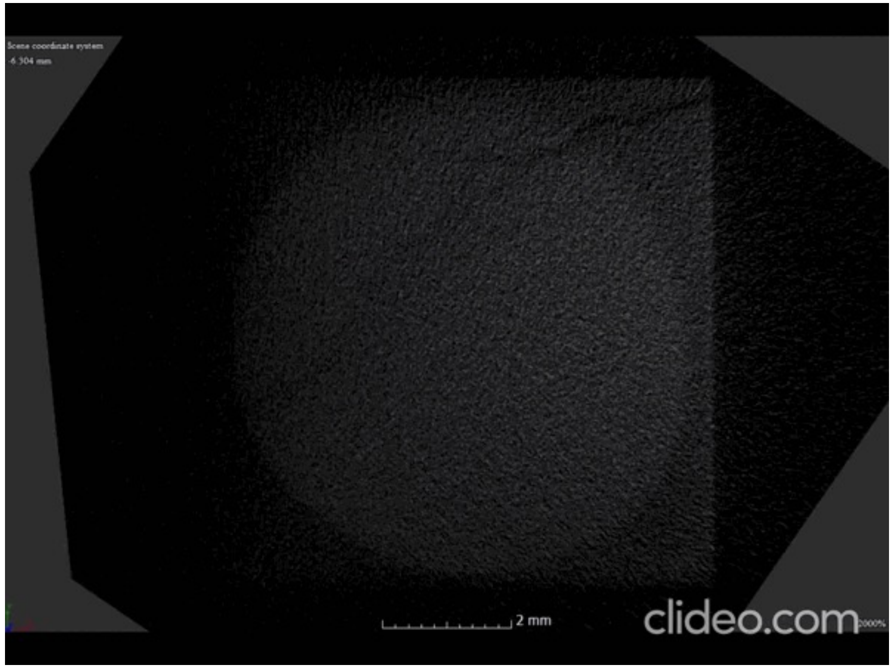
R. Génin, *PhD Thesis* (2025)

X-ray tomography

- Non-destructive 3D imaging technique based on differential X-ray absorption
- Reconstructs internal structures of materials with micron to sub-micron resolution
- Contrast arises from density and atomic number differences
- Applicable to a wide range of materials: metals, polymers, composites, biomaterials
- Enables quantitative analysis: porosity, cracks, inclusions, grain morphology
- The Beer-Lambert law:

$$N = N_0 \exp(-\mu_\rho \rho t)$$

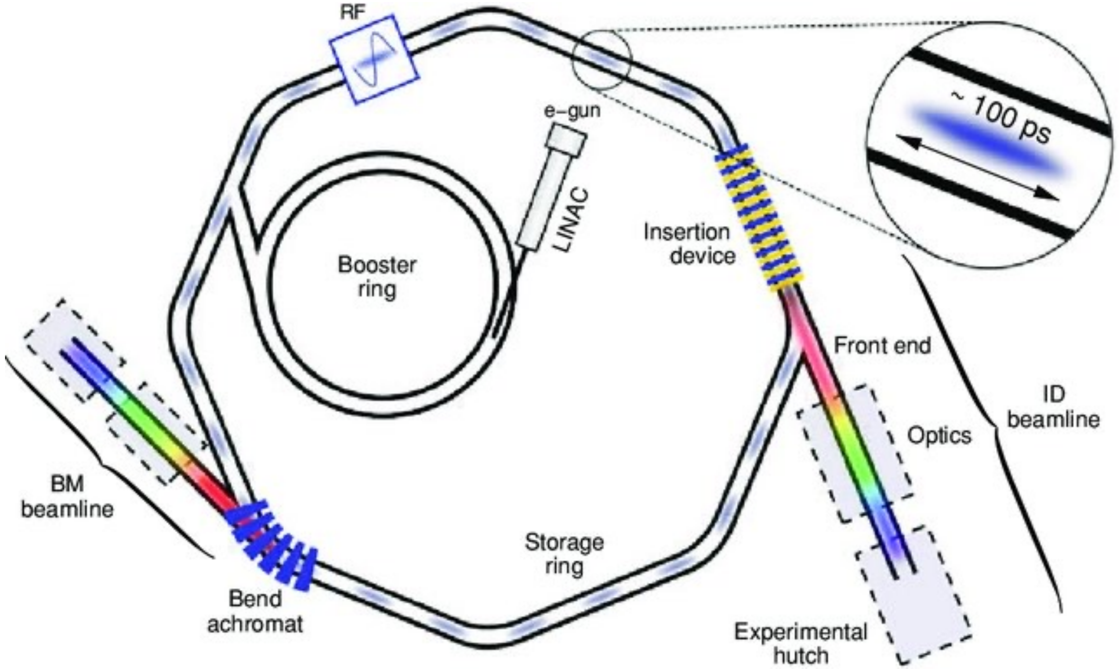
(N: number of photons; N_0: initial number of photons; ρ: density; t: thickness; μ_ρ: mass attenuation coefficient)

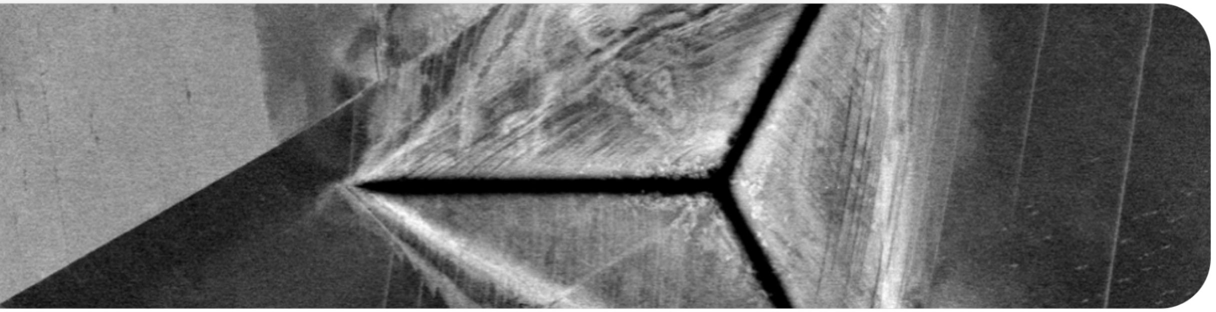


A. Monnet, *PhD work* (2025)

Large x-ray facilities

PSI





Electron microscopy

Micro- Nano-meter scale

The 2 types of electron microscopes

Transmission electron microscope (TEM)

- Invention: 1931 by Ernst RUSKA et Max KNOLL
- Specimen : thin foils (~ 100 nm)
- Price: ~ 1 M€
- Rare in industry

↳Nanometric/atomic scale

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Scanning electron microscope (SEM)

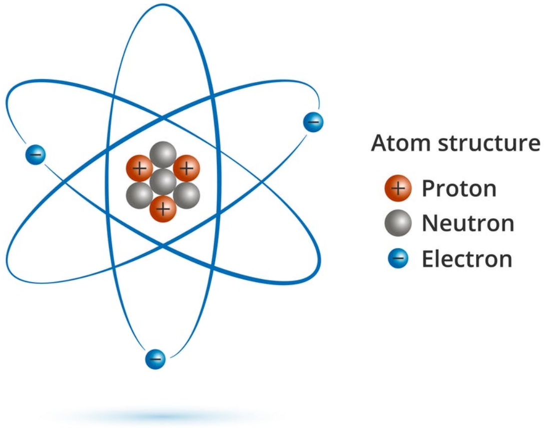
- Invention: 1937 by Manfred VON ARDENNE but developed in the 1960s
- Specimen : bulk
- Price: ~ 500 k€
- Comon in industry

↳Meso-/micro-/nano-metric scale

↳ **The two microscopes are complementary.**

Focus on the electron

Particle



Joseph John THOMSON
(1856–1940)

- $m_e = 9.11\times10^{-31}$ **kg**
- $e = -1.60\times10^{-19}$ **C**

J.J. Thomson, *Philos. Mag.* (1897)
L. De Broglie, *PhD thesis.* (1924)

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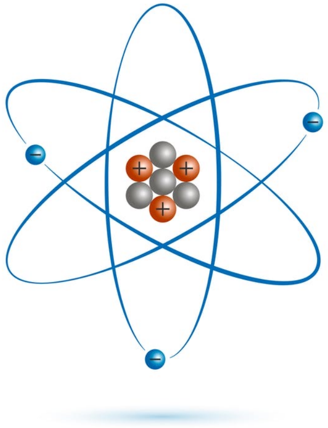


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Wave


Louis DE BROGLIE
(1892–1987)
1 Nobel Prize (1929)

- All moving particle has wave properties with the wavelength λ being related to the momentum p by:

$$\lambda = \frac{h}{p} = \frac{h}{mv}$$

Electrons act not only as particles but as waves too.

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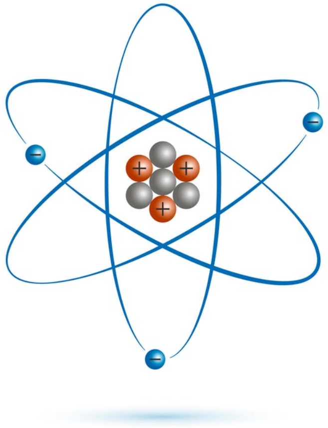

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- Energy of an electron in an electric field of a voltage V:

$$E = eV = \frac{mv^2}{2} \Leftrightarrow v = \sqrt{\frac{2eV}{m}} \Rightarrow \lambda = \frac{h}{\sqrt{2meV}}$$

$$\rightarrow \lambda = \frac{h}{\sqrt{2meV}\left(1 + \frac{eV}{2mc^2}\right)}$$

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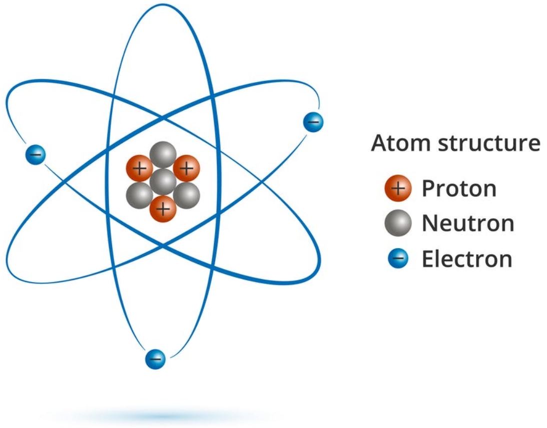

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$\lambda(30\ \mathrm{kV}) = 7\ \mathrm{pm}$
$\lambda(200\ \mathrm{kV}) = 2.5\ \mathrm{pm}$

J.J. Thomson, *Philos. Mag.* (1897)
L. De Broglie, *PhD thesis.* (1924)

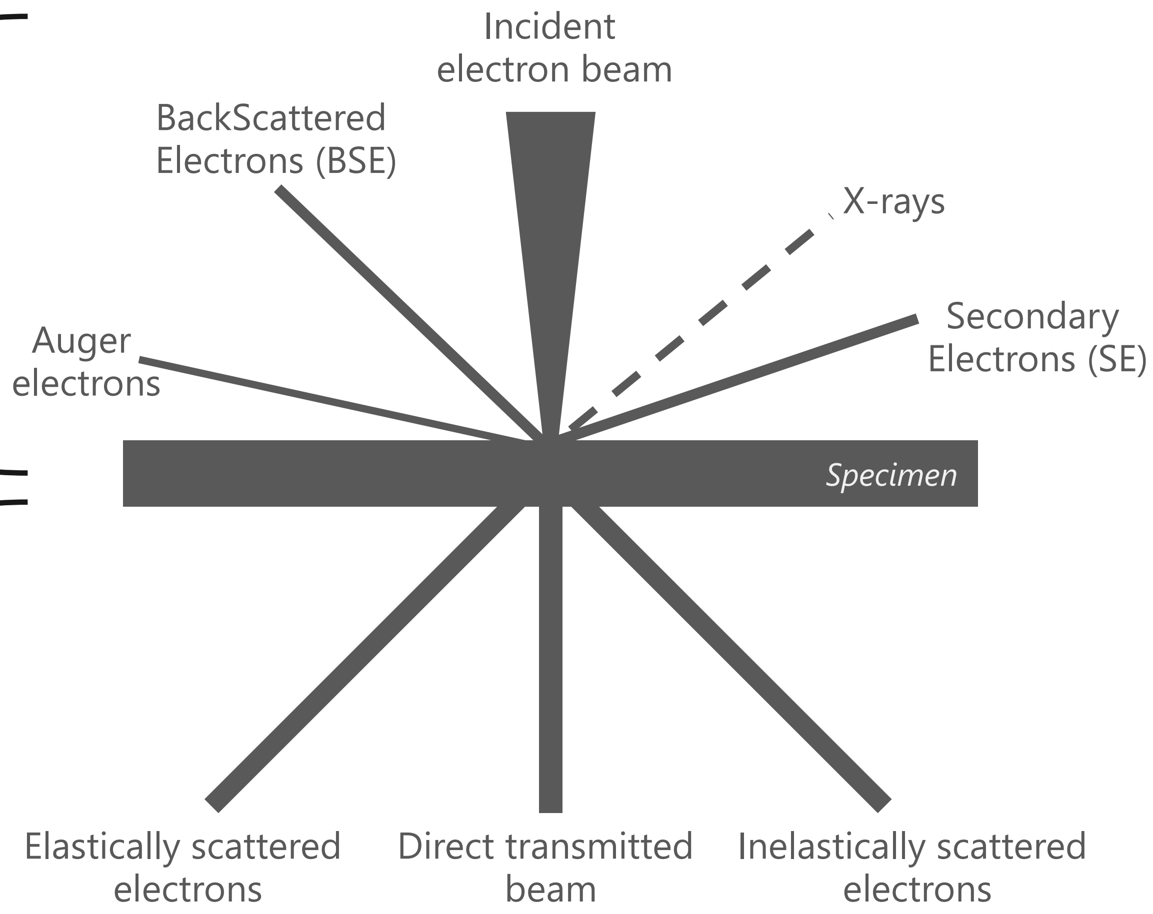
Electron-matter interaction

Reflection

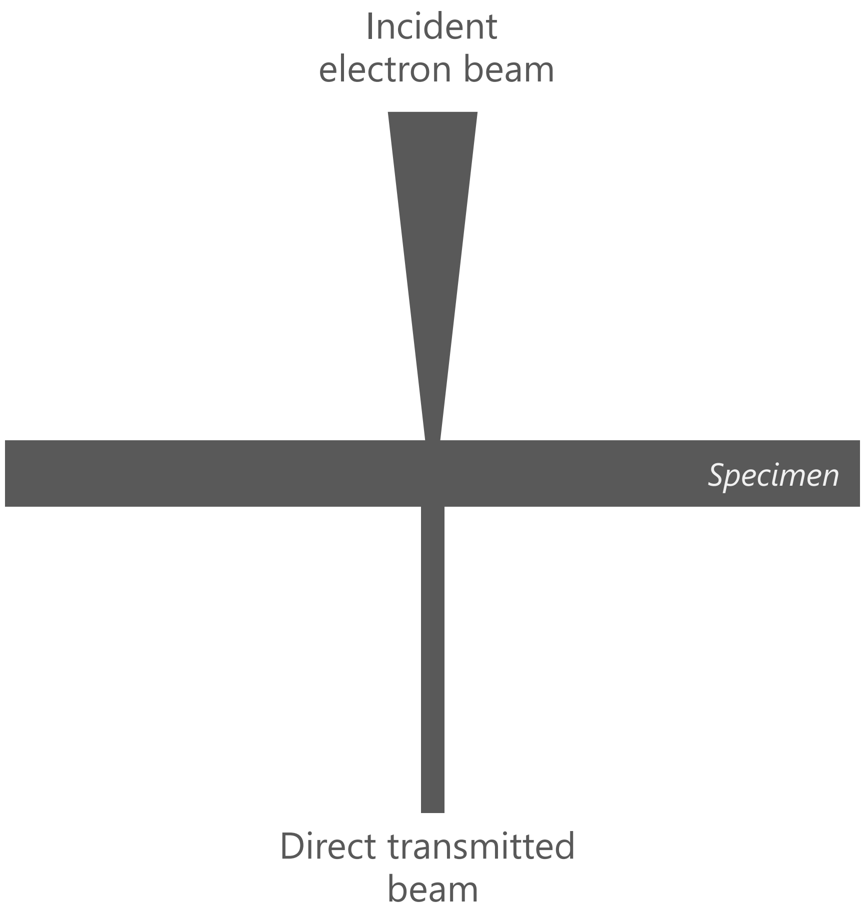
⇒ SEM
Bulk specimens

Transmission

⇒ TEM (+SEM)
Thin foils (~ 100 nm)



Electron-matter interaction



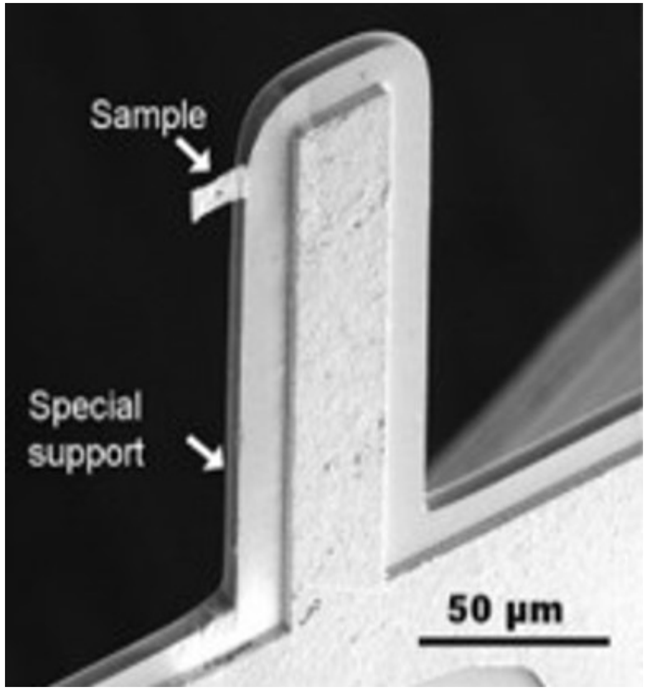
Transmission

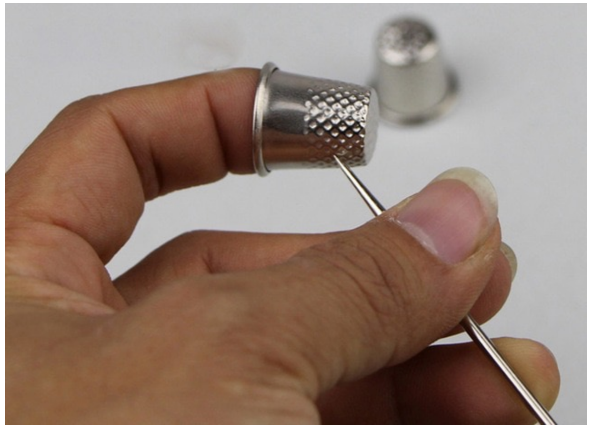
⇒ TEM (+SEM)

Thin foils (~ 100 nm)

Specimen thickness

Thickness ≈ 100 nm @ 200 kV (for conventional TEM)



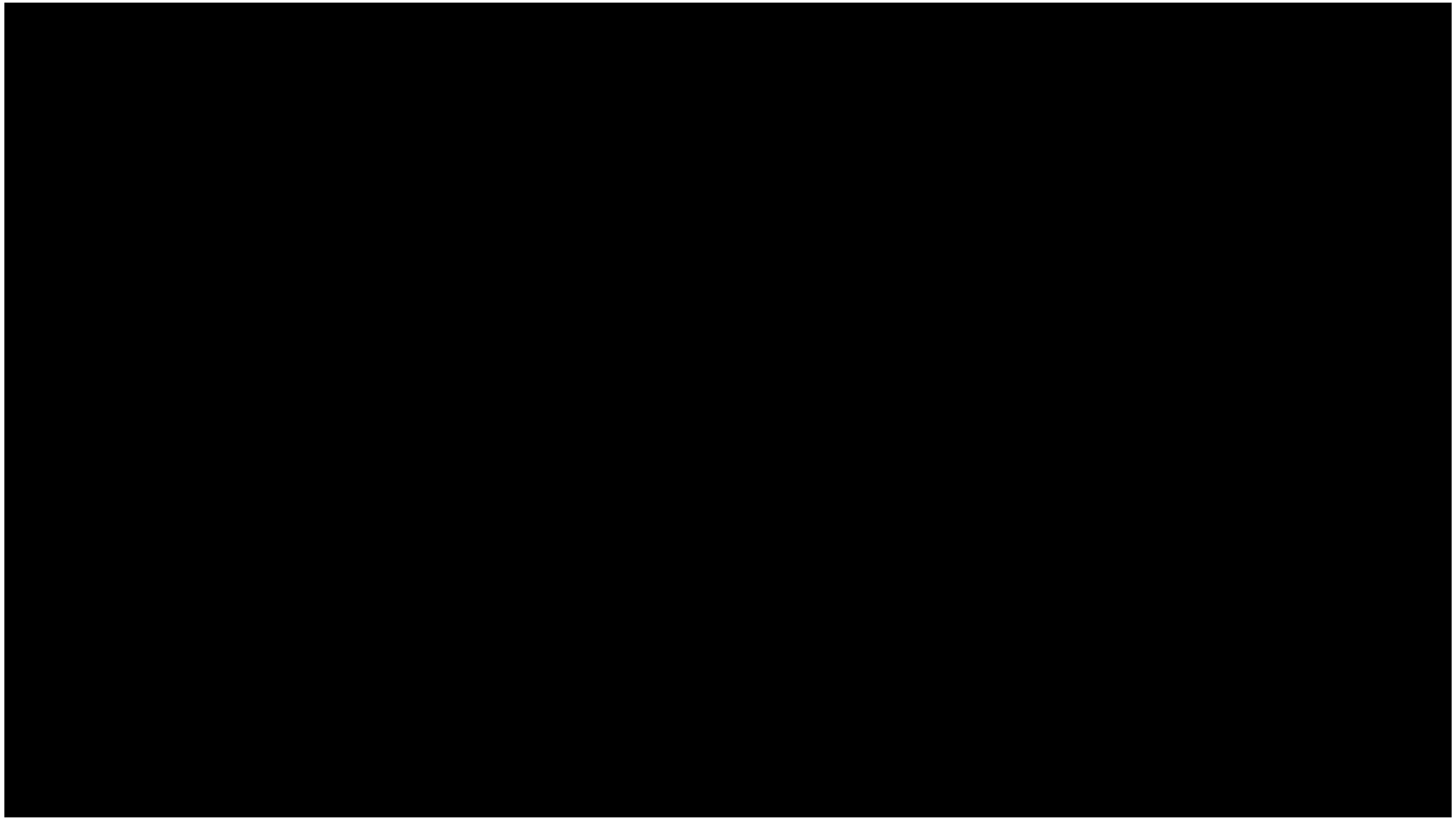


If one is able to assemble all zones studied by TEM since its development in the 40's, they would not even fill a thimble!

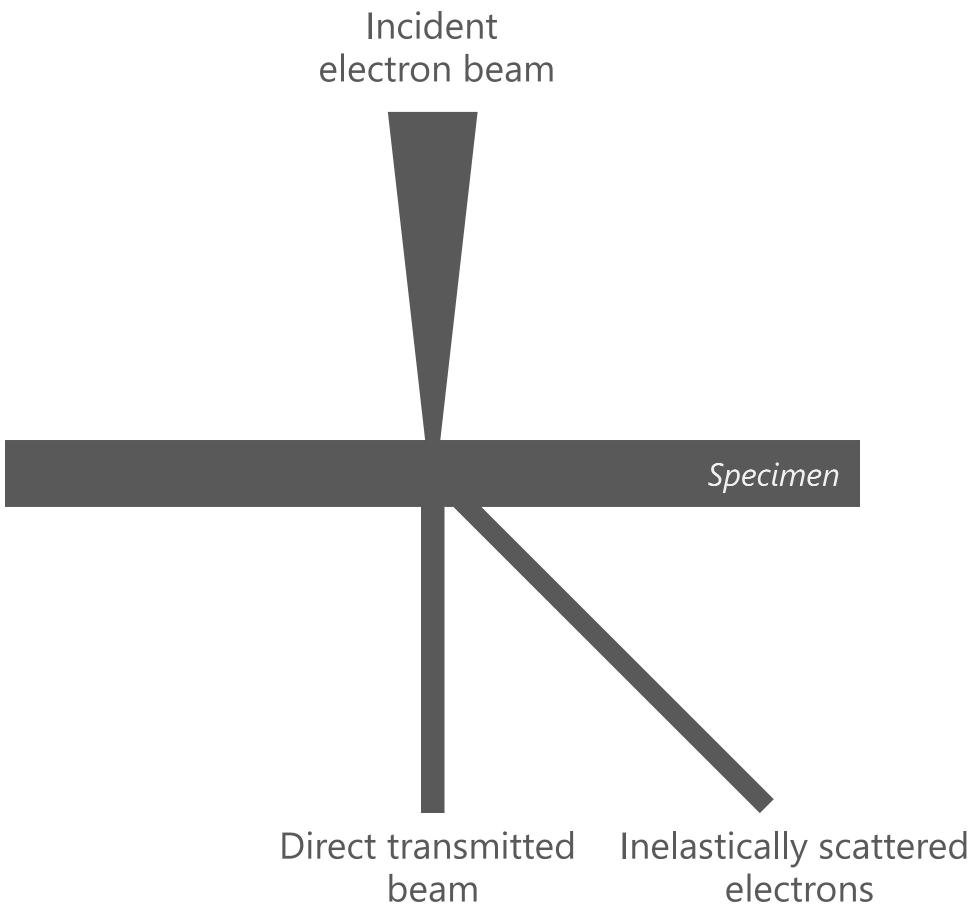
↳ Preparing TEM specimens is both an Art and a Science…

J. Ayache, J. Beaunier, J. Boumendil, G. Ehret and D. Laub, *Springer-Verlag New York. (2010)*

How to machine a TEM specimen?



Electron-matter interaction



Transmission

⇒ TEM (+SEM)

Thin foils (~ 100 nm)

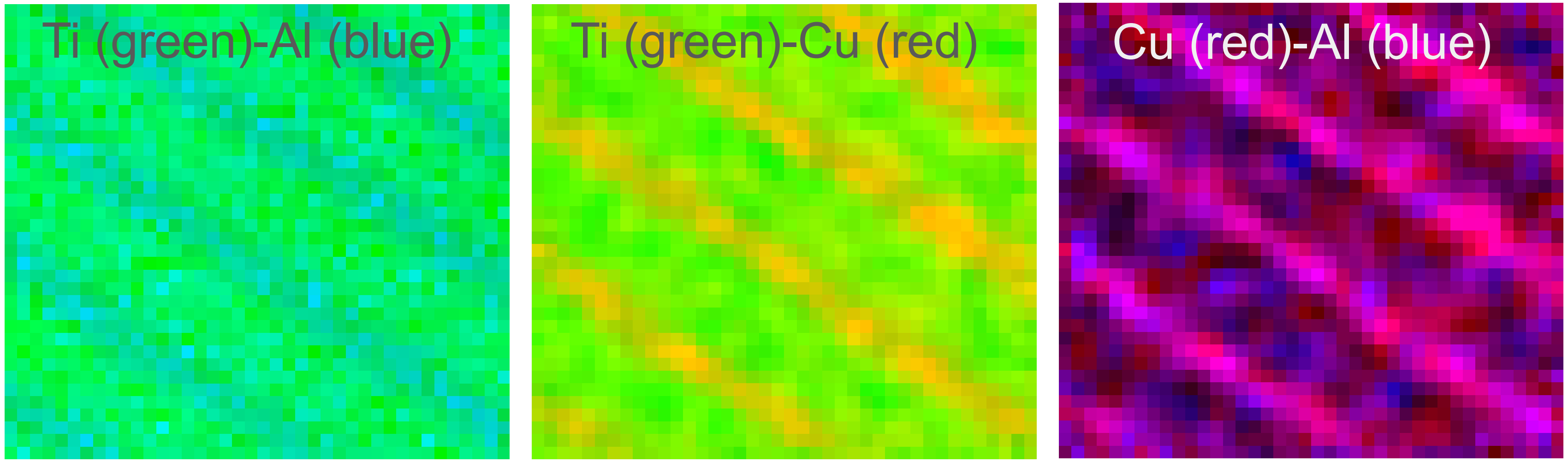
Inelastically scattered electrons

The energy is transferred from the electron to the sample.

$$\Rightarrow E_0 > E_{e^-}$$

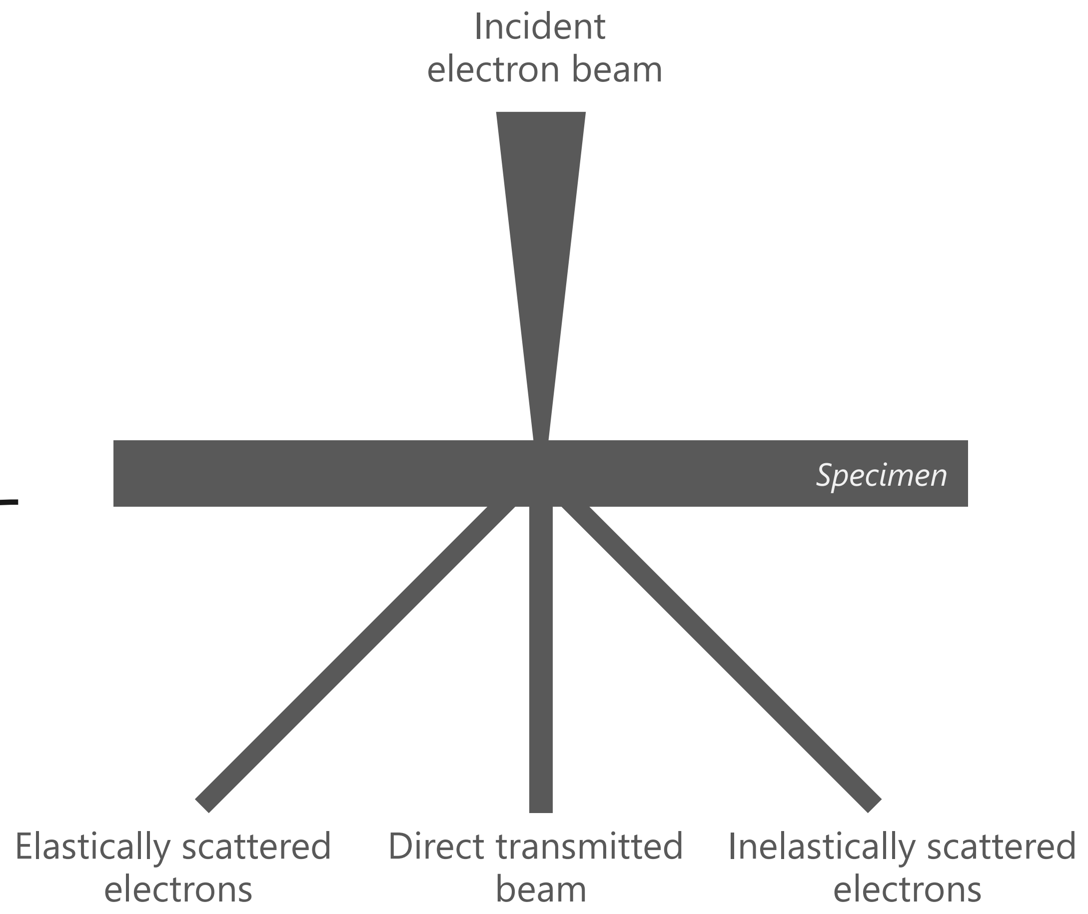
The energy loss is characteristic of the atoms encountered by the electron as it passes through the specimen.

Electron Energy Loss Spectroscopy (EELS)



W. Yu, J. Guénolé, J. Ghanbaja, M. Vallet, A. Guitton, *Scripta Mater.. (2021)*

Electron-matter interaction



Transmission

⇒ TEM (+SEM)

Thin foils (~ 100 nm)

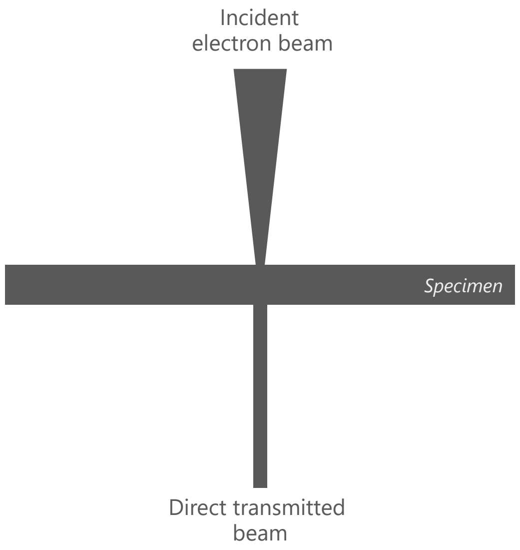
Elastically scattered electrons

No energy is transferred from the electron to the sample.

$$\Rightarrow E_0 \approx E_{e^-}$$

In the direction of the transmitted beam

- The electron passes through the specimen without any interaction.



↳ Such electrons contribute to the direct transmitted beam

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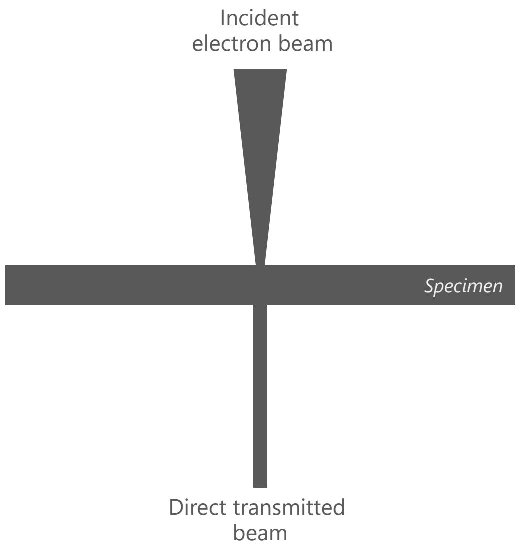

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In other directions

- The electrons are deflected from their path by Coulomb interactions with the positive potential of the atomic nuclei, screened by the surrounding electron cloud.

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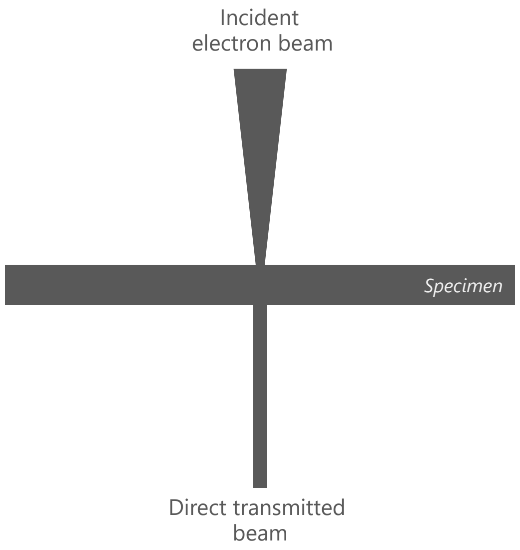

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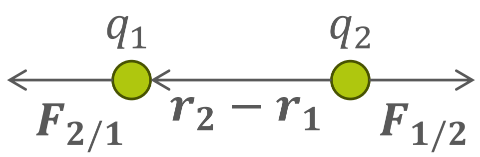
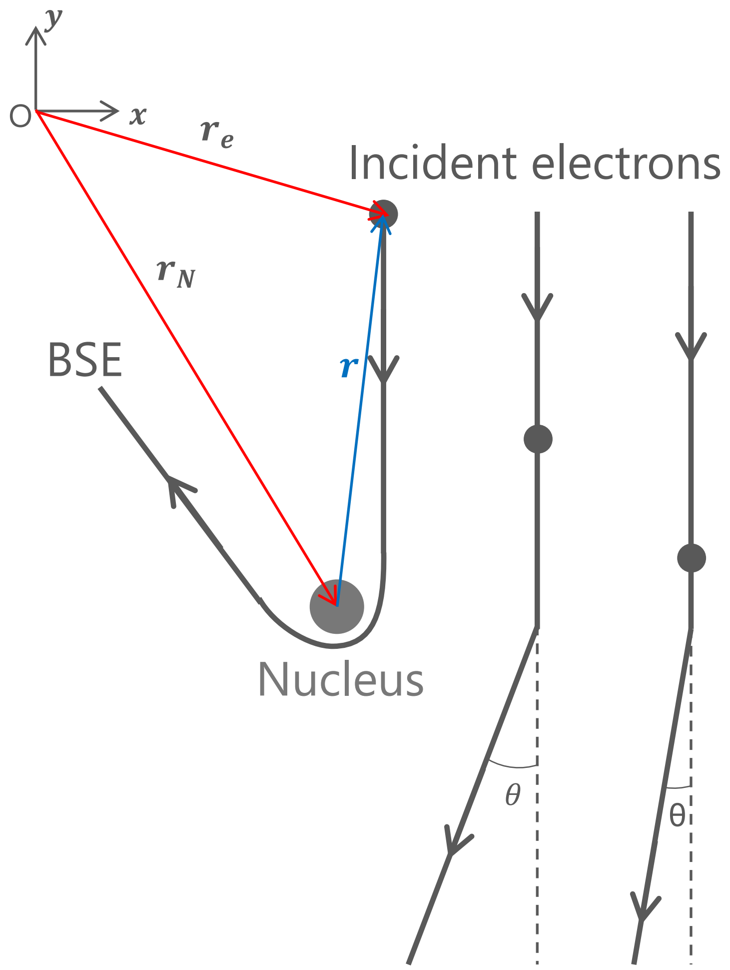


$$F_{1/2} = \frac{1}{4\pi\varepsilon_0}\frac{q_1 q_2 (r_2 - r_1)}{\|r_2 - r_1\|^3}$$

↳ Elastically scattered electrons are mainly exploited in TEM to generate contrast and diffraction patterns

Elastically scattered electrons



- Electrons are described as particles
- The electron path is deflected because of the Coulomb force:

$$\boldsymbol{F}_{N/e} = \frac{1}{4\pi\varepsilon_0}\frac{q_N q_e(\boldsymbol{r_e} - \boldsymbol{r_N})}{\|\boldsymbol{r_e} - \boldsymbol{r_N}\|^3} = \frac{1}{4\pi\varepsilon_0}\frac{q_N q_e}{\boldsymbol{r}^2}$$

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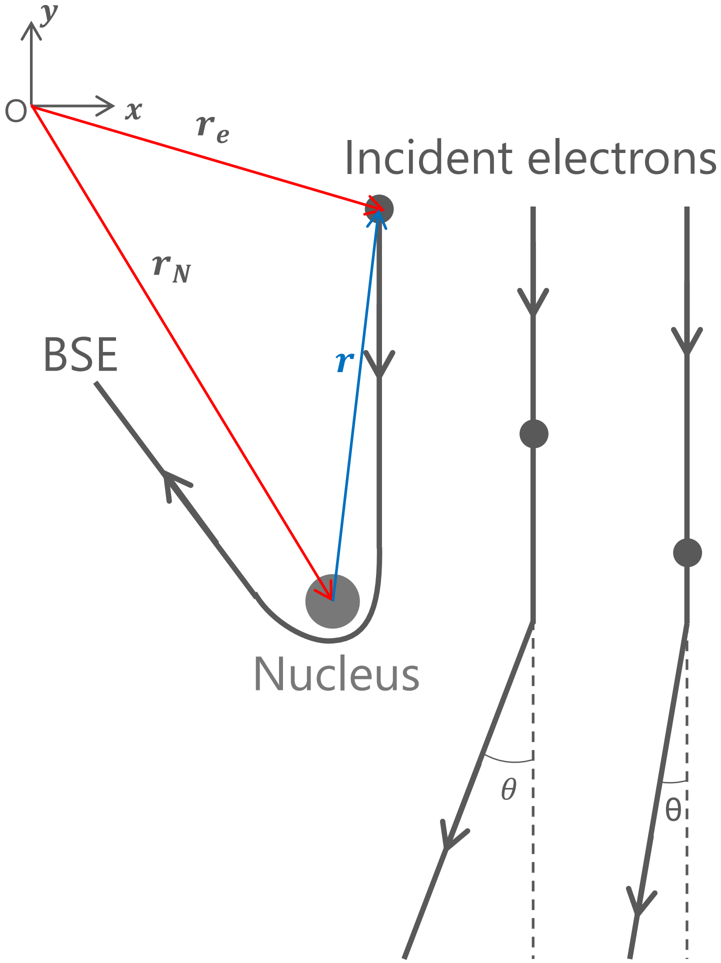

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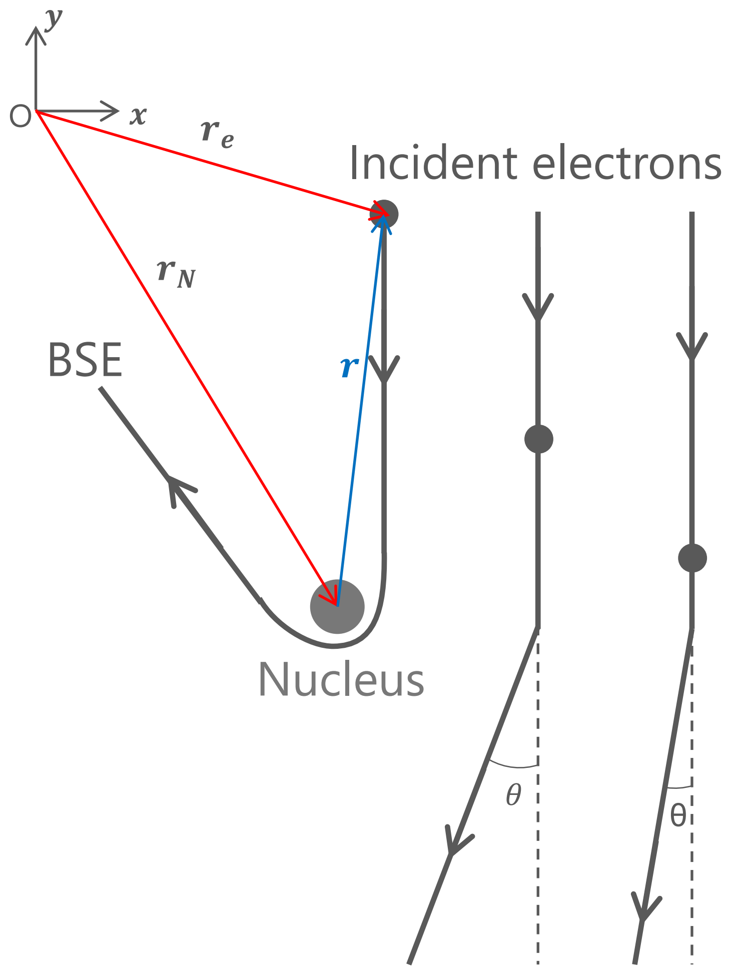
  - Scattered angle θ

$$\Rightarrow r \searrow \Rightarrow F_{N/e} \nearrow \Rightarrow \theta \nearrow$$

> If $\theta > 90°$
> $\Rightarrow$ BackScattered electrons (BSE)

Elastically scattered electrons



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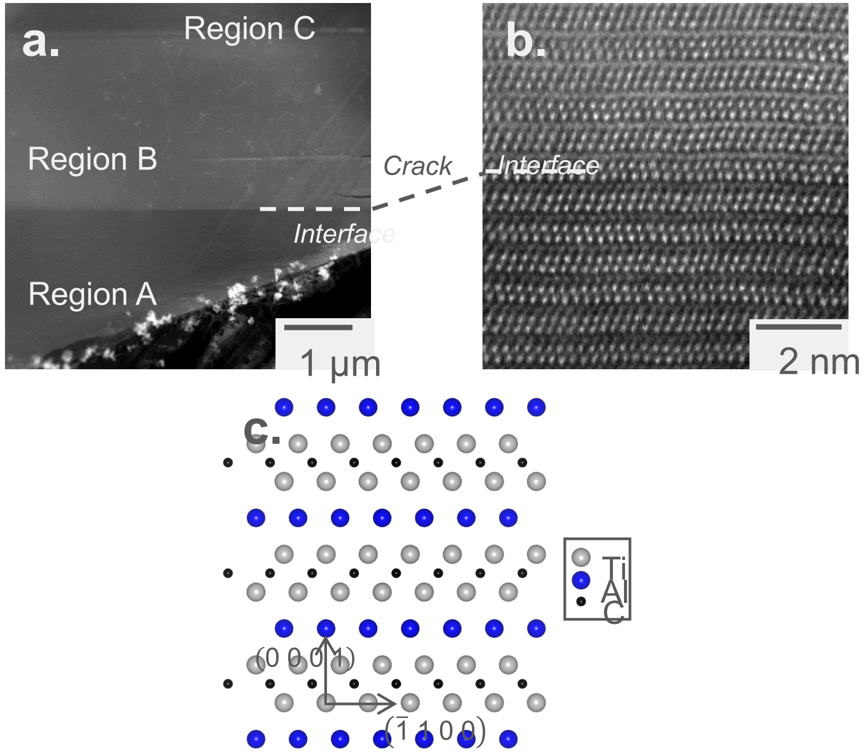
- Influence of Z:

$$q_N \nearrow \Rightarrow F_{N/e} \nearrow$$
$$q_N \nearrow \Rightarrow Z \nearrow$$
$$Z \nearrow \Rightarrow \theta \nearrow$$

> ⇒ High Z produces higher intensity at large θ

Intensity at large θ

Inside the (S)TEM



W. Yu, et al, *Scripta Mater..* (2021)

Intensity at large θ

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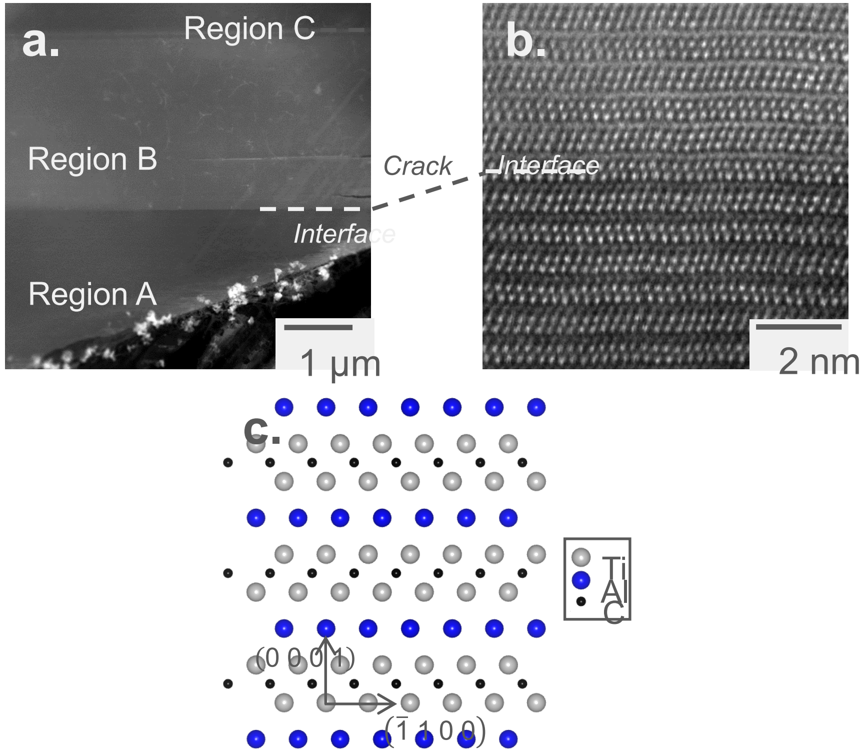
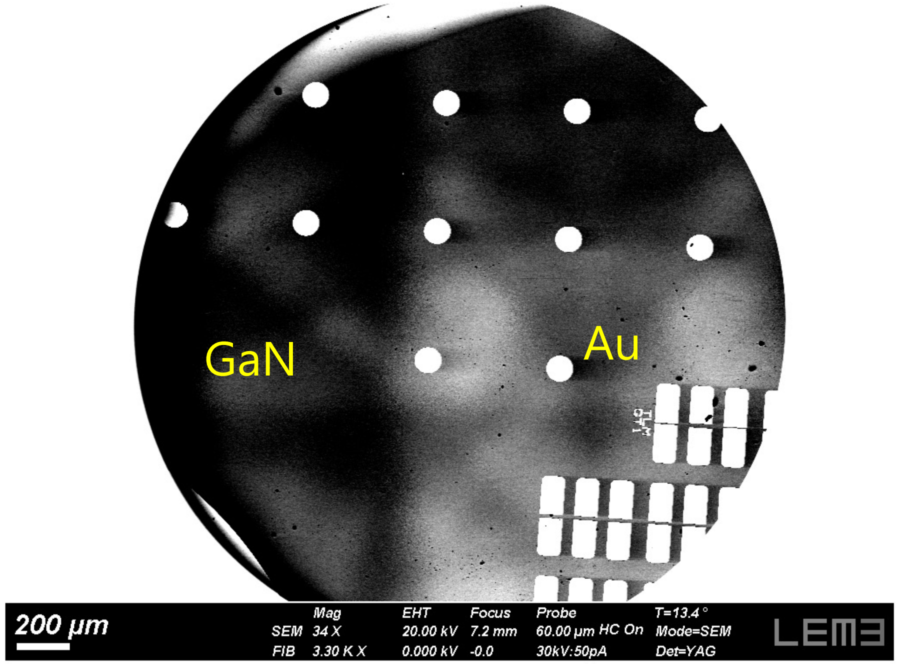


Inside the SEM

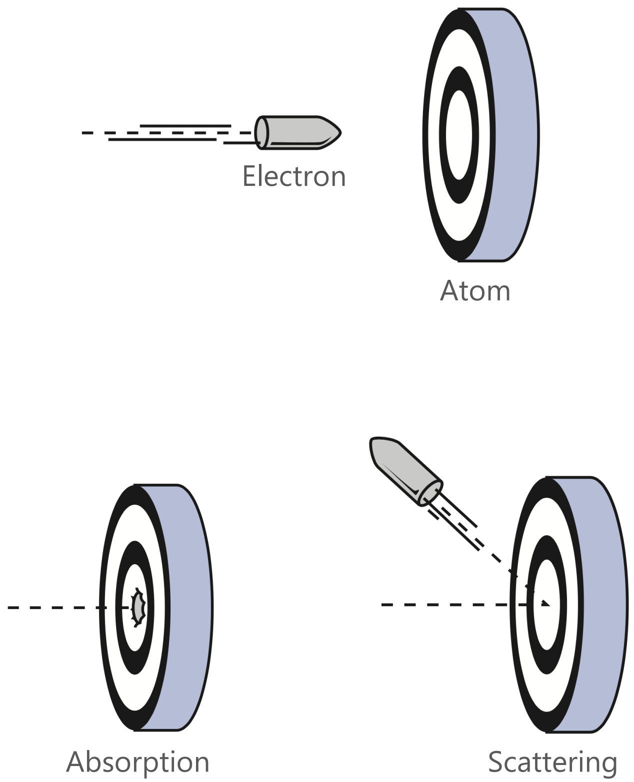


Bright contrast ⇒ high Z; Dark contrast ⇒ low Z

W. Yu, et al, *Scripta Mater.*. (2021)
A. Mandal, *Postdoctoral work* (2024)

The cross section of interaction

Cross section the probability that a certain interaction will occur between two particles for a given set of parameters.



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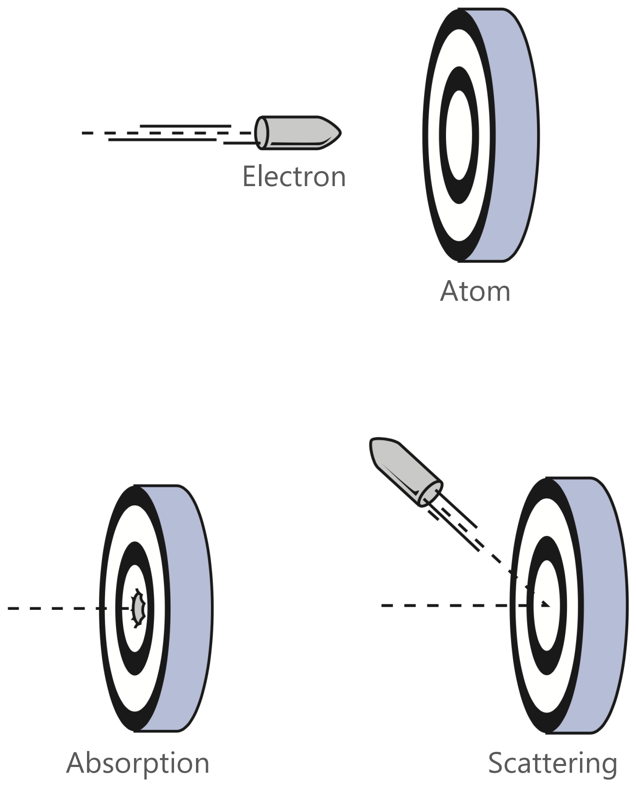

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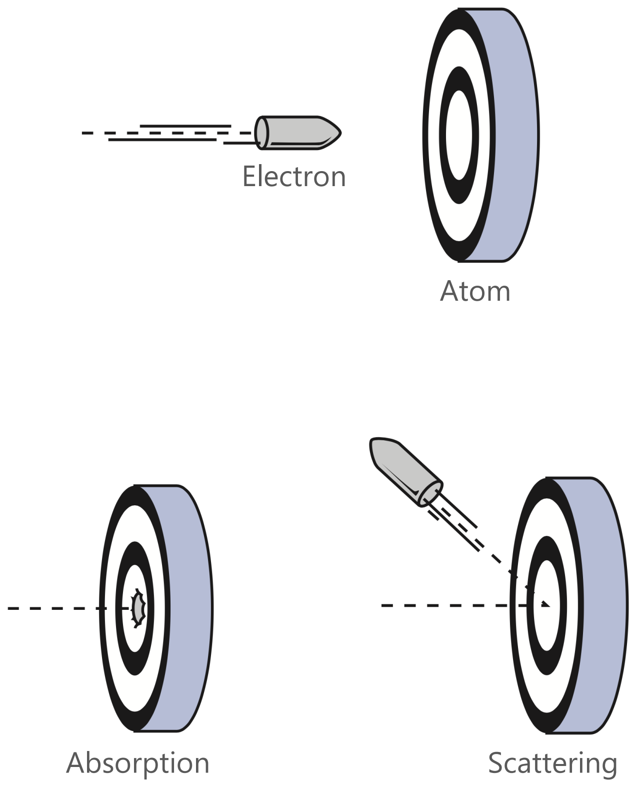

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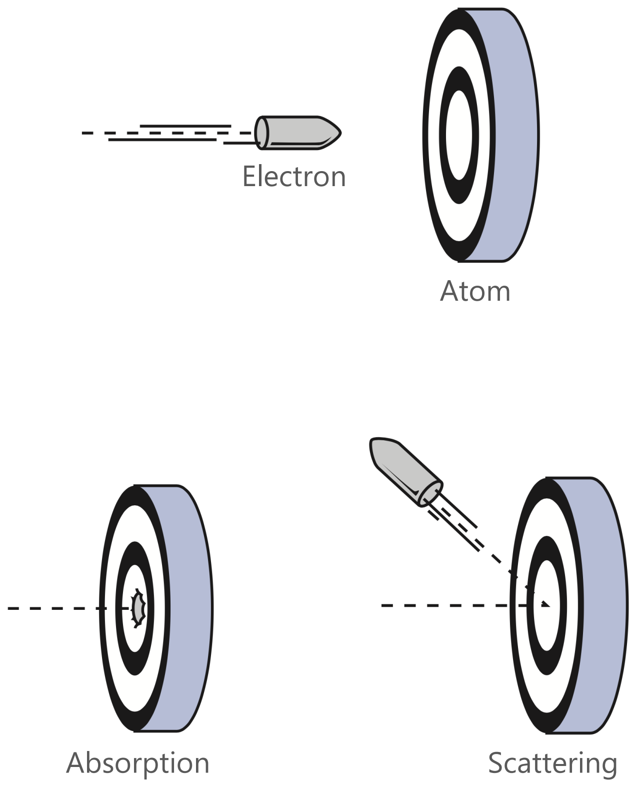

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(n: number of nuclei per volume; M: molar mass; ρ: density; N_a: Avogadro's number)

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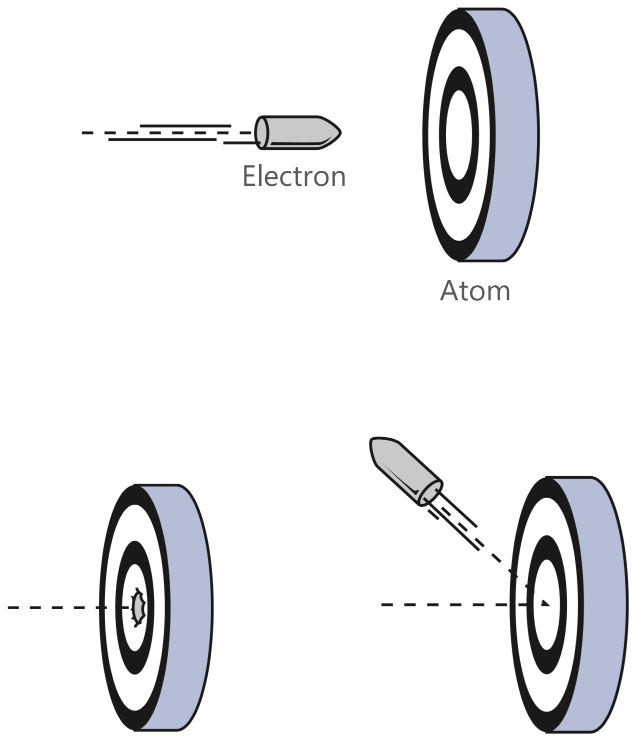

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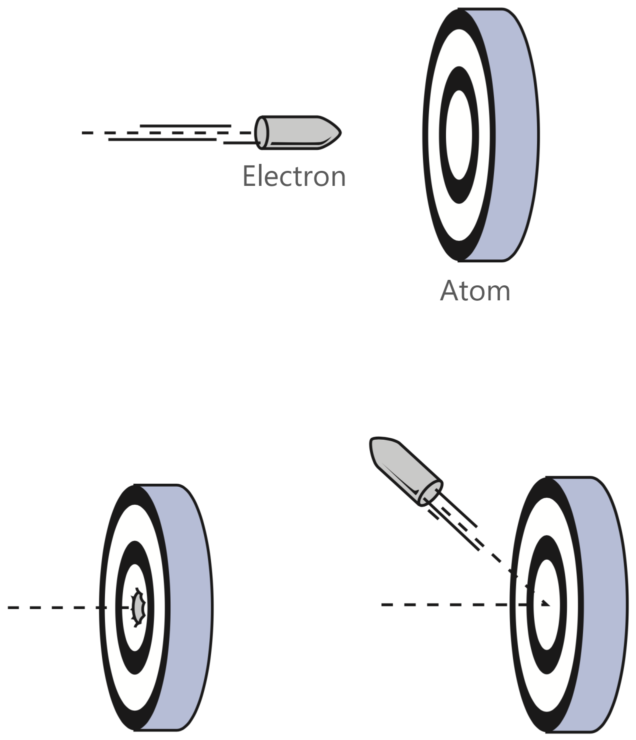

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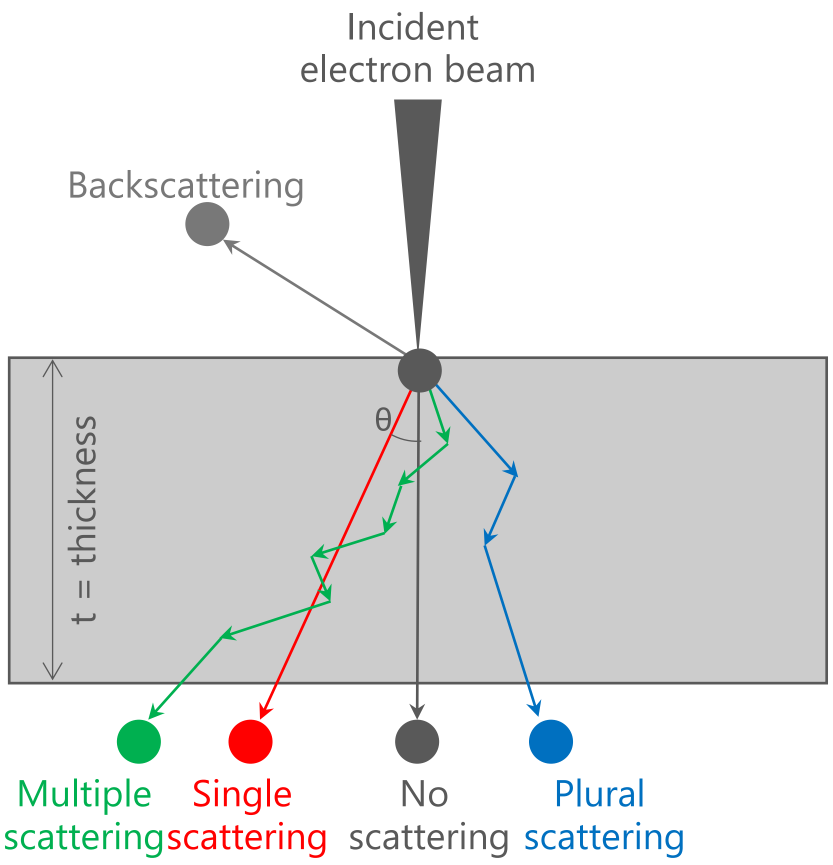
- **Mean free path:**
  - How far on average an electron will travel before it interacts with an atom
    $$\bar{l}[\mathrm{m}] = \frac{1}{\Sigma}$$

The cross section of interaction



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  - Only one interaction (either elastic or inelastic)
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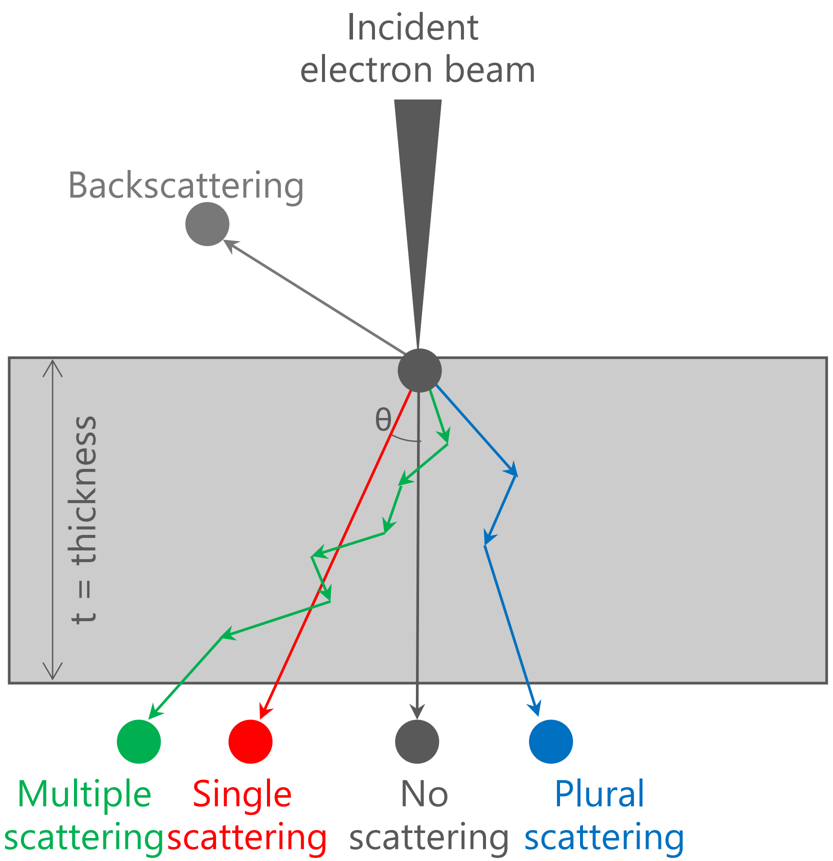
  - Ideal for quantitative analysis, since the signal is directly related to a single interaction event.

The cross section of interaction



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- Thick samples or in regions of slightly higher density.

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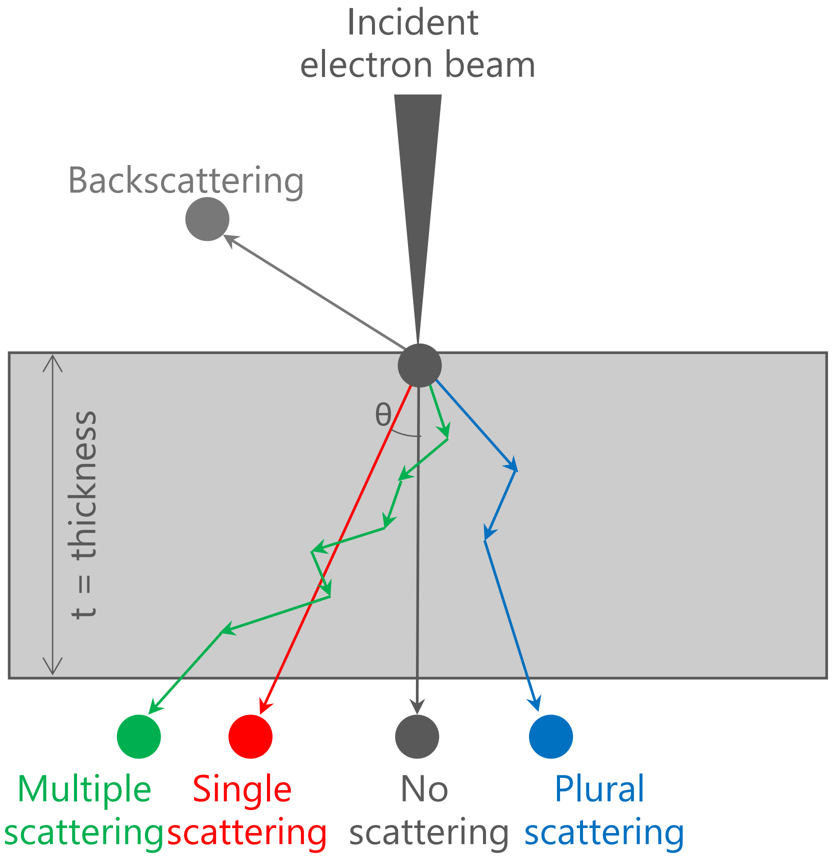

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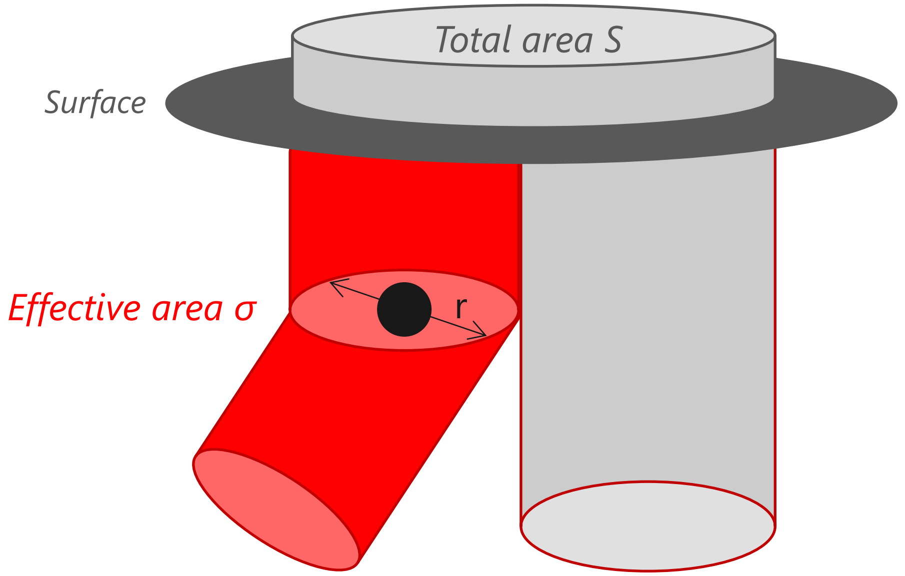
  - Thick samples or in regions of slightly higher density.
- **Multiple scattering:**
  - Many scattering events.
  - The path becomes complex, and the original direction and energy of the electron are heavily modified.
  - Common in thick or dense samples, especially in SEM where BSE and SE often result from multiple scattering.
  - Makes quantitative analysis more difficult, and signal interpretation more complex.

The cross section of interaction

- **Probability of scattering:**

$$P_{Scat} = \frac{\sigma_{tot}}{S} = \frac{\pi r^2}{S}$$

$P_{scat} \nearrow$ when $\sigma_{tot} \nearrow$



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❖ **Cross section:**

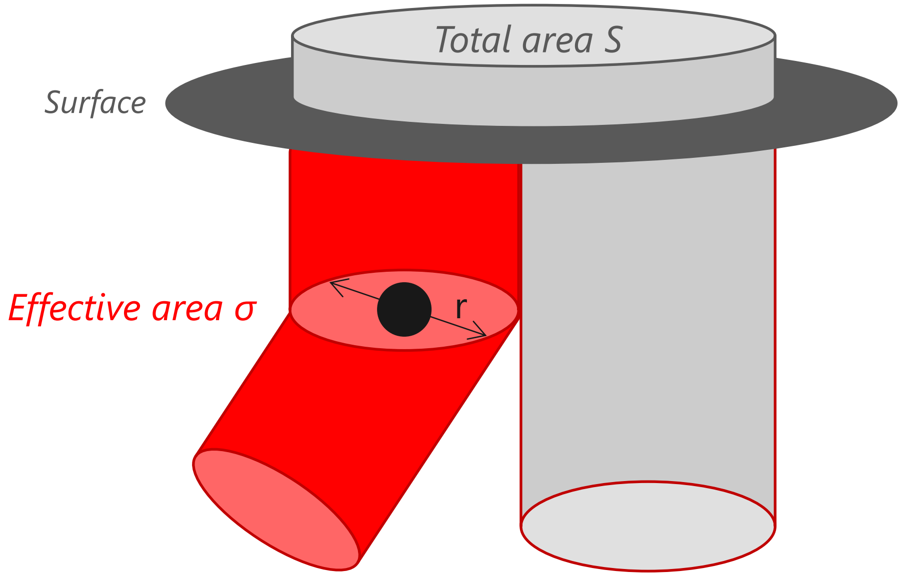
$$\sigma_{tot} = \sigma_{elastic} + \sigma_{inelastic}$$

$$\sigma_{elastic} = \pi r_{elastic}^2$$

$$r_{elastic} = \frac{Ze}{V\theta}$$

$$\Rightarrow P_{scat} \nearrow \text{ when } Z \nearrow$$

$$\theta \nearrow \Rightarrow P_{scat} \searrow$$



The cross section of interaction

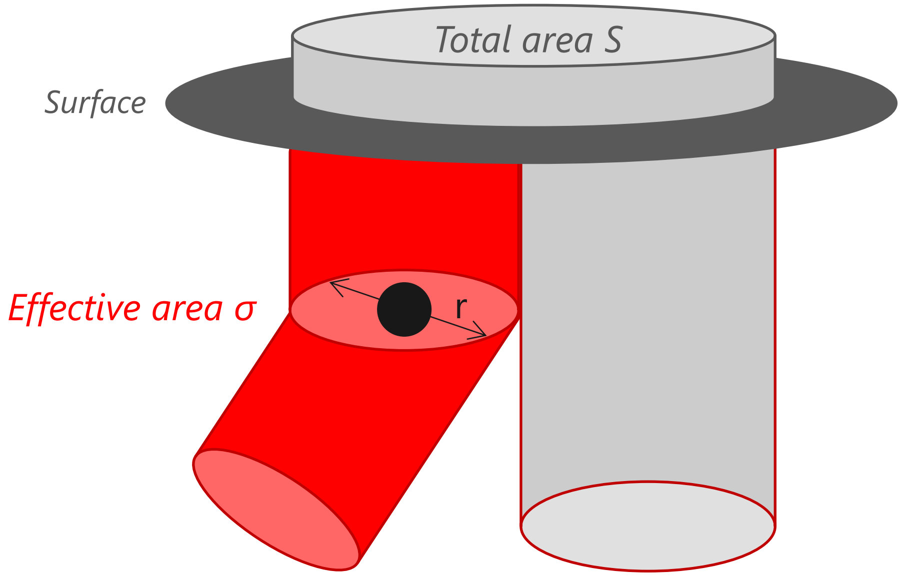
❖**Cross section for a specimen:**

$$\Sigma = n\sigma = \frac{N_a \rho}{M}\sigma_{tot}$$

$$Q_T = t\Sigma = \frac{N_a \rho t}{M}\sigma_{tot}$$

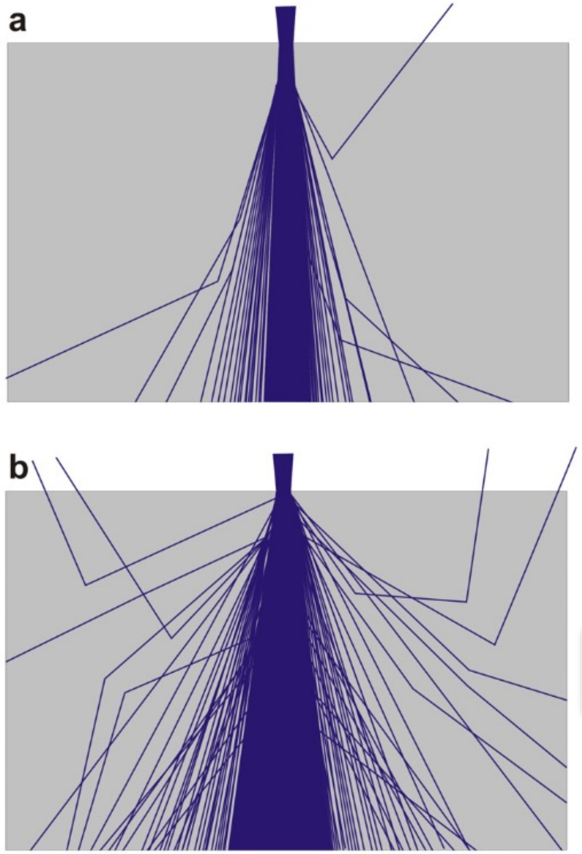
(t: thickness)

$\Rightarrow P_{scat} \nearrow$ when $t \nearrow$
$\Rightarrow P_{scat} \nearrow$ when $\rho \nearrow$



Interaction volume

Thin foil



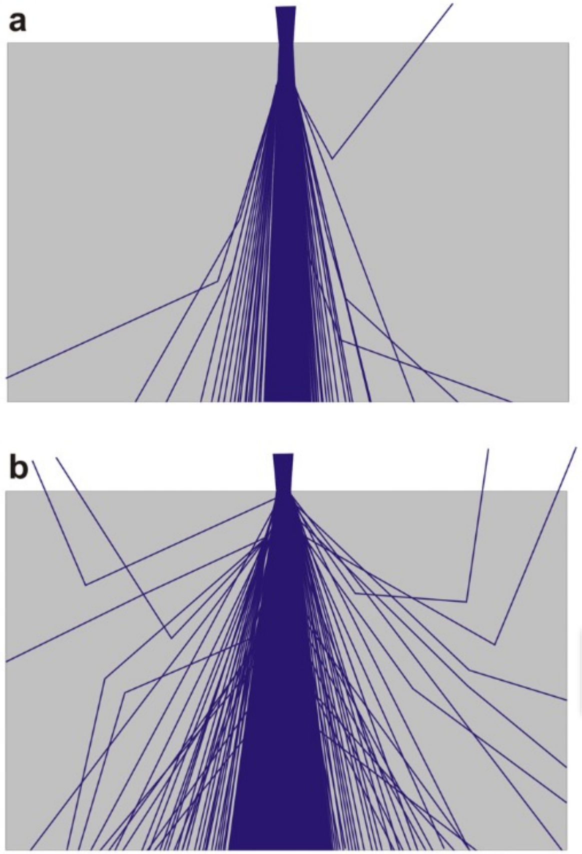
Low Z

High Z

Interaction volume

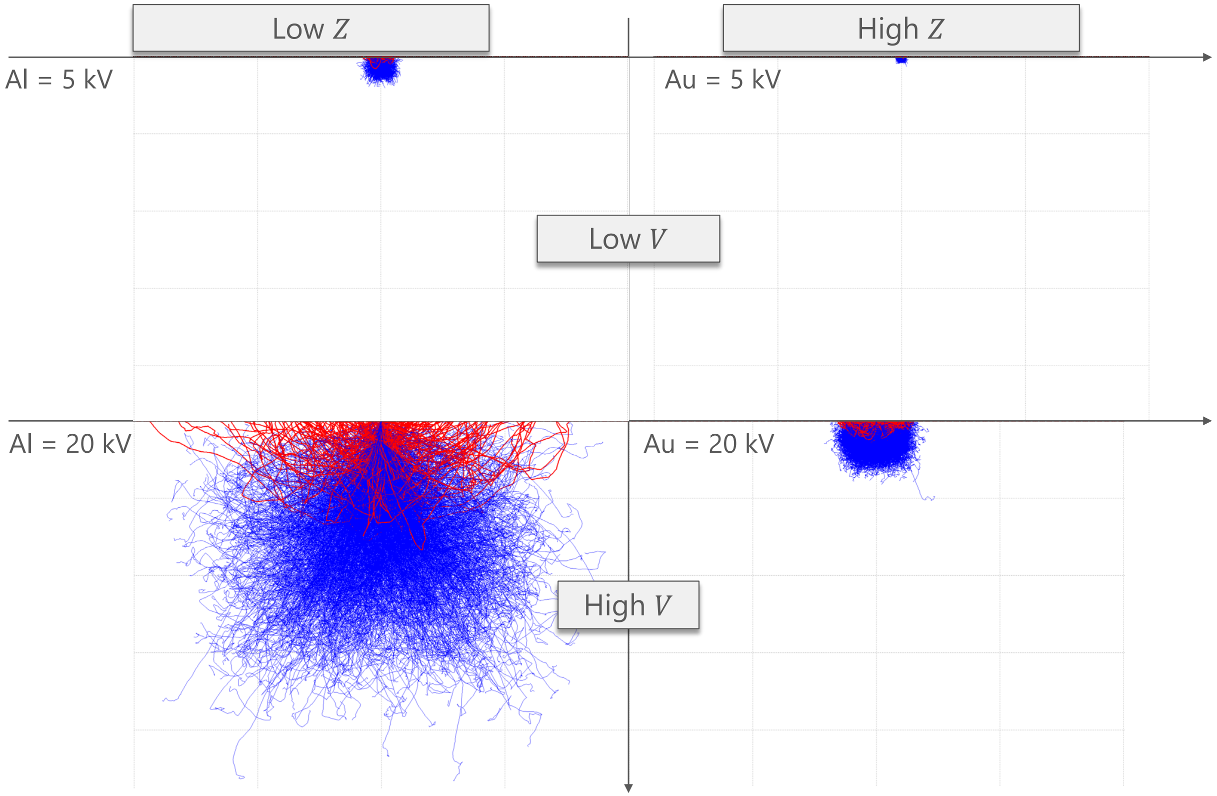
Thin foil

Low Z



High Z

Bulk specimen



E. Lavallée, *PhD work (2025)*

The 2 types of electron microscopes

Transmission electron microscope (TEM)

- Invention: 1931 by Ernst RUSKA et Max KNOL
- Specimen : thin foils (~ 100 nm)
- Price: ~ 1 M€
- Rare in industry

↳Nanometric/atomic scale

Scanning electron microscope (SEM)

- Invention: 1937 by Manfred VON ARDENNE but developed in the 1960s
- Specimen : bulk
- Price: ~ 500 k€
- Comon in industry

↳Meso-/micro-/nano-metric scale

↳ **The two microscopes are complementary.**

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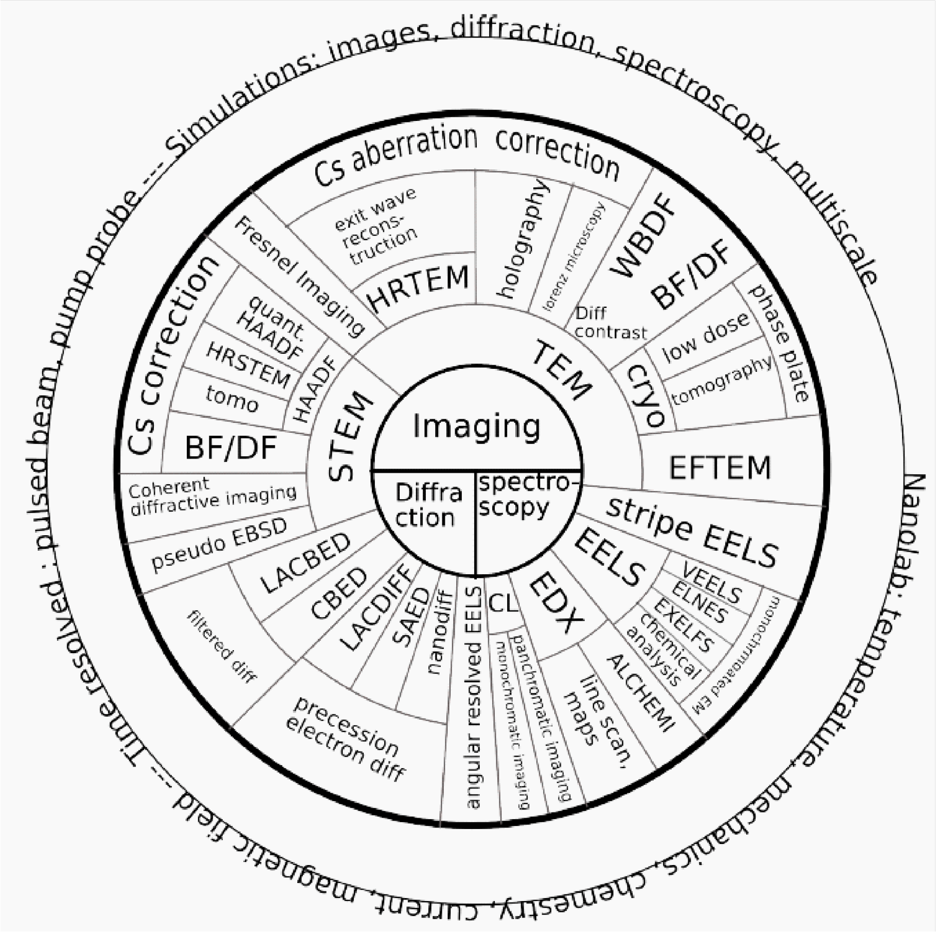

Basics of TEM contrast

Probability of scattering event:

$$Q_T = t\Sigma = \frac{N_a \rho t}{M} \sigma_{tot}$$

Basics of TEM contrast

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$$Q_T = t\Sigma = \frac{N_a \rho t}{M} \sigma_{tot}$$

Mass contrast:

$Q_T \nearrow$ *when* $\rho \nearrow$

You shall not study W by TEM…

High ρ

Low ρ

Basics of TEM contrast

Probability of scattering event:

$$Q_T = t\Sigma = \frac{N_a \rho t}{M}\sigma_{tot}$$

Mass contrast:

$Q_T \nearrow$ *when* $\rho \nearrow$

You shall not study W by TEM…

High ρ	Low ρ

Thickness contrast:

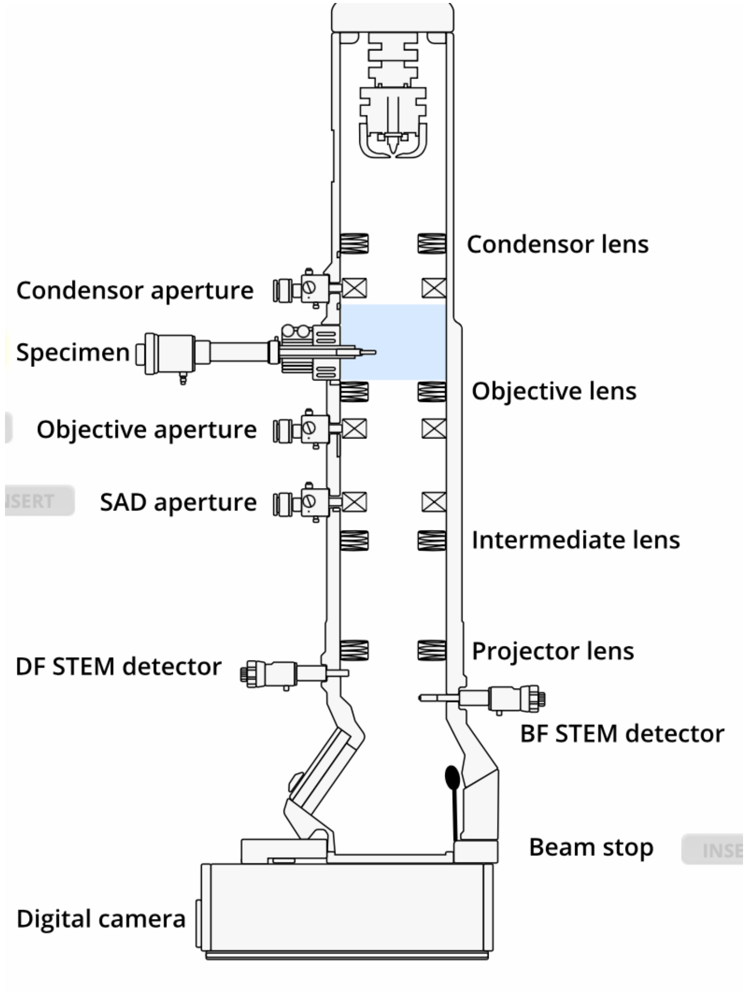
$Q_T \nearrow$ *when* $t \nearrow$

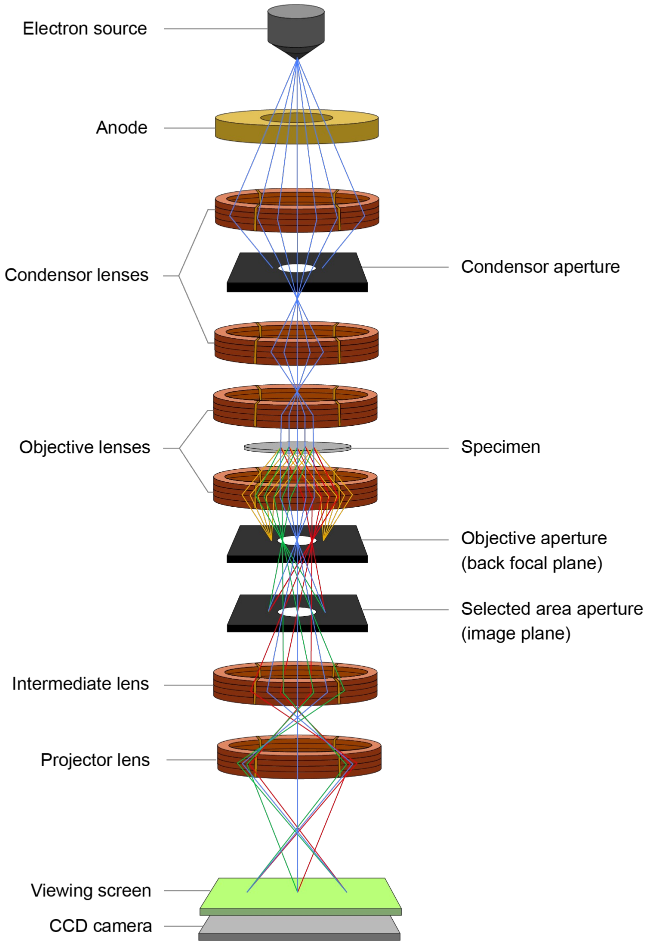
High t	Low t

You shall not study thick TEM foils…

↳It is the local scattering power that determines the contrast of TEM images

Basics of TEM

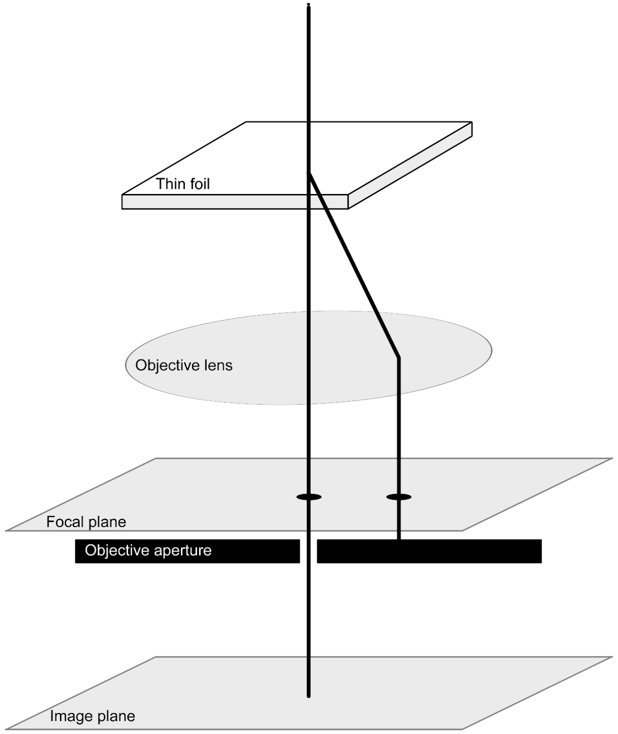


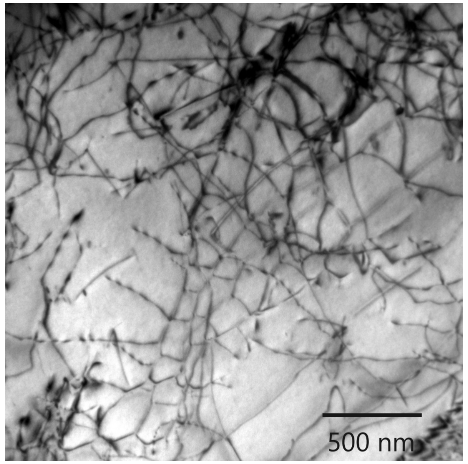


J. Aubourg, *PhD Thesis (2024)*

Basics of TEM

Bright field

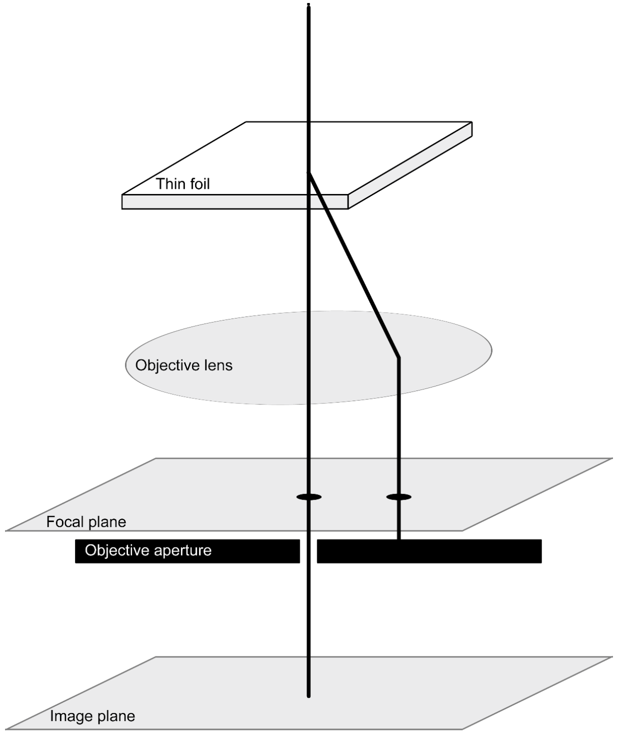


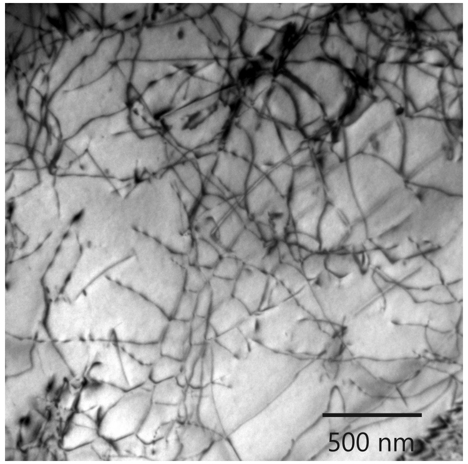


↳ Dark contrasts on a bright background

Basics of TEM

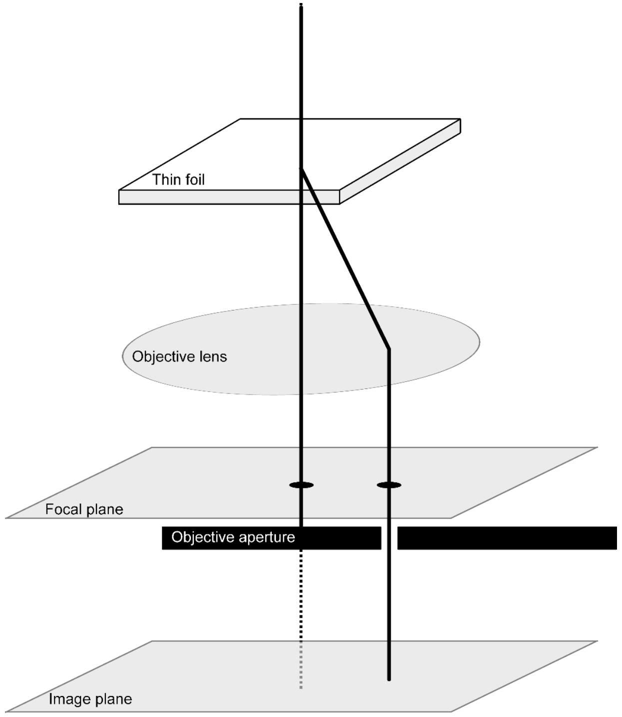
Bright field

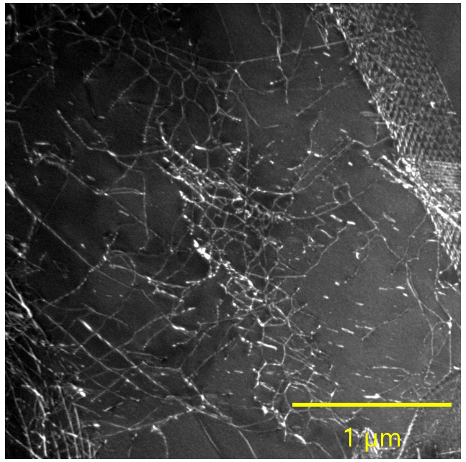




↳ Dark contrasts on a bright background

Dark field

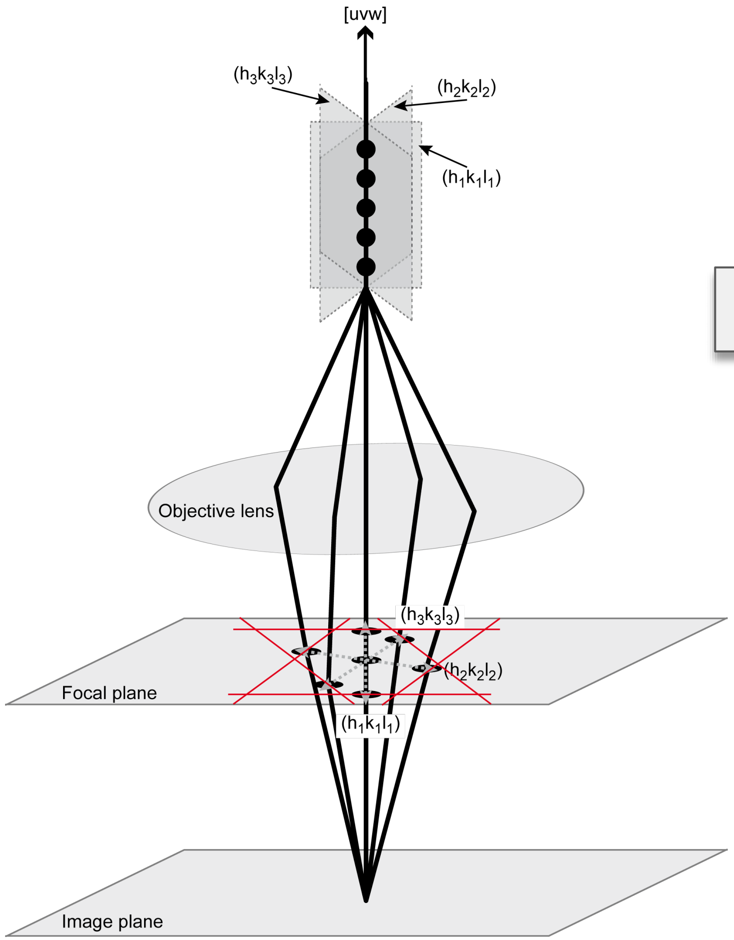




↳ Bright contrasts on a dark background

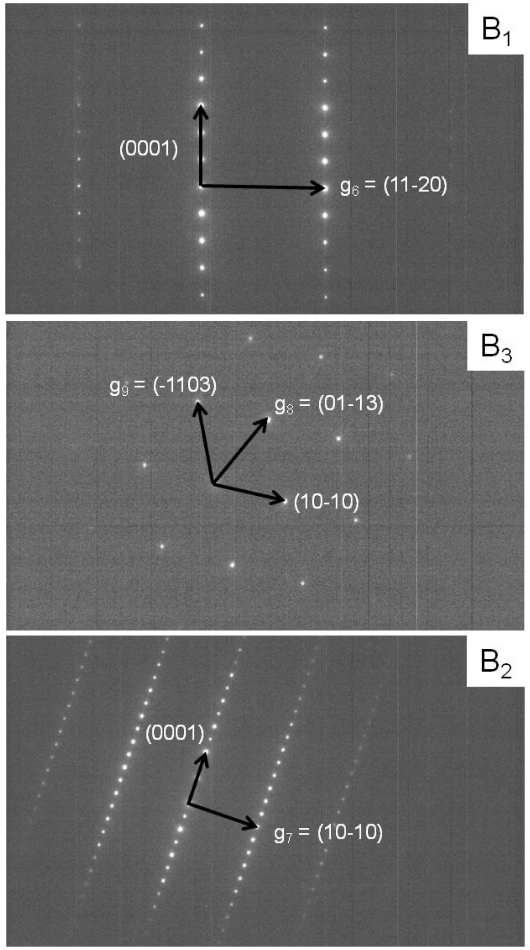
Basics of TEM contrast

Diffraction mode



Spot pattern

Information on the crystallographic structure



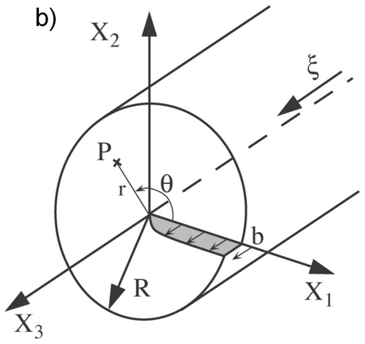
Beams are not convergent in the reality…

A. Guitton, *PhD Thesis* (2013)

Elastic theory of dislocations

- Burgers vector: $\boldsymbol{b}$
- Dislocation line direction: $\boldsymbol{\xi}$
  - Screw dislocation ($\boldsymbol{b} \parallel \boldsymbol{\xi}$)

$$\boldsymbol{R_S} = \frac{b\theta}{2\pi}$$



P.M. Anderson, J.P. Hirth, & J. Lothe, *Cambridge University Press* (2017)

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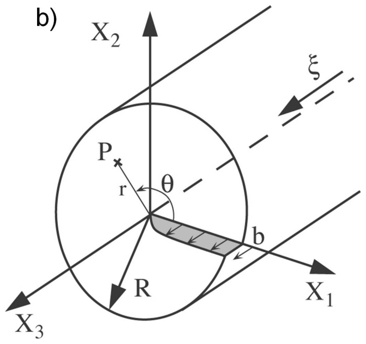
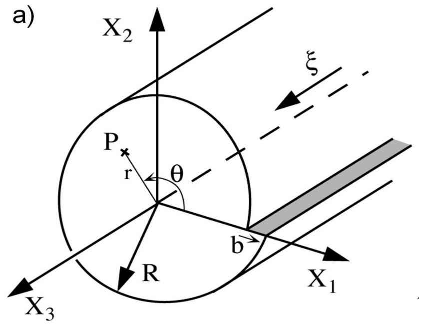


  - Edge dislocation ($\boldsymbol{b} \perp \boldsymbol{\xi}$)

$$\boldsymbol{R_e} = \underbrace{\frac{\boldsymbol{b}\theta}{2\pi}\left[\theta + \frac{\sin 2\theta}{2(1-\nu)}\right]}_{\parallel b} + \underbrace{\frac{\boldsymbol{b}\times\boldsymbol{\xi}}{2\pi}\left[\frac{1-2\nu}{2(1-\nu)}\ln r + \frac{\cos 2\theta}{4(1-\nu)}\right]}_{\perp b}$$



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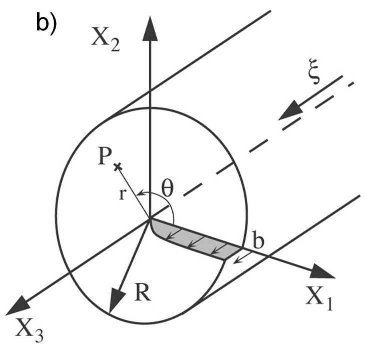

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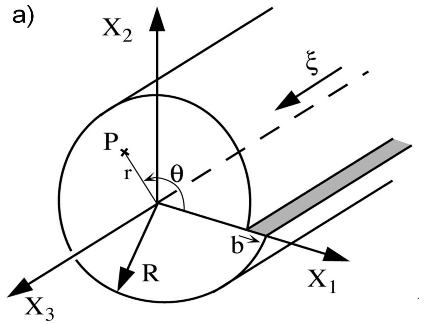

  - Mixed dislocation ($\boldsymbol{b} \angle \boldsymbol{\xi}$)

Combination of $\boldsymbol{R_S}$ and $\boldsymbol{R_e}$

P.M. Anderson, J.P. Hirth, & J. Lothe, *Cambridge University Press* (2017)

Contrast of unperfect crystal

- Perfect crystal
  - A crystal is composed of several unit cells located at positions $\boldsymbol{R_m}$, a Bravais vector
  - The atoms inside each unit cell are positioned at $\boldsymbol{r_j}$

$$\boldsymbol{r_{jm}} = \boldsymbol{R_m} + \boldsymbol{r_j}$$

$$\boldsymbol{R_m} = u\boldsymbol{a_1} + v\boldsymbol{a_2} + w\boldsymbol{a_3}$$

  - The total diffracted wave:

$$\psi'(\boldsymbol{r}, t) \propto \sum_j f_j \exp[2\pi i \boldsymbol{K} \cdot \boldsymbol{r_j}] \sum_m \exp[2\pi i \boldsymbol{K} \cdot \boldsymbol{R_m}]$$

  - With diffraction conditions ($\boldsymbol{K} = \boldsymbol{G}$):

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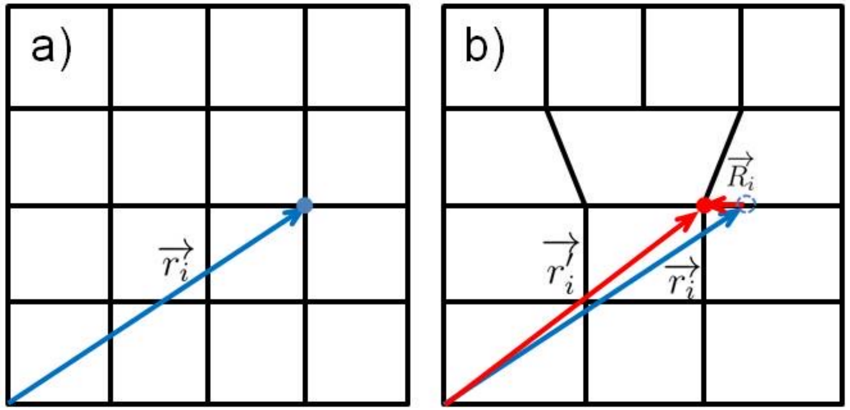

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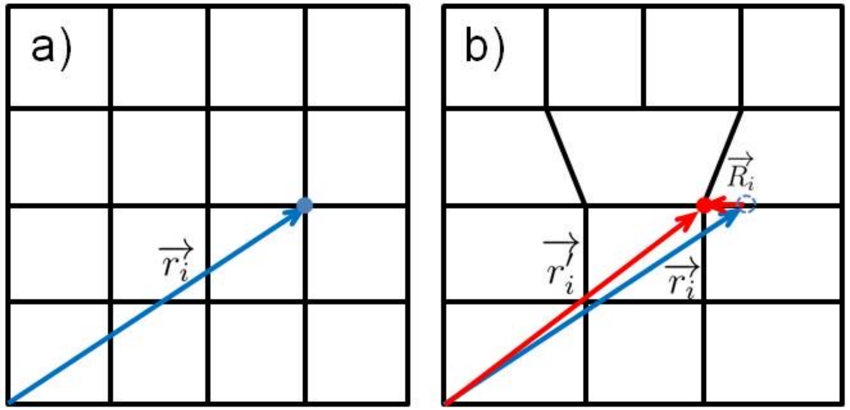

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  - With diffraction conditions ($\boldsymbol{K} = \boldsymbol{G}$):

$$\psi'(\boldsymbol{r}, t) \propto \sum_j f_j \exp[2\pi i \boldsymbol{G} \cdot \boldsymbol{r_j}] \sum_m \exp[2\pi i \boldsymbol{G} \cdot \boldsymbol{R_m}]$$

- Unperfect crystal
  - The atoms inside each unit cell are now positioned at $\boldsymbol{r_j} + \boldsymbol{R_j}$

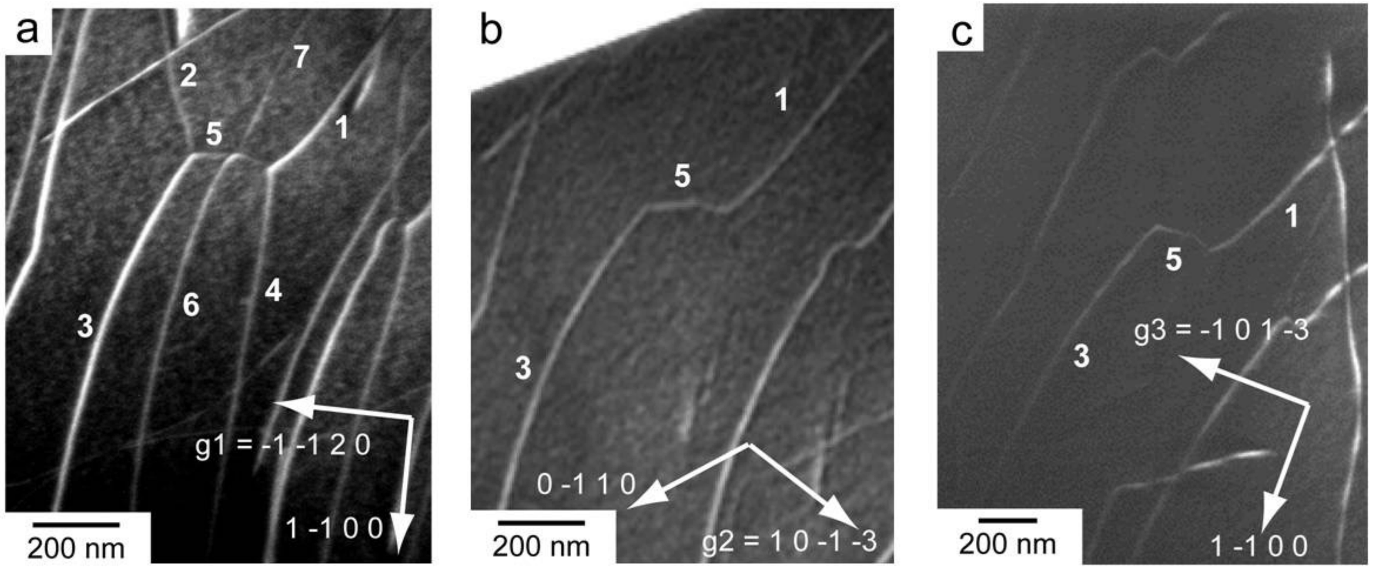
$$\psi'_d(\boldsymbol{r}, t) \propto \sum_j f_j \exp[2\pi i \boldsymbol{G} \cdot (\boldsymbol{r_j} + \boldsymbol{R_j})] = \sum_j f_j \exp[2\pi i \boldsymbol{G} \cdot \boldsymbol{r_j}] \times \sum_j f_j \exp[2\pi i \boldsymbol{G} \cdot \boldsymbol{R_j}] = \sum_j f_j \exp[2\pi i \boldsymbol{G} \cdot \boldsymbol{R_j}]$$

($\boldsymbol{G} \cdot \boldsymbol{r_j} = 2\pi$ by definition of $\boldsymbol{G}$)

➪ $\sum_j f_j \exp[2\pi i \boldsymbol{G} \cdot \boldsymbol{R_j}]$ characterizes the defects

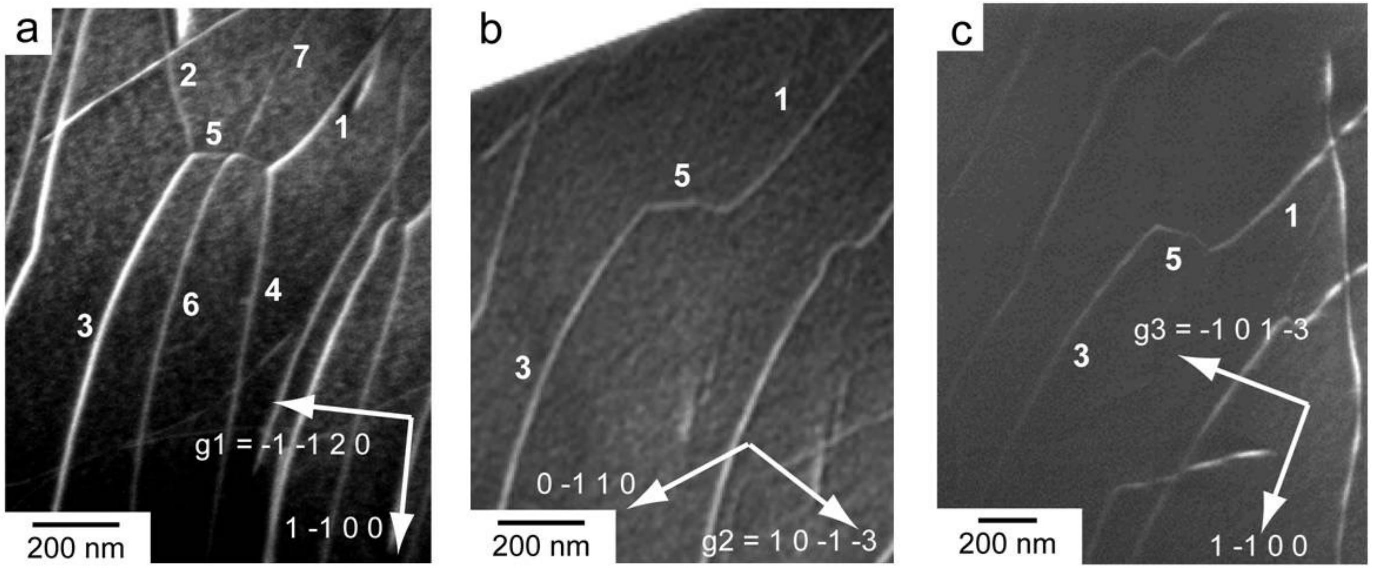
➪ If $\boldsymbol{G} \cdot \boldsymbol{R_j} = 0$ the defect will be out of contrast

Example of extinction condition



A. Guitton et al., *Philocs Mag.*(2012)

Example of extinction condition

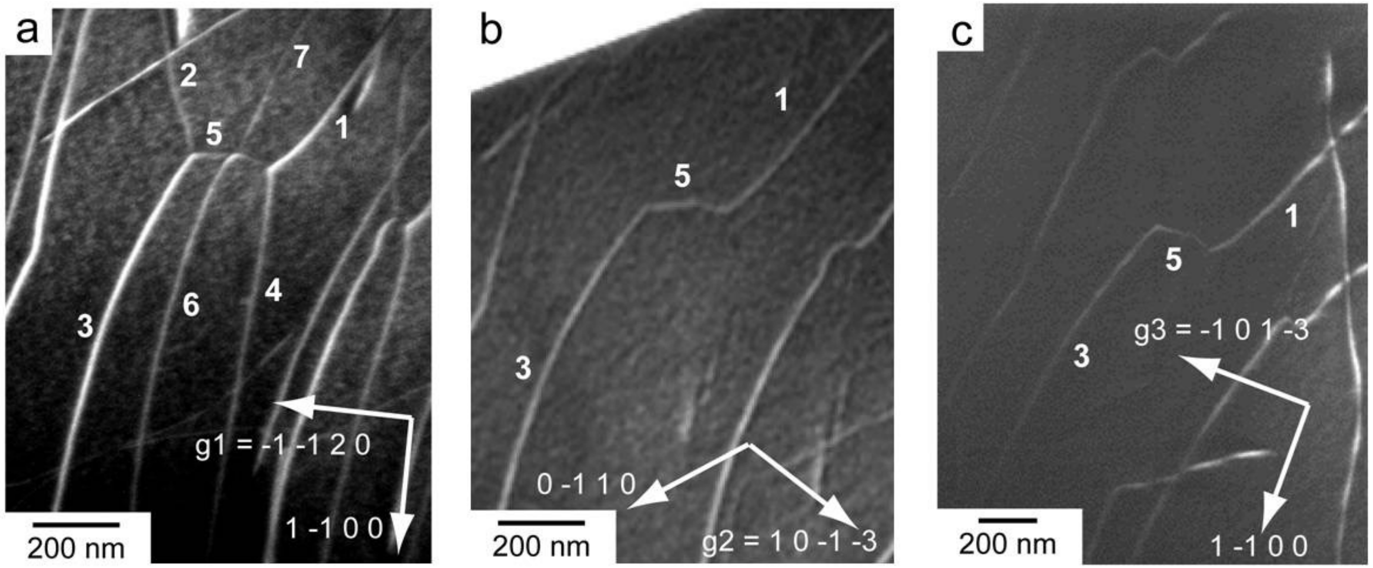


❖ The dislocation 2 is obviously out of contrast for both $g_2 = (1\,0\,\bar{1}\,\bar{3})$ and $g_3 = (\bar{1}\,0\,1\,\bar{3})$

$$\begin{cases} g_2 \cdot b_2 = 0 \\ g_3 \cdot b_2 = 0 \end{cases} \Rightarrow \begin{cases} (1\,0\,\bar{1}\,\bar{3}) \cdot [h\;k\;i\;l] = 0 \\ (\bar{1}\,0\,1\,\bar{3}) \cdot [h\;k\;i\;l] = 0 \end{cases} \Rightarrow \begin{cases} h - i - 3l = 0 \\ -h + i - 3l = 0 \\ -(h+k) = i \end{cases}$$

A. Guitton et al., *Philocs Mag.*(2012)

Example of extinction condition



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$$\boldsymbol{b_2} = \pm\, a/3[\bar{1}\,2\,\bar{1}\,0]$$

A. Guitton et al., *Philocs Mag.*(2012)

The 2 types of electron microscopes

Transmission electron microscope (TEM)

- Invention: 1931 by Ernst RUSKA et Max KNOL
- Specimen : thin foils (~ 100 nm)
- Price: ~ 1 M€
- Rare in industry

↳Nanometric/atomic scale

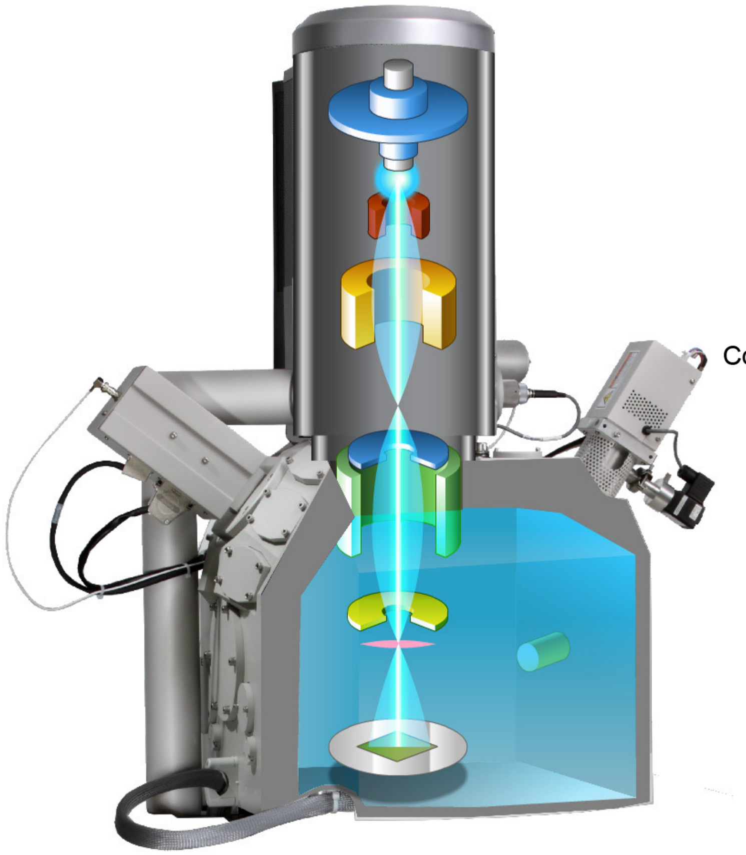
Scanning electron microscope (SEM)

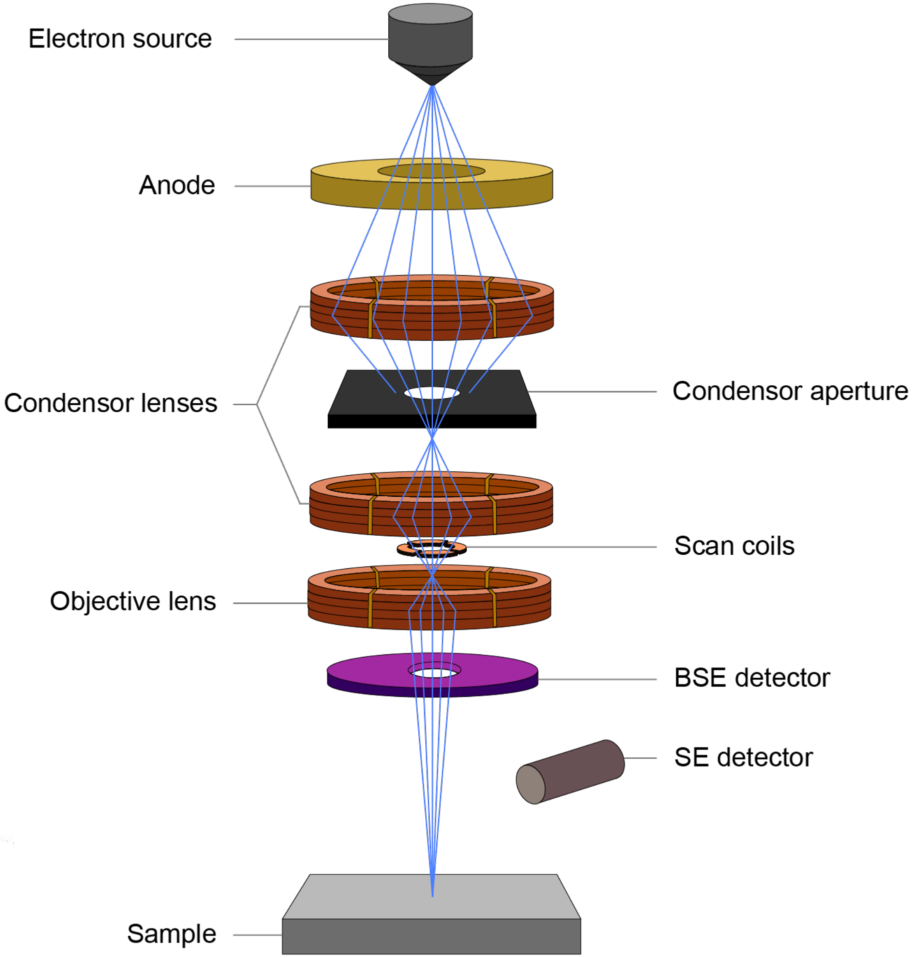
- Invention: 1937 by Manfred VON ARDENNE but developed in the 1960s
- Specimen : bulk
- Price: ~ 500 k€
- Comon in industry

↳Meso-/micro-/nano-metric scale

↳ **The two microscopes are complementary.**

Basics of SEM

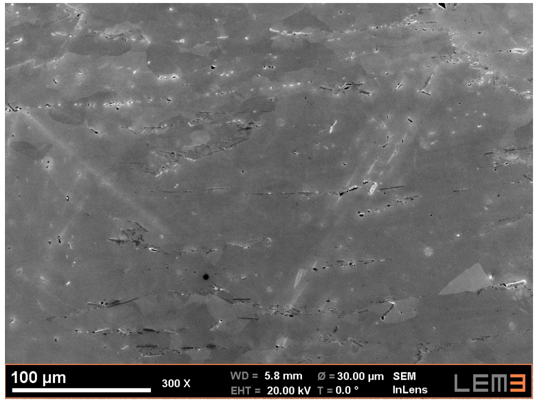




J. Aubourg, *PhD Thesis (2024)*

SEM imaging

Secondary electrons (SE)



- Inelastic scattering between incident electrons and outer-shell electrons of sample atoms ⇒ emission of SE
- Inelastic scattering involving inner-shell electrons, followed by electronic transitions → emission of characteristic X-rays

  ⇒ EDS

↳ Topographic contrast

SEM imaging

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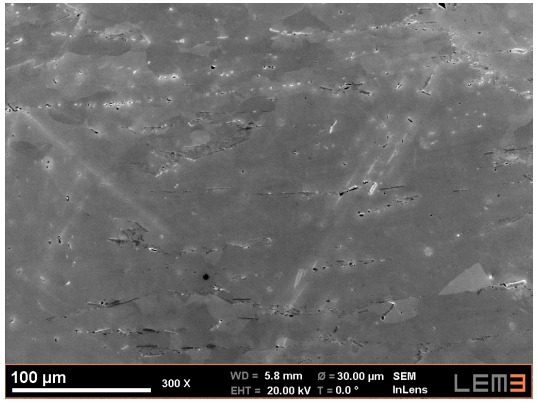

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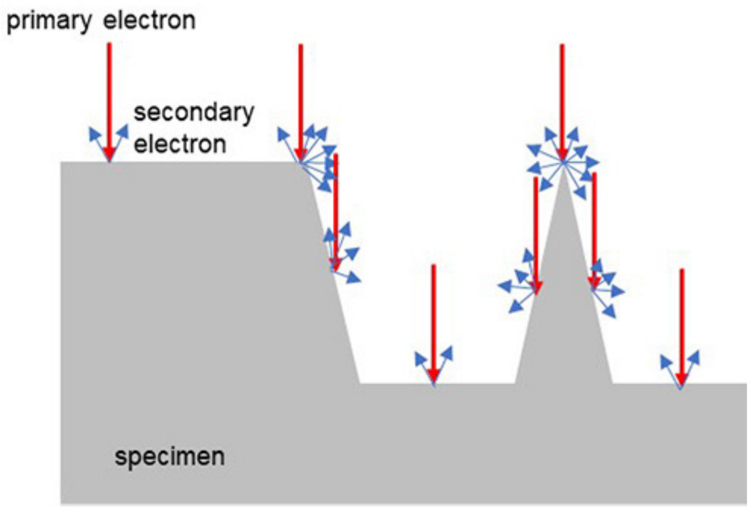
↳Topographic contrast

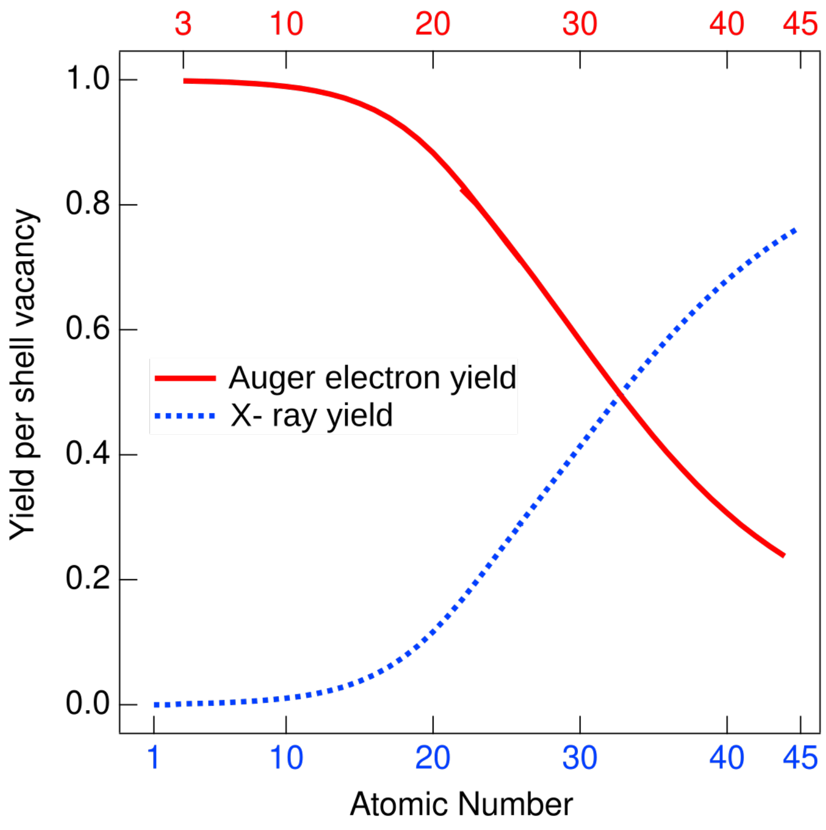
The edge effect

❖ Areas at edges and corners appear very bright in SE images due to increased SE emission.



↳SEM is not 3D, but it gives a strong 3D impression.

Energy Dispersive X-ray Spectroscopy (EDS)



❖ **Inner-shell ionization:**

- When the high-energy electron beam hits the sample, it can eject inner-shell electrons (usually from K or L shells) in the atoms.

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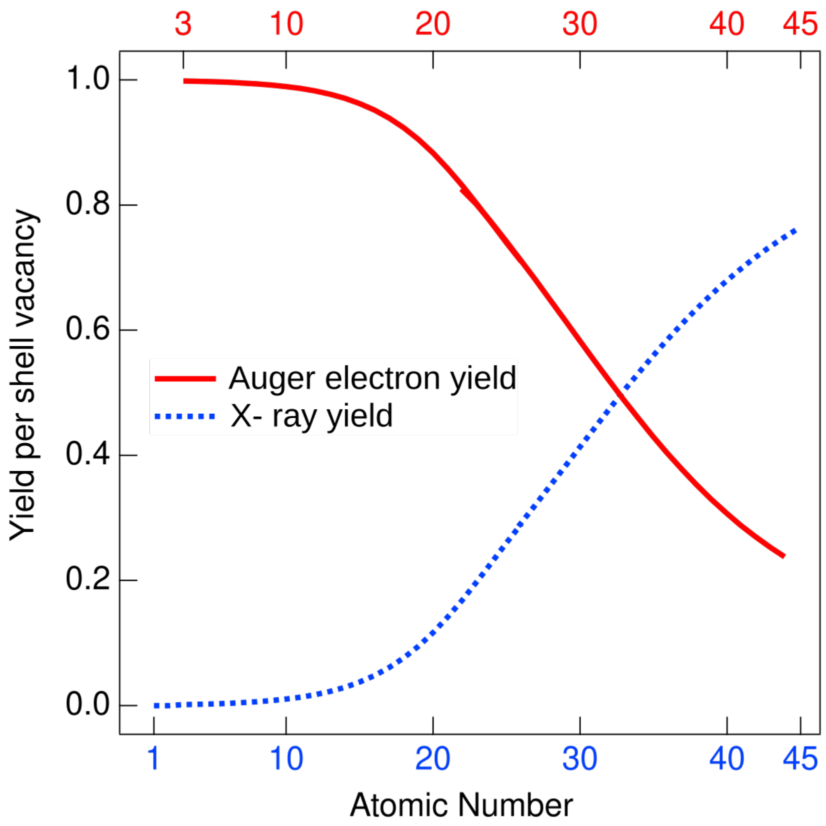

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- An electron from a higher-energy shell falls down to fill the vacancy. This transition releases energy in the form of an X-ray photon.

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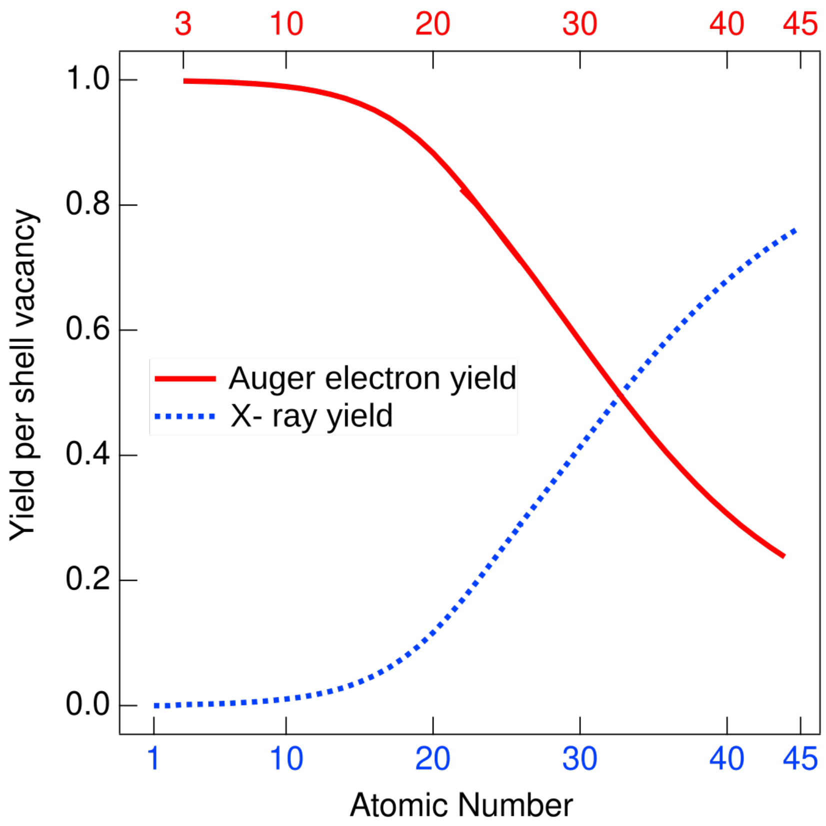

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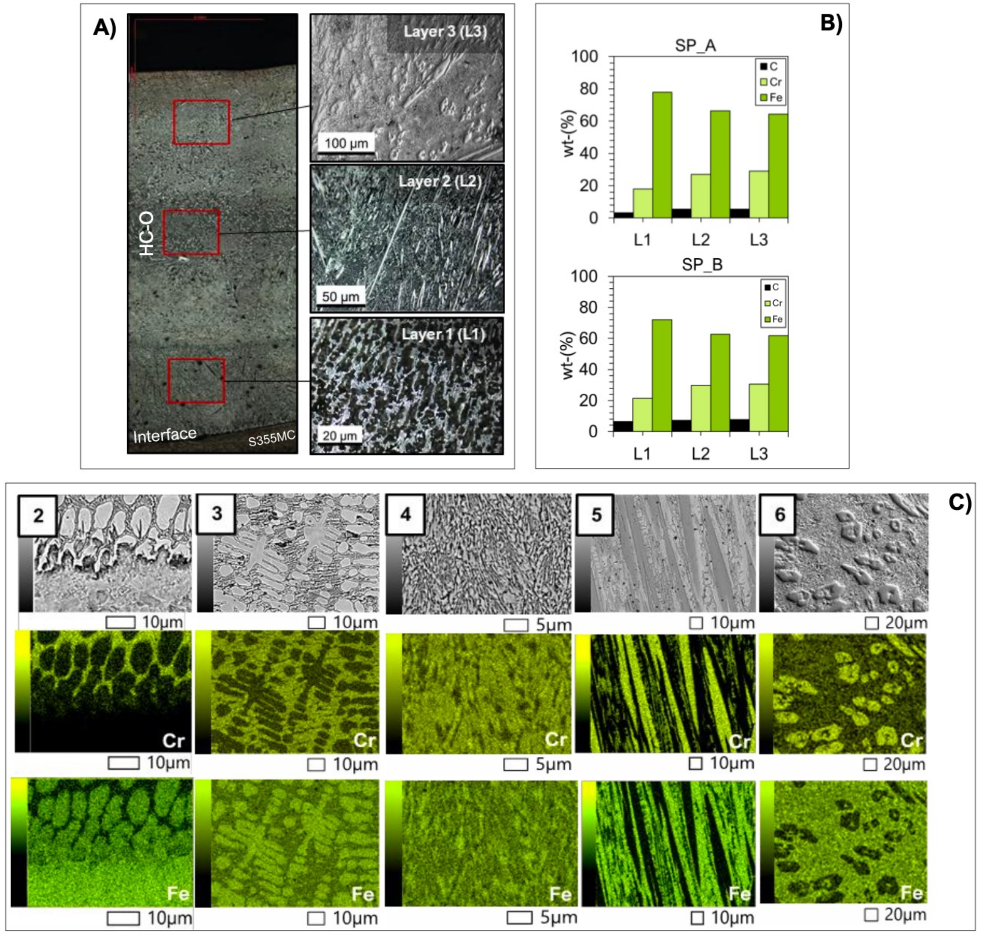

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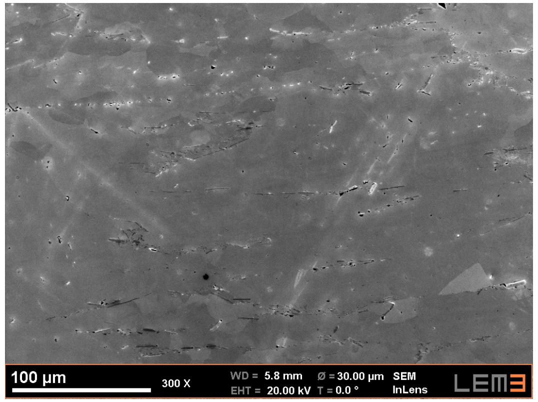
- **Detection:**
  - An EDS detector measures the energy of these X-rays, allowing us to:
    - ✓ Identify the elements present (qualitative analysis)
    - ✓ Estimate their relative amounts (semi-quantitative analysis but not accurate for low Z elements)

A. Monnet et al., *Mater. Charac.* (2026)

SEM imaging

Secondary electrons (SE)



- Inelastic scattering between incident electrons and outer-shell electrons of sample atoms ⇒ emission of SE
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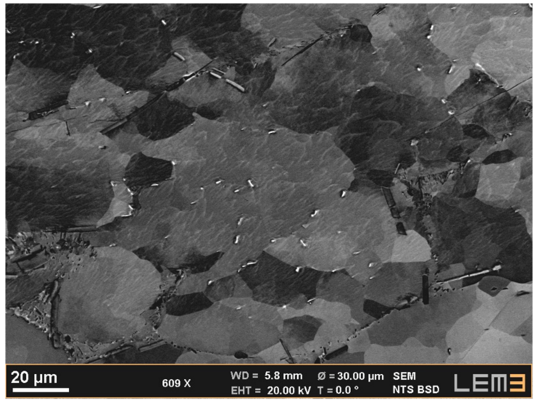
  ⇒ EDS

↳Topographic contrast

BackScattered Electrons (BSE)



- Elastic scattering: the incident electron is deflected by the electrostatic field of the atomic nucleus, without significant energy loss.

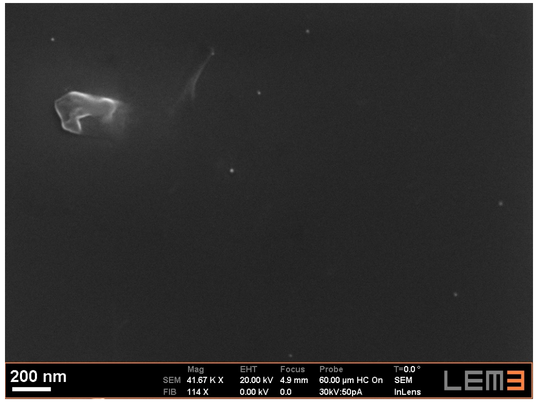
  ⇒ EBSD, ECCI

↳Topographic contrast
↳Chemistry contrast
↳Orientation contrast

↳ The two imaging modes are complementary.

SEM imaging

Secondary electrons (SE)



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Fate of incident electrons in SEM

- **X-rays:**
  - ~0.1–1% of incident electrons generate X-rays

- **Secondary electrons (SE):**
  - ~5–40%
- **Backscattered electrons (BSE):**
  - ~10–50%
  - The BSE cross section increases with Z

- **Other energy losses (heat, phonons, electronic excitations)**

D.C. Joy et al, *J. App. Phys.*. (1982)

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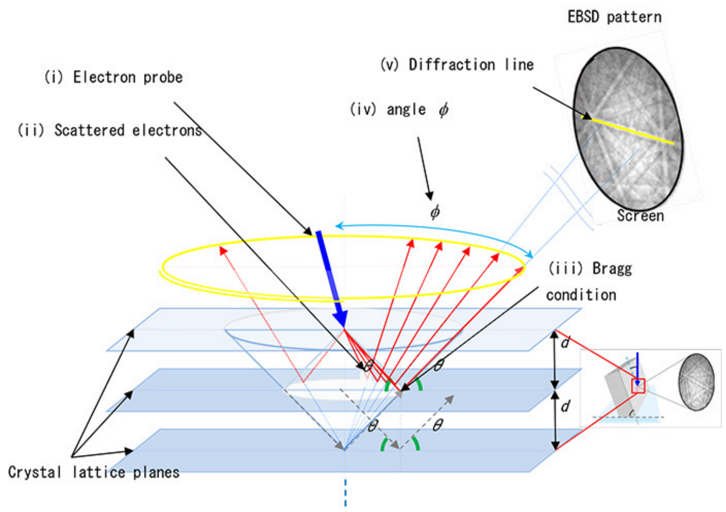
- **Other energy losses (heat, phonons, electronic excitations)**

- **Relative contributions within the BSE signal**
  - Orientation contrast (electron channeling): ~2–5% of the BSE contrast
  - Atomic number contrast (Z-contrast)
  - ~10–30% of the BSE contrast
  - Topographic contrast: ~65–90% of the BSE contrast

D.C. Joy et al, *J. App. Phys.*. (1982)

Basics of the Electron BackScattered Diffraction (EBSD)



❖ Generation of Kikuchi patterns:

- The incident electron beam interacts with the crystal lattice, generating BSE through inelastic and elastic scattering.
- Some of these BSE undergo coherent elastic diffraction by the crystal planes, fulfilling the Bragg condition.
- This diffraction produces Kikuchi bands, which appear as pairs of parallel lines corresponding to specific crystallographic planes.

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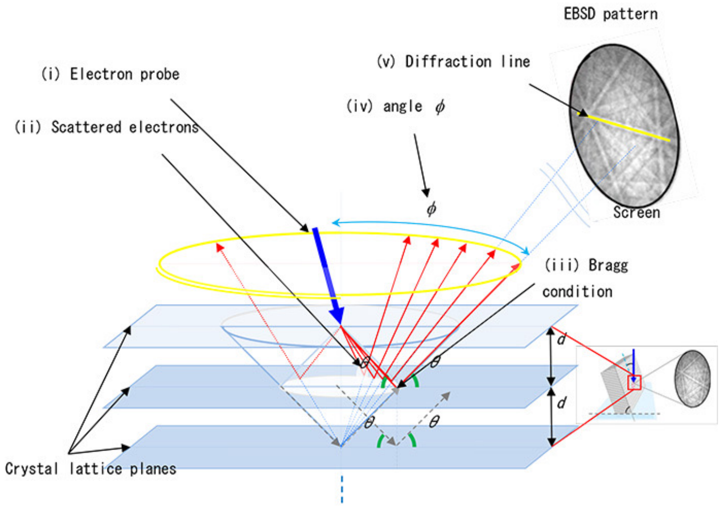

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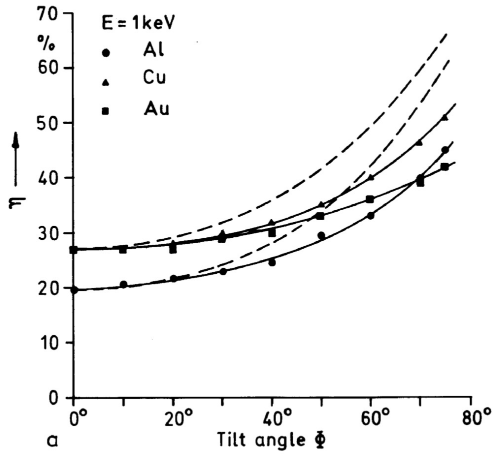
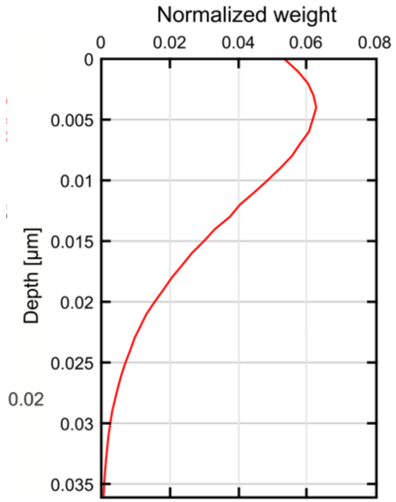
↳The BSE yield increases with increasing tilt angle

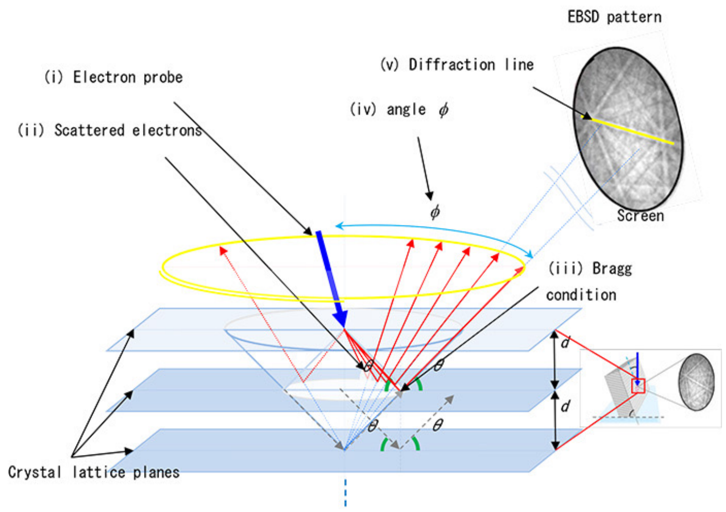


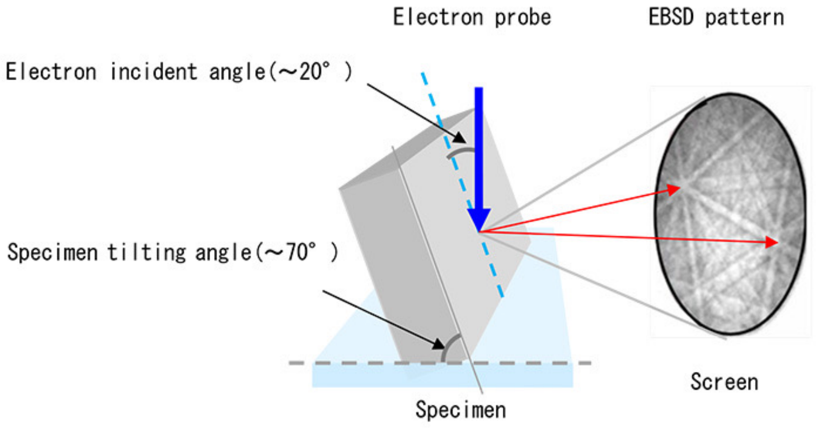


↳The probing depth in GaN at 15 keV is ~35 nm below the surface.

www.jeol.co.jp
L. Reimer, *Springer* (1998); C. Ernould et al., *Mater. Charact.* (2022)

Basics of the Electron BackScattered Diffraction (EBSD)





❖ Generation of Kikuchi patterns:

- The incident electron beam interacts with the crystal lattice, generating BSE through inelastic and elastic scattering.
- Some of these BSE undergo coherent elastic diffraction by the crystal planes, fulfilling the Bragg condition.
- This diffraction produces Kikuchi bands, which appear as pairs of parallel lines corresponding to specific crystallographic planes.

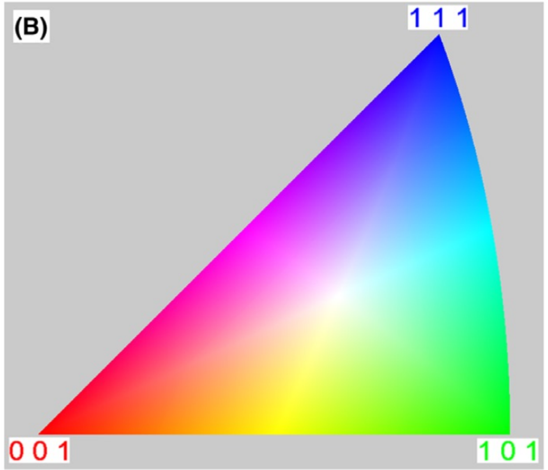
❖ Basics of the EBSD:

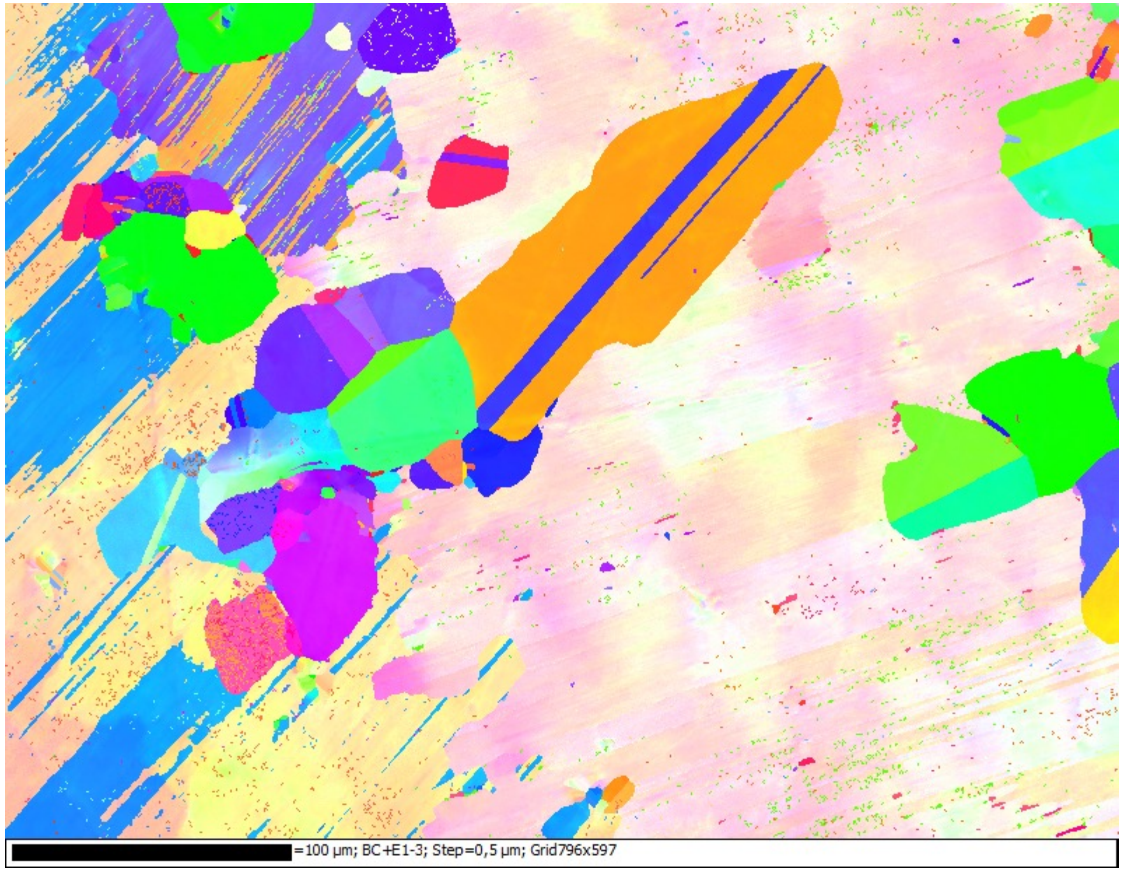
- EBSD involves directing a focused electron beam onto a tilted crystalline specimen
- The BSE form Kikuchi patterns, which are captured by a phosphor screen and recorded using a camera.
- These patterns are analyzed to extract information about the material's grain structure, orientation, and phase.

www.jeol.co.jp

Example of extinction condition

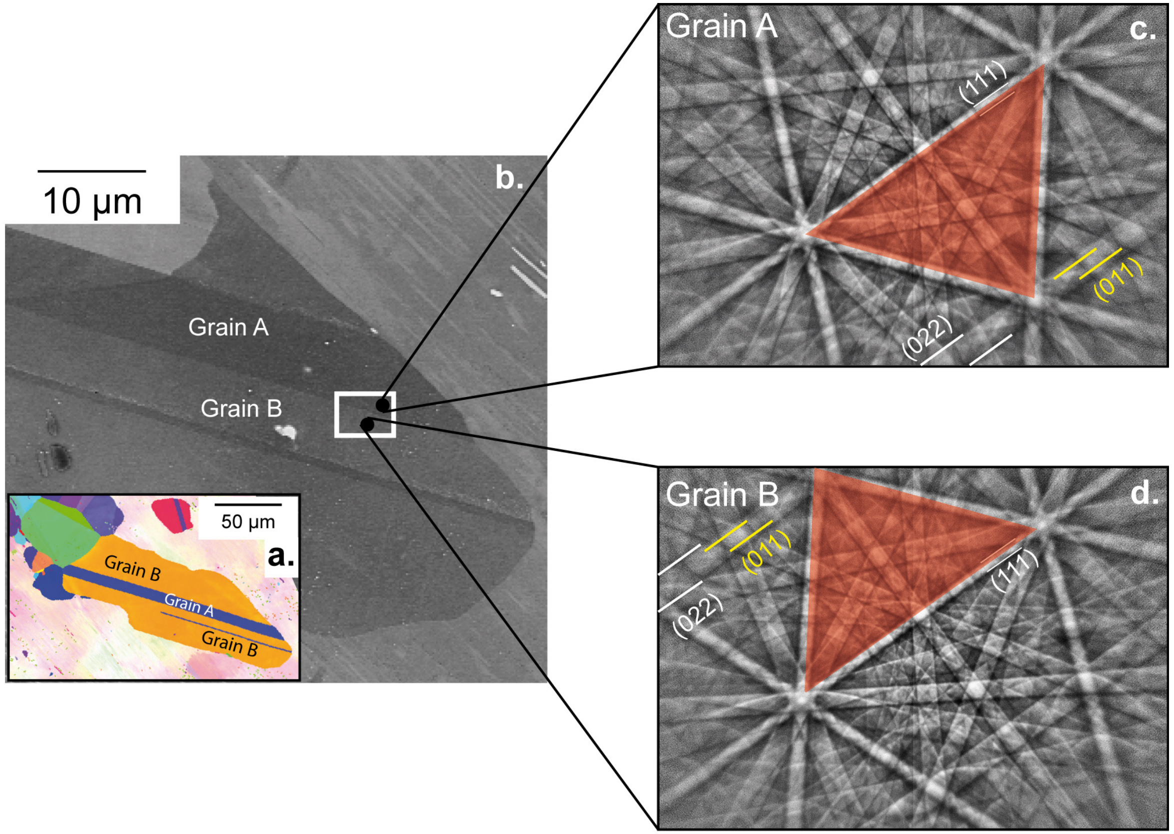
- **Capabilities of EBSD:**
  - Orientation measurements
  - Local misorientation measurements
  - Grain identification
  - Phase identification
  - Statistics on the microstructure





A. Guitton et al., *Materials (2018)*

What is the nature of the boundary?



A. Guitton et al., *Materials (2018)*

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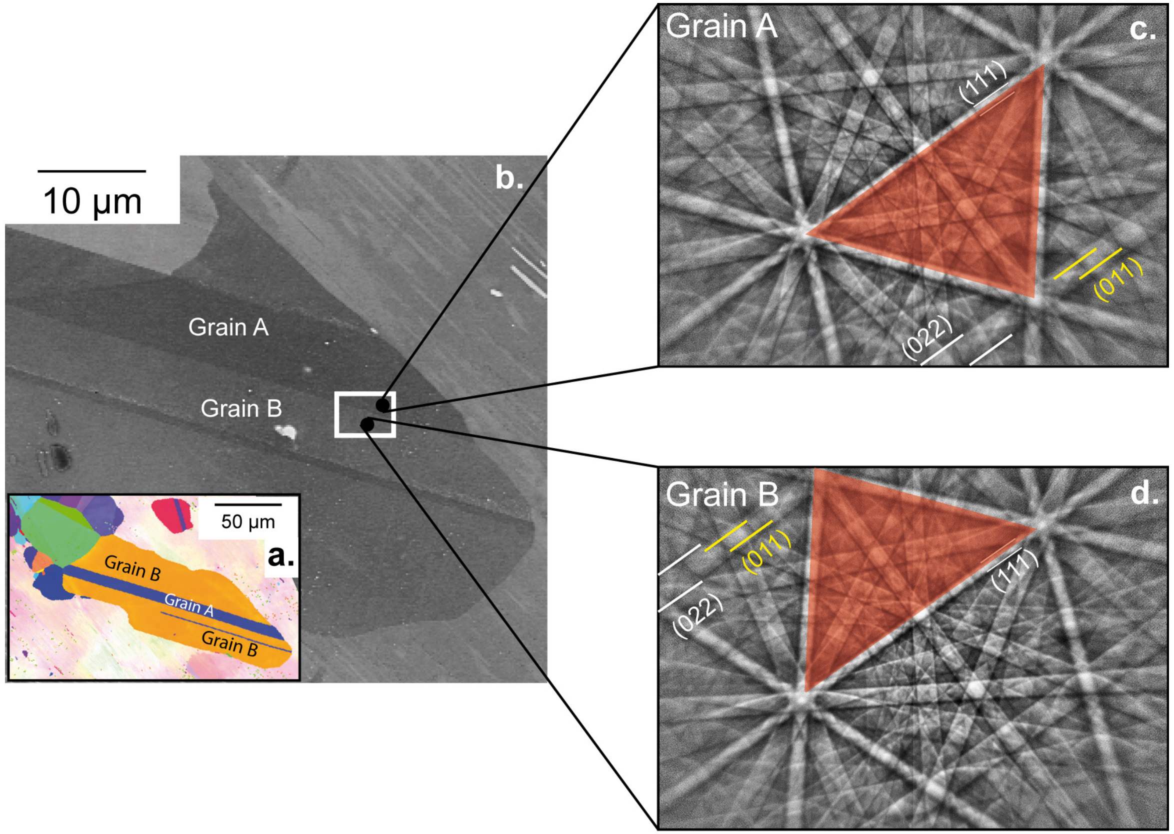
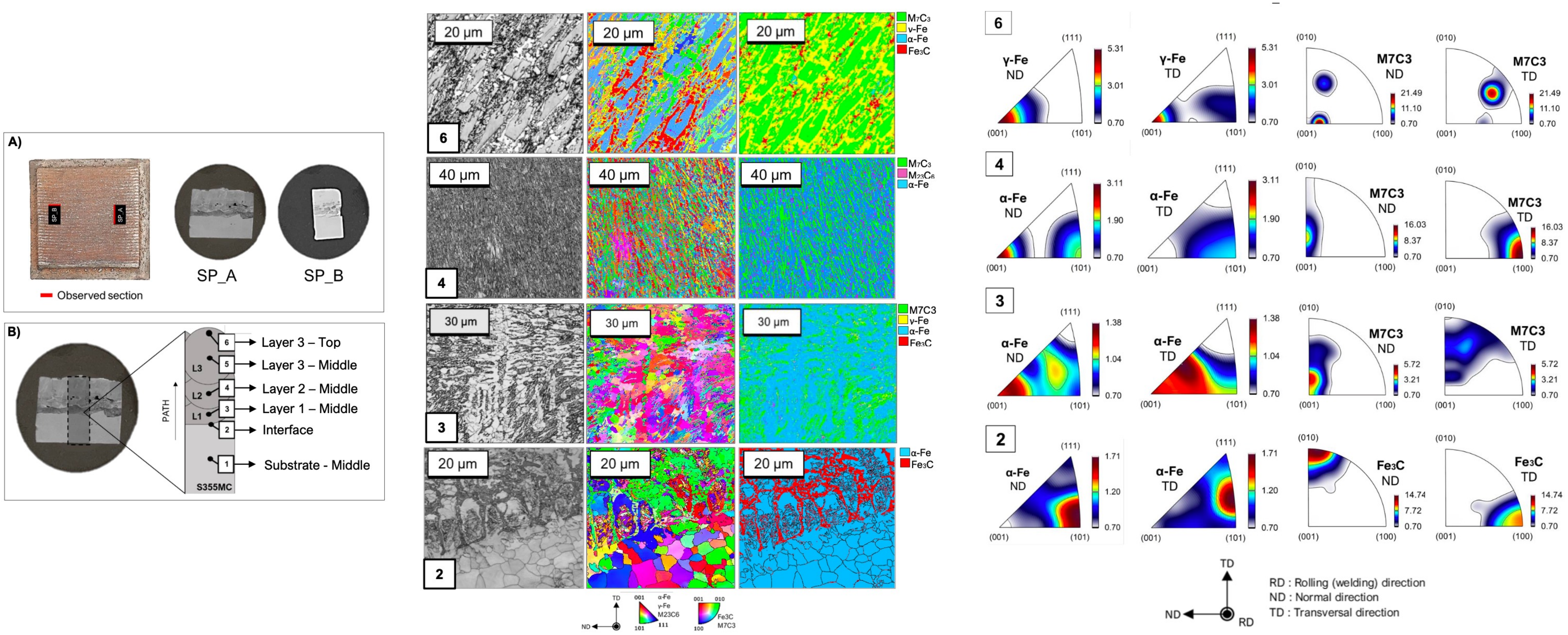


This is a (1 1 1) twin boundary.

A. Guitton et al., *Materials (2018)*

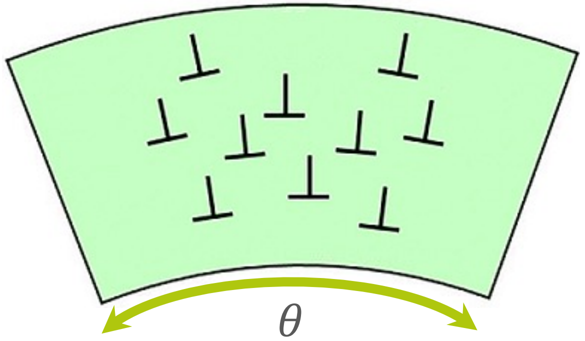
Texture measurements by EBSD



A. Monnet et al., *Mater. Charac.* (2026)

Different types of dislocation densities

Geometrically necessary dislocations (GND)



⇒ Lattice curvature at long range

$$\rho_{\mathrm{GND}} \approx \frac{1}{\|\boldsymbol{b}\|}\|\boldsymbol{\alpha}\|; \|\boldsymbol{\alpha}\| = \sqrt{\alpha_{ij}.\alpha_{ij}}$$

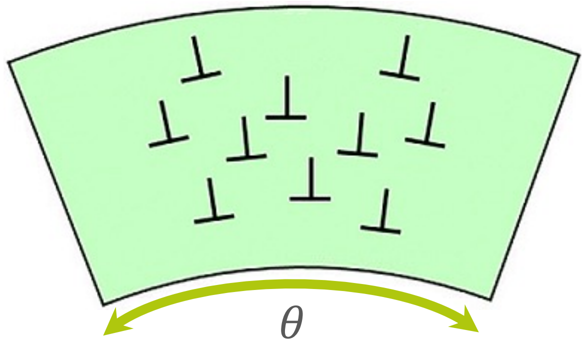
$$\boldsymbol{\alpha} = curl\,\boldsymbol{\varepsilon} + \mathrm{tr}(\boldsymbol{\kappa_e}) \cdot \mathbb{I} - \boldsymbol{\kappa_e}^T$$

$$\kappa_{ij} \cong \frac{\Delta\omega_i}{\Delta x_j};\ \varepsilon_{ij} = \frac{1}{2}\left(\frac{\partial u_i}{\partial x_j} + \frac{\partial u_j}{\partial x_i}\right)$$

A. Mandal et al., *Microsc. Microanal.* (i2025)

Different types of dislocation densities

Geometrically necessary dislocations (GND)



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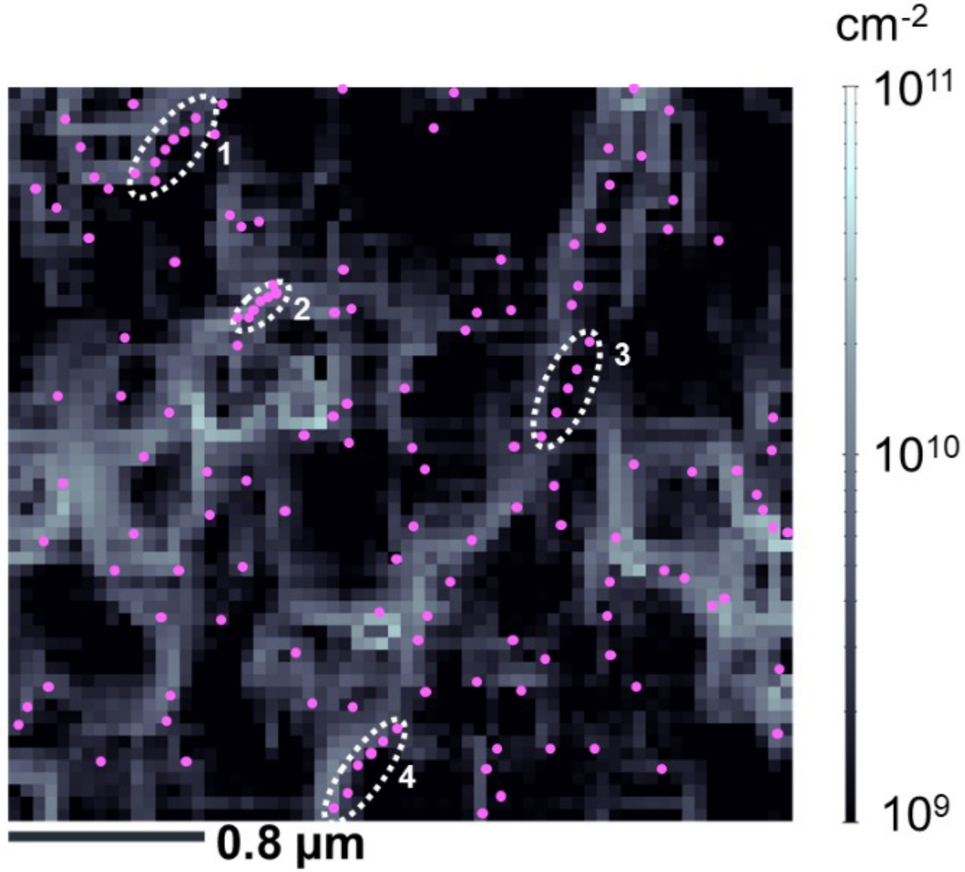
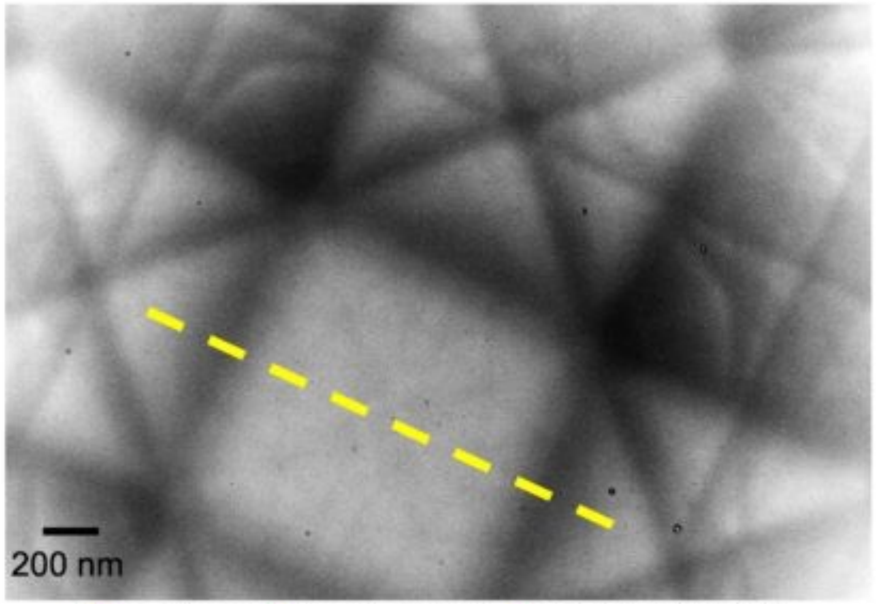
Norm of the Nye tensor (‖α‖)



A. Mandal et al., *Microsc. Microanal.* (i2025)

Origin of the Electron Channeling Pattern (ECP)

- Kikuchi-like bands are observed at low magnification on a bulk single crystal.



Electron Channeling Pattern "ECP" obtained from Si single crystal.

D. G. Coates, *Philos. Mag.* (1967); G.R. Booker et al., *Philos. Mag.* (1967)

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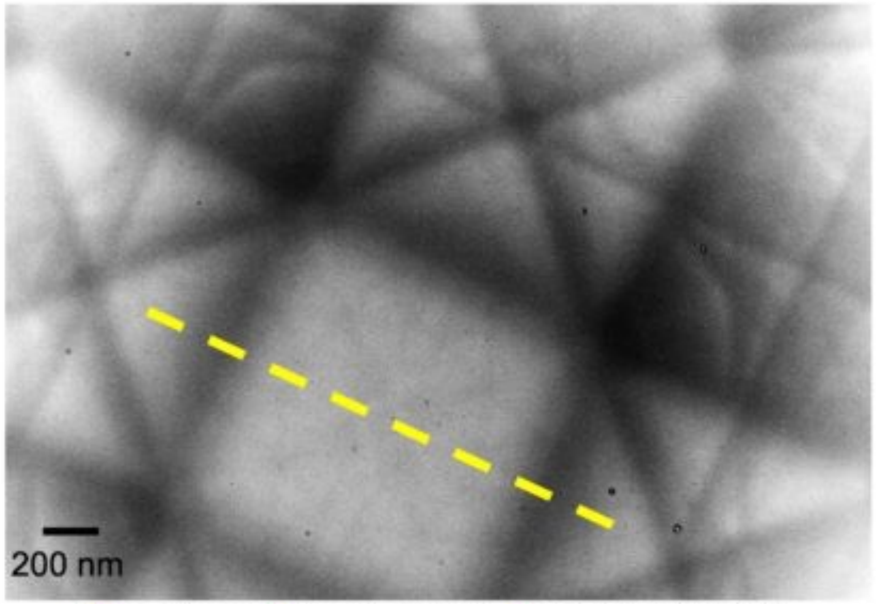
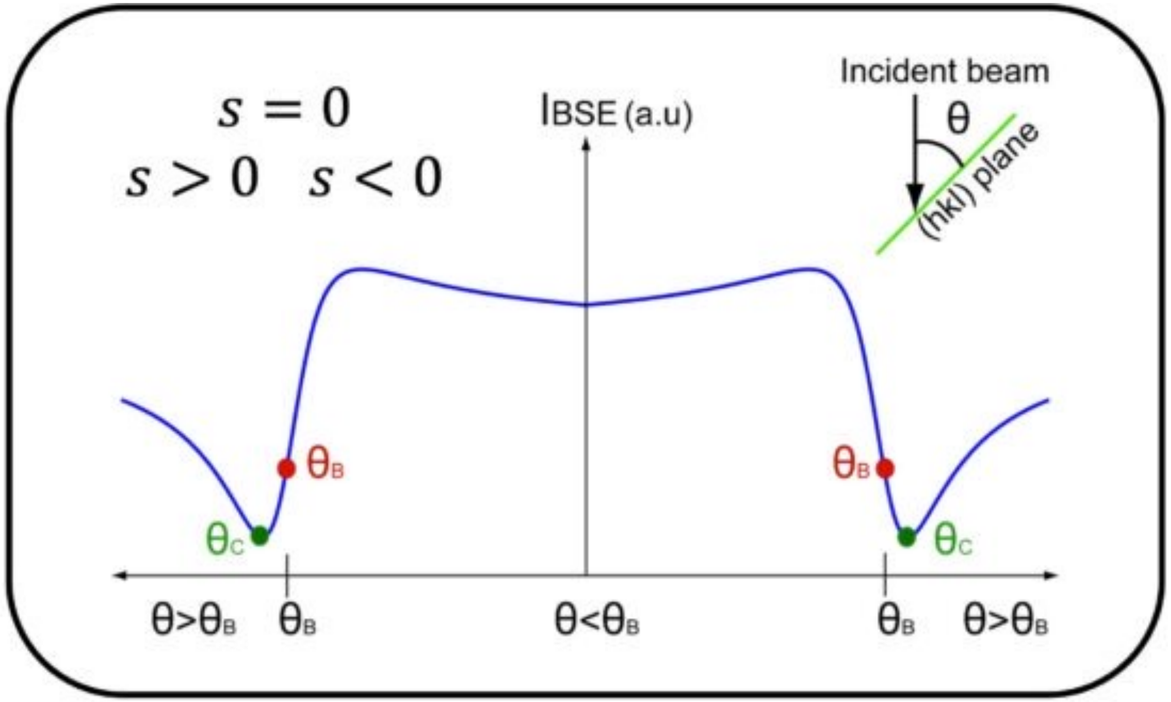


Electron Channeling Pattern "ECP" obtained from Si single crystal.

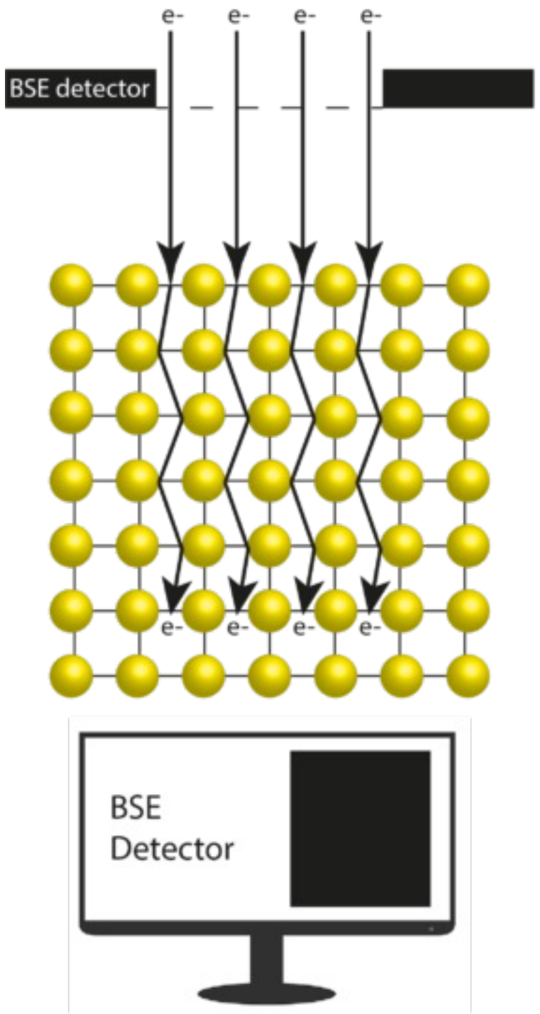
- BSE yield depends on the specimen orientation relative to the incident beam.



Channeling condition ≠ Bragg condition

D. G. Coates, *Philos. Mag.* (1967); G.R. Booker et al., *Philos. Mag.* (1967)

What is the Electron Channeling Contrast Imaging?



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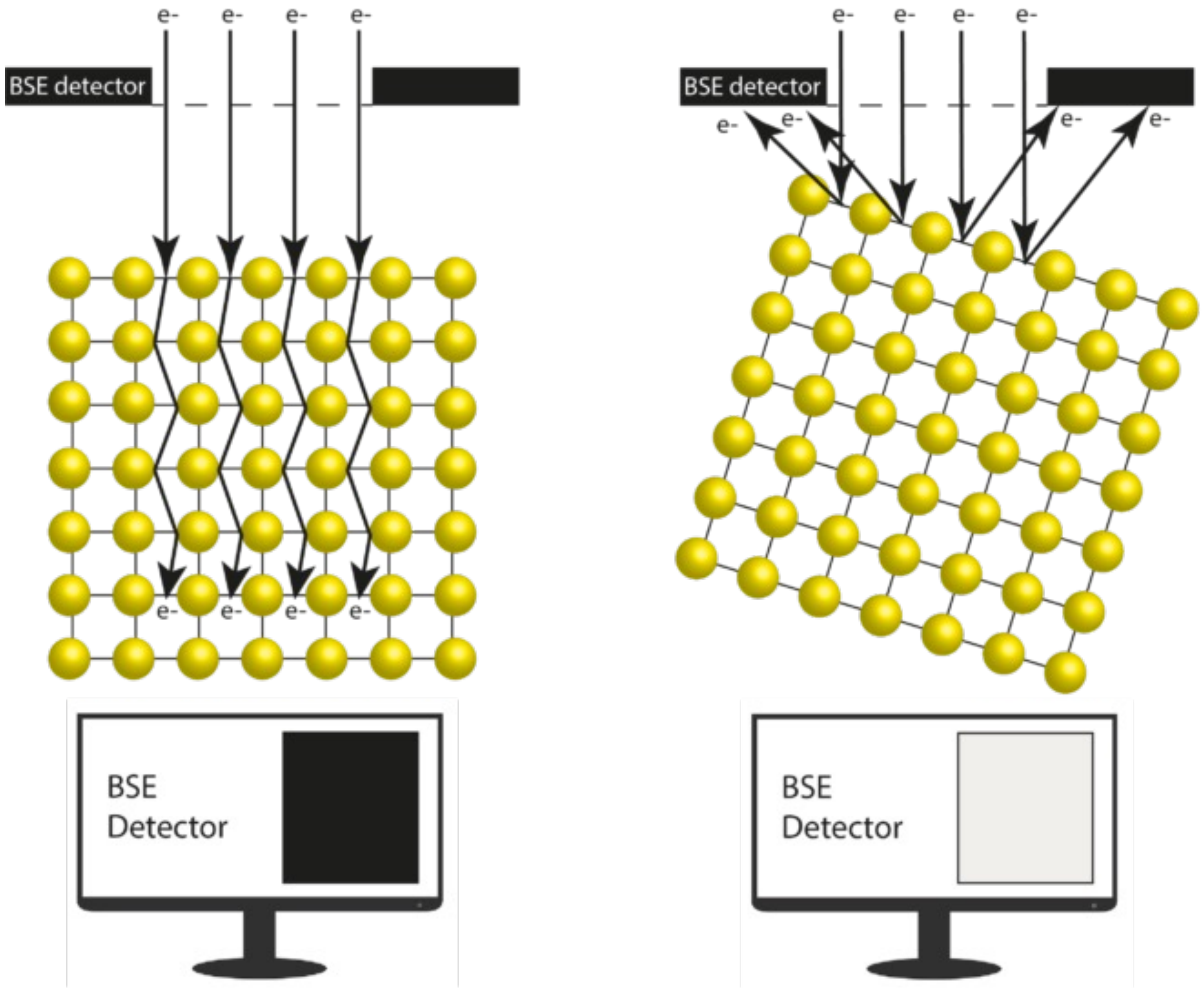
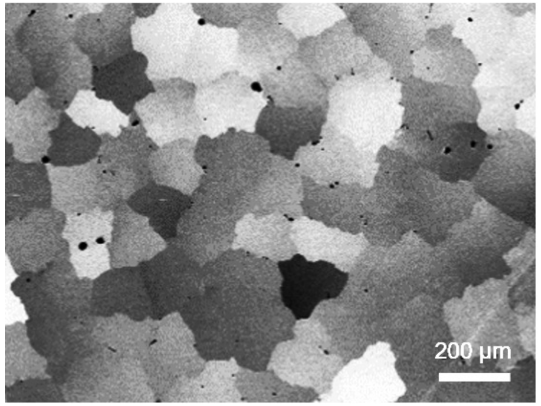


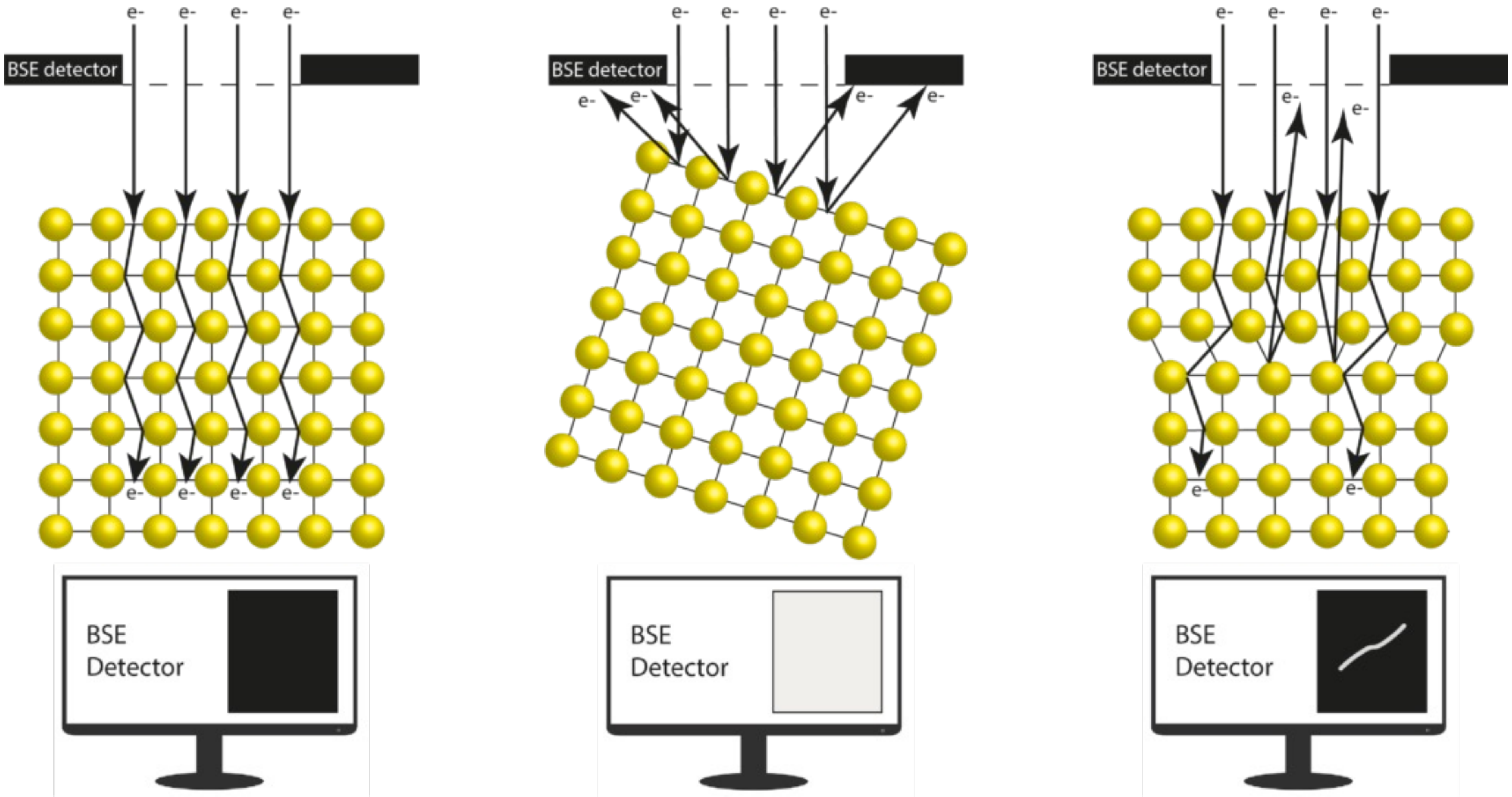
This is not a polycrystal!

F. Habiyaremye et al., *MSEA* (2021)



Single crystal of CrCoNi with dendrites
(mean misorientation: <1°)

What is the Electron Channeling Contrast Imaging?



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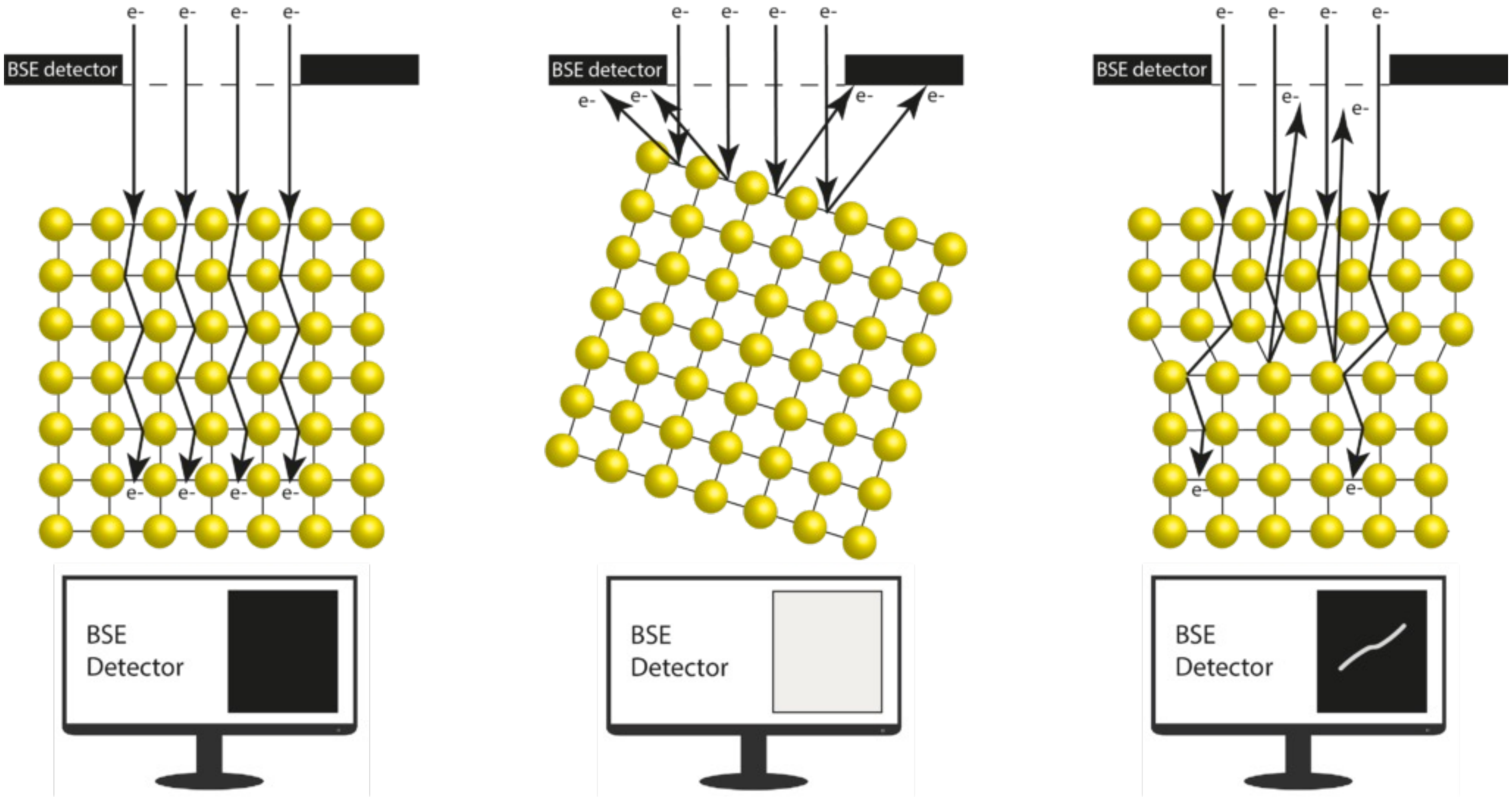
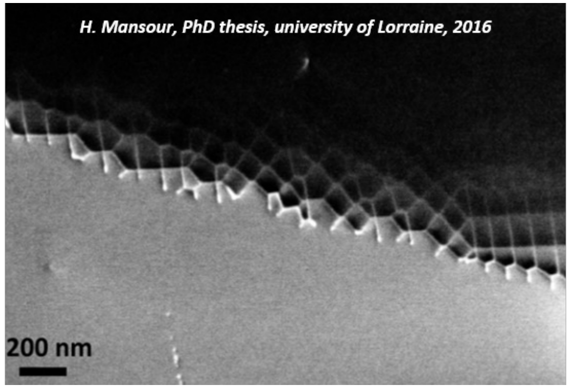


Dislocation $\Rightarrow \begin{cases} \boldsymbol{g}\cdot\boldsymbol{b} = 0 \\ \boldsymbol{g}\cdot\boldsymbol{b}\times\boldsymbol{\xi} = 0 \end{cases}$

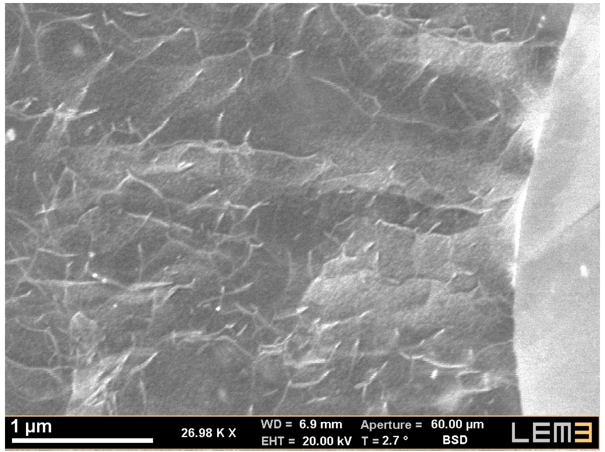
Stacking fault $\Rightarrow \boldsymbol{g}\cdot\boldsymbol{R} = 0$

Nature of the crystalline defects

Detailed characterization of crystalline defects

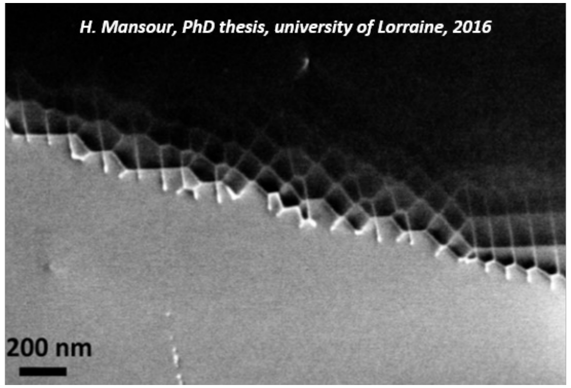


Ceramics (UO_2)

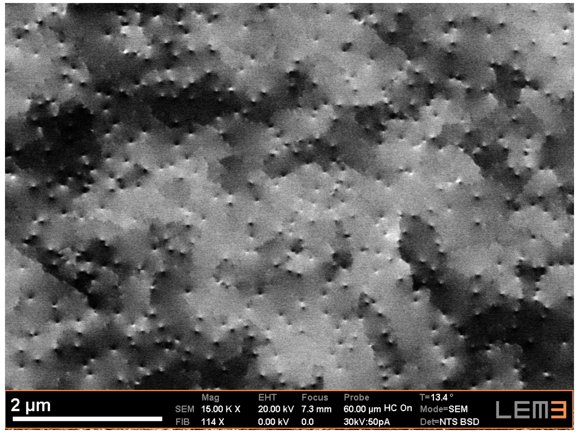


Ceramics (MAX phase)

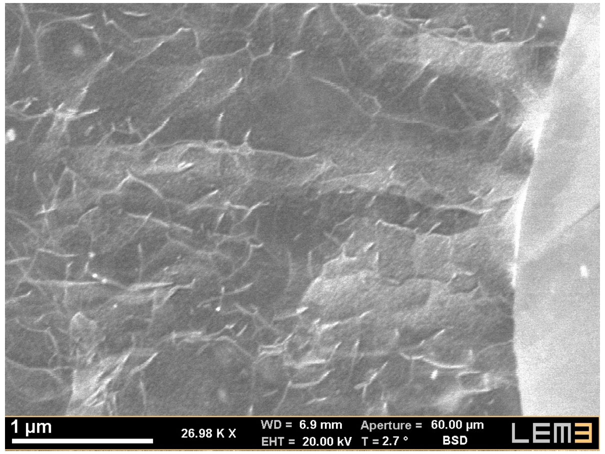
Detailed characterization of crystalline defects



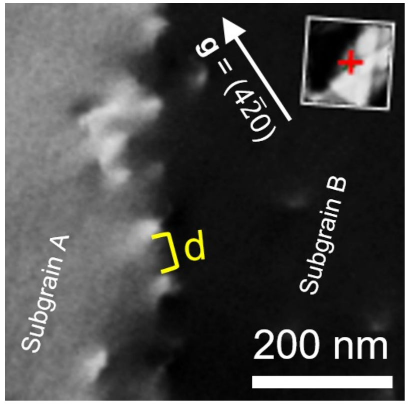
Ceramics (UO_2)



Semi-conductors (GaN)

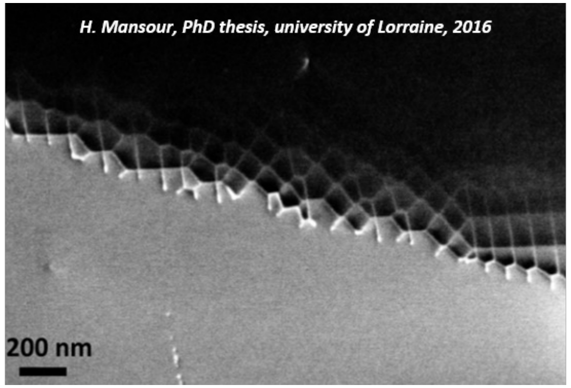


Ceramics (MAX phase)

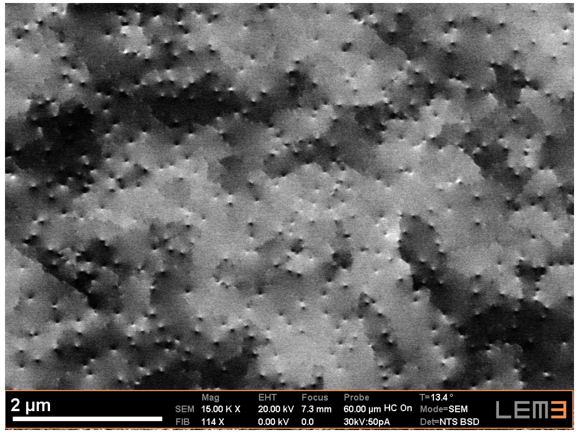


Metals (CrCoNi)

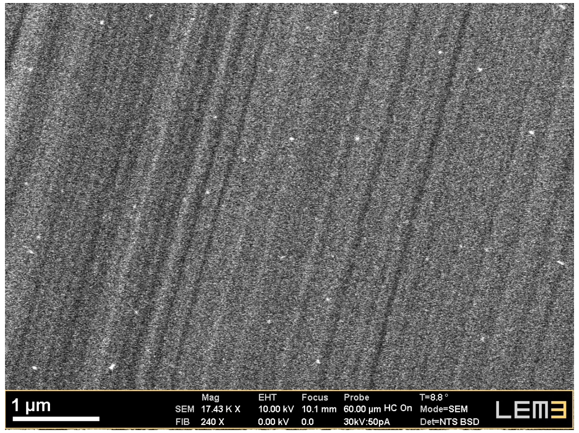
Detailed characterization of crystalline defects



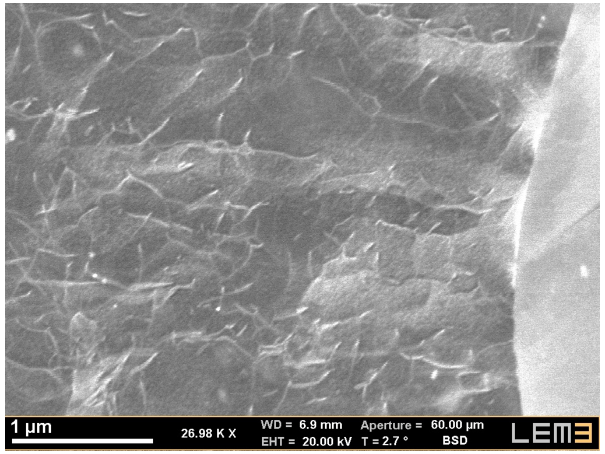
Ceramics (UO_2)



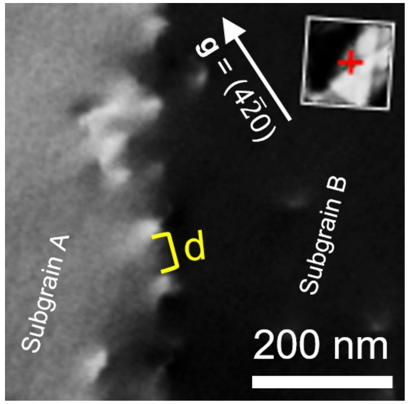
Semi-conductors (GaN)



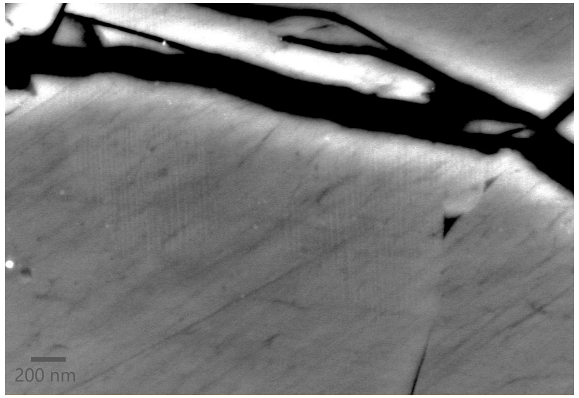
Diamond



Ceramics (MAX phase)

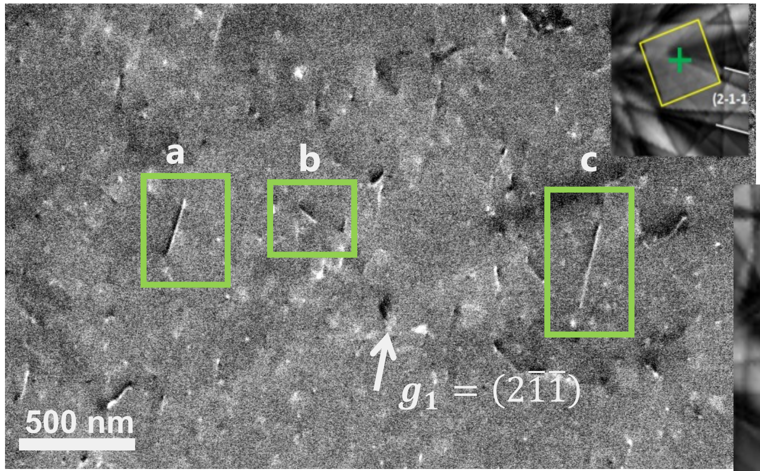


Metals (CrCoNi)



Rock (meteorite)

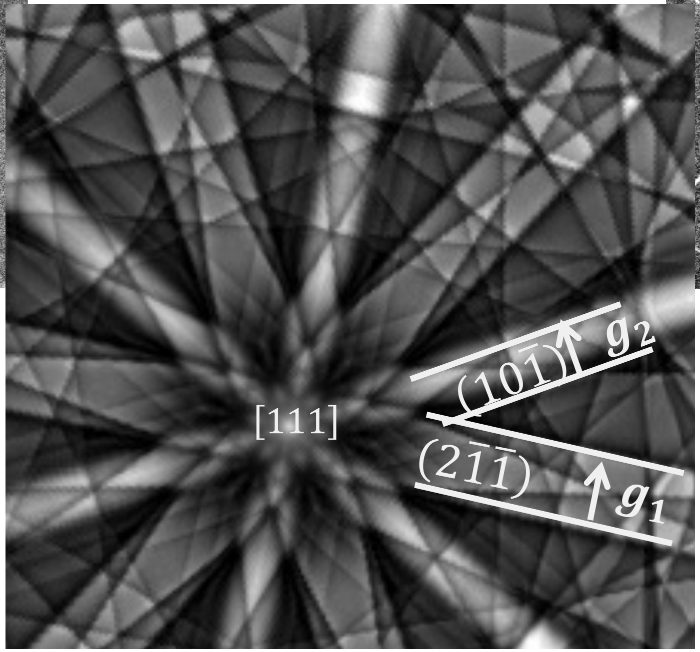
How to characterize dislocations by ECCI?

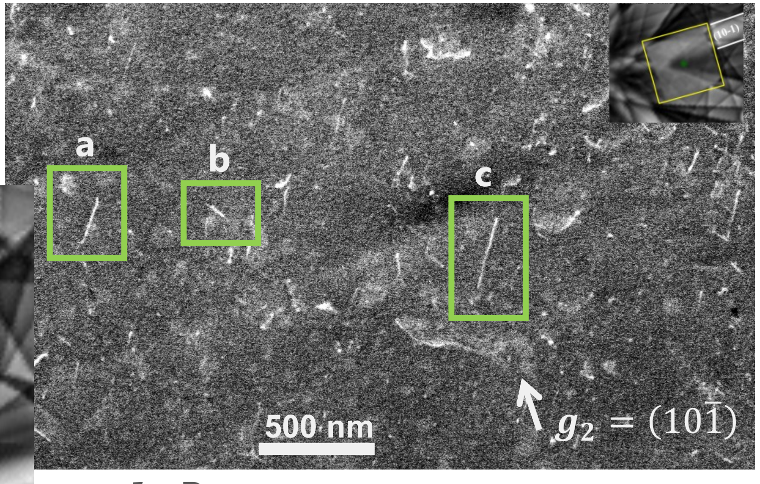


$\boldsymbol{g}$: diffraction vector

$$\boldsymbol{b} = \frac{1}{2}\langle 111 \rangle$$

$$\boldsymbol{g} \cdot \boldsymbol{b} \neq 0$$



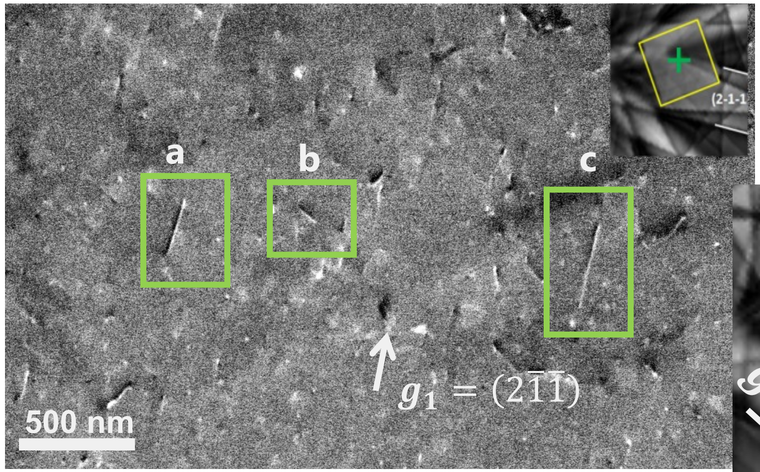


$\boldsymbol{b}$: Burgers vector

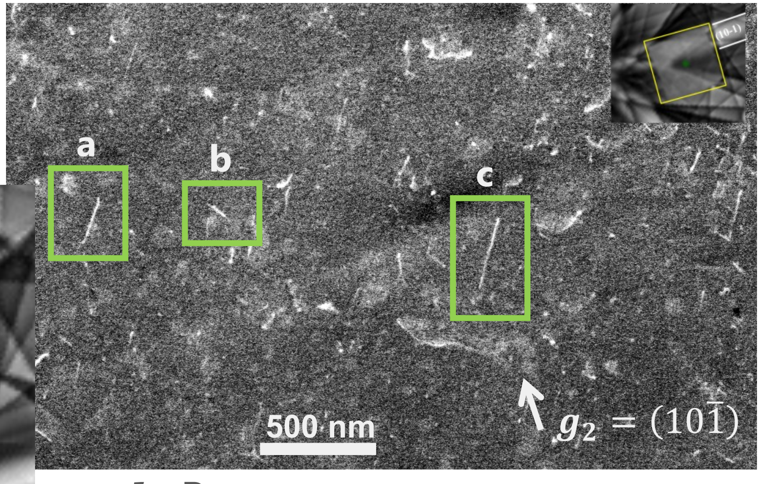
H. Kriaa et al., *Sc. Rep.* (2017)

How to characterize dislocations by ECCI?

$$\boldsymbol{b} = \frac{1}{2}\langle 111\rangle$$



$\boldsymbol{g}\cdot\boldsymbol{b} \neq 0$



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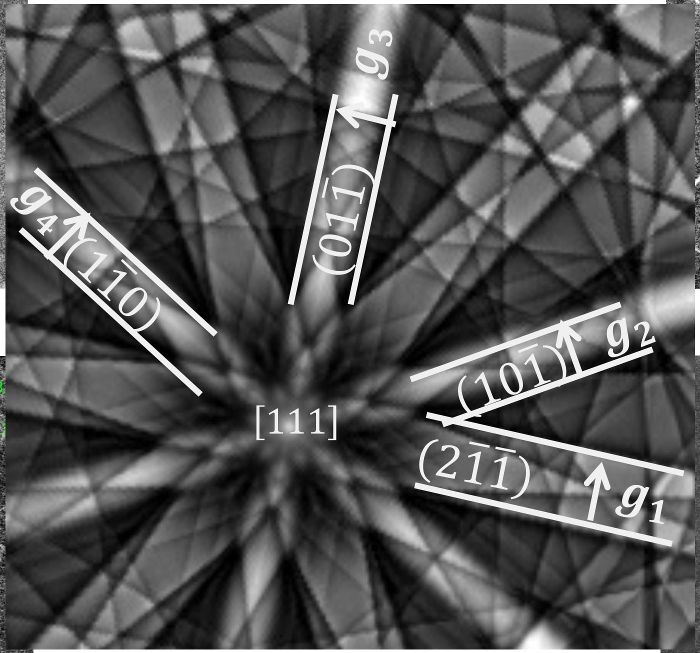
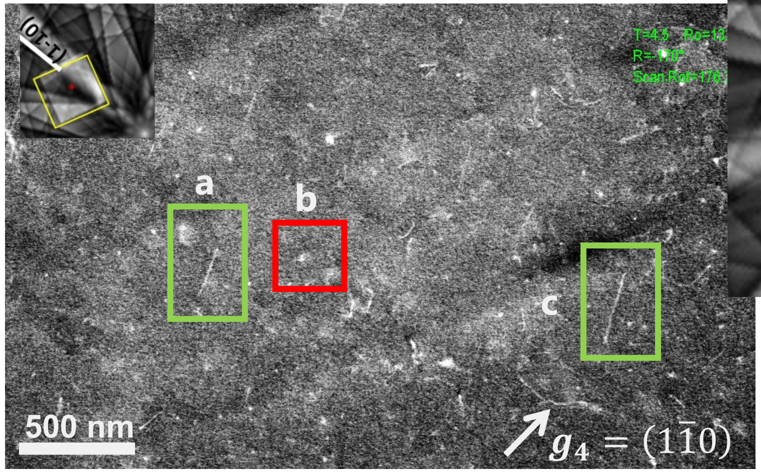


$\boldsymbol{b}$: Burgers vector



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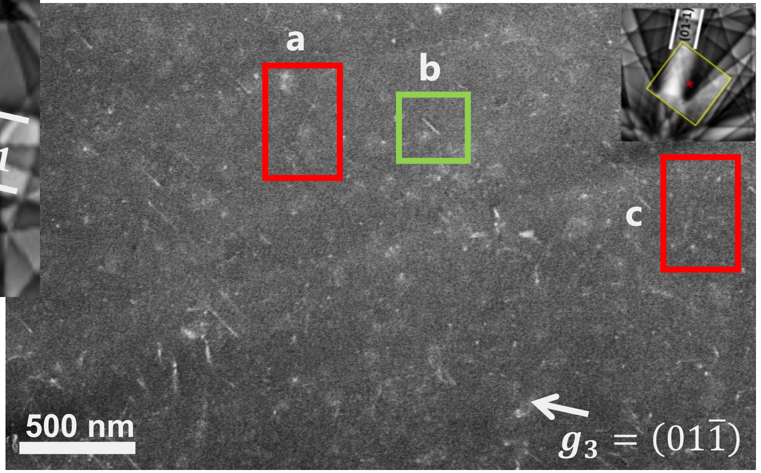

H. Kriaa et al., *Sc. Rep.* (2017)

How to characterize dislocations by ECCI?

Experimental invisibility criteria: $\boldsymbol{g} \cdot \boldsymbol{b} = \mathbf{0}; \boldsymbol{g} \cdot (\boldsymbol{b} \times \boldsymbol{\xi}) = \mathbf{0}$

	$\boldsymbol{g_1} = (2\bar{1}\bar{1})$	$\boldsymbol{g_2} = (10\bar{1})$	$\boldsymbol{g_3} = (01\bar{1})$	$\boldsymbol{g_4} = (1\bar{1}0)$	$\boldsymbol{g_5} = (11\bar{2})$	Burgers vector $\boldsymbol{b}$	Line direction $\boldsymbol{\xi}$	$\alpha = (\boldsymbol{b}, \boldsymbol{\xi})$
Dislocation a	✓	✓	✗	✓	✓	±1/2[-111]	[011]	35°
Dislocation b	✓	✓	✓	✗	✓	±1/2[11-1]	[110]	35°
Dislocation c	✓	✓	✗	✓	✓	±1/2[-111]	[011]	35°

Note: ✓: visibility; ✗: invisibility, α: angle between $\boldsymbol{b}$ and $\boldsymbol{\xi}$

H. Kriaa et al., *Sc. Rep.* (2017)

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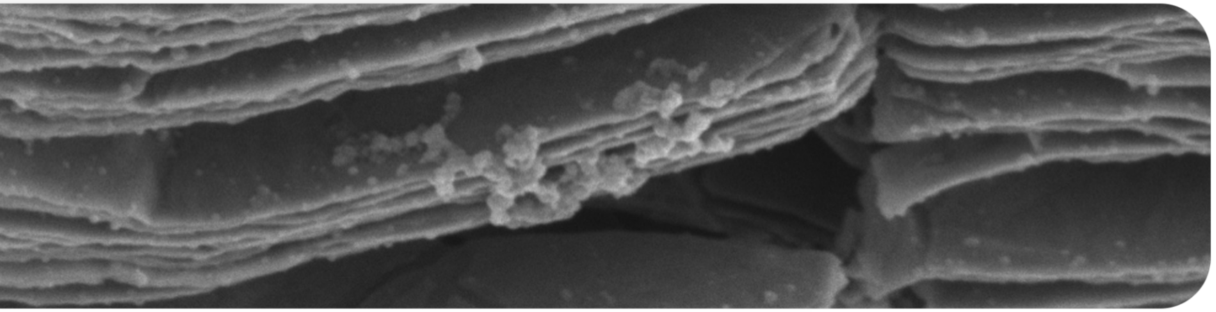
Note: ✓: visibility; ✗: invisibility, α: angle between $\boldsymbol{b}$ and $\boldsymbol{\xi}$

Comprehensive analysis of crystalline defects in SEM on bulk samples

- Burgers vector $\boldsymbol{b}$ / Fault vector $\boldsymbol{R}$
- Line direction $\boldsymbol{\xi}$

Nevertheless, near the surface
(≈ hundred nanometers below the surface)

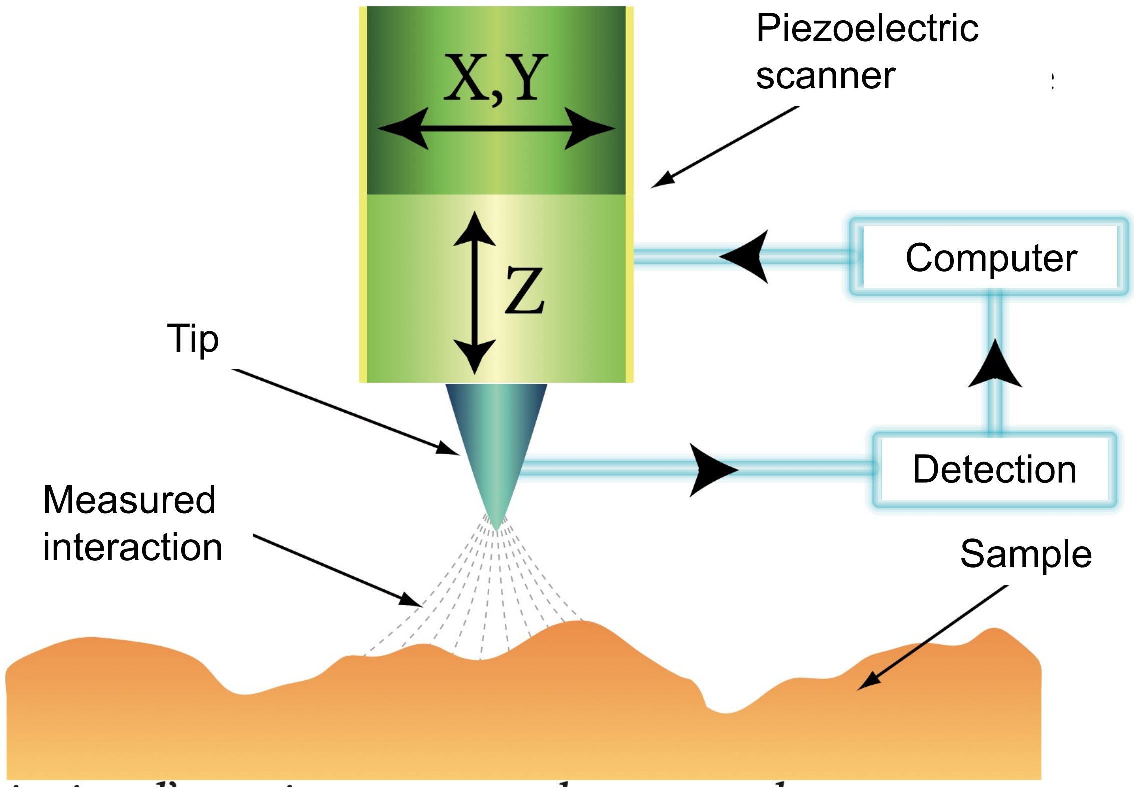
H. Kriaa et al., *Sc. Rep.* (2017)



Near-field scanning microscopy

Nanometer scale

Basics

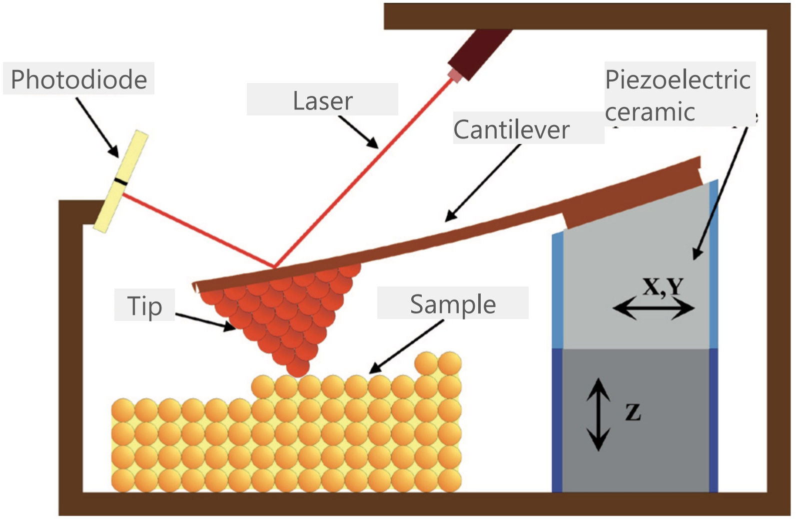


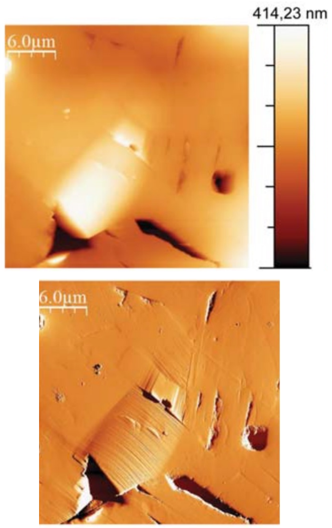
- Bypass the diffraction limit of wave microscopy
- Rely on evanescent waves captured in the near-field (within ~20–100 nm of the sample surface)
- Use a sharp tip to scan very close to the sample
- Topographic information
- Enable imaging of non-conductive or biological samples
- Require precise tip-sample distance control

C. Tromas, *Cours – Microscopie champ proche* (2010)

Two types of near-field microscopes

Atomic force Microscope (AFM)



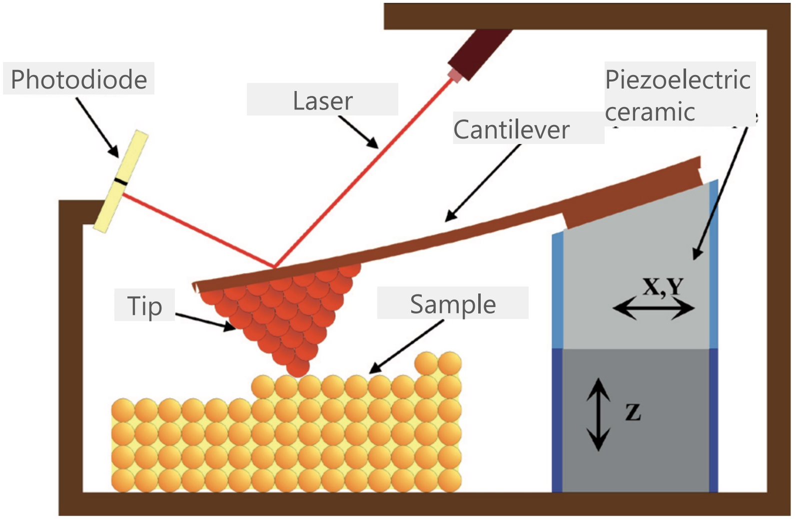


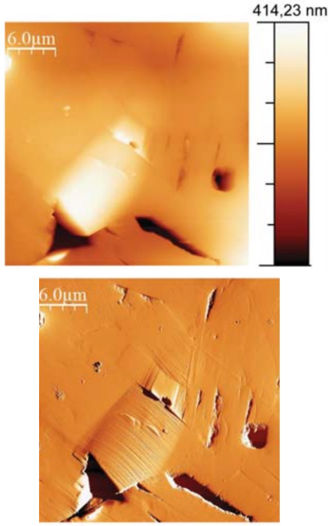
↳Interaction forces between the tip and the surface
↳ All materials
↳ nm resolution
↳Quantification in z

C. Tromas, *Cours – Microscopie champ proche* (2010)
A. Guitton, *PhD Thesis* (2013)

Two types of near-field microscopes

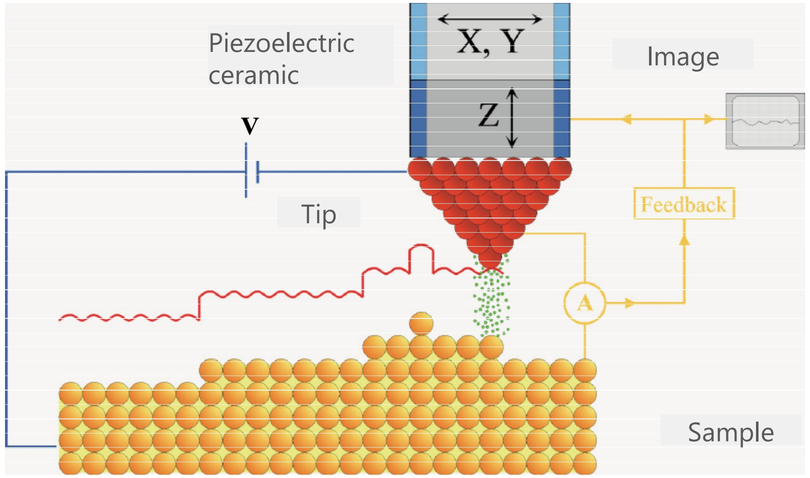
Atomic force Microscope (AFM)

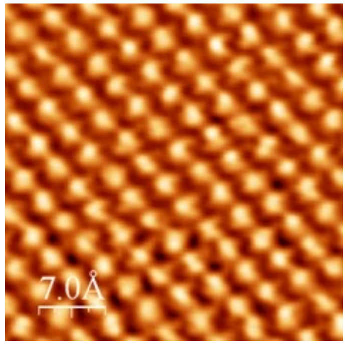




↳Interaction forces between the tip and the surface
↳ All materials
↳ nm resolution
↳Quantification in z

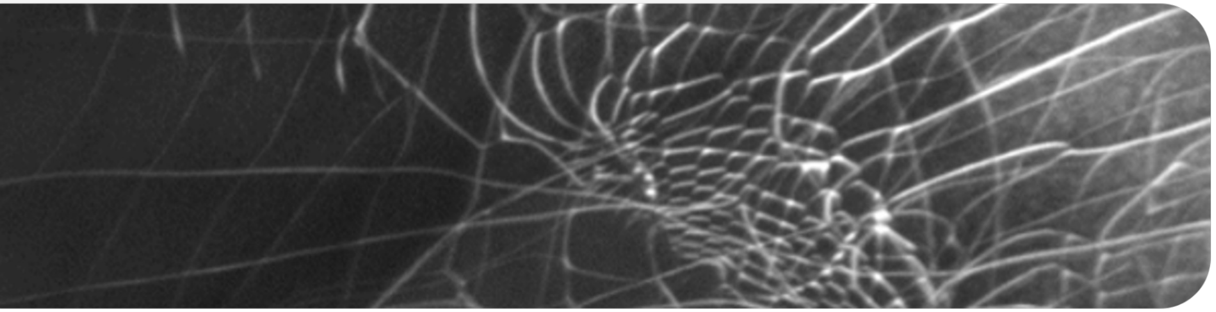
Scanning Tunneling Microscope (STM)





↳Tunnel current
↳ Only electrical conductors
↳ Atomic resolution
↳Quantification in z

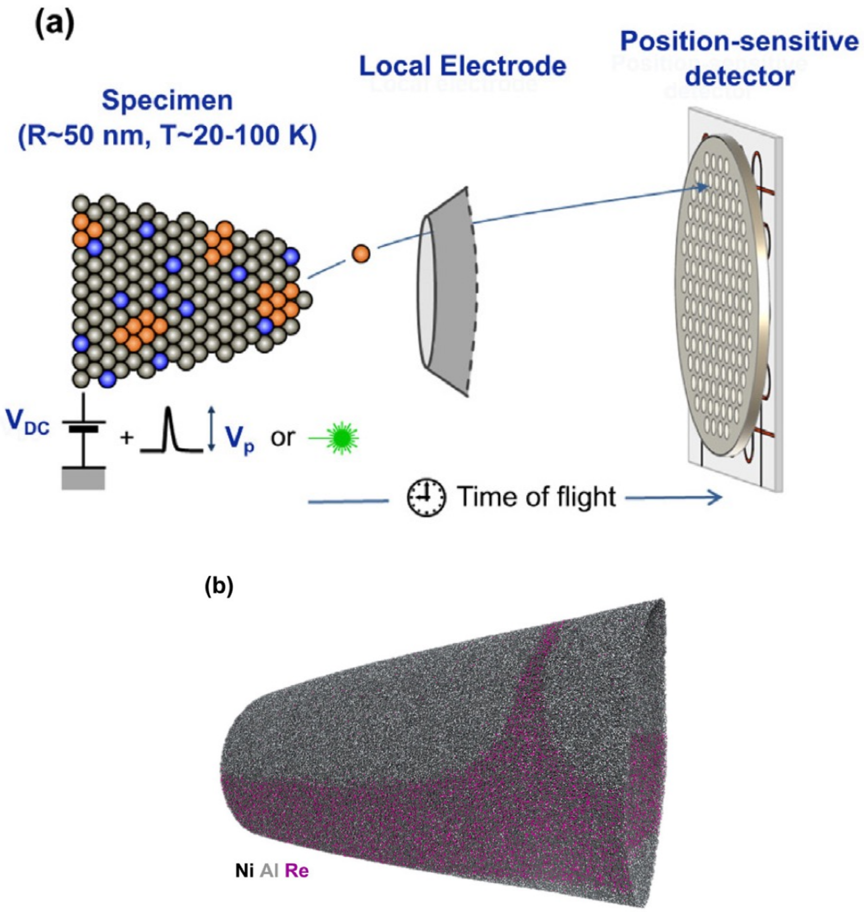
C. Tromas, *Cours – Microscopie champ proche* (2010)
A. Guitton, *PhD Thesis* (2013)



Atom Probe Tomography (APT)

Atomic scale

Basics of APT



❖ Definition:

- APT is a microanalytical technique that provides 3D compositional imaging and quantitative analysis at the atomic scale.

❖ Principle:

- Utilizes field evaporation induced by a high electric field to remove atoms from a sharp specimen tip.
- These atoms are then identified based on their time of flight.

❖ Spatial resolution:

- Offers near-atomic spatial resolution, enabling the visualization of individual atoms within a material.

A. Prakash et al., *Acta Mater.* (2015)

Thanks for your listening!

If you need further information:

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Full Professor at Université de Lorraine

Phone (LEM3): +33 372 747 826

Email: antoine.guitton@univ-lorraine.fr

Website: www.antoine-guitton.fr

LABORATOIRE D'ÉTUDE DES MICROSTRUCTURES ET DE MÉCANIQUE DES MATÉRIAUX

UNIVERSITÉ DE LORRAINE

enim École Nationale d'Ingénieurs de Metz