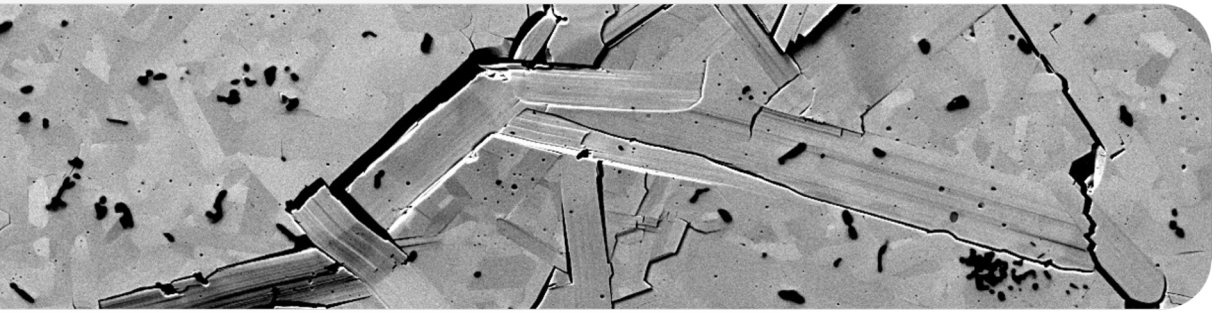


Characterization of micro-/nano-structures

Prof Antoine GUITTON

Université de Lorraine, CNRS, Arts et Métiers Institute of Technology, LEM3, F-57000 Metz,
antoine.guitton@univ-Lorraine.fr

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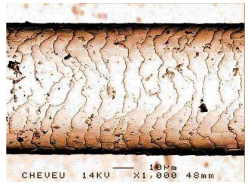


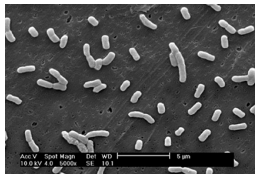
Some micro-/nano-structural features

www.antoine-guitton.fr

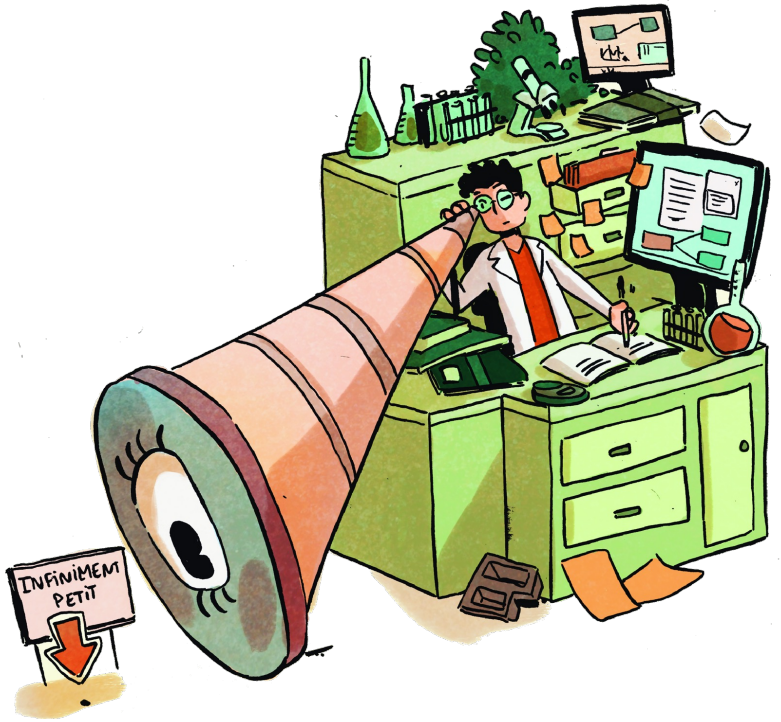
Some orders of magnitude

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





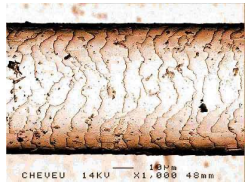
- Hair diameter = 50-100 µm
- Size of a bacteria = 0.1-10 µm

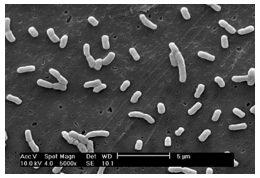


A. Guitton, Science en 4 Saisons – Hiver, *EDP Sciences (2023)*

Some orders of magnitude

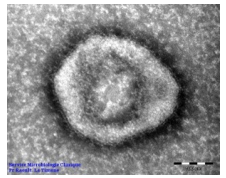
❖ Micrometer (1 µm = 0.000001 m = 10^{-6} m)

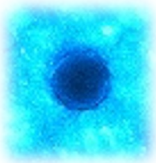




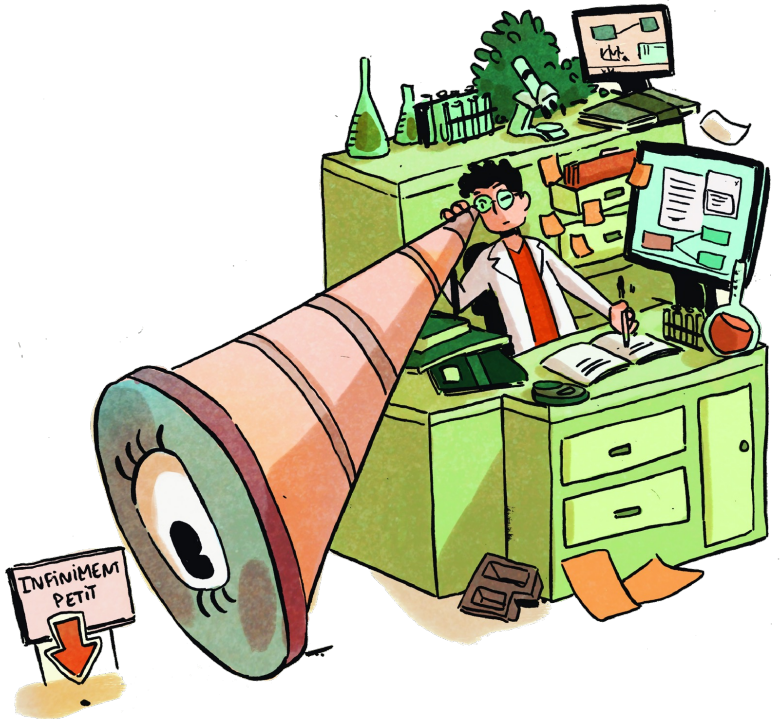
- Hair diameter = 50-100 µm
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❖ Nanometer (1 nm = 0.000000001 m = 10^{-9} m)





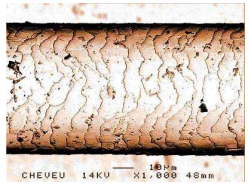
- Size of a virus = 20-450 nm

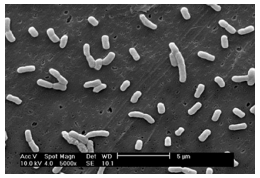


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Some orders of magnitude

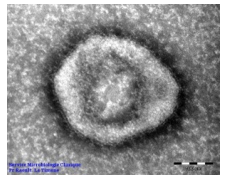
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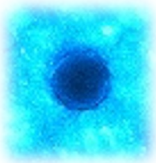




- Hair diameter = 50-100 µm
- Size of a bacteria = 0.1-10 µm

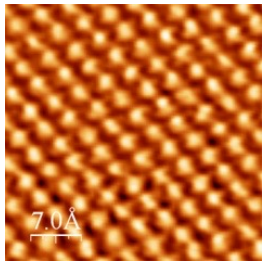
❖ Nanometer (1 nm = 0.000000001 m = 10^{-9} m)



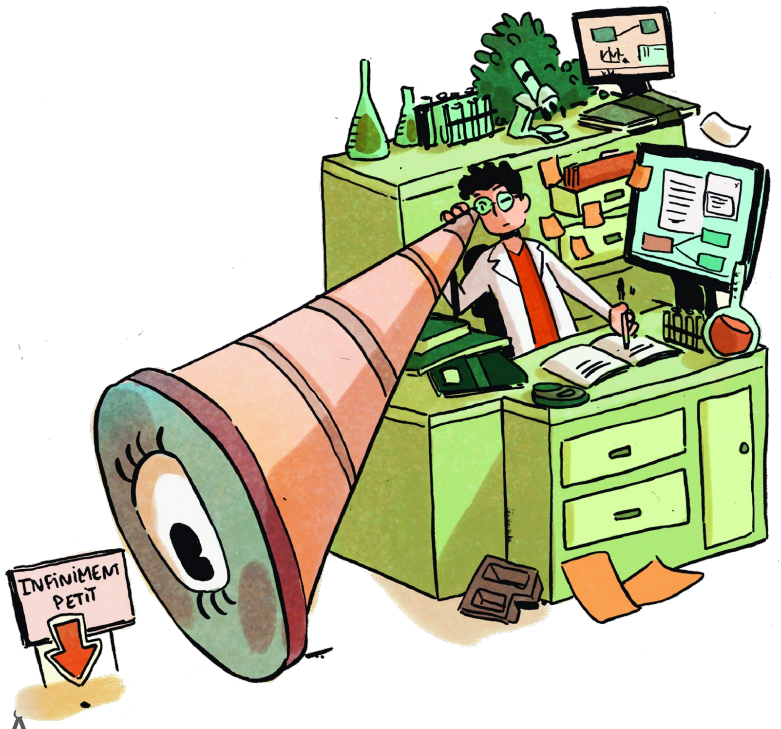


- Size of a virus = 20-450 nm

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



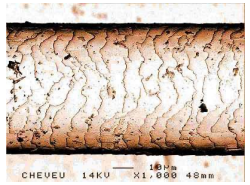
- Distance between atoms = 0.2 nm = 2 Å
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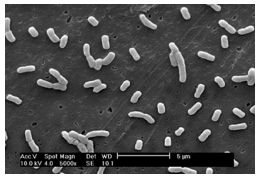


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Some orders of magnitude

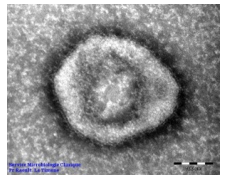
❖ Micrometer (1 µm = 0.000001 m = 10^{-6} m)

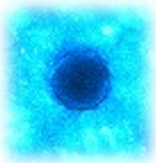




- Hair diameter = 50-100 µm
- Size of a bacteria = 0.1-10 µm

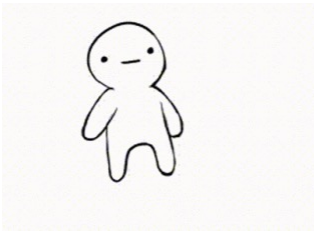
❖ Nanometer (1 nm = 0.000000001 m = 10^{-9} m)

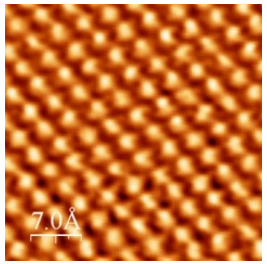




- Size of a virus = 20-450 nm

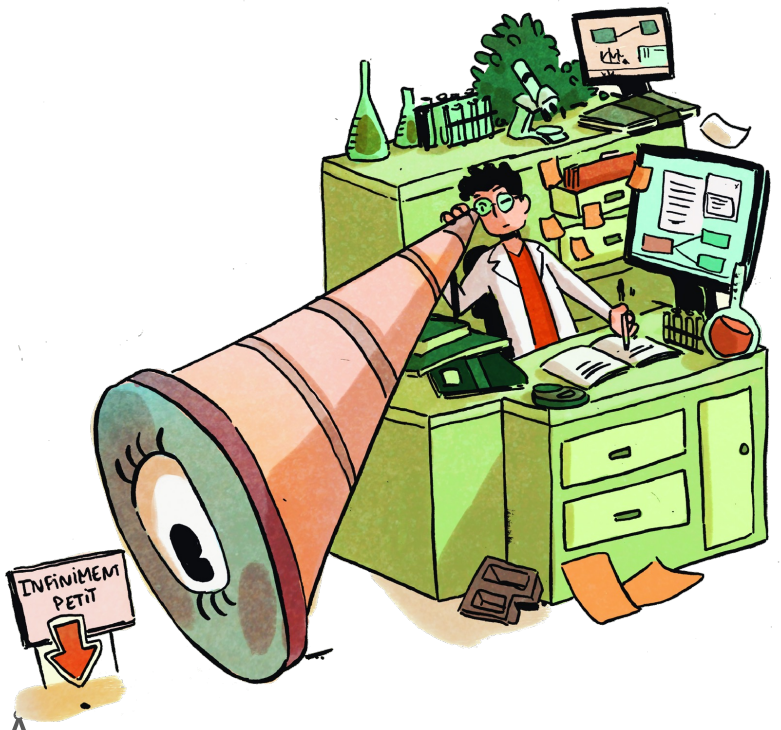
❖ Atomic scale (1 Å = 0.0000000001 m = 10^{-10} m)





- Distance between atoms = 0.2 nm = 2 Å
- Atom radius = 0.1 nm = 1 Å

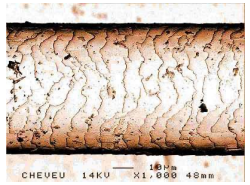
This is neither copper nor gold!!!

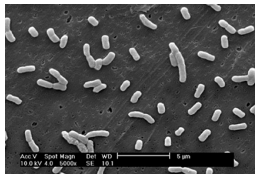


A. Guitton, Science en 4 Saisons – Hiver, *EDP Sciences (2023)*

Some orders of magnitude

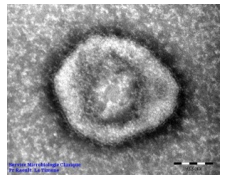
❖Micrometer (1 µm = 0.000001 m = 10^{-6} m)

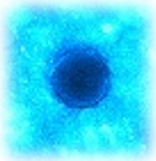




- Hair diameter = 50-100 µm
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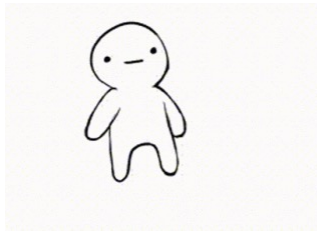
❖Nanometer (1 nm = 0.000000001 m = 10^{-9} m)

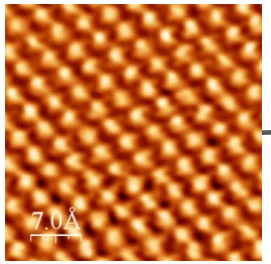




- Size of a virus = 20-450 nm

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

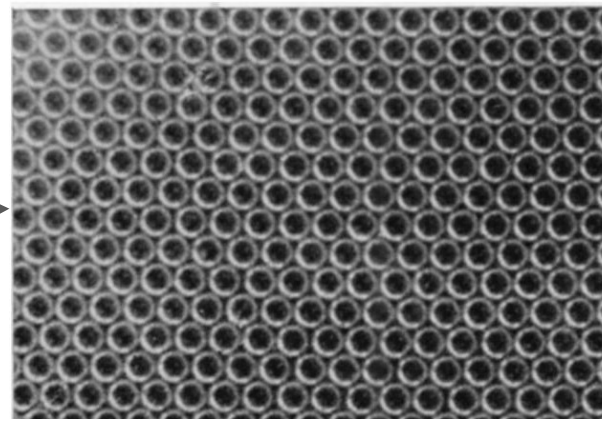




- Distance between atoms = 0.2 nm = 2 Å
- Atom radius = 0.1 nm = 1 Å

Stacking of spheres

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

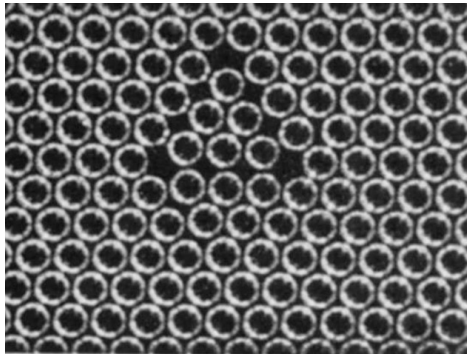


This is neither copper nor gold!!!

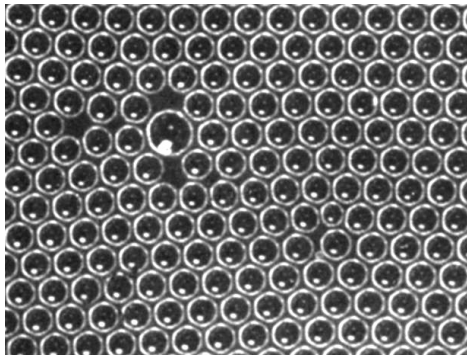
Defects in crystalline materials

0 dimension

Vacancy



Interstitial

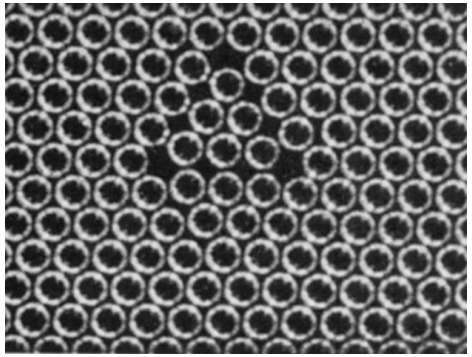


L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)

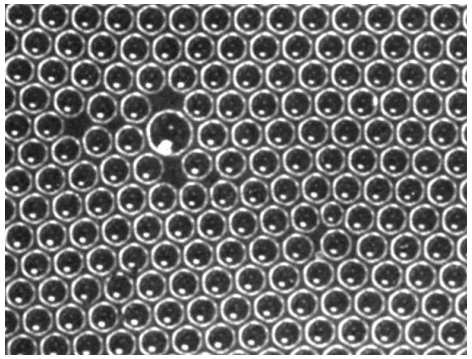
Defects in crystalline materials

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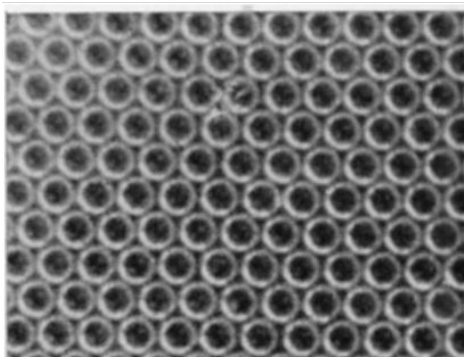


Interstitial



1 dimension

Dislocation

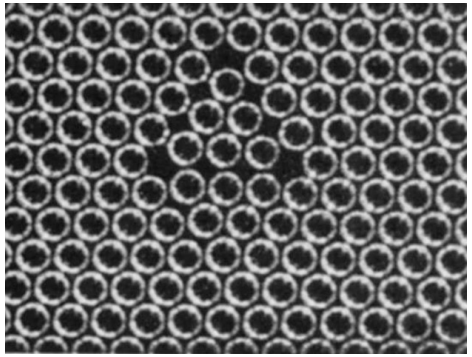


L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)

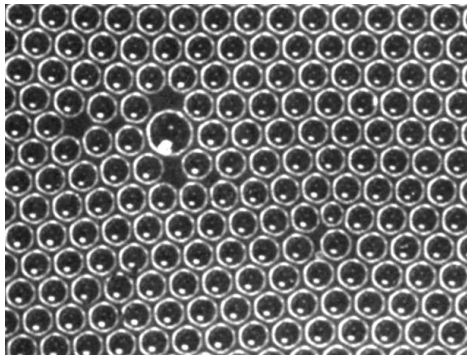
Defects in crystalline materials

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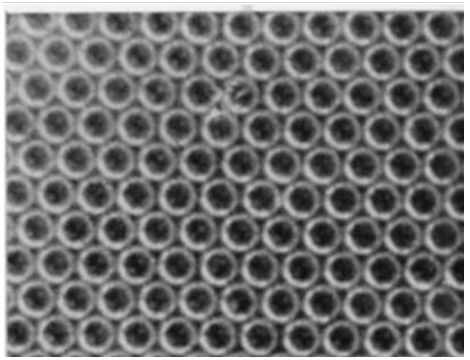


Interstitial



1 dimension

Dislocation

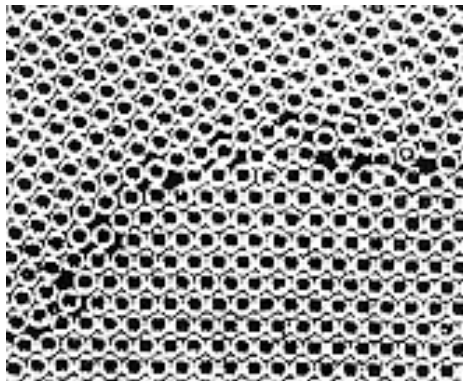


2 dimensions

Stacking fault



Grain boundaries

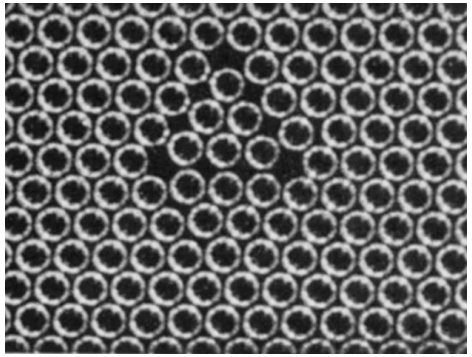


L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)

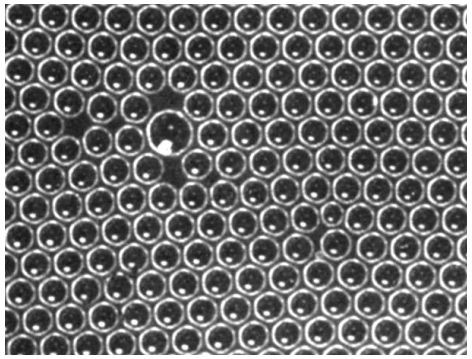
Defects in crystalline materials

0 dimension

Vacancy

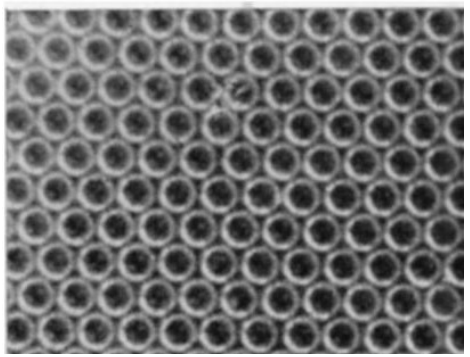


Interstitial



1 dimension

Dislocation

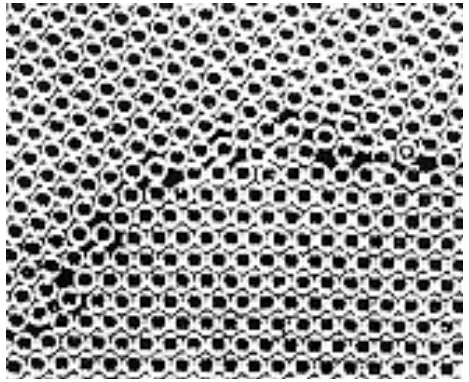


2 dimensions

Stacking fault



Grain boundaries



3 dimensions

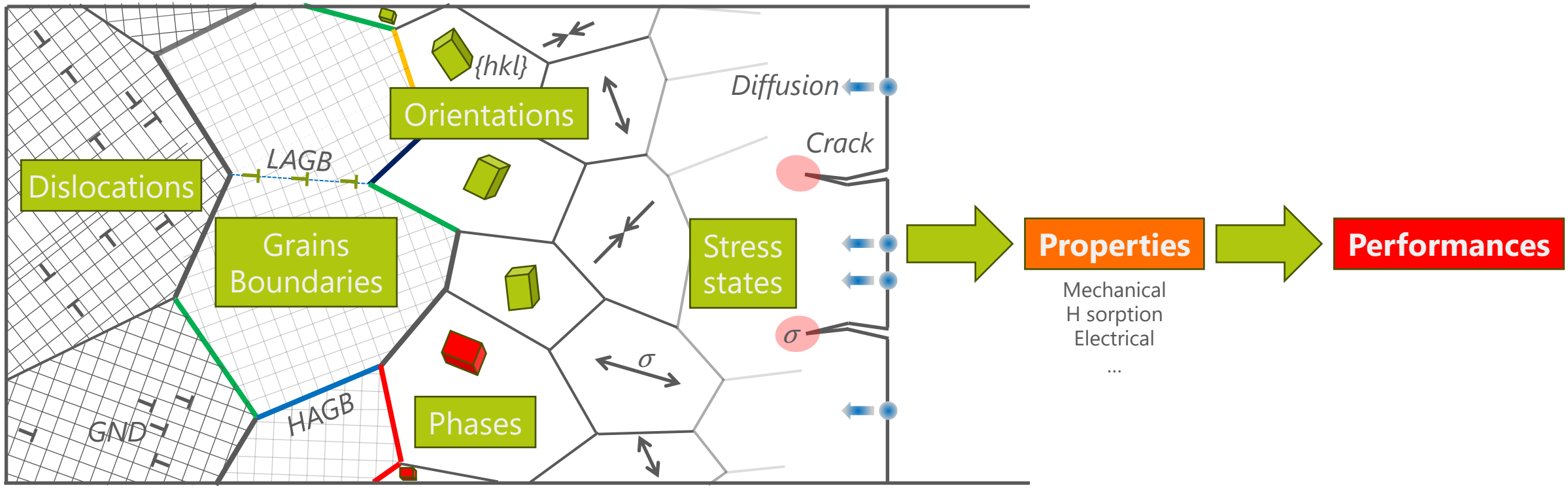
Inclusions, precipitates…

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

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

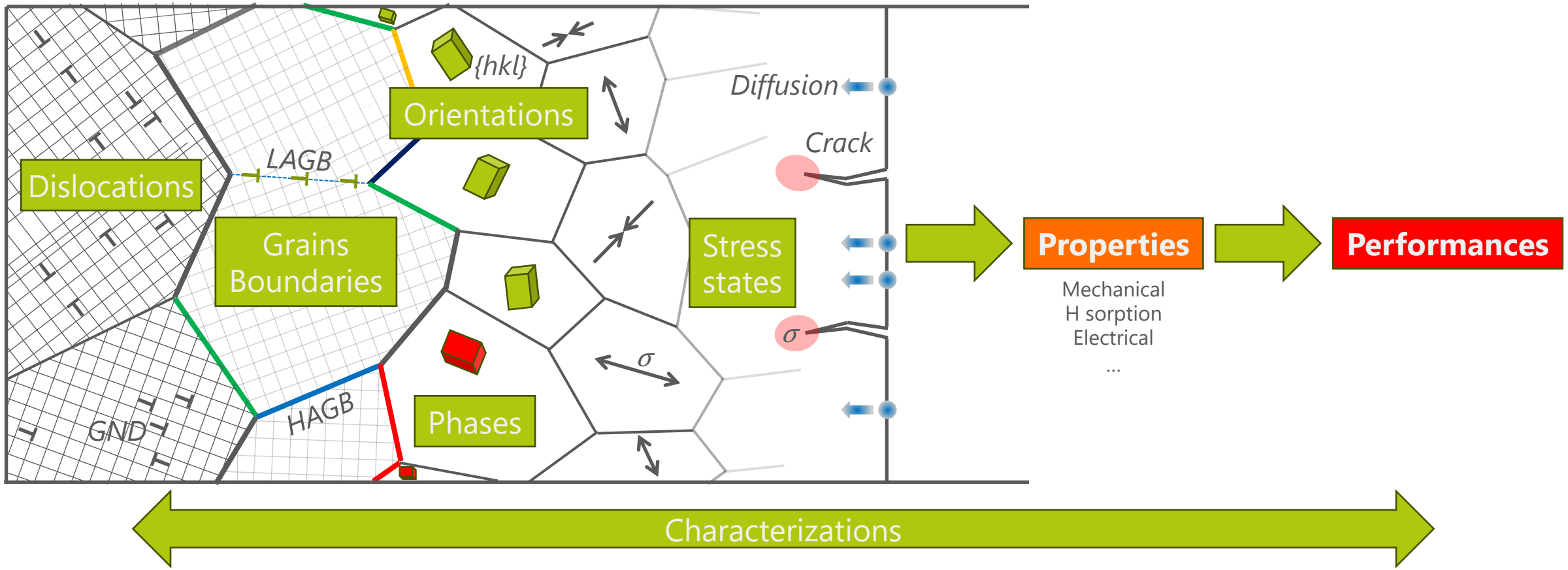
Micro-/nano-structures

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

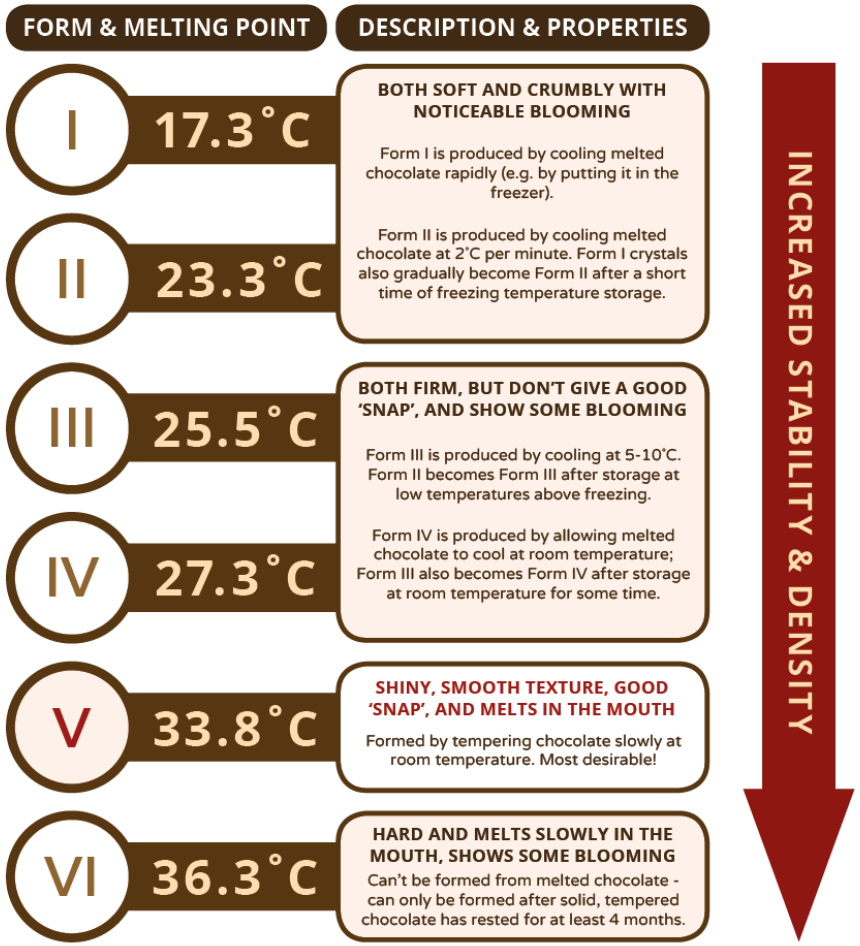


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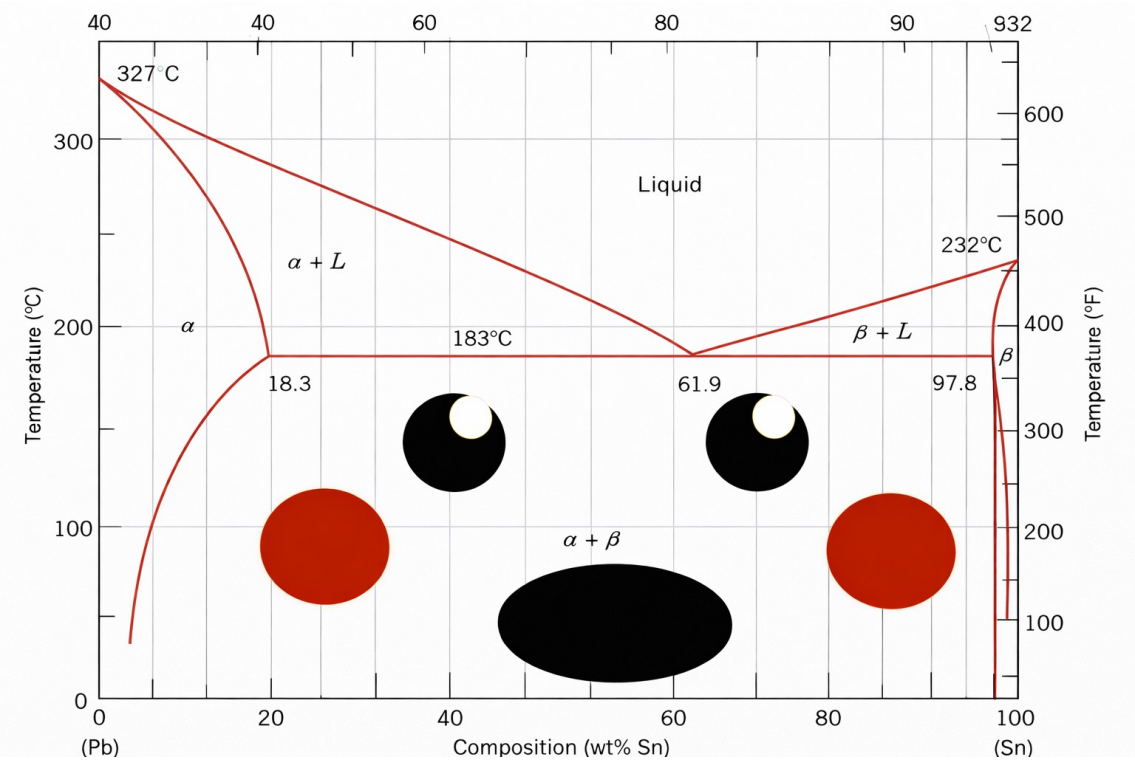
Phases



- **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)

Phases

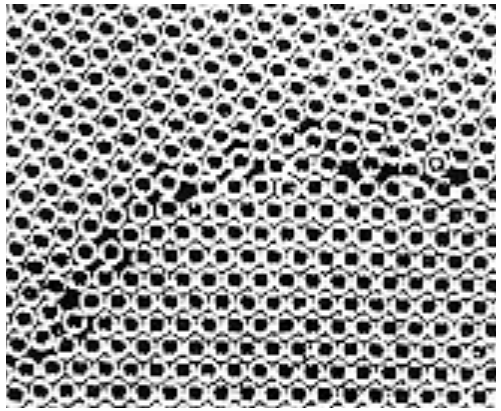


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    - ✓ Most desirable phase: Form V (β') – gives chocolate its smooth texture, glossy finish, and ideal snap.
- **Phase transitions:**
  - Heating and cooling control polymorphic transformation
- **Phase diagram:**
  - Graphically represents phase 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.

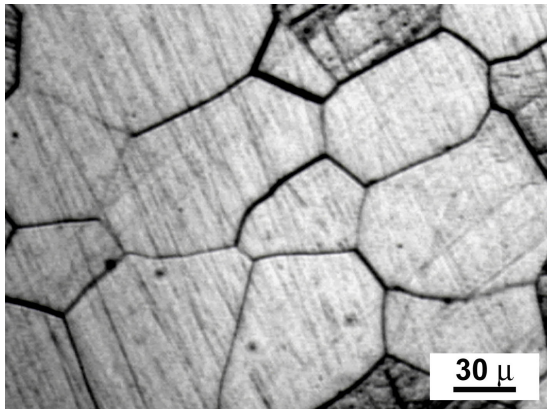
P. Fryer et al., *MRS Bulletin. (*2000)

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).

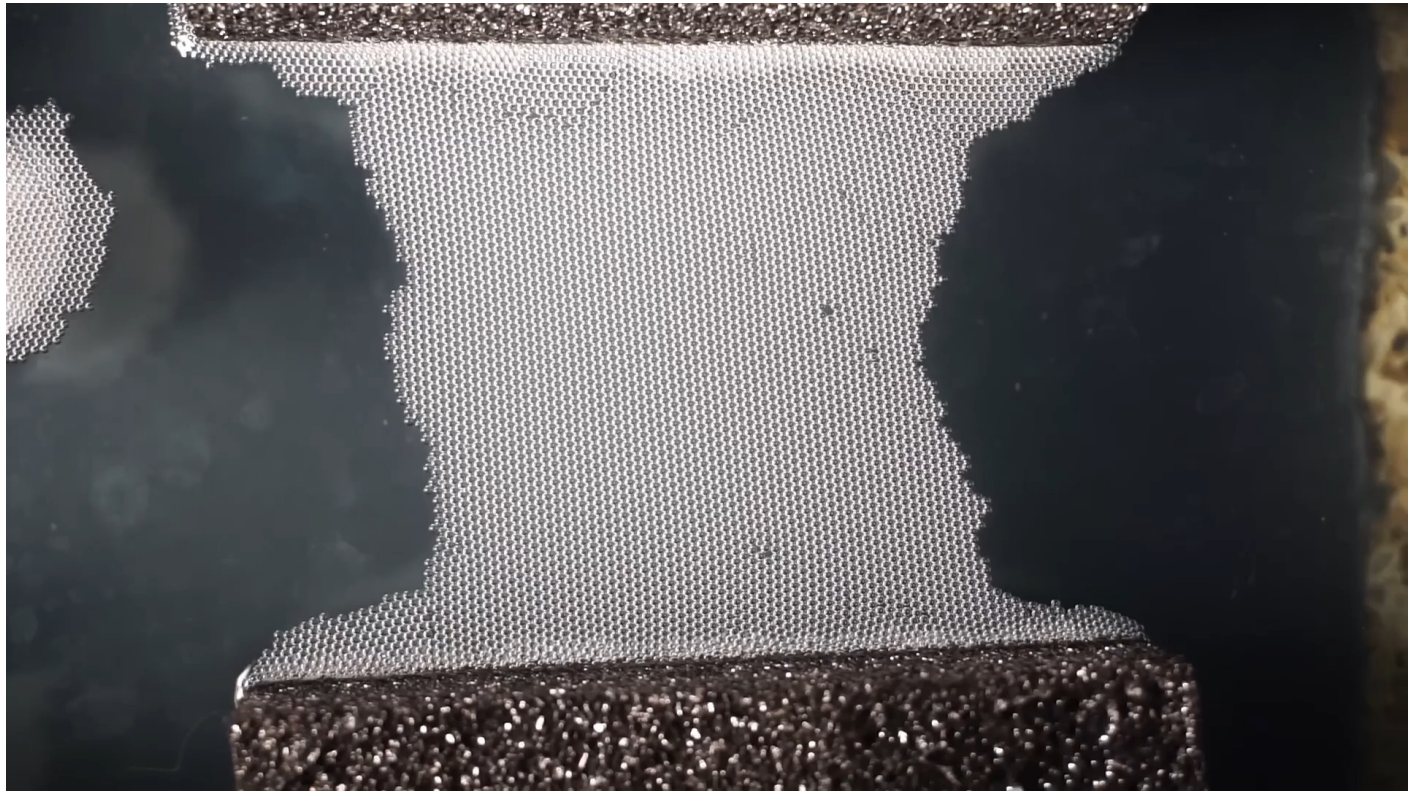
❖ Description:

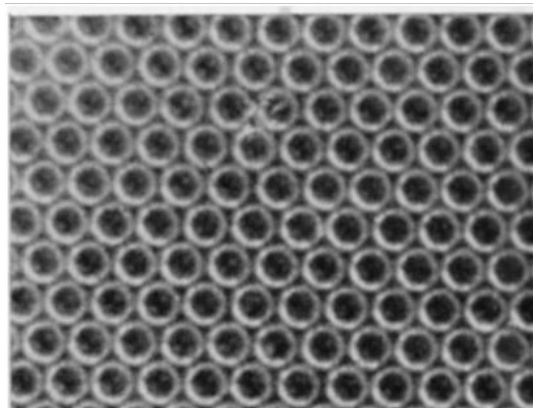
- Shape
- Size

❖ Texture:

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

Dislocations





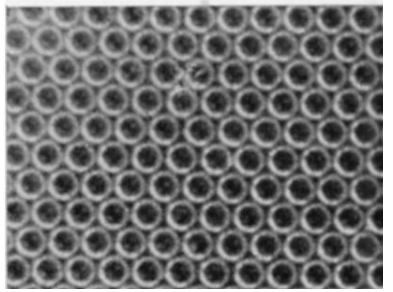
L. Bragg & J.F. Nye, *Proc. R. Soc. Lond. (*1947)

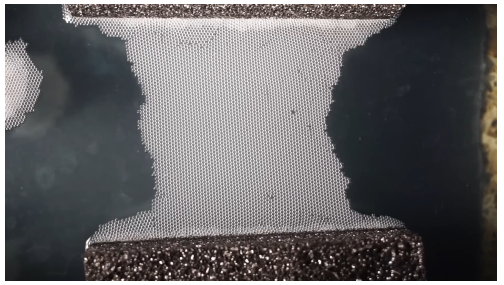
Dislocations

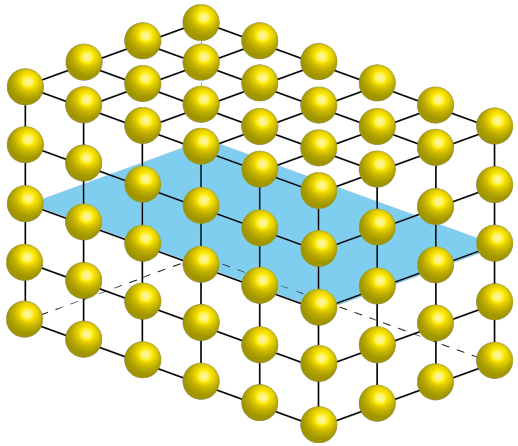
❖ **Definition:**

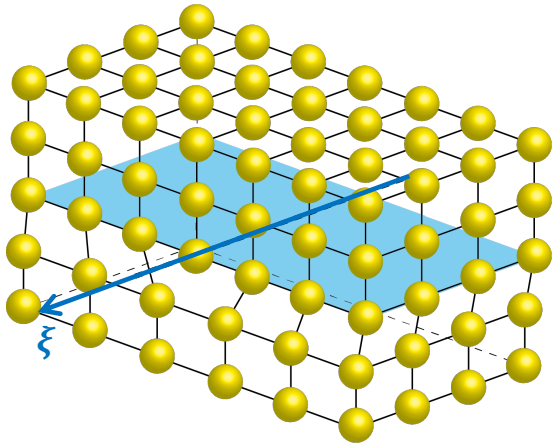
- Line defects in a crystalline material where atoms are misaligned.
- They represent a discontinuity in the crystal lattice that allows deformation to occur at lower stresses.
- Characterized by $\boldsymbol{b}$ and $\boldsymbol{\xi}$.

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







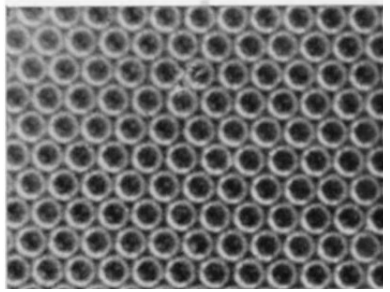


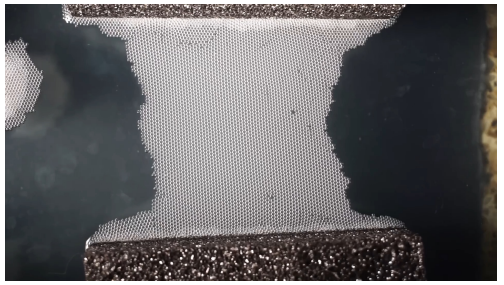
Dislocations

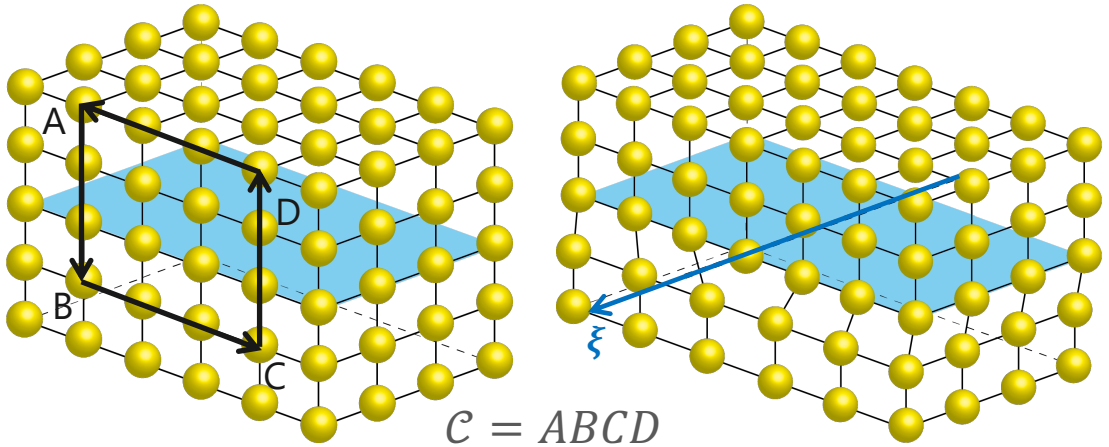
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- Line defects in a crystalline material where atoms are misaligned.
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L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)







$\mathcal{C} = ABCD$

Dislocations

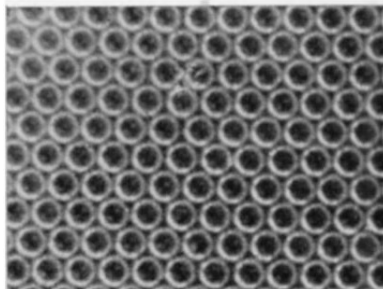
❖ Definition:

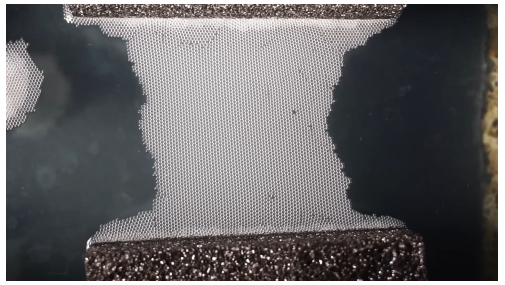
- Line defects in a crystalline material where atoms are misaligned.
- They represent a discontinuity in the crystal lattice that allows deformation to occur at lower stresses.
- Characterized by $\boldsymbol{b}$ and $\boldsymbol{\xi}$.

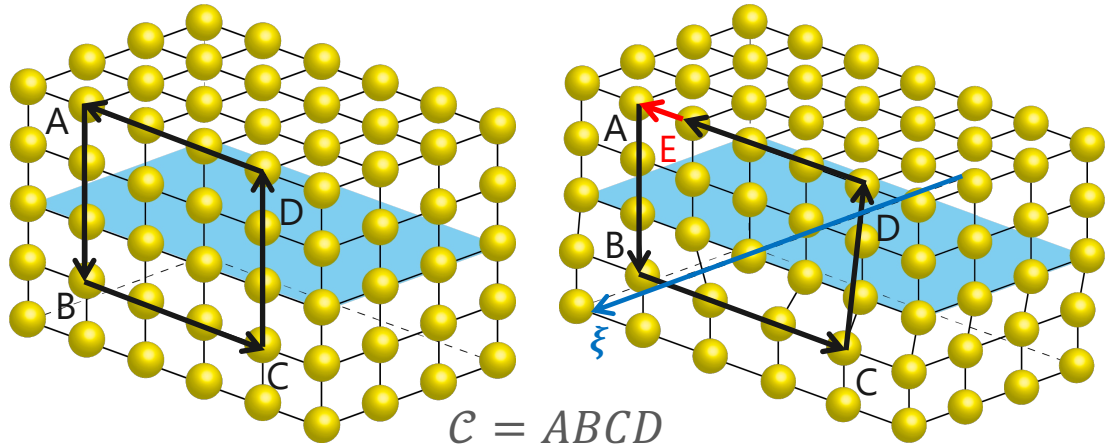
❖ Types:

- *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)







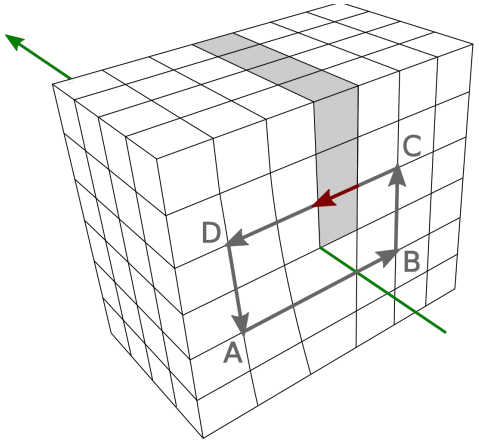
$$\mathcal{C} = ABCD$$

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

Dislocation types

Edge

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





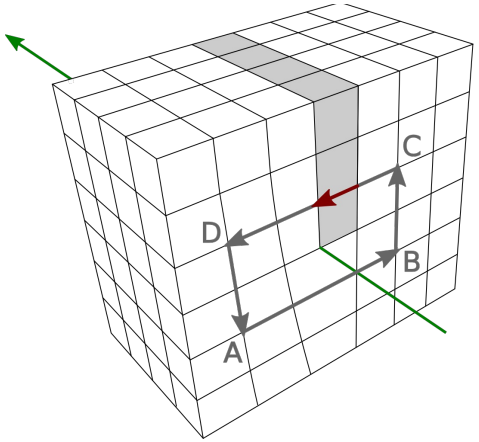
Symbol: ⊥

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

Dislocation types

Edge

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

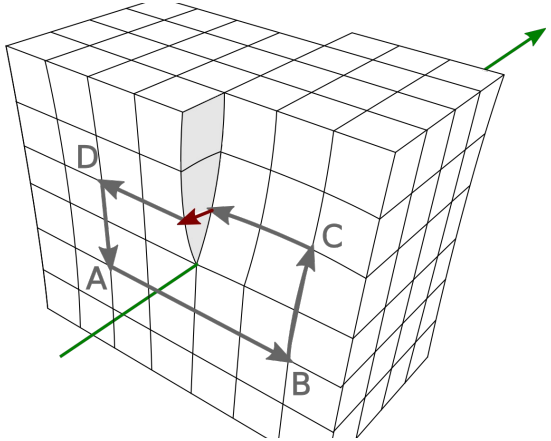




Symbol: ⊥

Screw

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



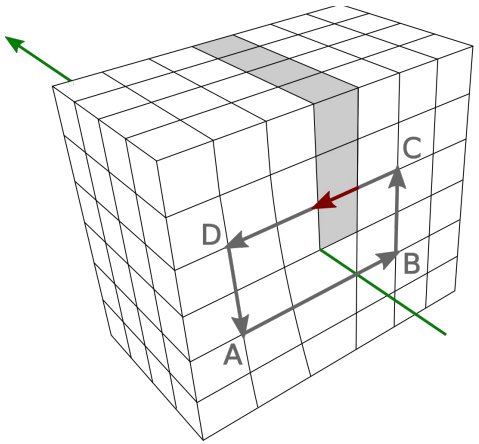


F. C. Frank, *Proc. Phys. Soc. A*, 62, 131–134 (1949)

Dislocation types

Edge

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

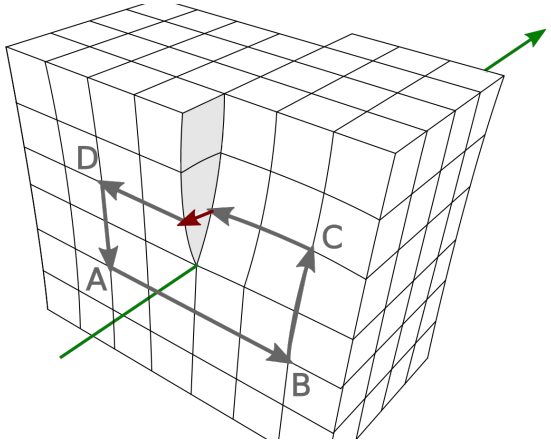




Symbol: ⊥

Screw

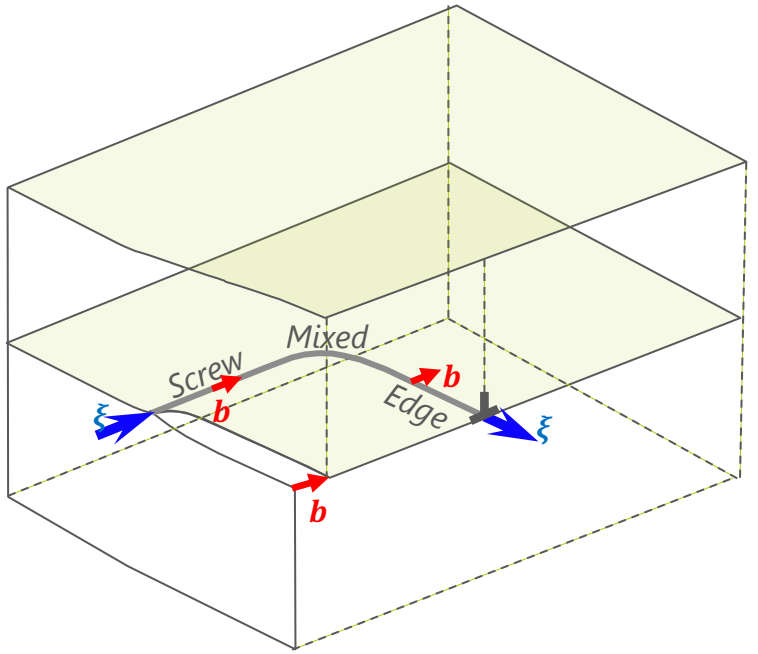
Results from a shear distortion, forming a helical ramp in the crystal lattice ($\boldsymbol{b} \parallel \boldsymbol{\xi}$).





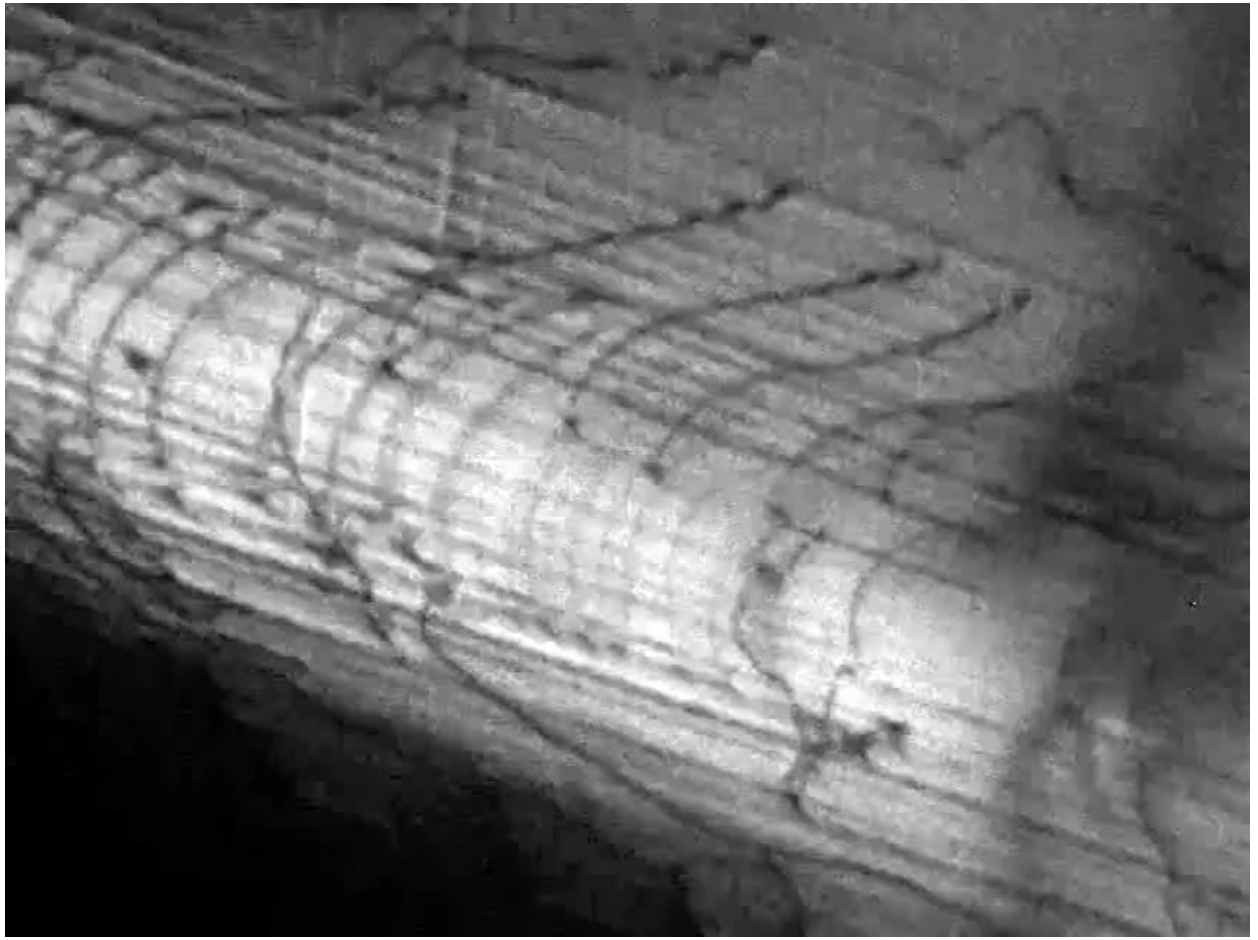
Mixed

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



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

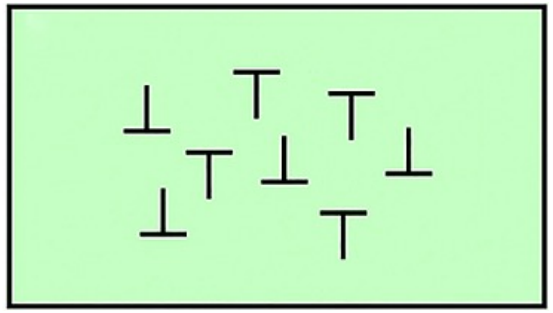
Dislocation glide -in 304 stainless steel



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

Different types of dislocation densities

Statistically stored dislocations (SSD)

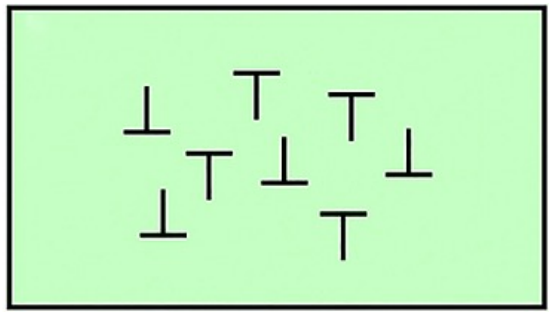


⇒ No lattice curvature at long range

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

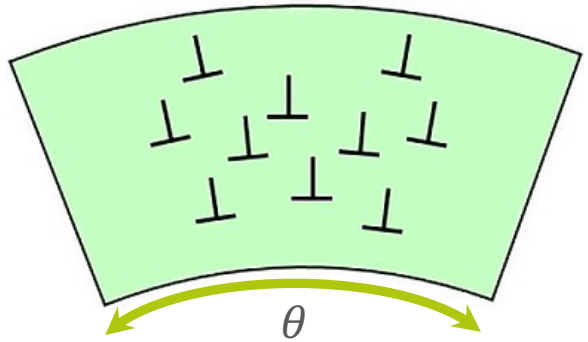
Different types of dislocation densities

Statistically stored dislocations (SSD)



⇒ No lattice curvature at long range

Geometrically necessary dislocations (GND)



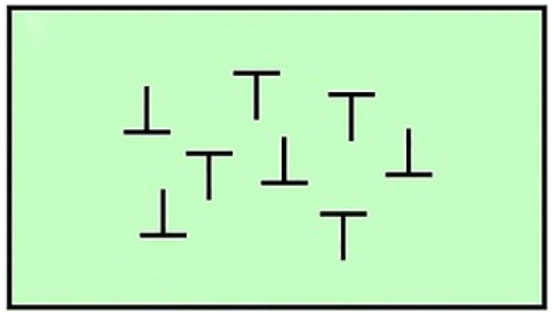
⇒ Lattice curvature at long range

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

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

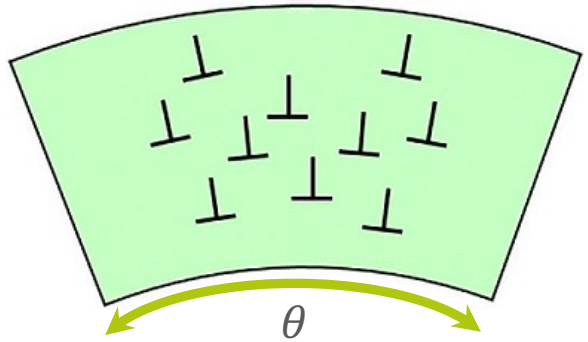
Different types of dislocation densities

Statistically stored dislocations (SSD)



⇒ No lattice curvature at long range

Geometrically necessary dislocations (GND)



⇒ Lattice curvature at long range

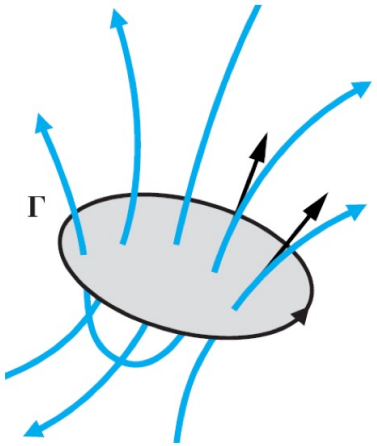
$$\rho_{\mathrm{GND}} \approx \frac{1}{\|\boldsymbol{b}\|}\|\boldsymbol{\alpha}\|;\ \|\boldsymbol{\alpha}\| = \sqrt{\alpha_{ij}.\alpha_{ij}}$$

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

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

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

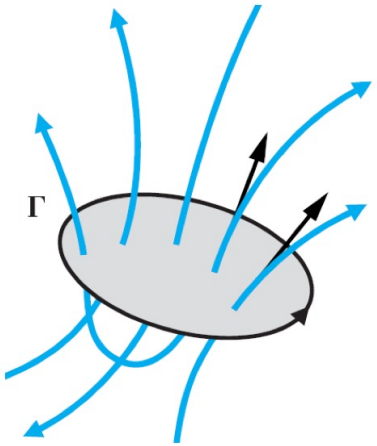
The Nye tensor





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

The Nye tensor





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

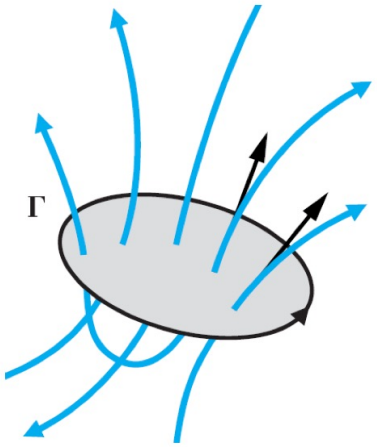
$$\boldsymbol{B} = \left(\oint_{\Gamma} \boldsymbol{u}(\boldsymbol{x}) \boldsymbol{n} \mathrm{d}S \right) \boldsymbol{b} = \oint_{\Gamma} \boldsymbol{\alpha} \boldsymbol{n} \mathrm{d}S$$

Where

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

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

The Nye tensor



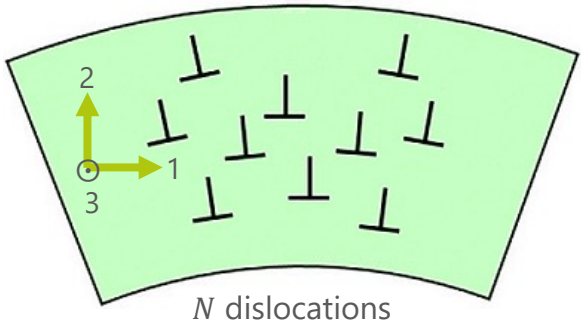
- The net Burgers vector on the closed circuit Γ limiting the surface S

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

Where

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





Edge dislocations (1 of 2 types)

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

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

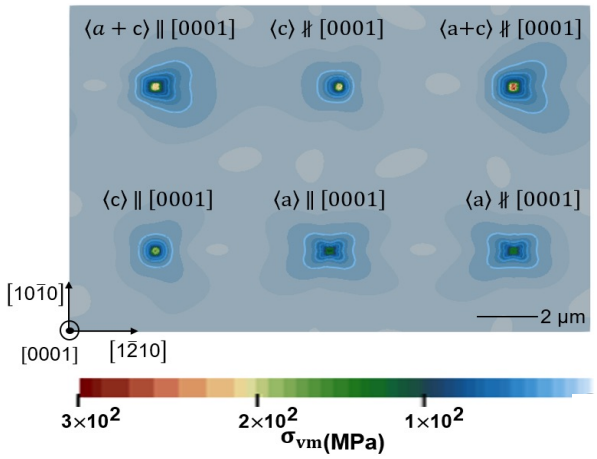
Screw dislocations

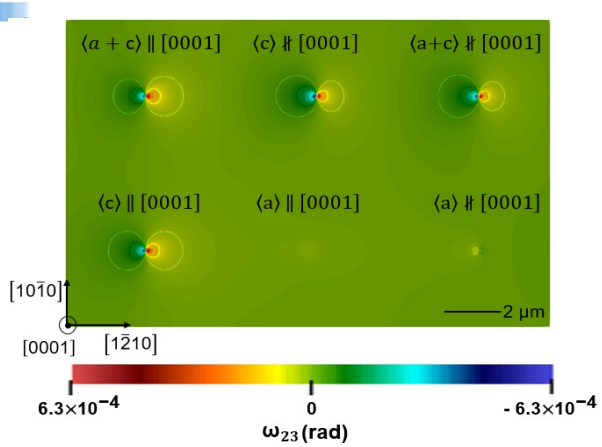
$$\boldsymbol{B}_{screw} = N b \boldsymbol{e_3} = \boldsymbol{\alpha} \boldsymbol{e_3} S$$

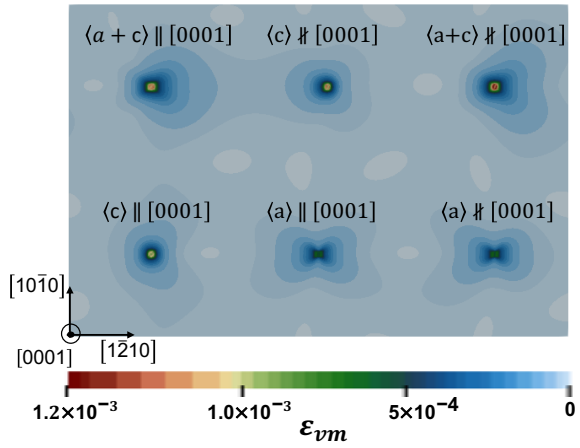
$$\boldsymbol{\alpha} = \frac{N}{S} \boldsymbol{b} \otimes \boldsymbol{u}(\boldsymbol{x}) = \frac{N}{S} b \boldsymbol{e_3} \otimes \boldsymbol{e_3} = \rho_{GND} \boldsymbol{e_3} \otimes \boldsymbol{e_3}$$

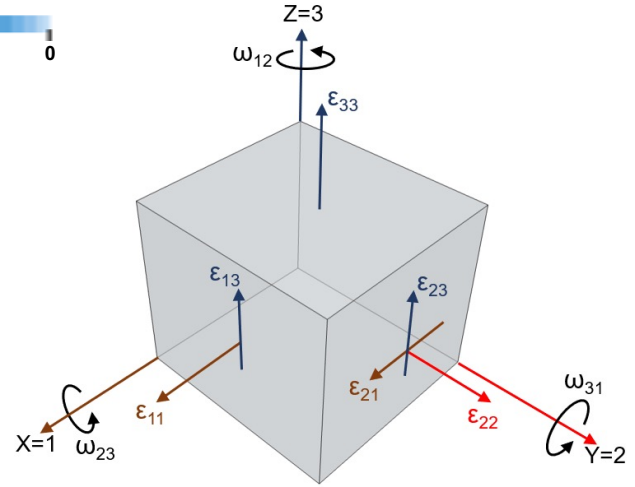
J.F. Nye, *Acta Metal.* (1953)

Elastic fields of dislocations





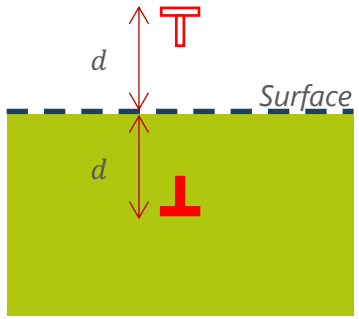




↳Dislocations induce elastic fields, which are key to their detection and analysis.

F. El Ajjouri et al., *Philos. Mag.* (2026)

Effect of free surfaces on dislocations



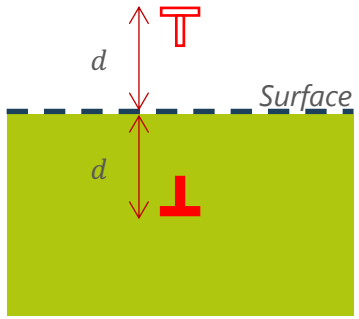
❖ **Surface presence alters the elastic field of a dislocation loop.**

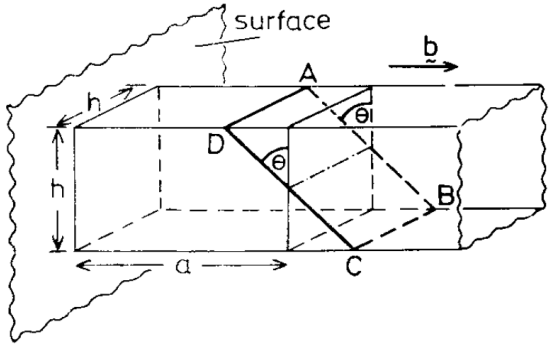
⇒ An image force appears to satisfy the zero-traction boundary condition at the surface.

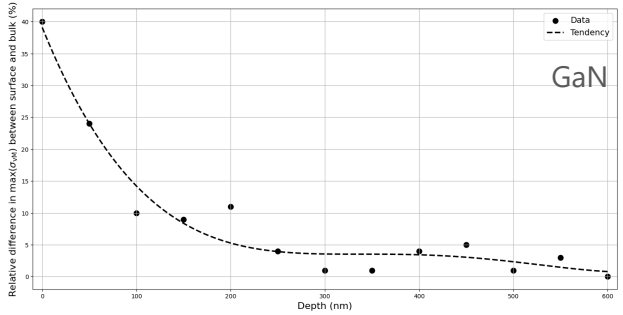
⇒ Physically, it corresponds to the attraction between the real dislocation and a virtual dislocation of opposite Burgers vector ($-\boldsymbol{b}$) placed symmetrically on the other side of the surface.

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

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.

- **Surface effects are short-ranged:**
  - Significant influence when depth $a \leq 2h - 3h$

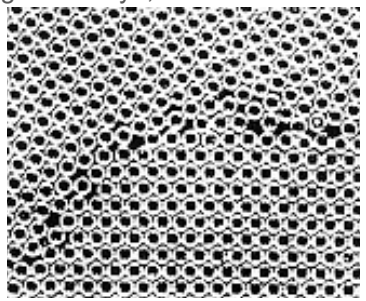
    a: distance from loop center to surface; h: loop size (length of one side of rectangular loop)
  - For $a > 3h$, the dislocation behaves as in an infinite medium.

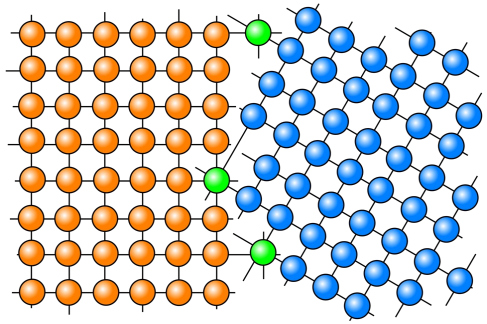
↳The behavior of dislocations is influenced by the presence of free surfaces

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

Grain boundaries

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





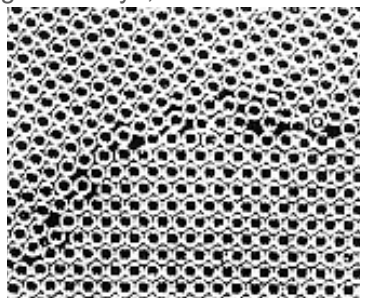
❖ Definition:

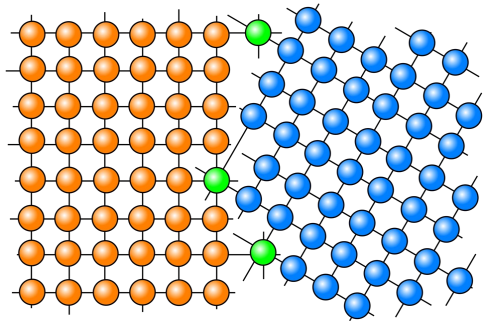
- GBs are interfaces where crystals of different orientations meet in a polycrystalline material.
- Five parameters to describe a GB
  - ✓ 1 disorientation angle (θ) → 1 parameter
  - ✓ Unitary rotation vector ($\boldsymbol{w}$) → 2 parameters
  - ✓ Unitary vector for the GB plane normal ($\boldsymbol{n}$)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

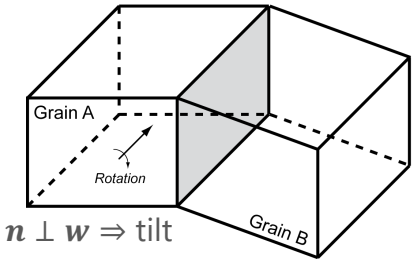
L. Priester, *Springer Science & Business Media.* (2012)

Grain boundaries

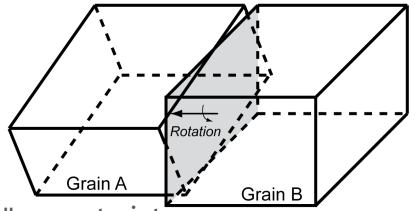
L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)







$\boldsymbol{n} \perp \boldsymbol{w} \Rightarrow$ tilt



$\boldsymbol{n} \parallel \boldsymbol{w} \Rightarrow$ twist

❖ Definition:

- GBs are interfaces where crystals of different orientations meet in a polycrystalline material.
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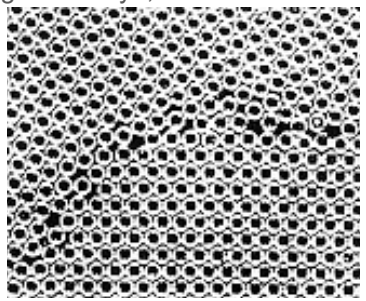
❖ Types:

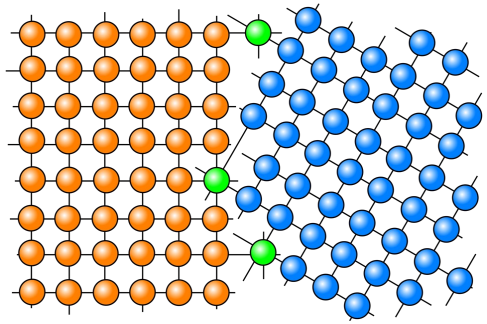
- *Tilt GB:* $\boldsymbol{n} \perp \boldsymbol{w}$
- *Twist GB:* $\boldsymbol{n} \parallel \boldsymbol{w}$
- *Mixed GB:* combination of tilt and twist components.

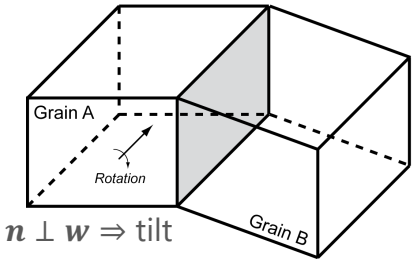
L. Priester, *Springer Science & Business Media.* (2012)

Grain boundaries

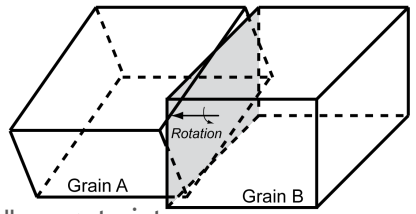
L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)



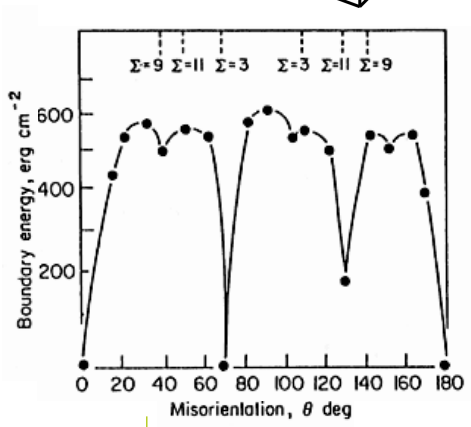


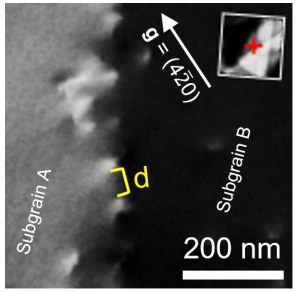


$\boldsymbol{n} \perp \boldsymbol{w} \Rightarrow$ tilt



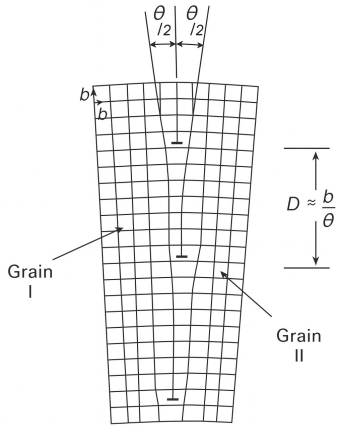
$\boldsymbol{n} \parallel \boldsymbol{w} \Rightarrow$ twist





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

$\Rightarrow \theta = 0.24 \pm 0.02°$



❖ 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

❖ Types:

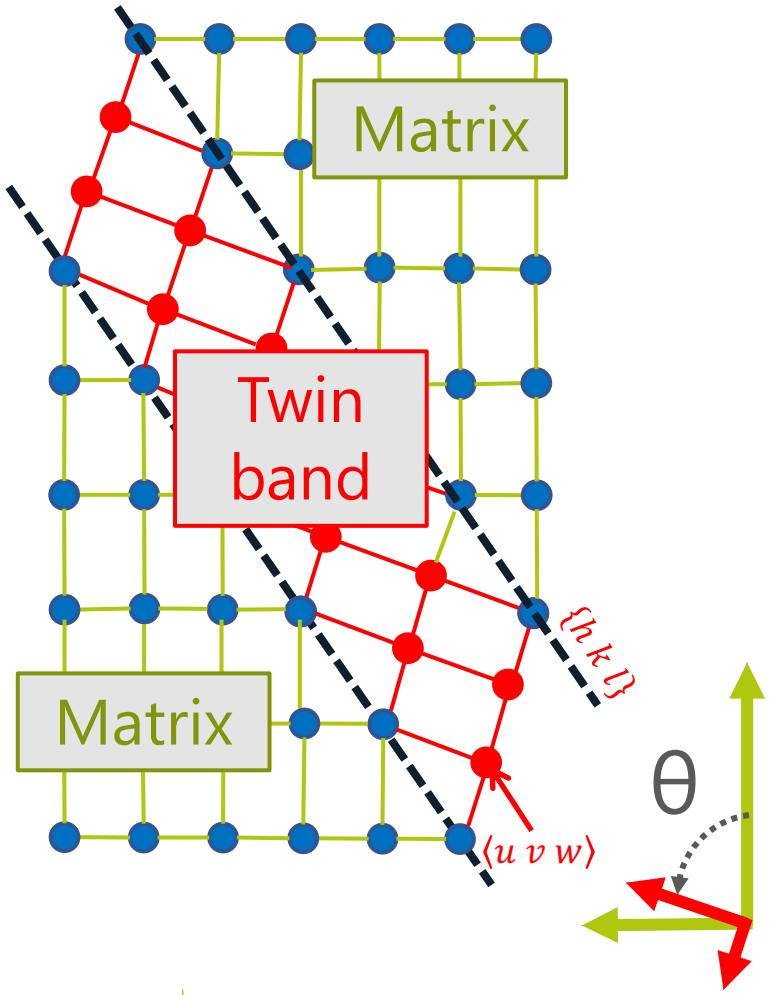
- *Tilt GB:* $\boldsymbol{n} \perp \boldsymbol{w}$
- *Twist GB:* $\boldsymbol{n} \parallel \boldsymbol{w}$
- *Mixed GB:* combination of tilt and twist components.
- Low-Angle GB (LAGB): $\theta \leqslant 15°$

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

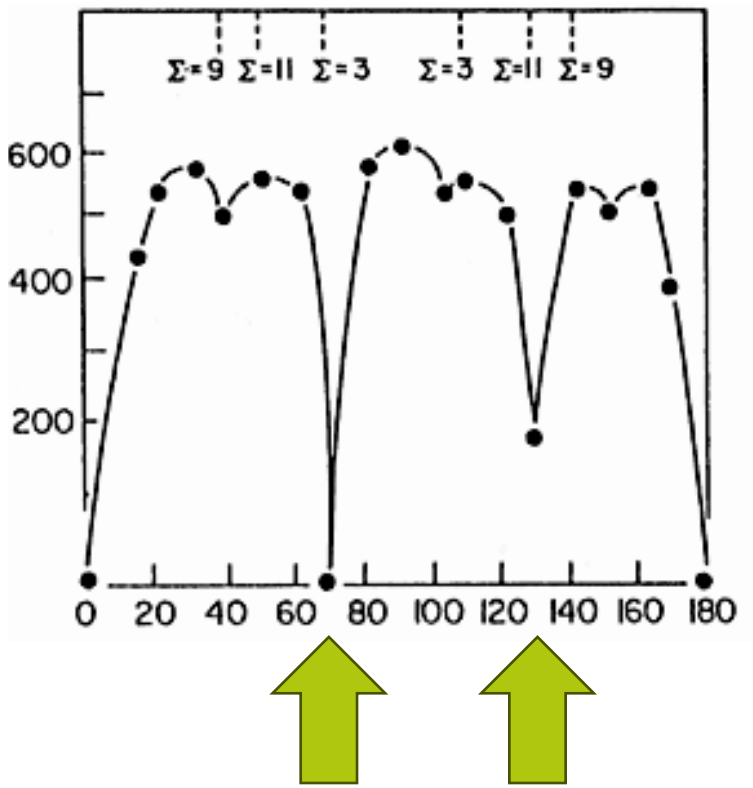
- High-Angle GB (HAGB): $\theta \geqslant 15°$

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

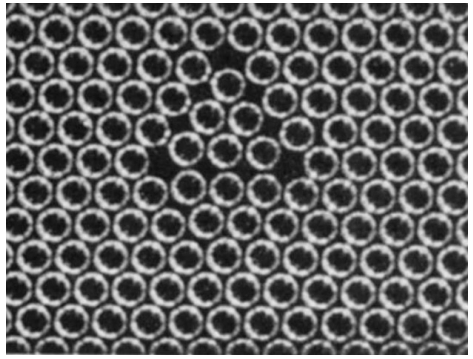
❖ Definition:

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

❖ Types:

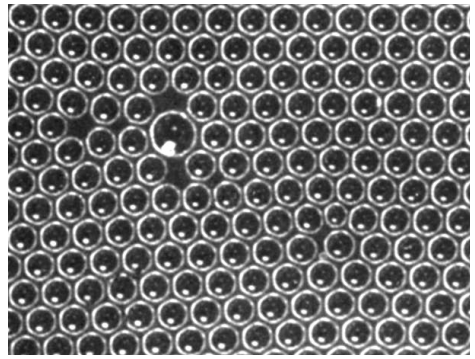
- *Vacancies:* Missing atoms in the lattice.
- *Interstitials:* Extra atoms positioned in the interstitial sites between the regular lattice points.
- *Substitutional defects:* Foreign atoms replacing host atoms in the lattice.

Vacancy



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

Interstitial



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

Punctual defects

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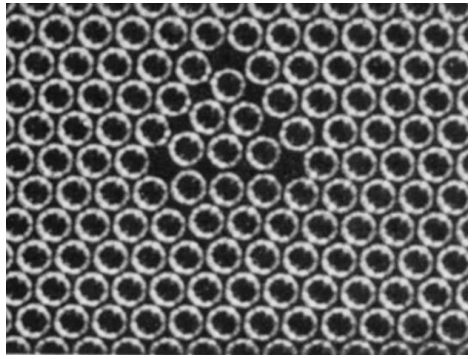
❖ **Types:**

- *Vacancies:* Missing atoms in the lattice.
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❖ **Diffusion:**

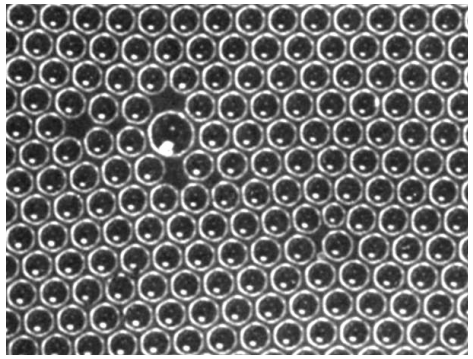
- They enable atomic diffusion, which is governed by Fick's Laws…

Vacancy



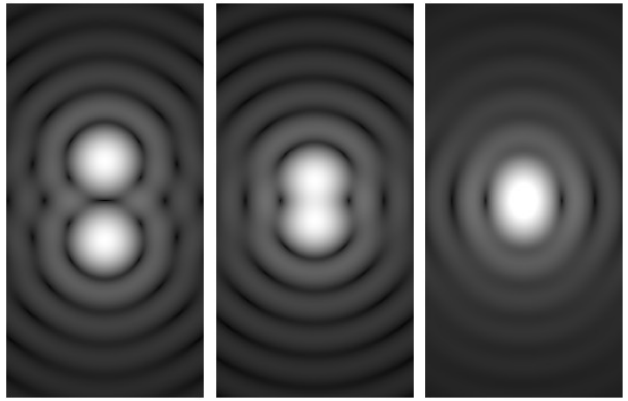
L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)

Interstitial



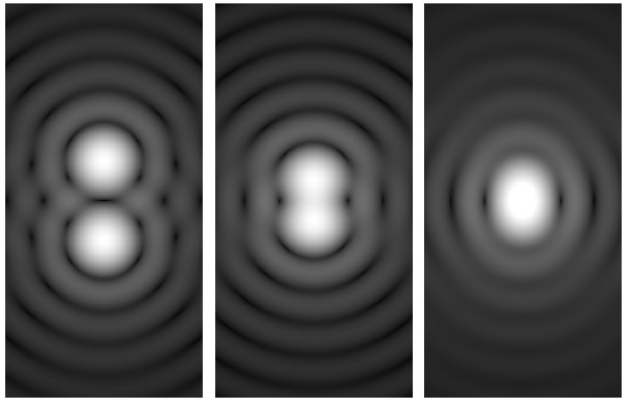
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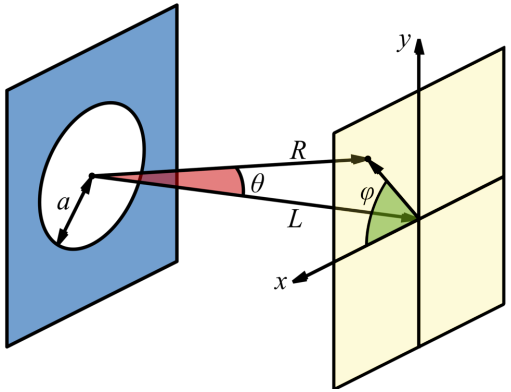
Limit of resolution



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

Limit of resolution



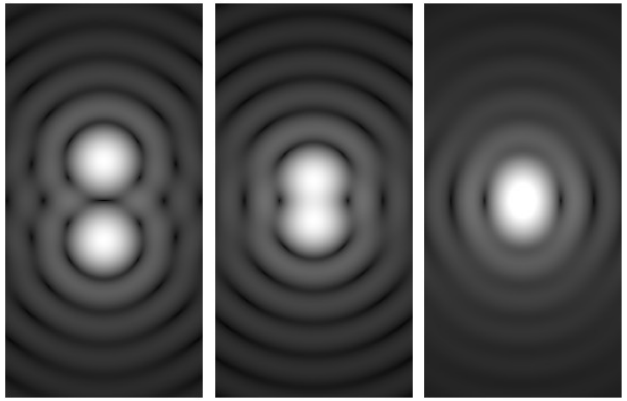


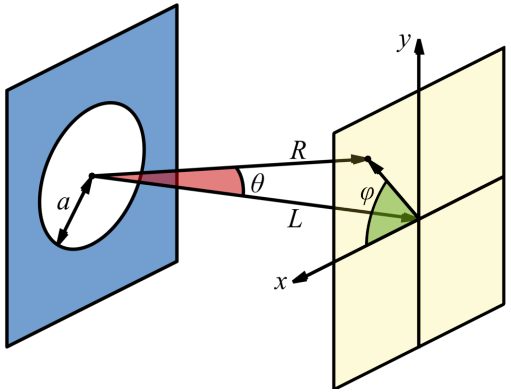
❖ Airy disk and Rayleigh criterion

- It refers to the ability to distinguish between very close details.

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

Limit of resolution





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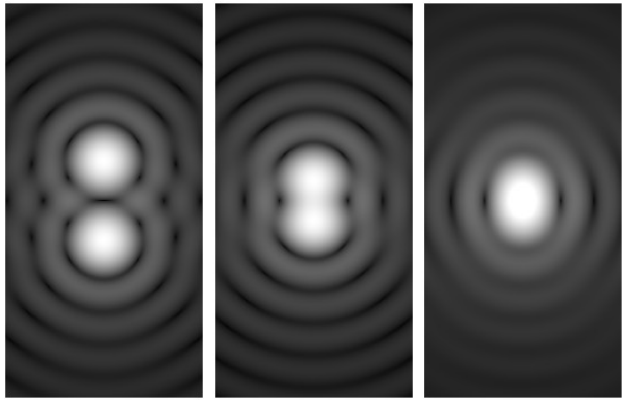
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$$\sin\theta = 0.61\frac{\lambda}{a} \sim \theta$$

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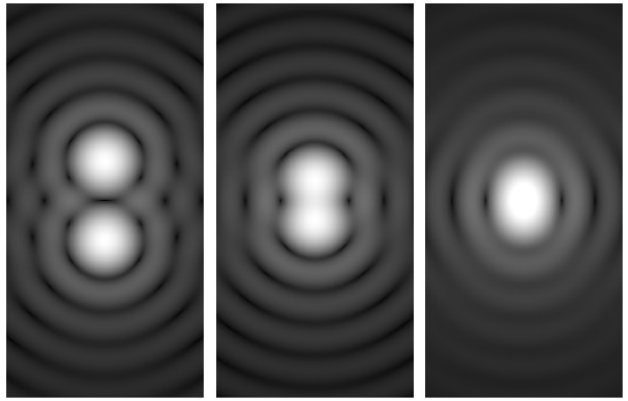
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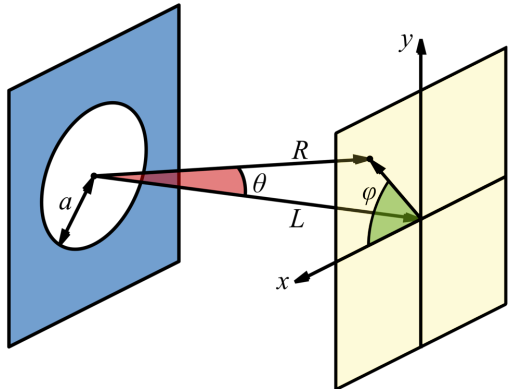
$$\sin\theta = 0.61\frac{\lambda}{a} \sim \theta$$

- The 1st minimum coincides with the maximum of the other.

$$\Rightarrow \Delta r = L\tan\left(0.61\frac{\lambda}{a}\right) \sim 0.61L\frac{\lambda}{a}$$

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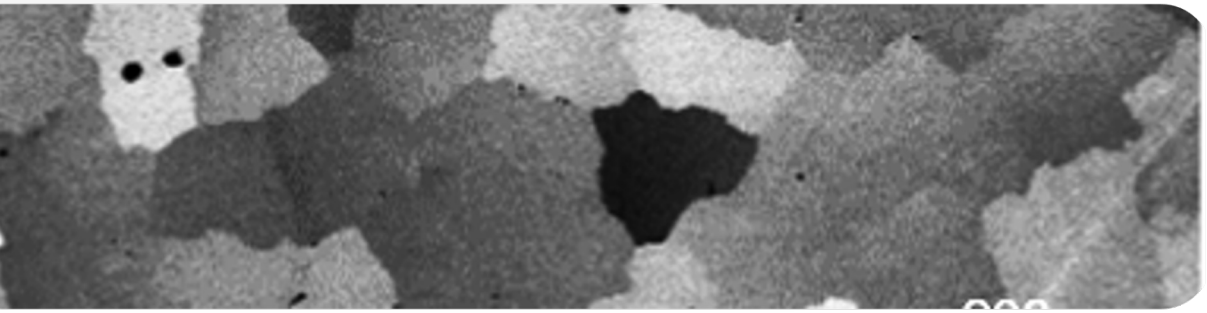
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↳ Two objects cannot be resolved if they are closer than one wavelength λ.



Characterization by X-rays diffraction

Meso- Micro-meter scale

www.antoine-guitton.fr

“Radiations studied by M. Röntgen”

❖ **On the evening of November 8, 1895, in Würzburg, Germany:**

- Röntgen made a screen fluoresce near a Crookes tube inside a black box.
- Then, he played with various objects revealing their transparency.



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

W.C. Röntgen, *Ann. Phys. (*1898)

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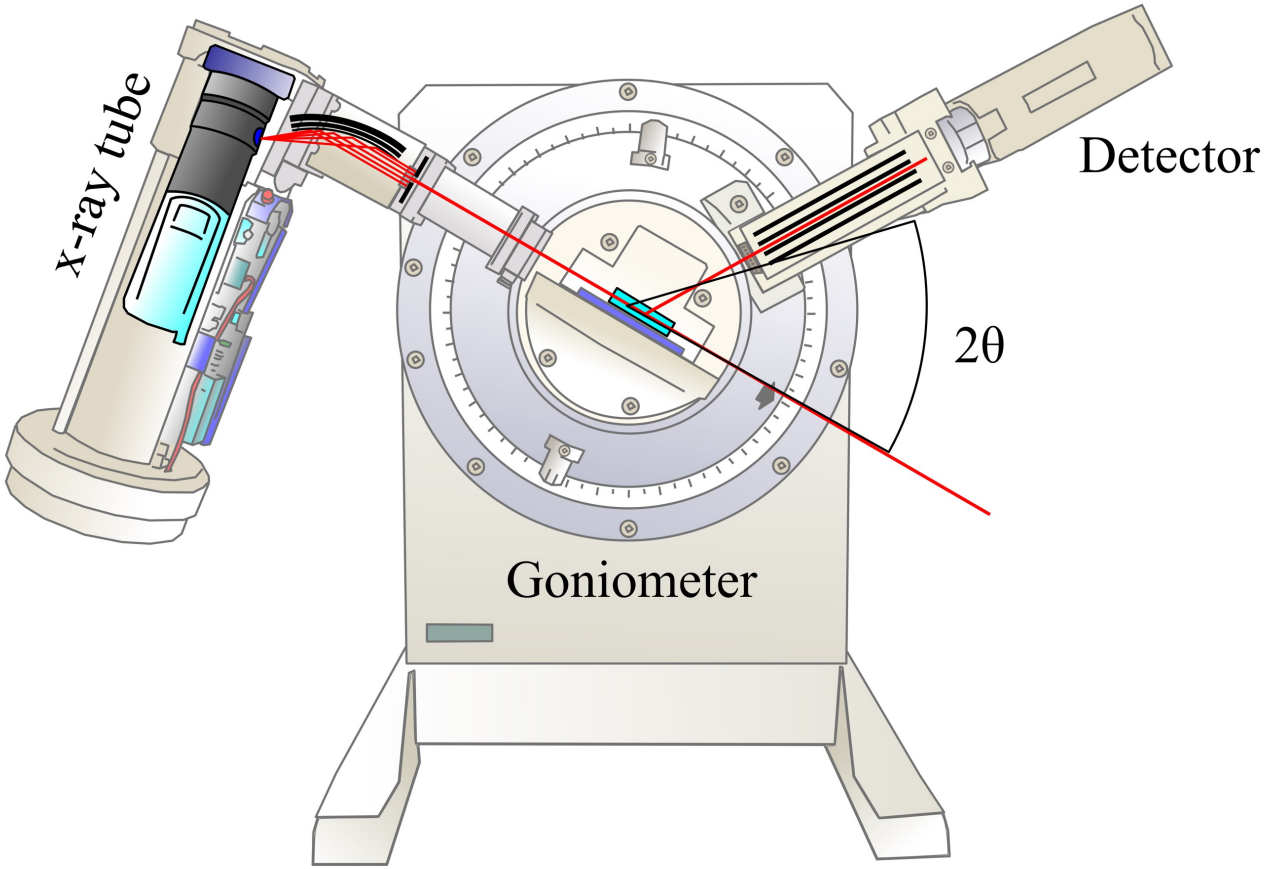


↳ Since Röntgen did not understand their nature, he called these rays, X-rays!

W.C. Röntgen, *Ann. Phys. (*1898)

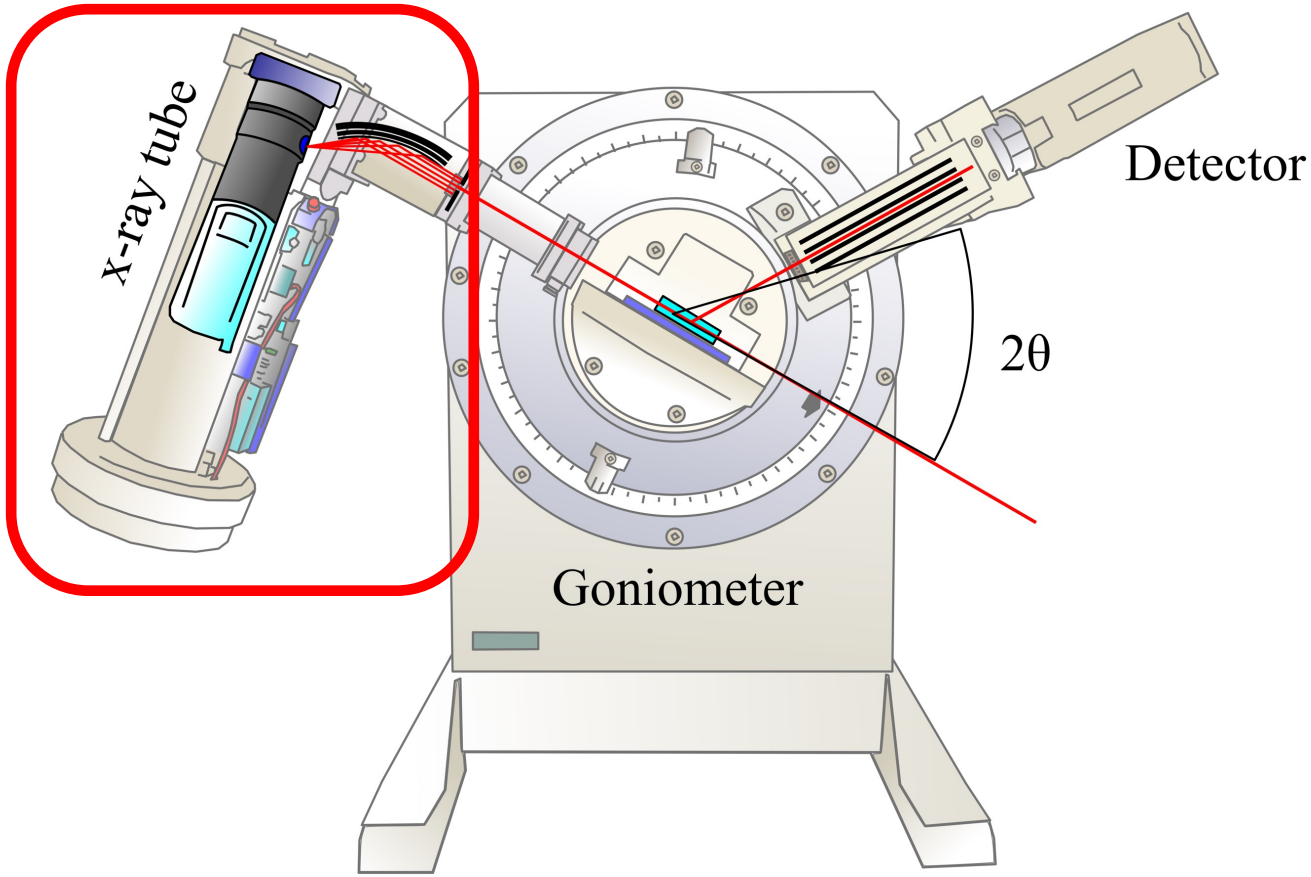
The experimental setup: the diffractometer

The diffractometer (Bragg-Brentano configuration)

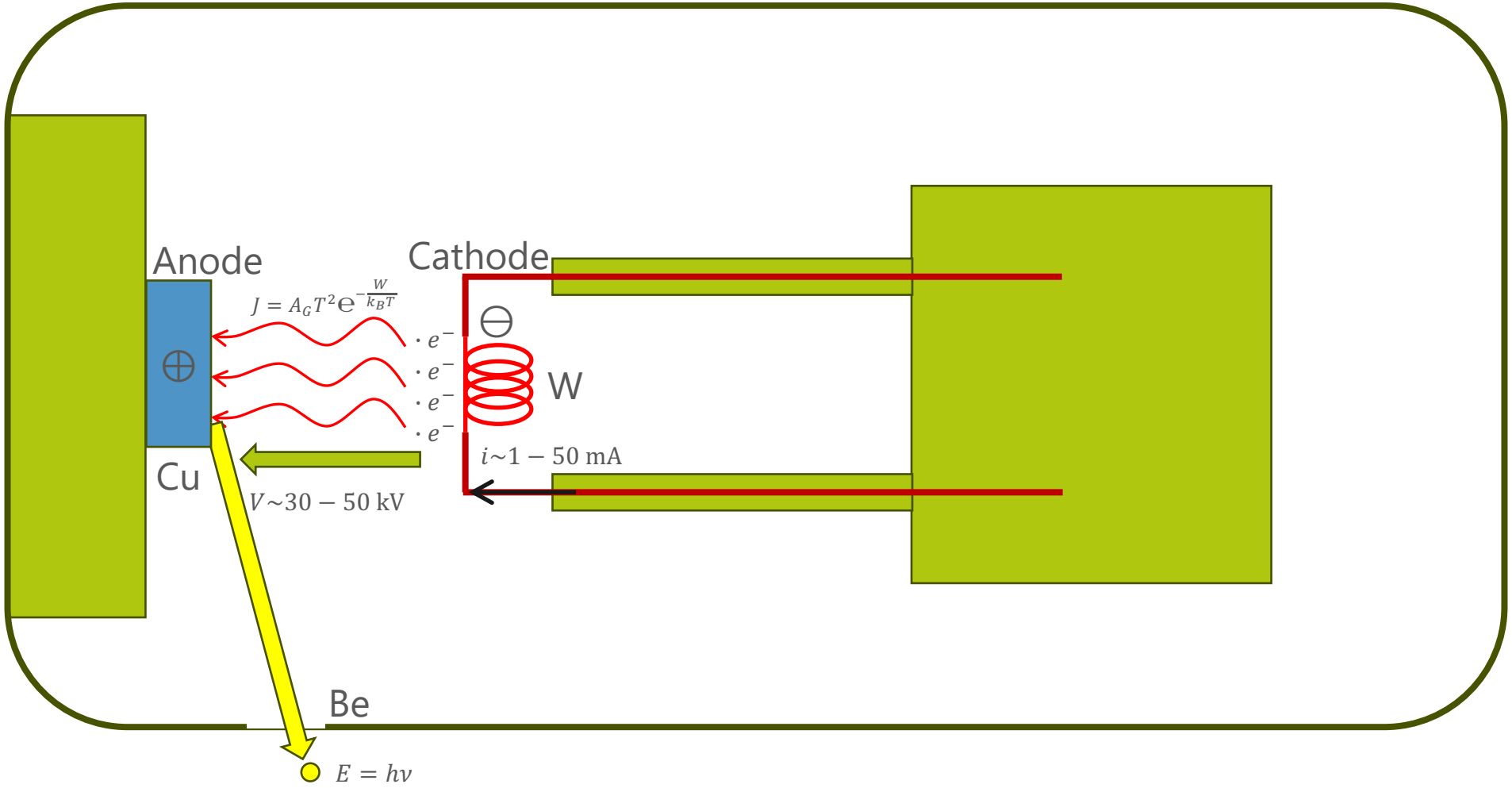


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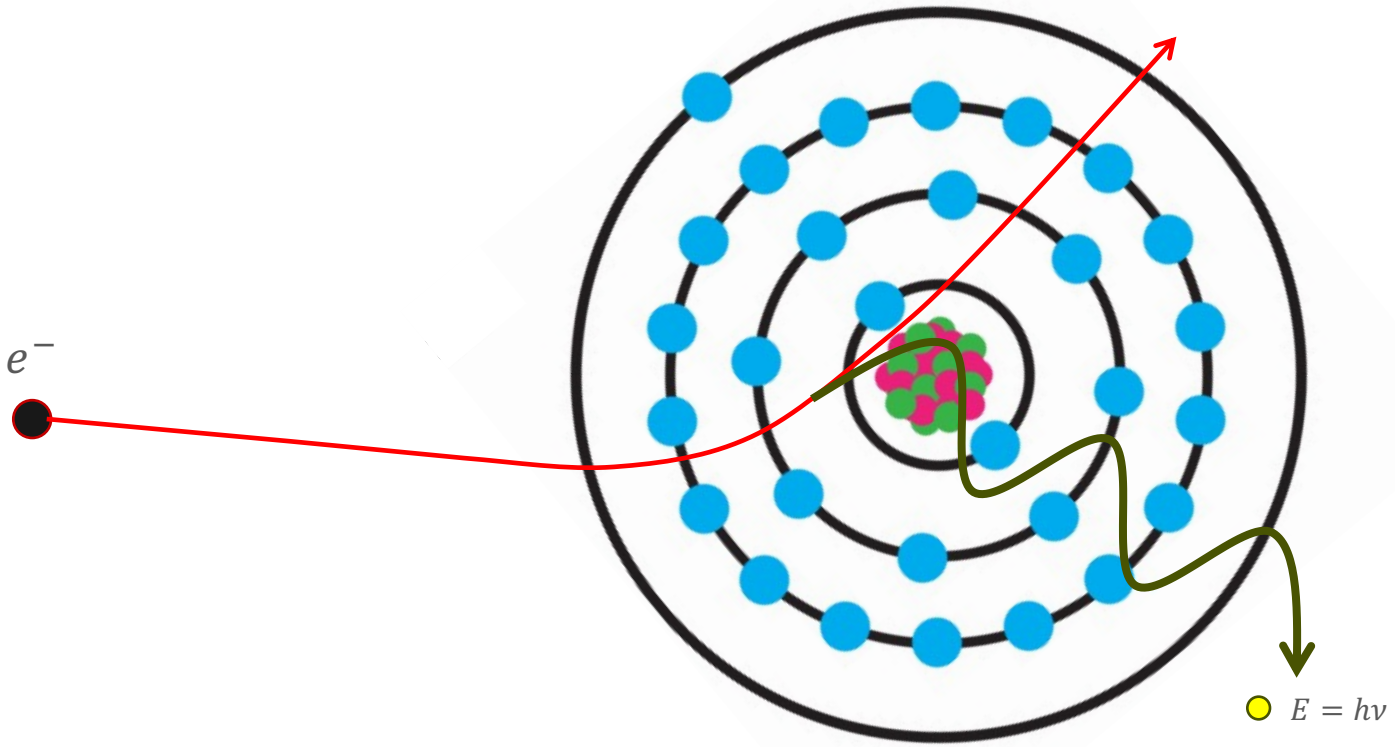
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X-ray production



X-ray production



Bremsstrahlung radiation

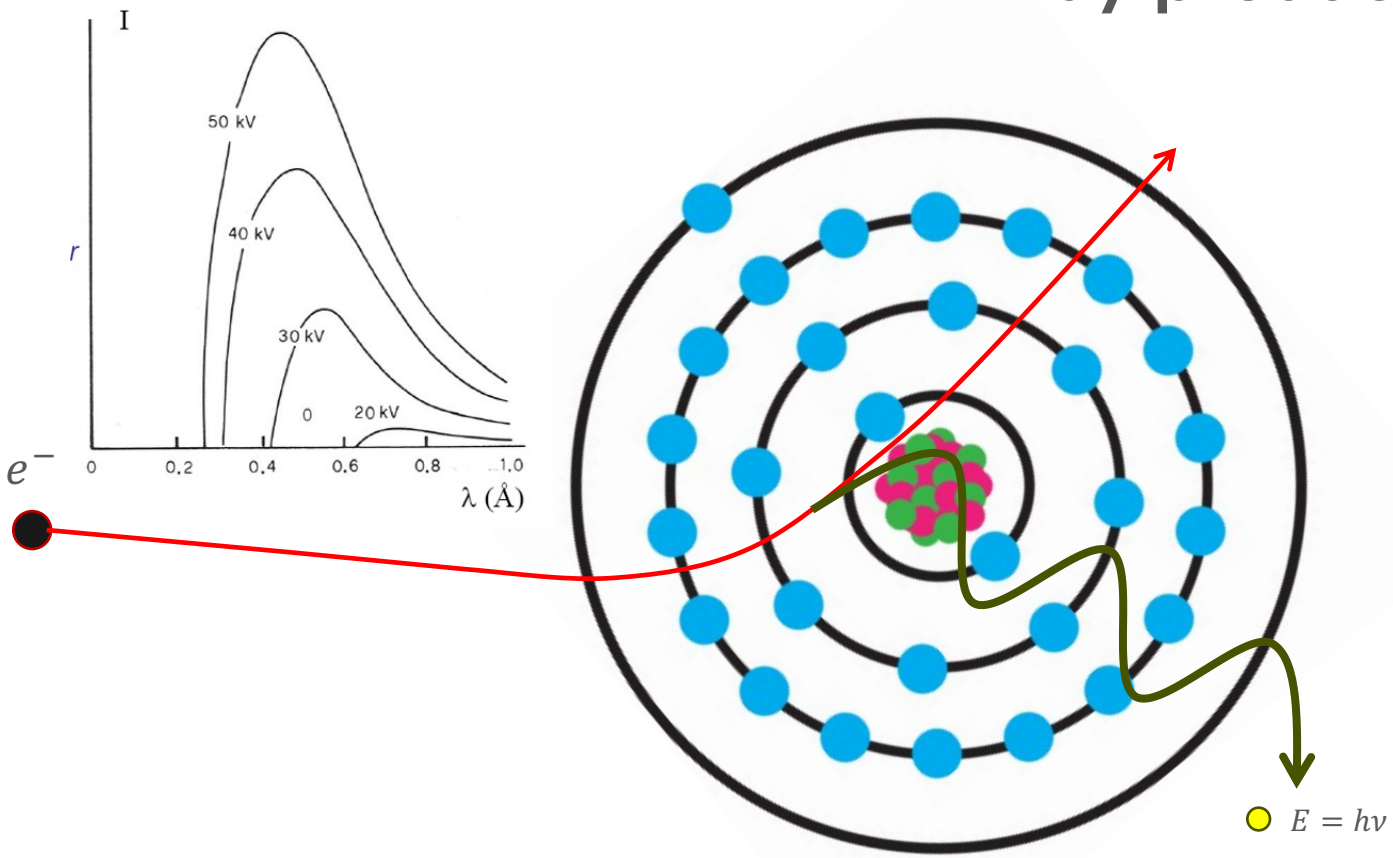
❖ **Definition:**

- 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



Bremsstrahlung radiation

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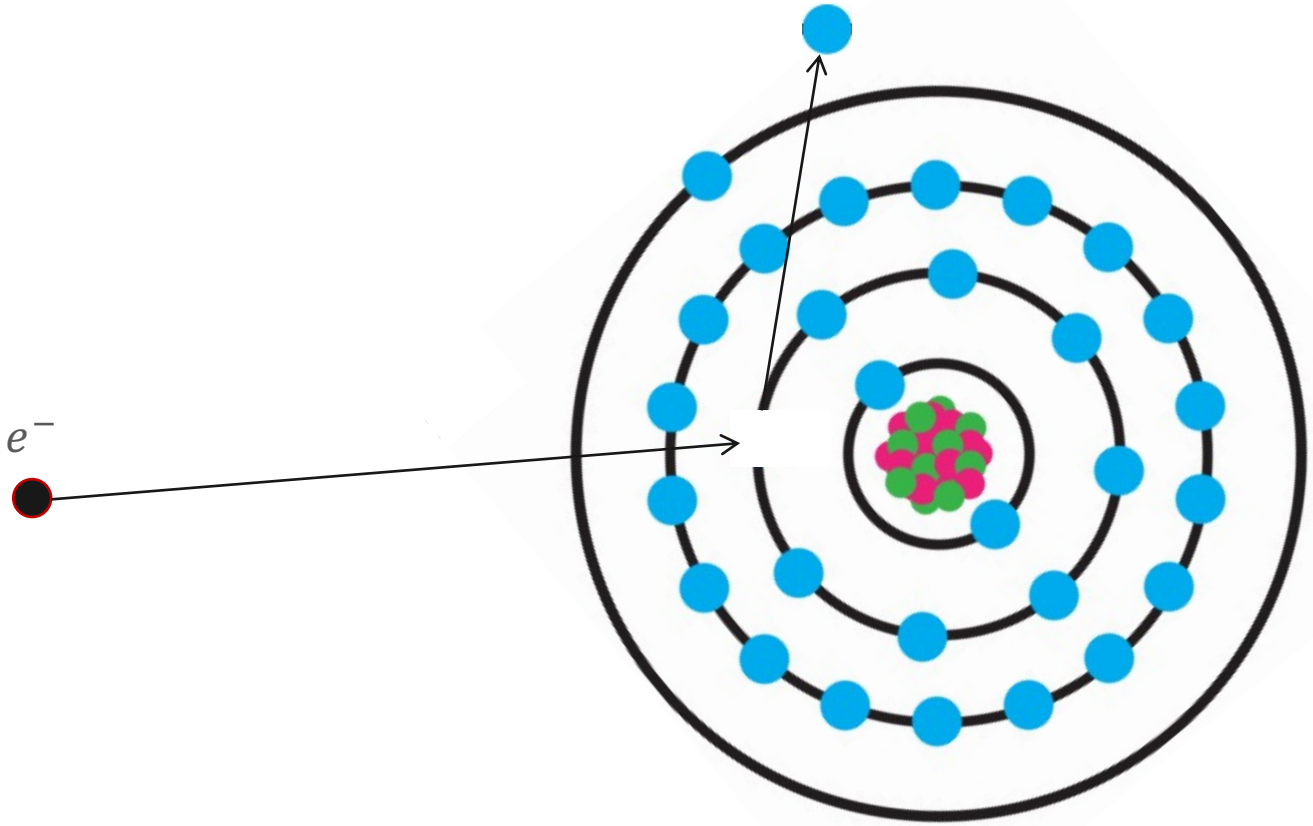
- Electrons lose kinetic energy when interacting with atomic nuclei.
- Lost energy is emitted as X-ray photons.

❖ 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.

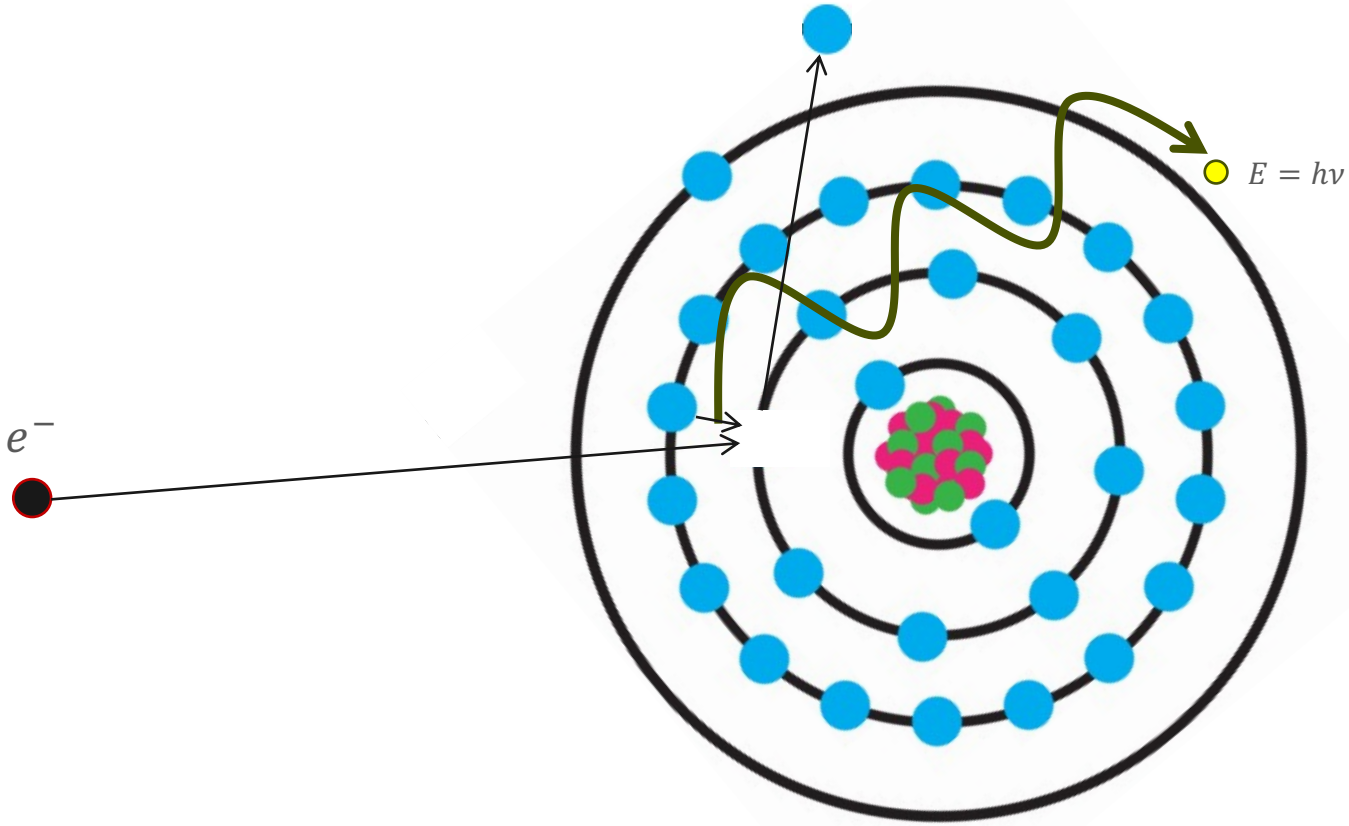
X-ray production



Characteristic radiation

- **Definition:**
  - X-rays emitted as an outer electron fills an inner-shell vacancy.
- **Mechanism:**
  - The incident electron removes an inner-shell electron.

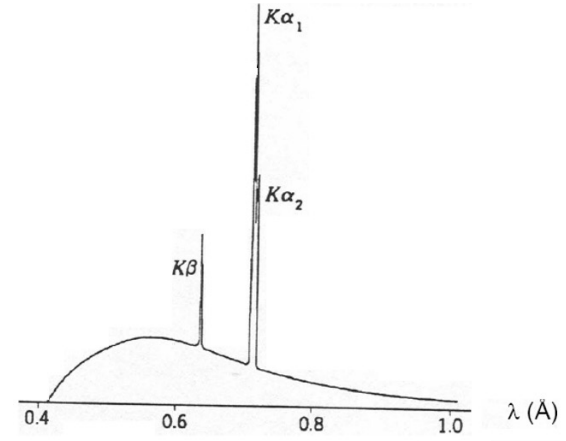
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X-ray production



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  - A higher-energy electron fills the vacancy.
  - The energy difference emits a defined X-ray photon.
- **Characteristic:**
  - Distinct peaks
  - Element-specific

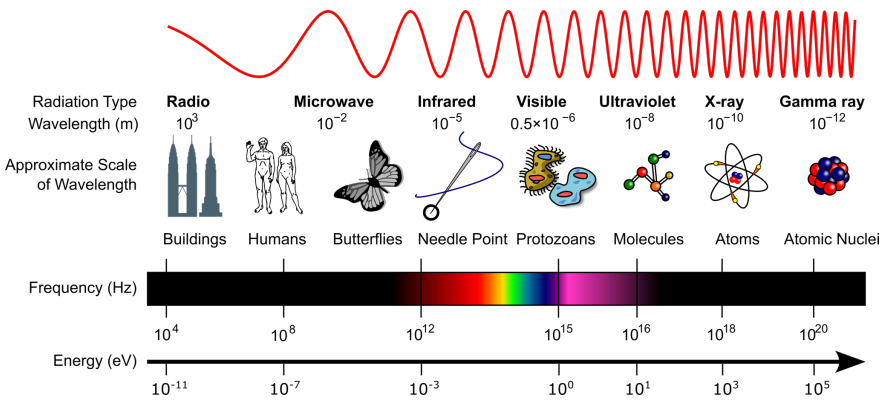
↳Characteristic X-rays enable elemental analysis, aiding in materials characterization.

What are x-rays?

Electromagnetic waves

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

$$1\ eV = 1.60 \times 10^{-19}\ \text{J}$$



$$E = h\nu = \hbar\omega$$

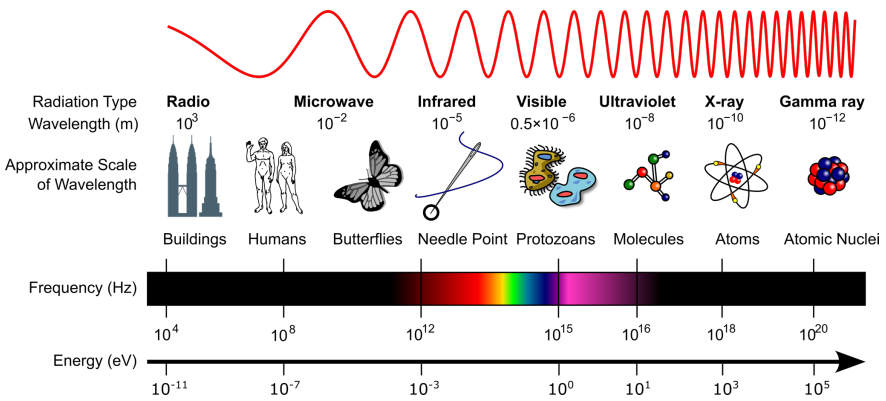
where h: Planck's constant; ν:frequency; ω: pulsation

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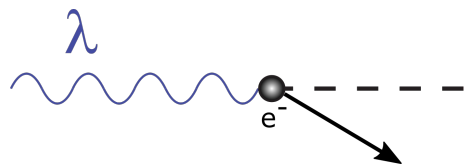


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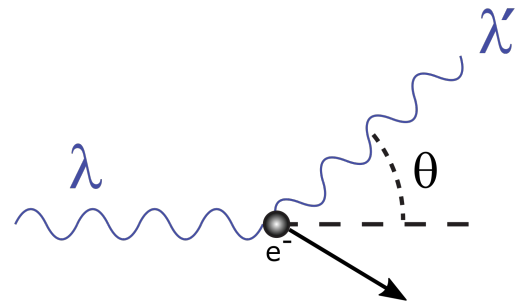
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Photon-matter interaction

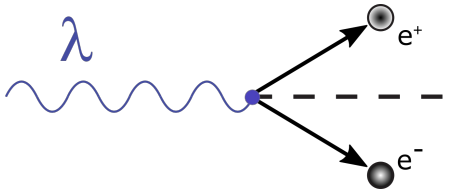
- **Photoabsorption effect (10 eV – 100 keV):** A photon transfers all its energy to an electron, ejecting it if the energy exceeds the electron's binding energy.



- **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.



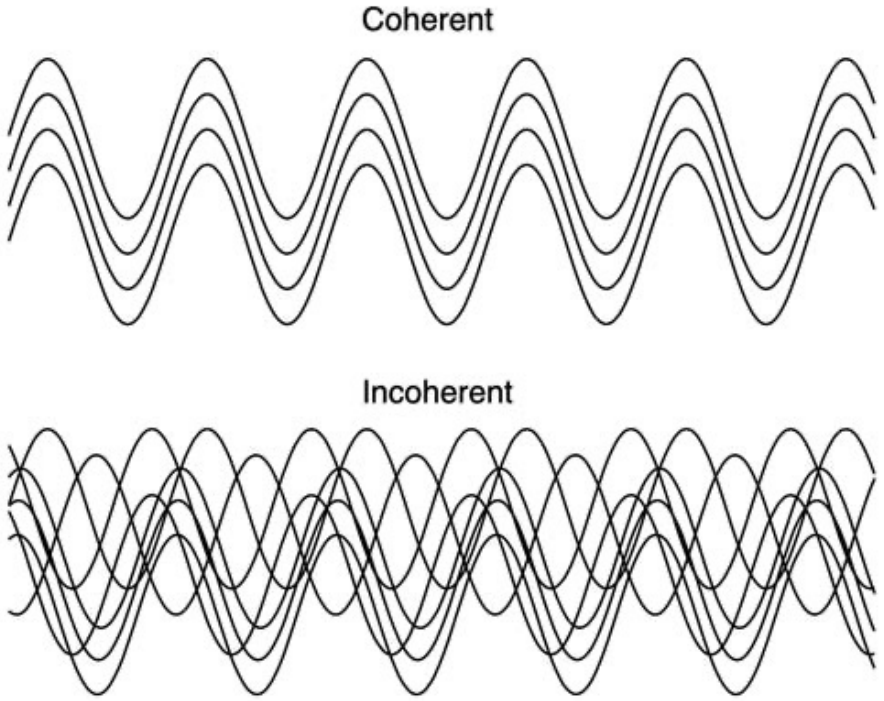
- **Pair production (>1 MeV):** A high-energy photon interacts with an atom's nucleus and converts into an electron-positron pair.



Coherency

❖ **Definition:**

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



Coherency

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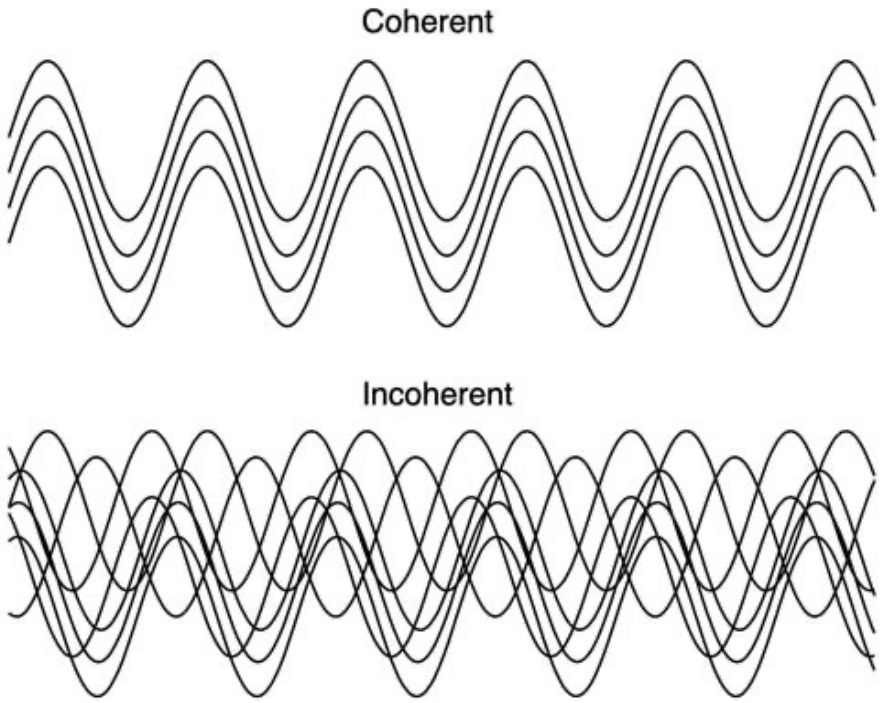
- Coherence describes the stability of an X-ray wave's phase in time and space, crucial for producing clear interference patterns.

❖ **Types of coherence:**

- Temporal coherence (energy stability)
  - ✓ Determines the ability of X-rays to interfere over time.
  - ✓ Related to the spectral width: narrow bandwidth → High temporal coherence.
  - ✓ Expressed by the coherence length:

$$l_c = \frac{c}{\Delta f}$$

  - ✓ Important for monochromatic X-ray sources in diffraction.



Coherency

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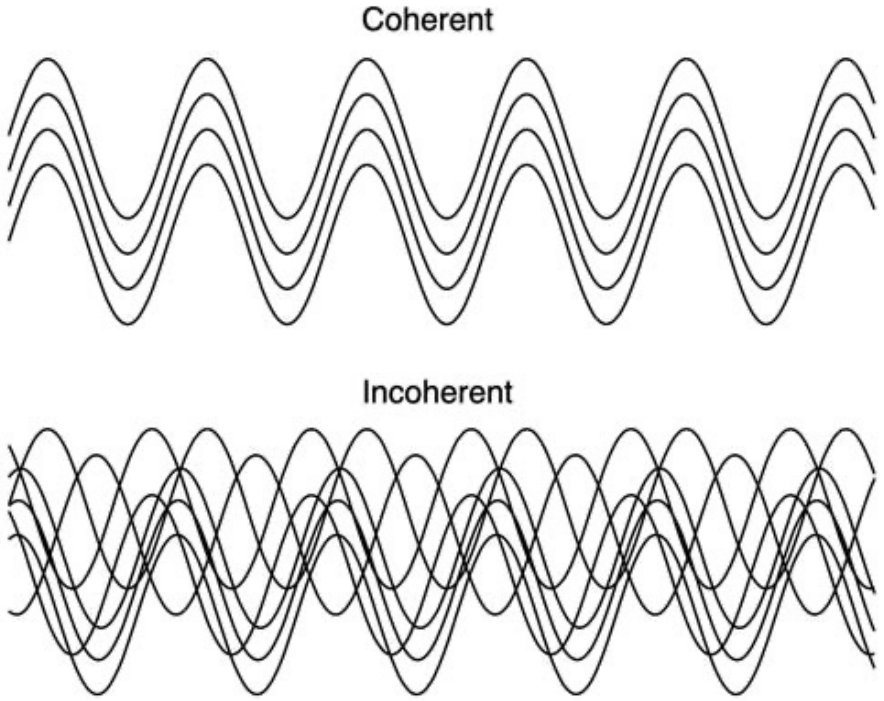
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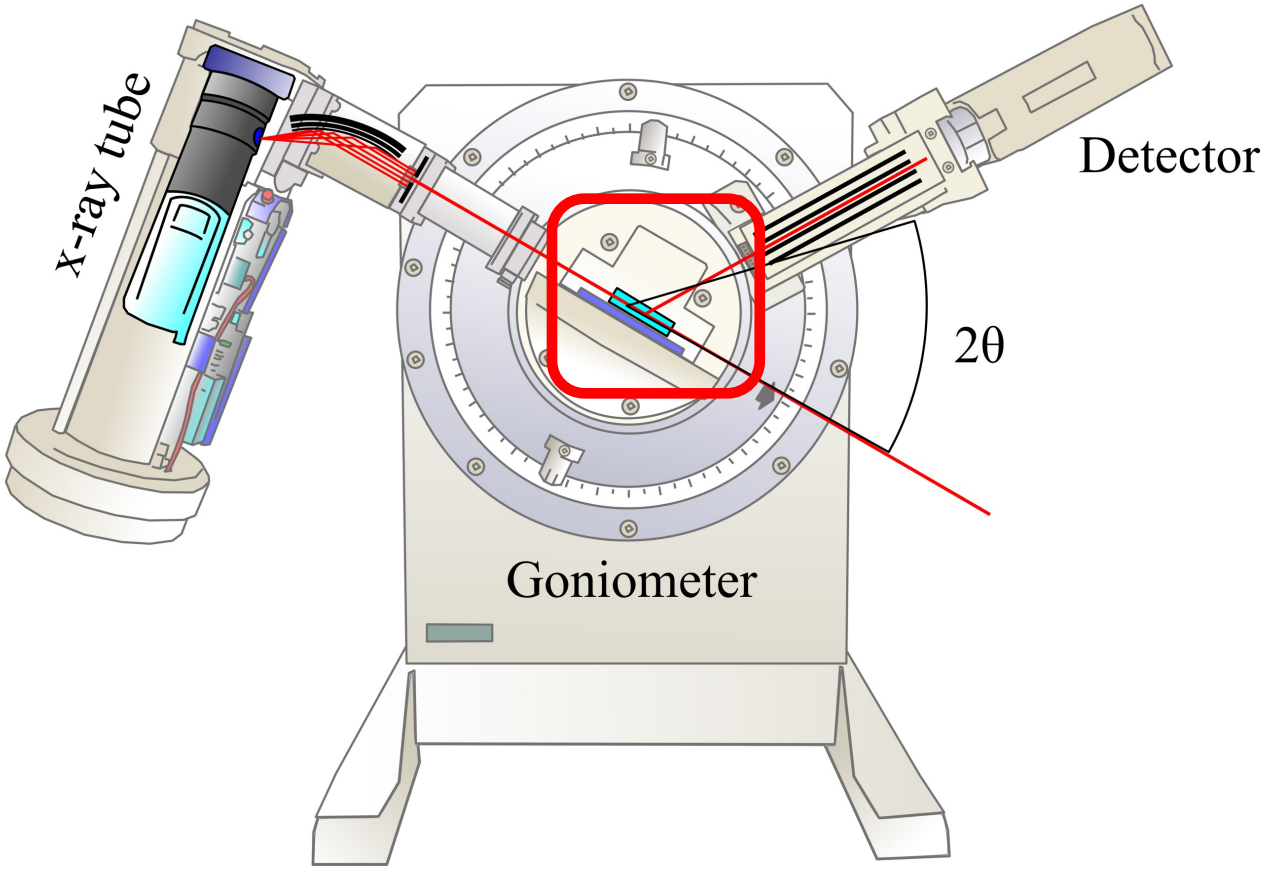
$$l_c = \frac{c}{\Delta f}$$

  - ✓ Important for monochromatic X-ray sources in diffraction.
- 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).

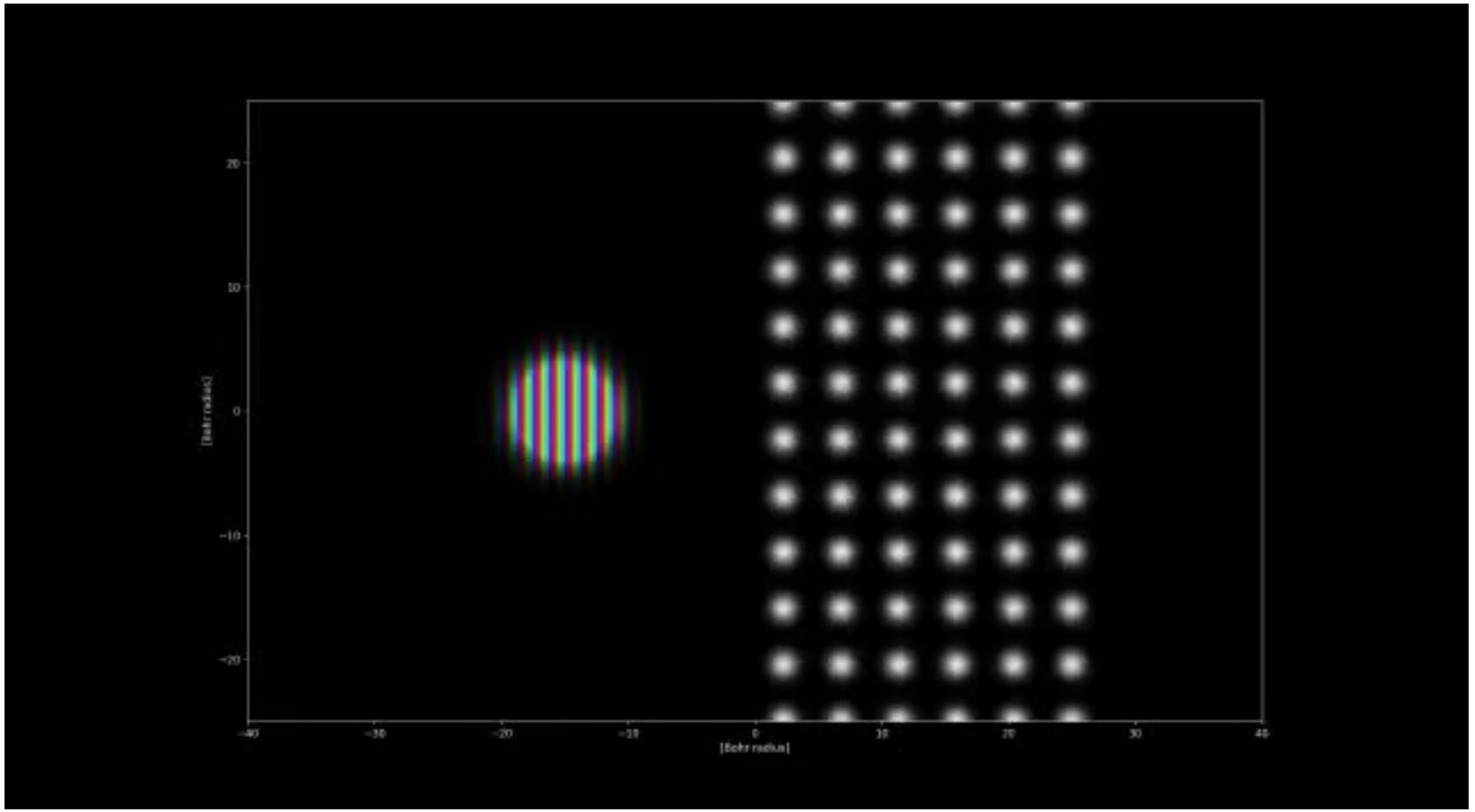


The experimental setup: the diffractometer

The diffractometer (Bragg-Brentano configuration)



The diffraction

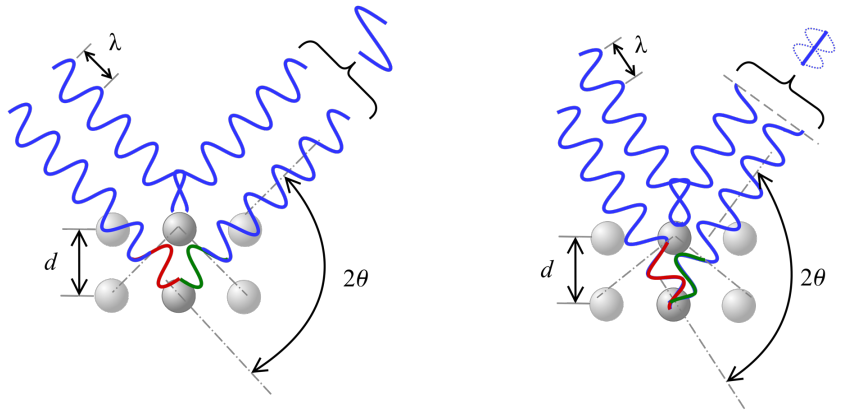


Interaction X-ray matter

- **Liquids or amorphous materials:**
  - X-rays are scattered in many directions, leading to broad, diffuse halos.

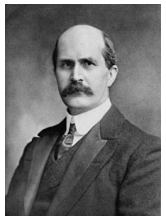
W.H. Bragg, W.L. Bragg, *Proc. R. Soc. Lond. A.* (1913)

Interaction X-ray matter



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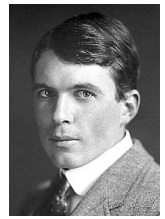
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(1862 – 1940)

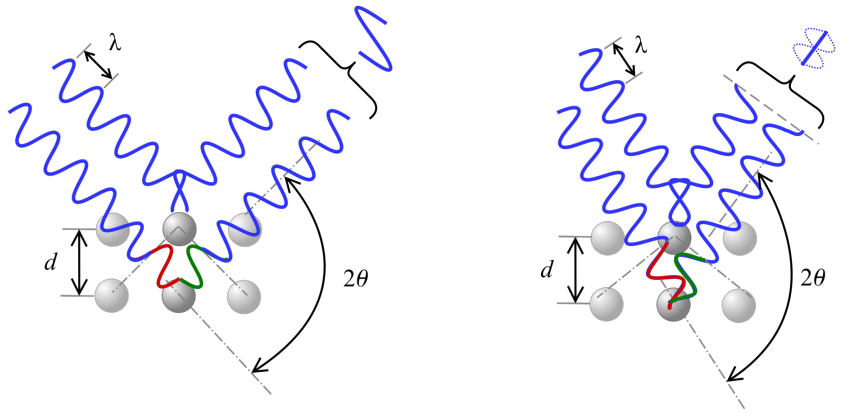
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2/2 Nobel Prize (1915)



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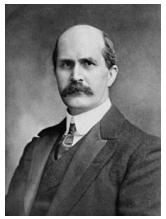
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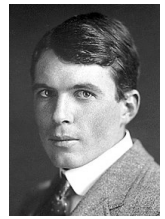
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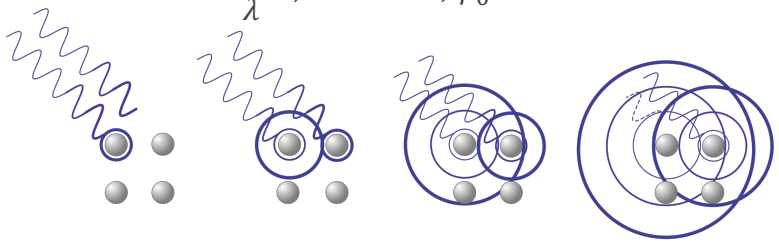
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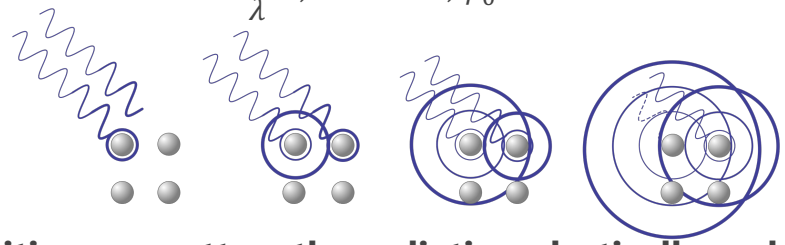


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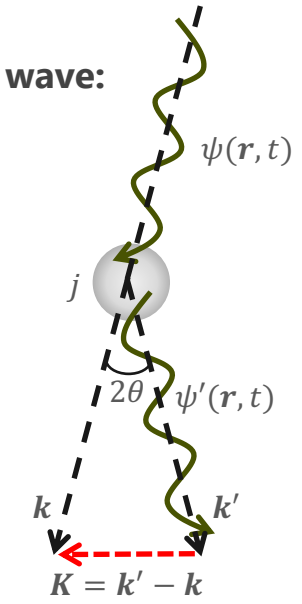
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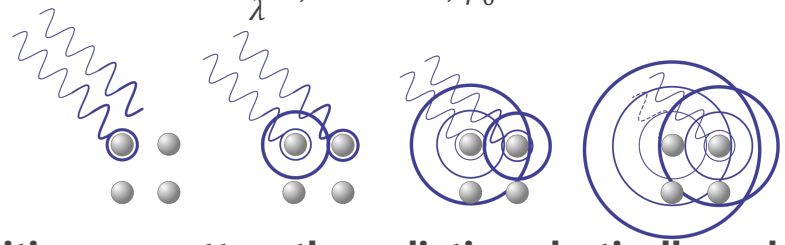


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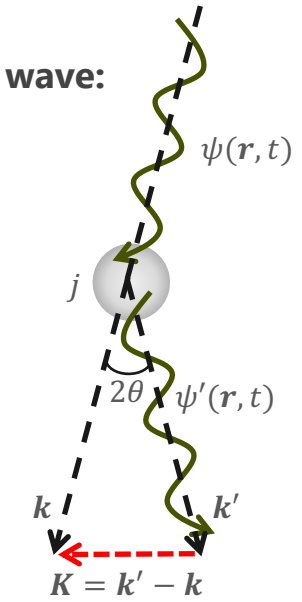
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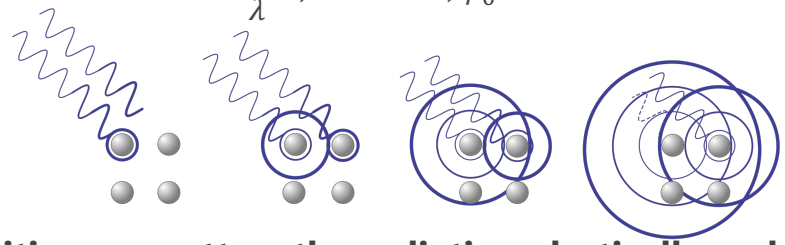
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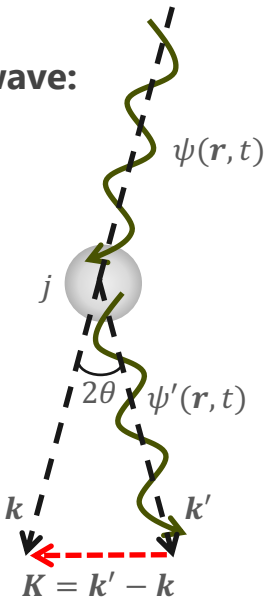
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$$I(\boldsymbol{K}) \propto |\psi'(\boldsymbol{r},t)|^2$$

Diffraction condition

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  - The atoms inside each unit cell are positioned at $\boldsymbol{r_j}$

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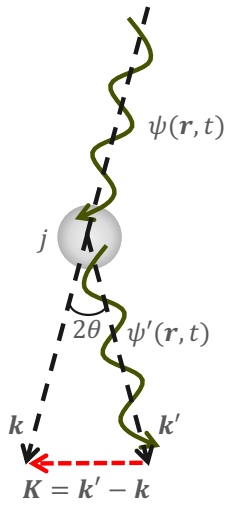
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- Constructive diffraction occurs when $\boldsymbol{K}$ corresponds to a vector of the reciprocal lattice.

$$\Rightarrow \boldsymbol{K} = \boldsymbol{G}$$

- We can show that: $\|\boldsymbol{G}\| = \frac{2\pi}{d}$

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



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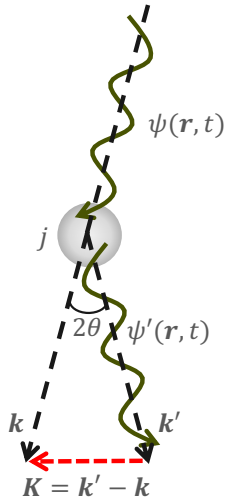
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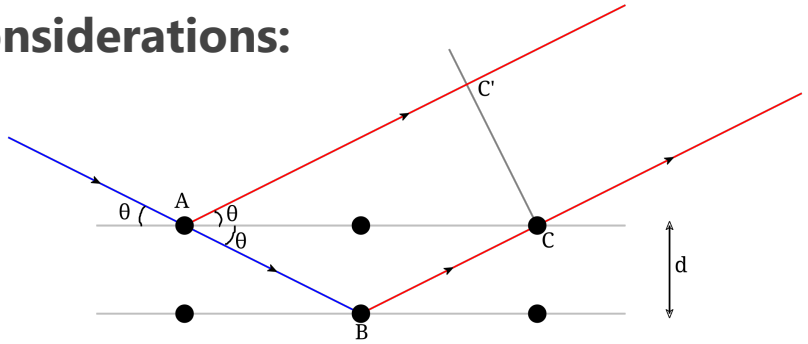
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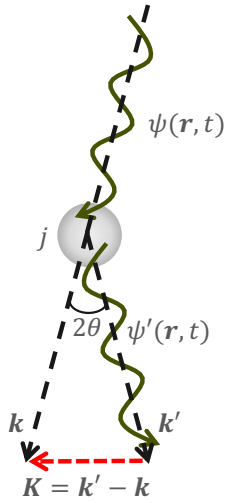
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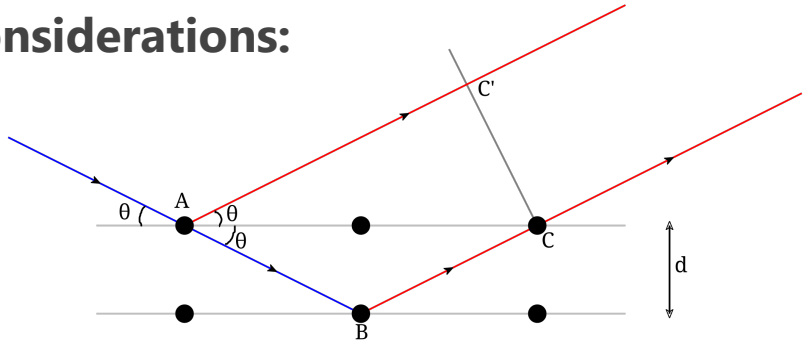
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❖ **From geometrical considerations:**



- 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 $\Rightarrow n\lambda = (AB) - (BC) - (AC')$

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

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:**

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

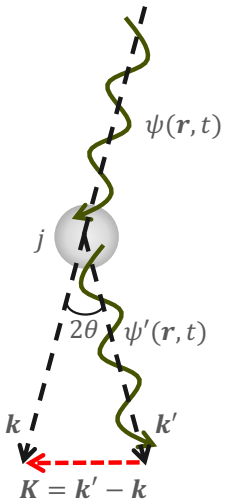
Elastic scattering $\Rightarrow \|\boldsymbol{k'}\| = \|\boldsymbol{k}\| = k$

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

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

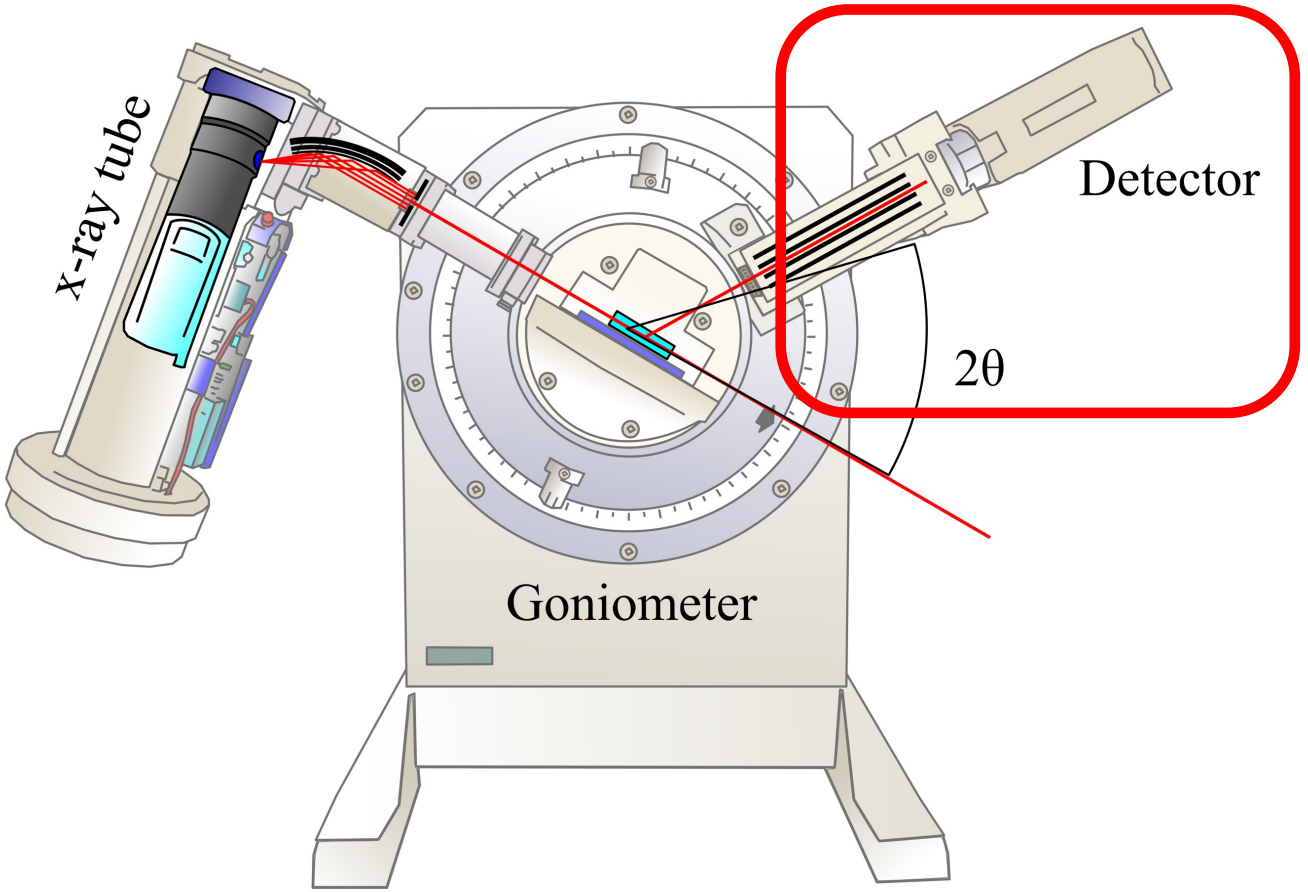
$$\Rightarrow \frac{2\pi}{d_{hkl}} = 4k^2 \sin^2 \theta = 4\left(\frac{2\pi}{\lambda}\right)^2 \sin^2 \theta$$

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



The experimental setup: the diffractometer

The diffractometer (Bragg-Brentano configuration)



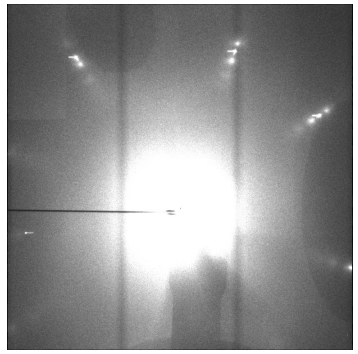
X-ray diffraction techniques

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**



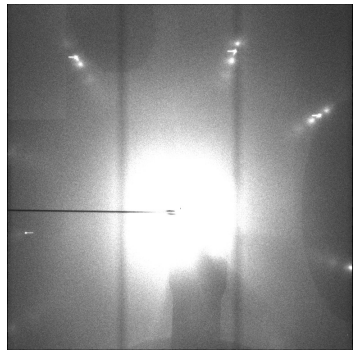
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Polychromatic beam

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

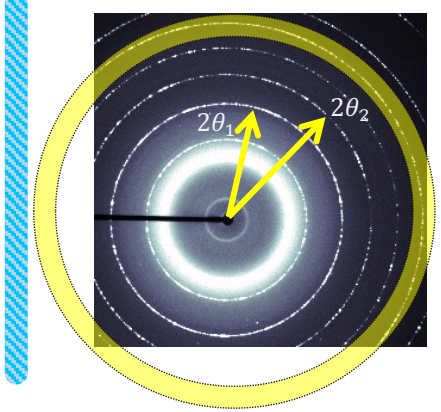
⇒ **Isolated spots corresponding to the $\{\lambda, \theta, d\}$ combinations**



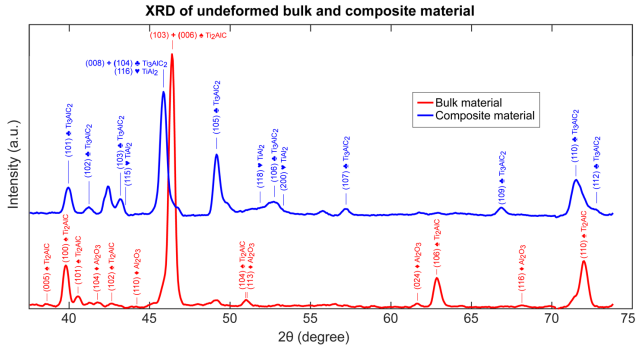
Monochromatic beam

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



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



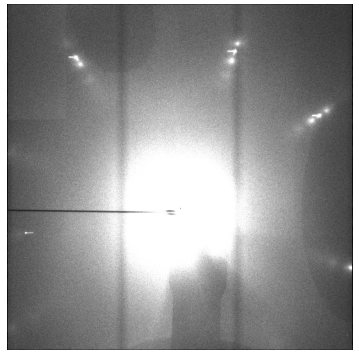
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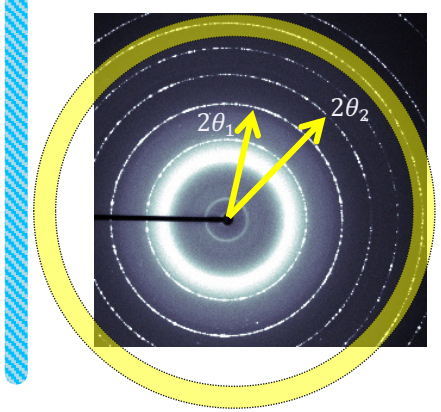
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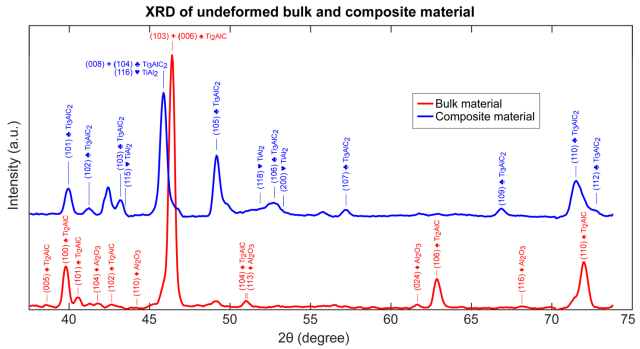
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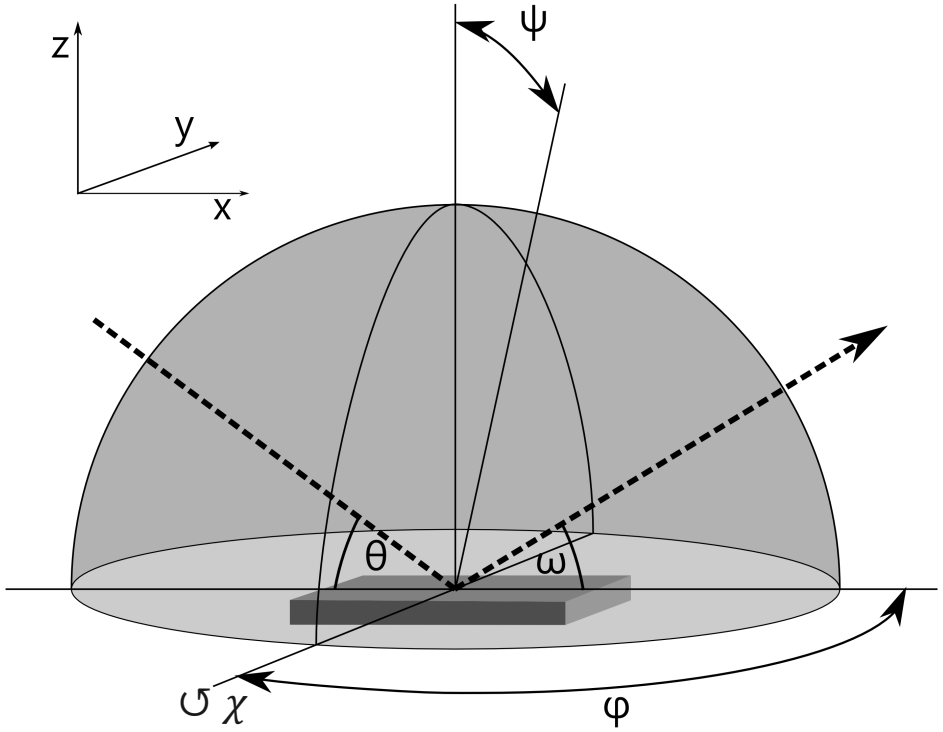


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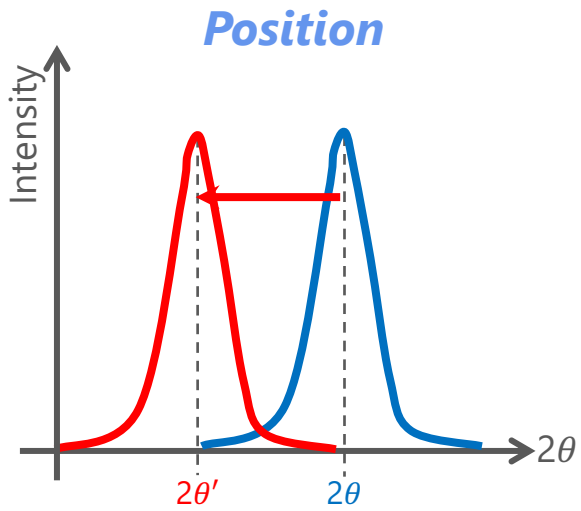
↳ One crystal, many wavelengths – that's Laue.
↳ Many crystals, one wavelength – that's powder.

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

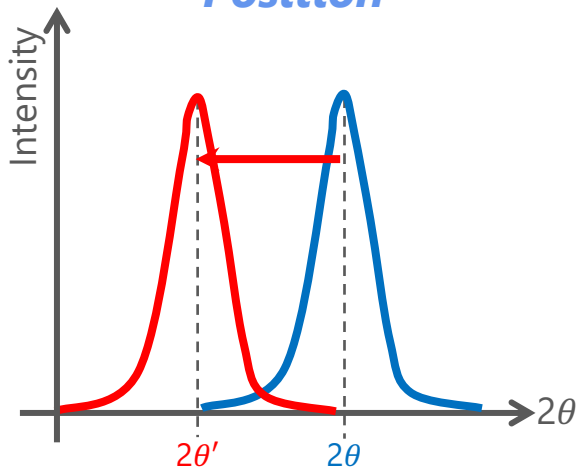


- **Peak shift:**
  - $n\lambda = 2d_{hkl}\sin\theta$
    - ✓ $2\theta \searrow \Rightarrow \sin\theta \searrow \Rightarrow d_{hkl} \nearrow \Rightarrow$ macrotraction
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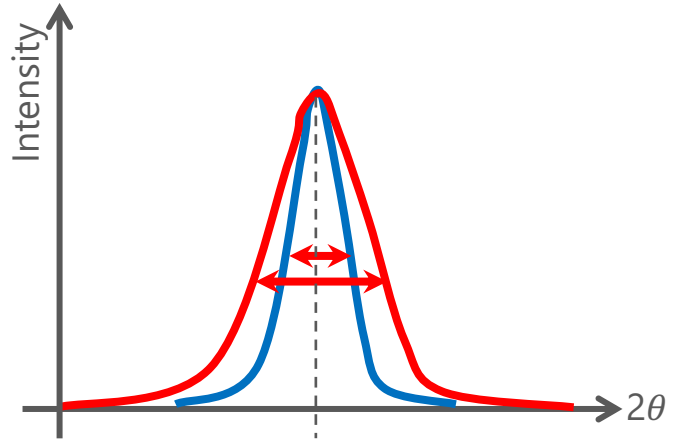
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- **Peak width:**
  - Crystallite size (L)

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

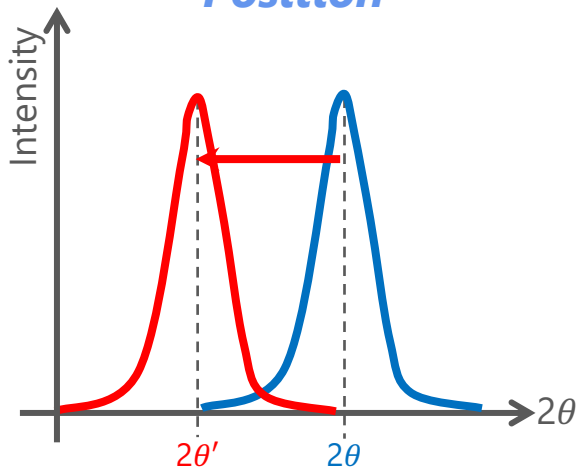
  - Lattice strain heterogeneities
    - ✓ Dislocation density (ρ)

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

    - ✓ Microstrain

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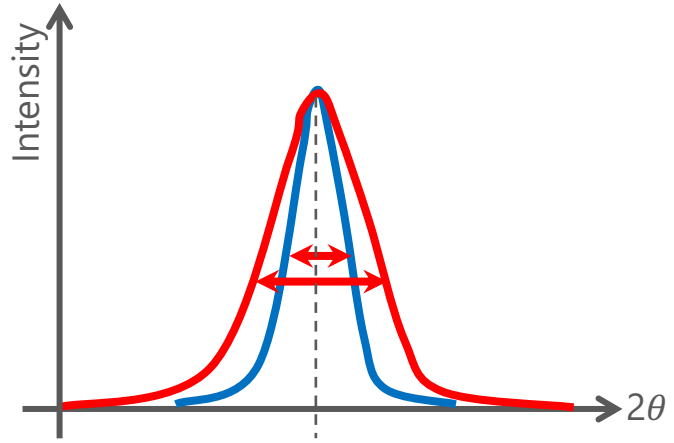
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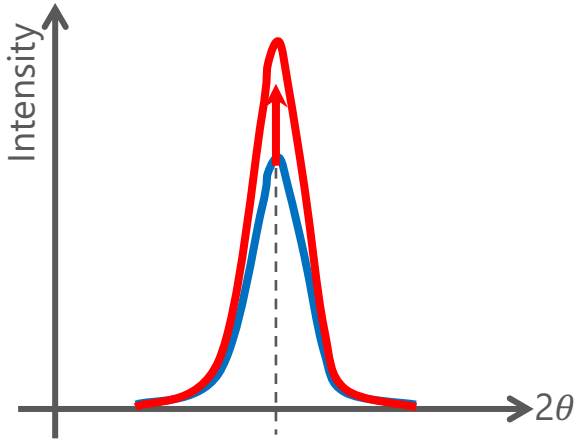
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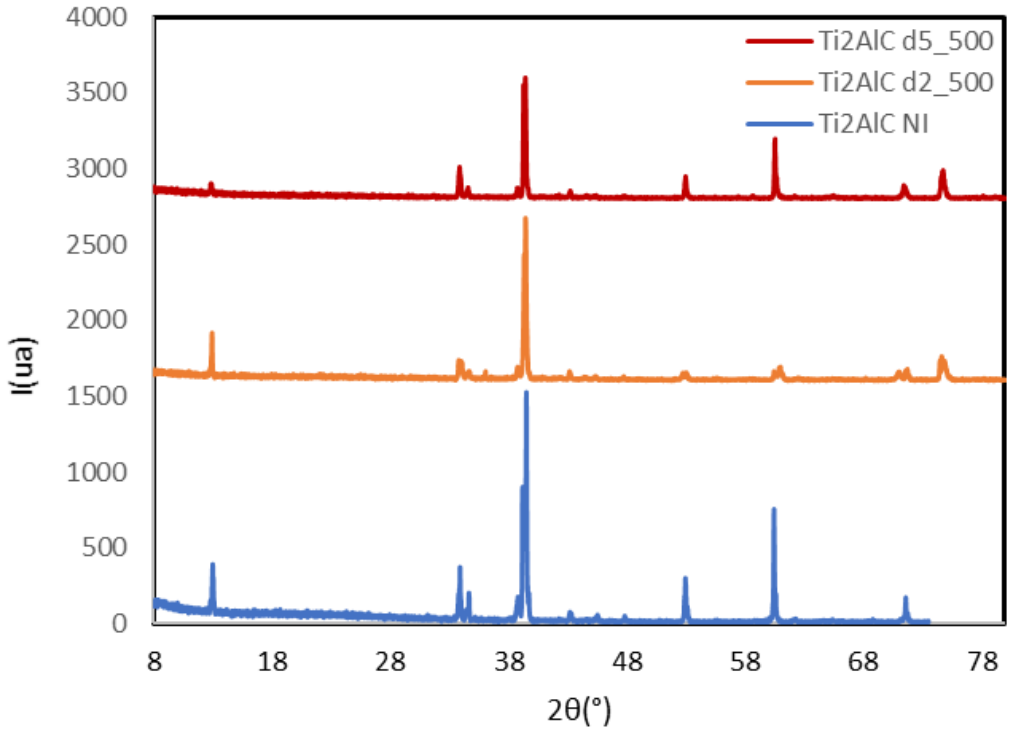
Height



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

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

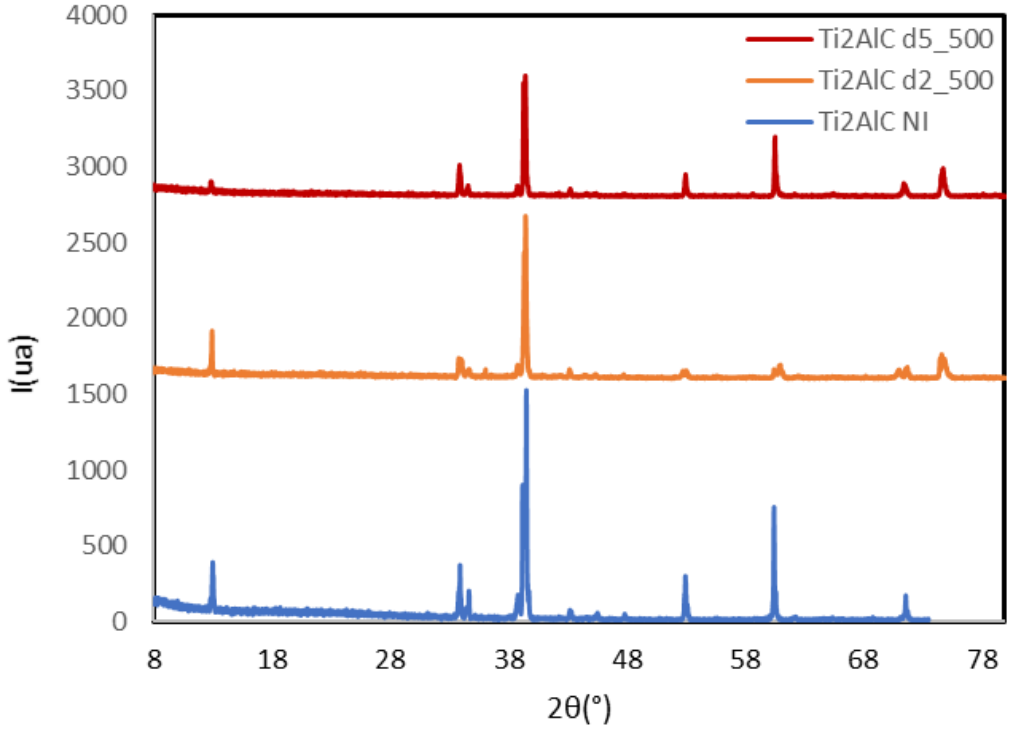
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Fluence 1	$2 \cdot 10^{15}$ cm^{-2}
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M. Josse, *PhD Thesis* (2025)

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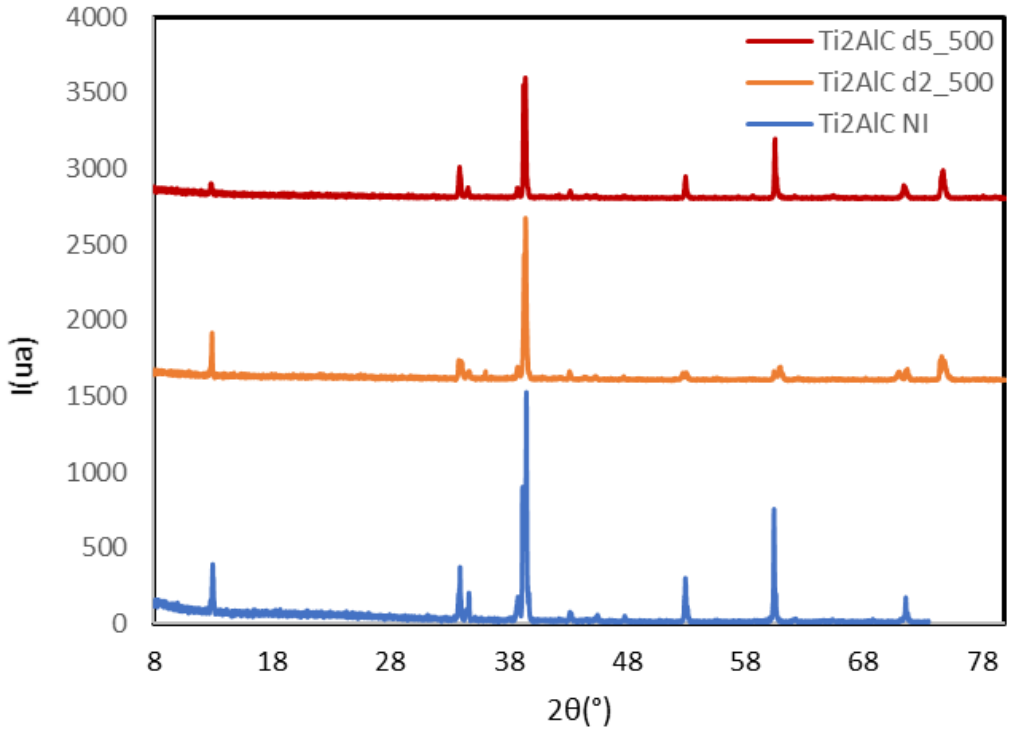
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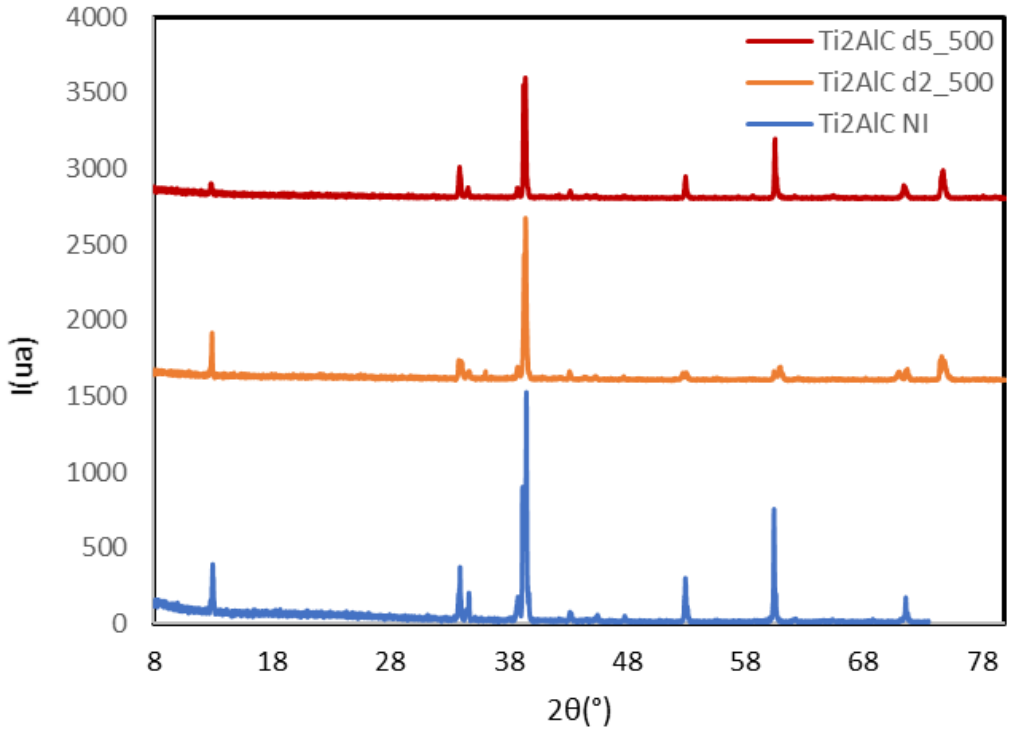


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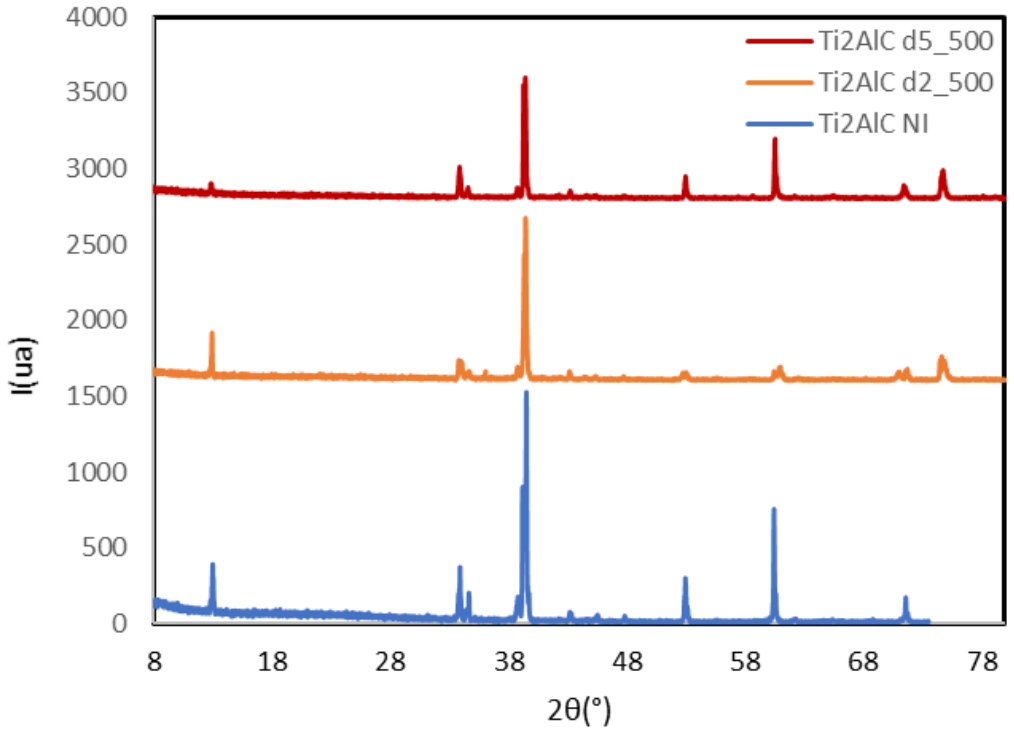


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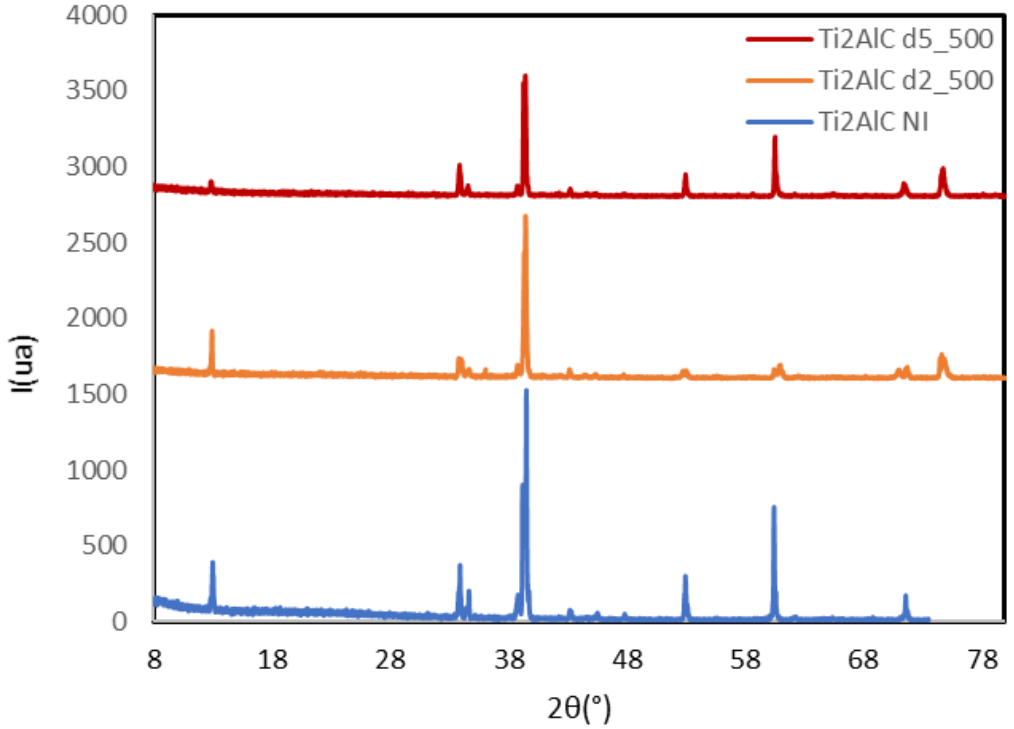


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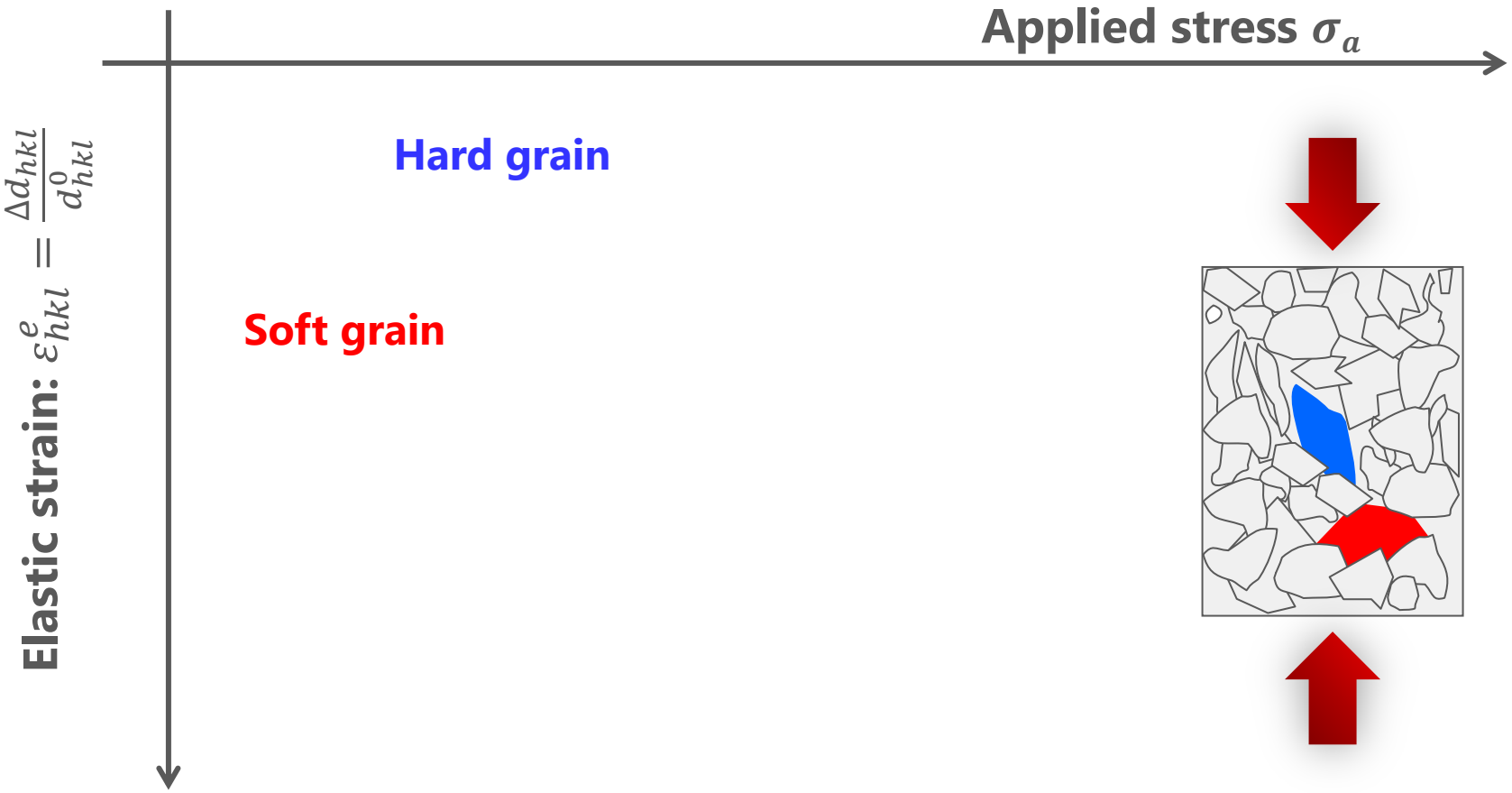


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↳The structure of Ti_2AlC seems preserved after irradiation, which is significant for radiation tolerance.

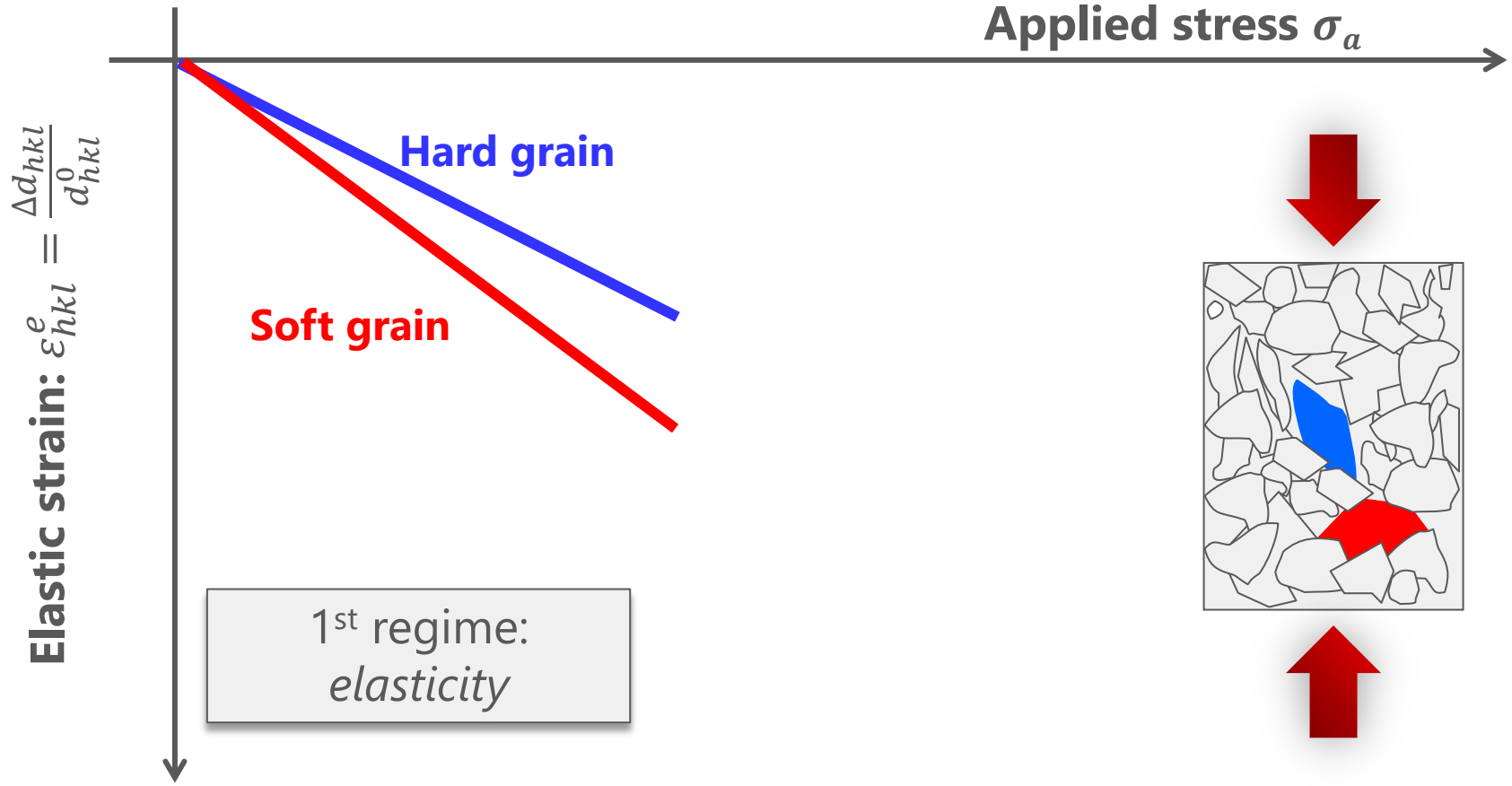
M. Josse, *PhD Thesis* (2025)

Evolution of the elastic strain



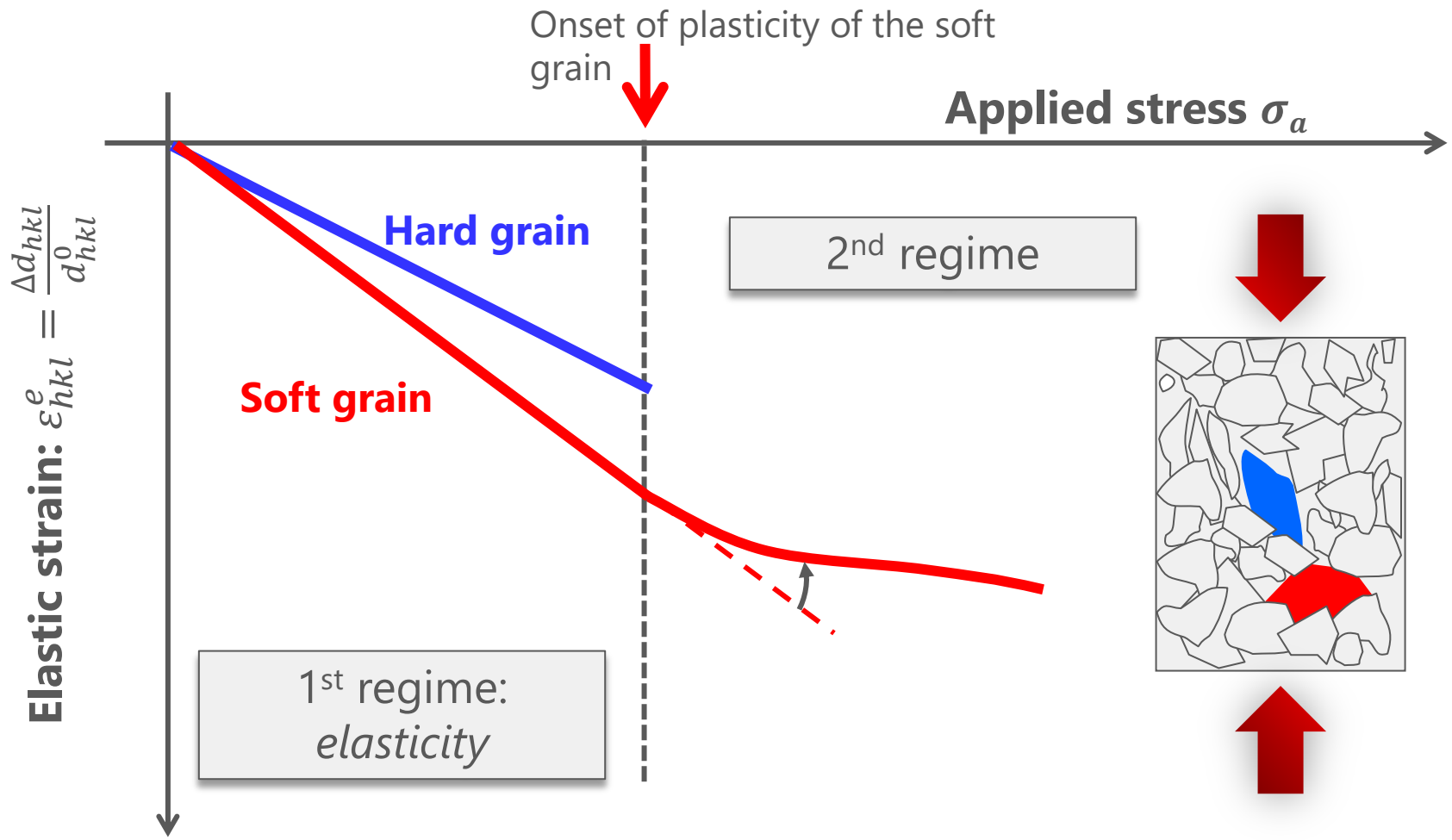
A. Guitton et al., *App. Phys. Let.*(2014)

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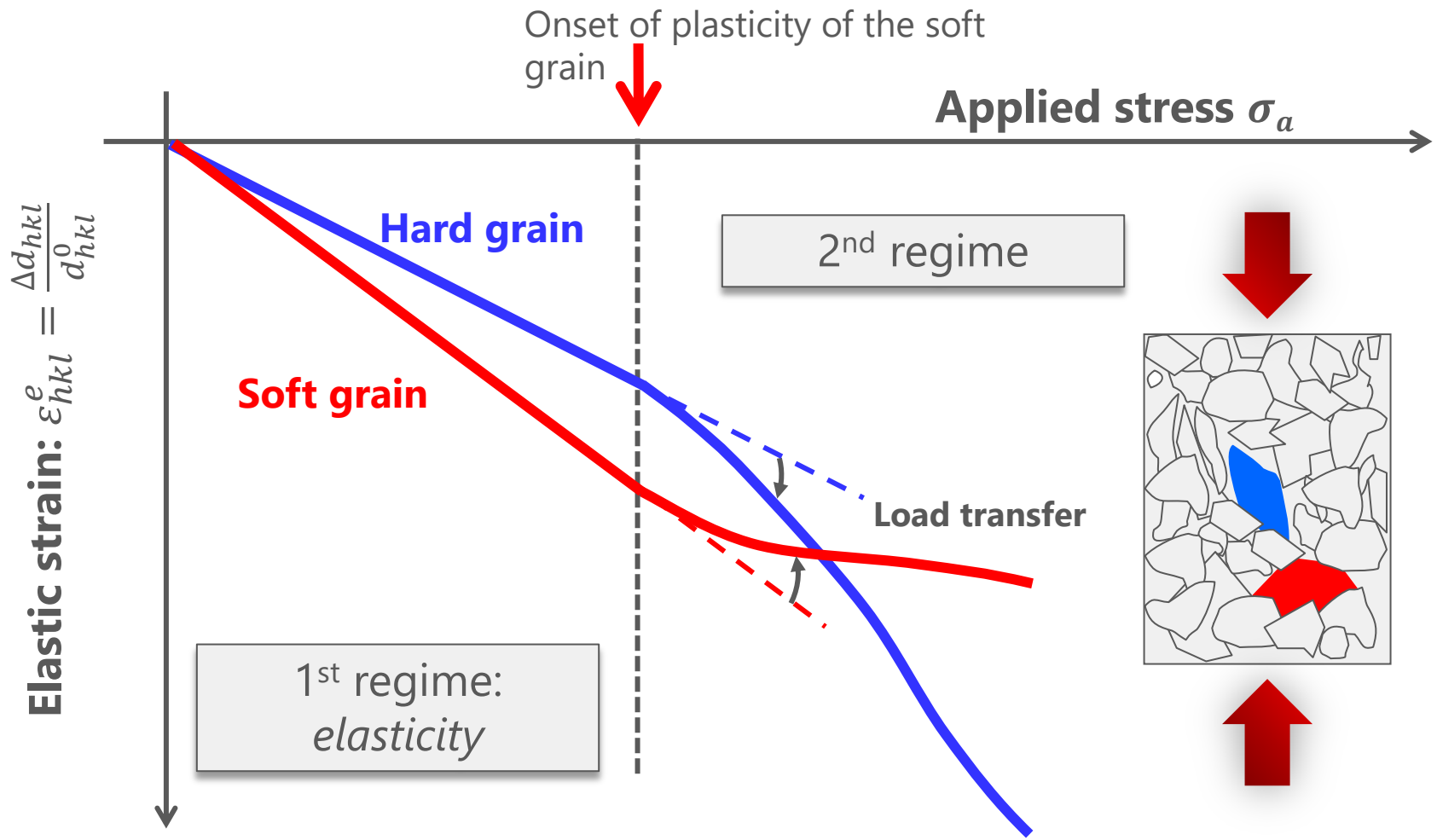
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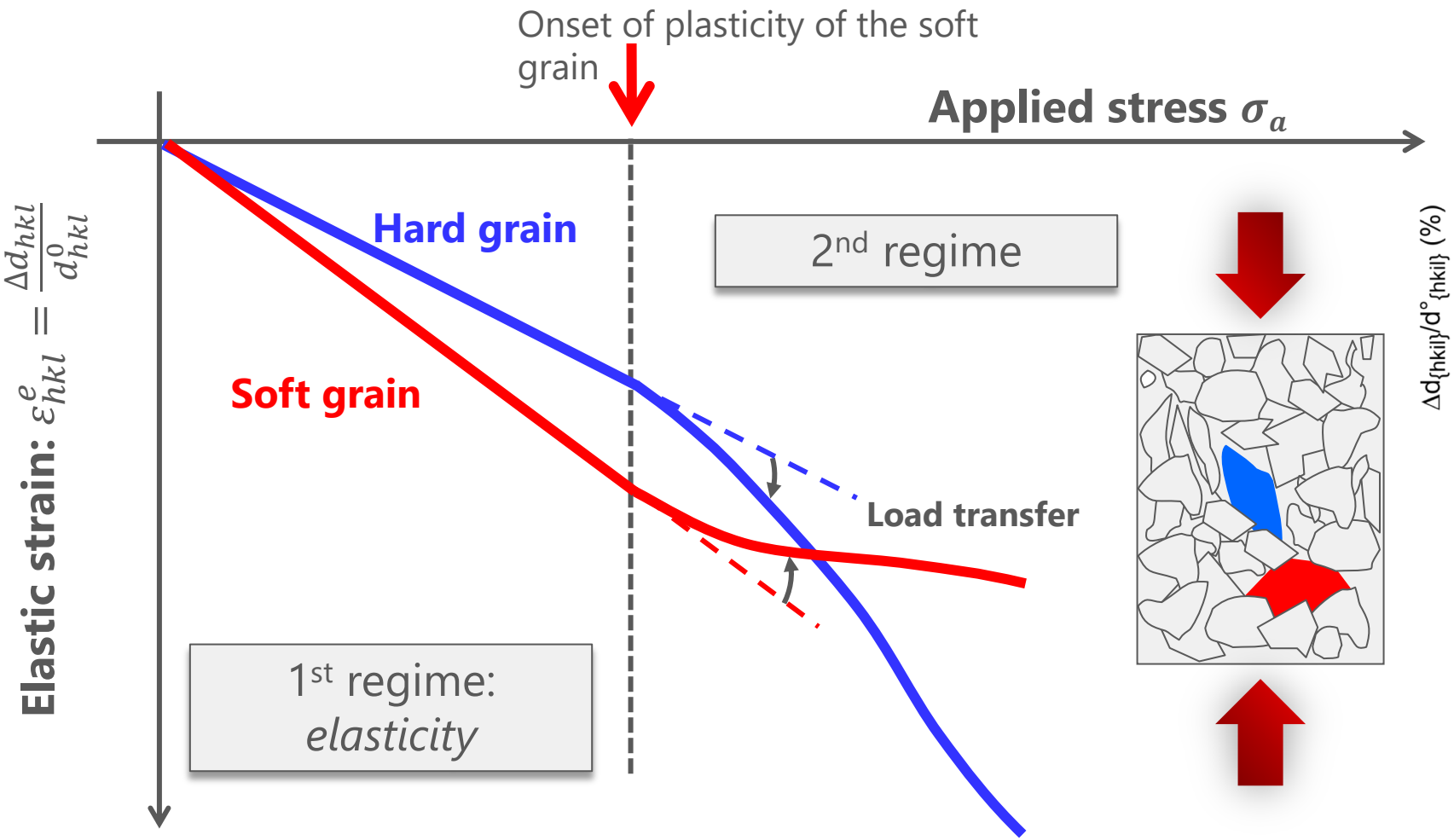
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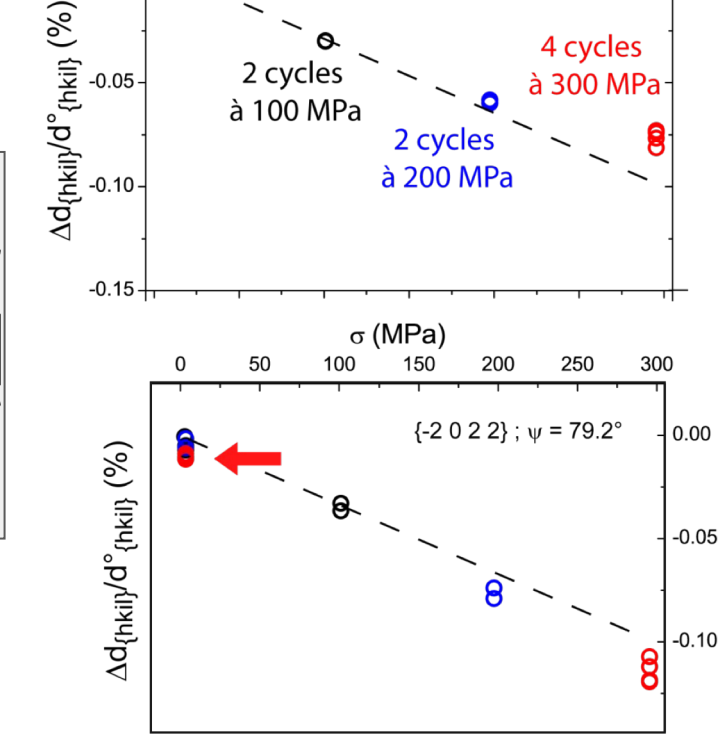
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A. Guitton et al., *App. Phys. Let.*(2014)

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Residual stresses

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

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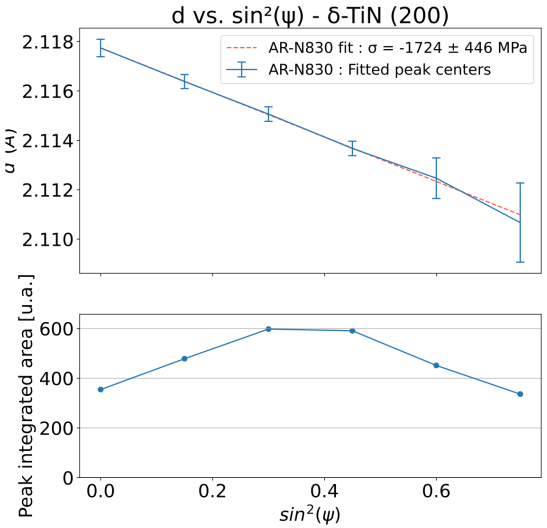
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↳ Plotting $\varepsilon_{hkl}^{\psi\varphi} = f(\sin^2\psi)$ yields a straight line; making residual stress measurable through its slope.

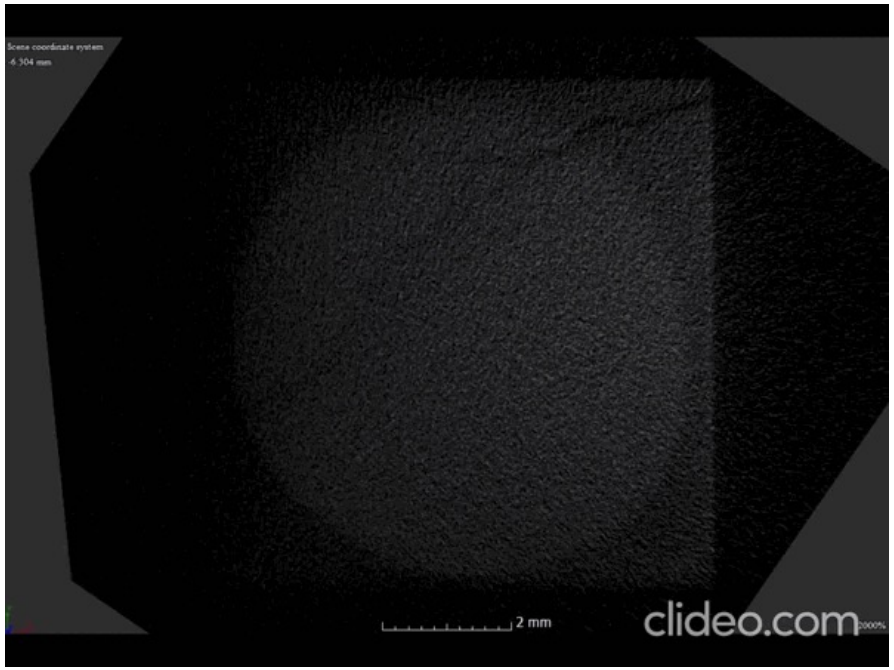
R. Génin, *PhD Thesis* (2025)

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:

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

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



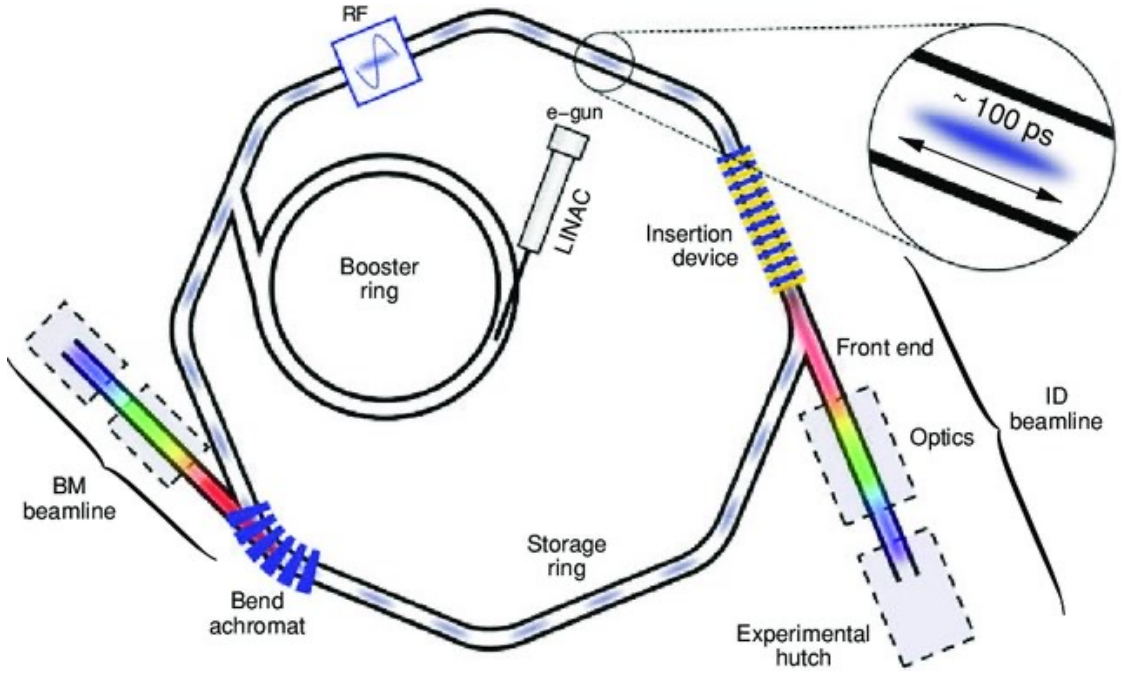
A. Monnet, *PhD work* (2025)

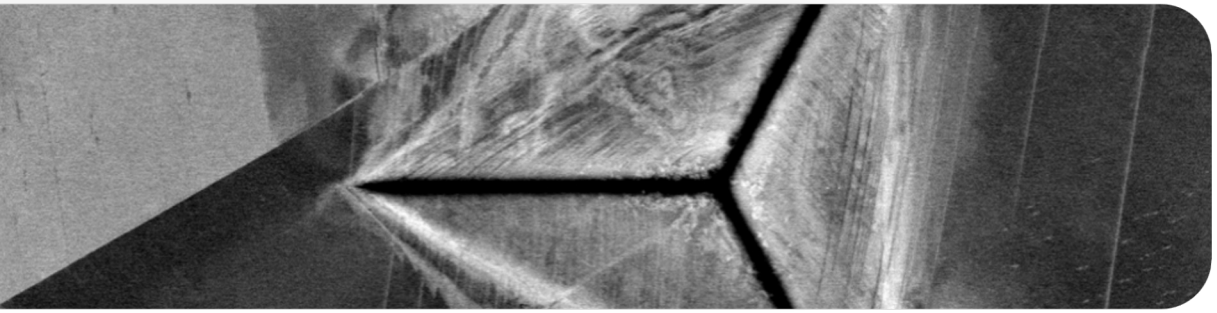
Large x-ray facilities



PSI







Electron microscopy

Micro- Nano-meter scale

www.antoine-guitton.fr

The 2 types of electron microscopes

Transmission electron microscope (TEM)



- Invention: 1931 by Ernst RUSKA et Max KNOL
- Specimen : thin foils (~ 100 nm)
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↳Nanometric/atomic scale

Scanning electron microscope (SEM)



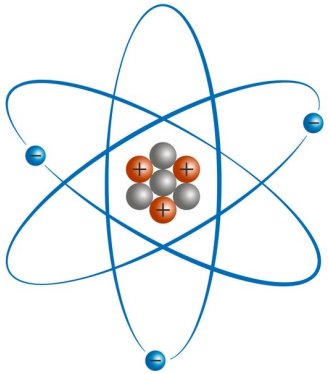
- Invention: 1937 by Manfred VON ARDENNE but developed in the 1960s
- Specimen : bulk
- Price: ~ 500 k€
- Comon in industry

↳Meso-/micro-/nano-metric scale

↳ The two microscopes are complementary.

Focus on the electron

Particle



Atom structure
- Proton
- Neutron
- Electron



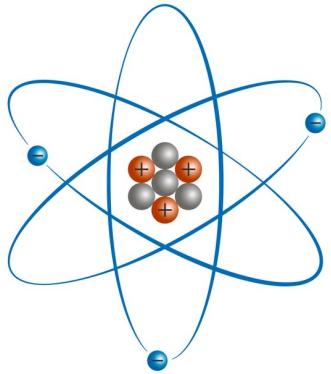
Joseph John THOMSON
(1856–1940)

- $m_e = 9.11 \times 10^{-31}$ kg
- $e = -1.60 \times 10^{-19}$ C

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

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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:

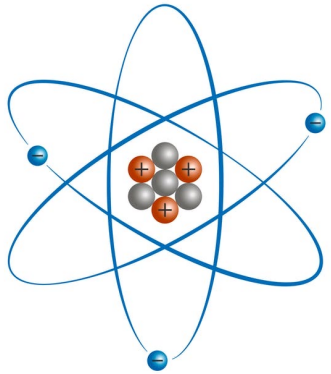
$$\lambda = \frac{h}{p} = \frac{h}{mv}$$

↳ Electrons act not only as particles but as waves too.

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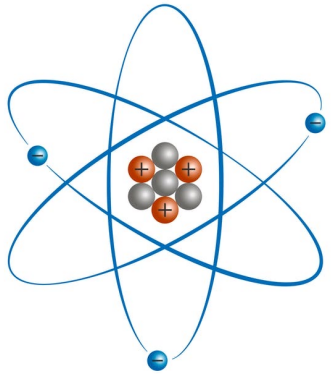
$$E = eV = \frac{mv^2}{2} \Leftrightarrow v = \sqrt{\frac{2eV}{m}} \Rightarrow \lambda = \frac{h}{\sqrt{2meV}}$$

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

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

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

Electron-matter interaction

Reflection

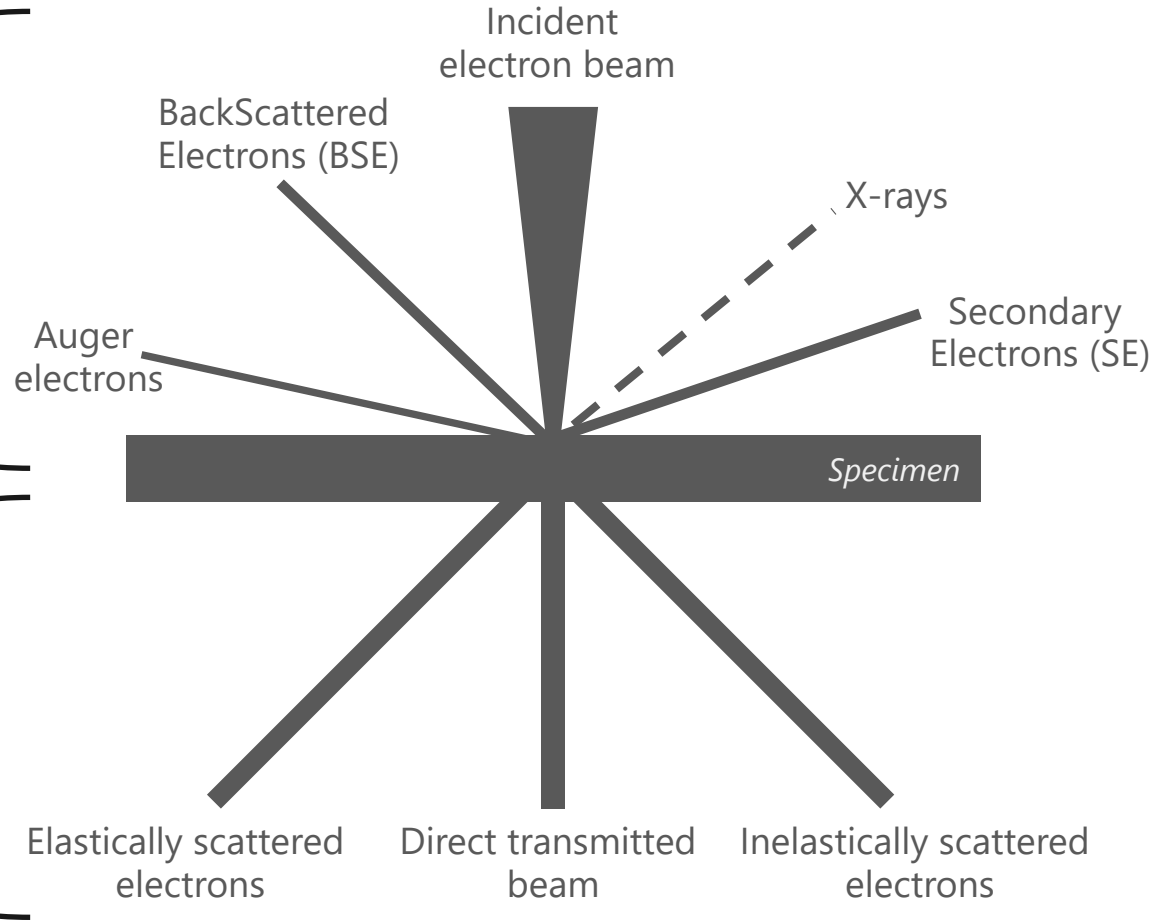
⇒ SEM

Bulk specimens

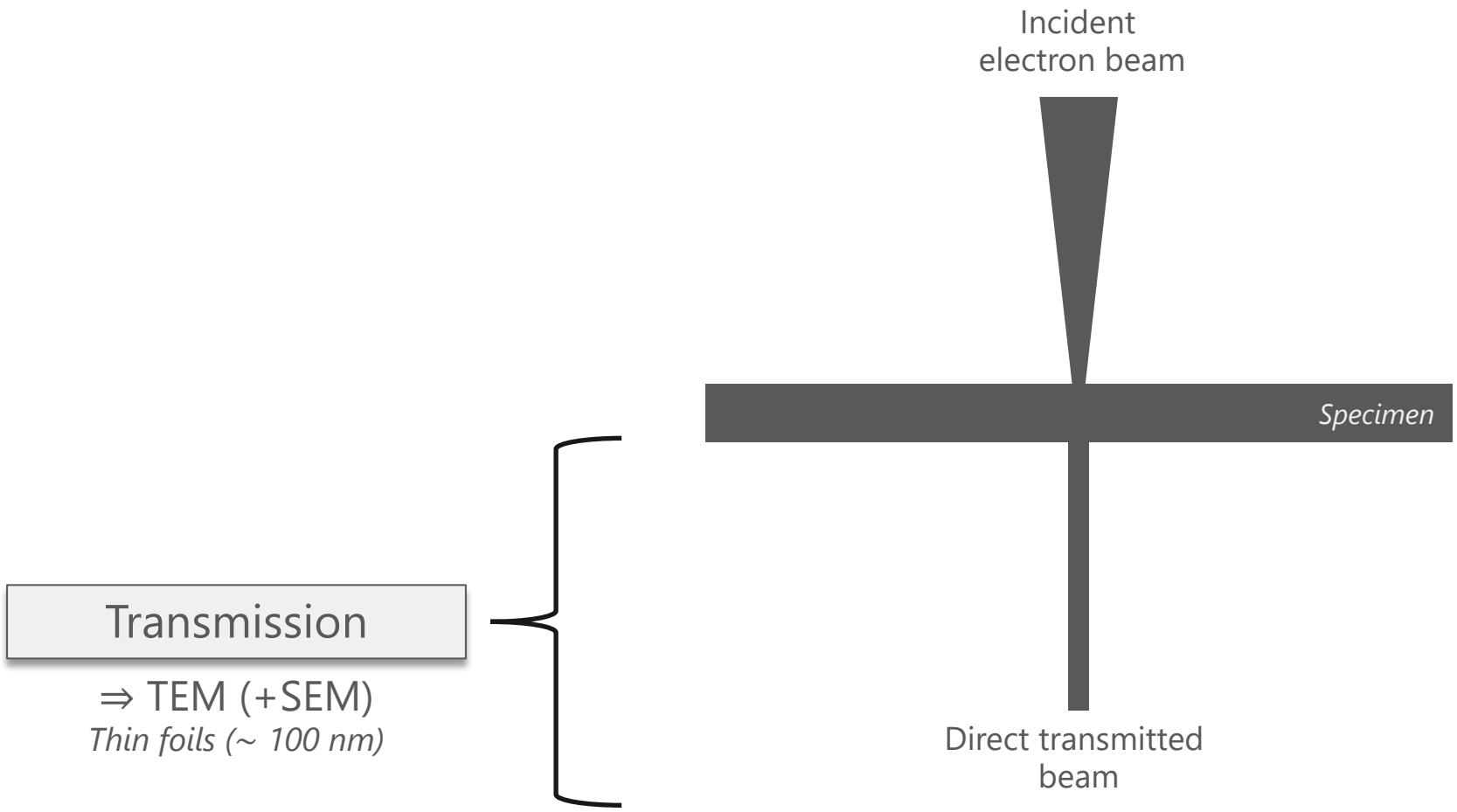
Transmission

⇒ TEM (+SEM)

Thin foils (~ 100 nm)



Electron-matter interaction



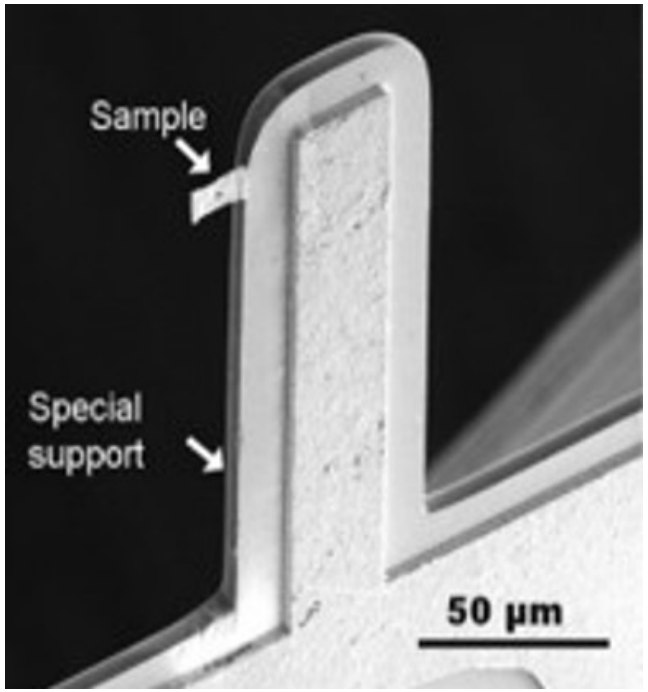
Transmission

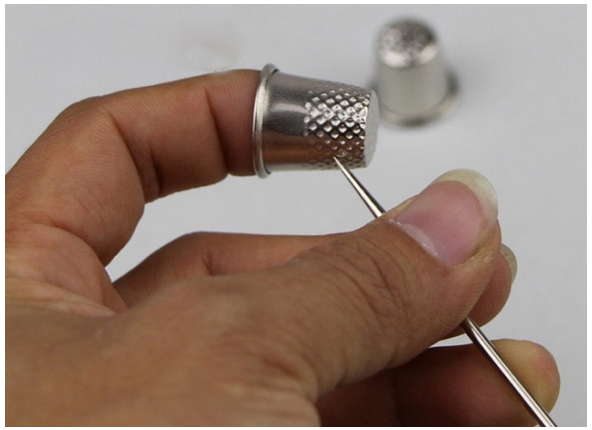
⇒ TEM (+SEM)

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?

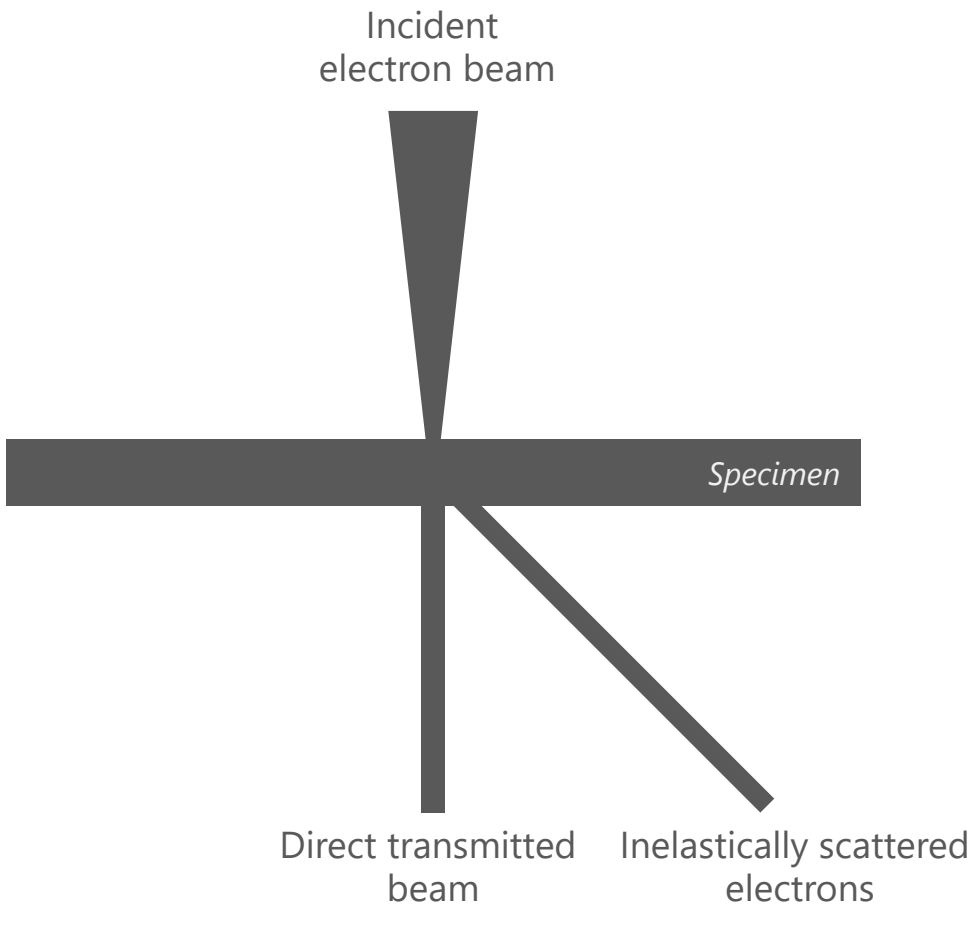


Electron-matter interaction

Transmission

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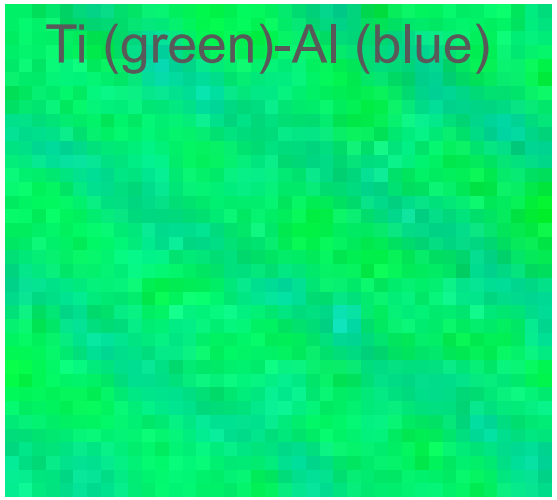
Inelastically scattered electrons

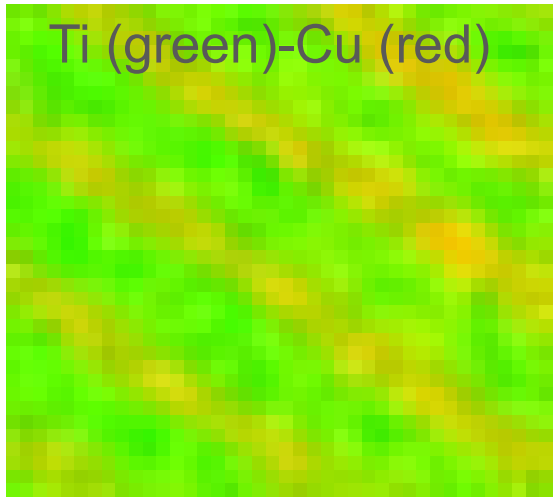
The energy is transferred from the electron to the sample.

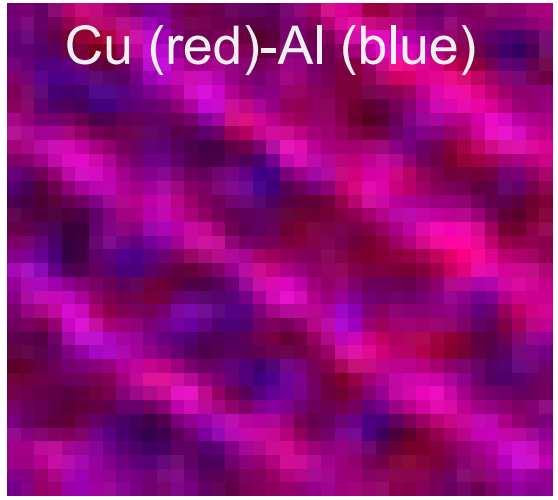
$$\Rightarrow E_0 > E_{e^-}$$

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

Electron Energy Loss Spectroscopy (EELS)

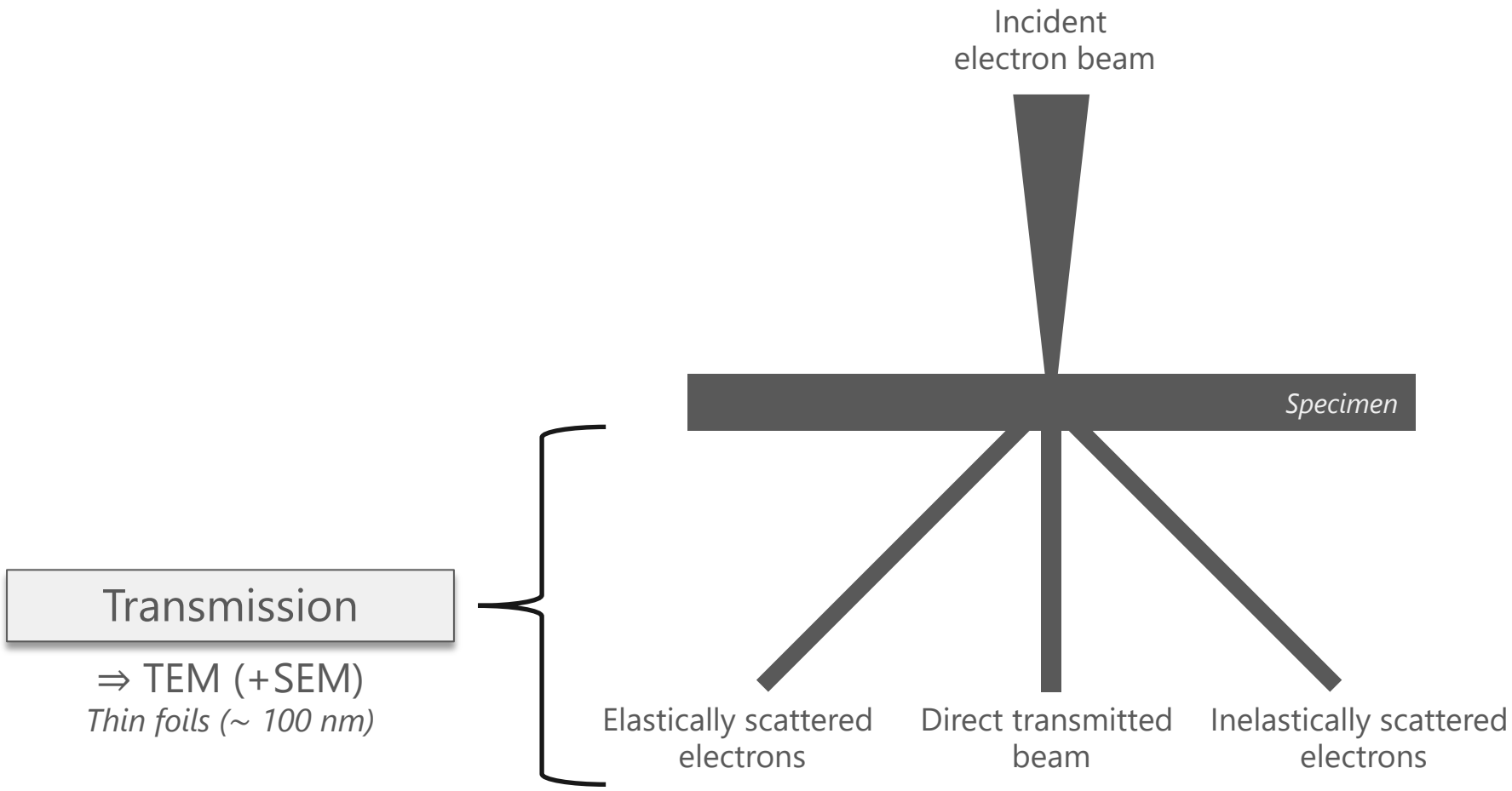






W. Yu, J. Guénolé, J. Ghanbaja, M. Vallet, A. Guitton, *Scripta Mater.. (2021)*

Electron-matter interaction



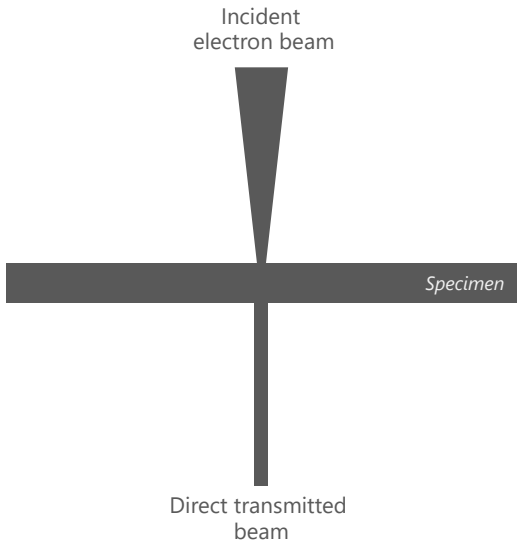
Elastically scattered electrons

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

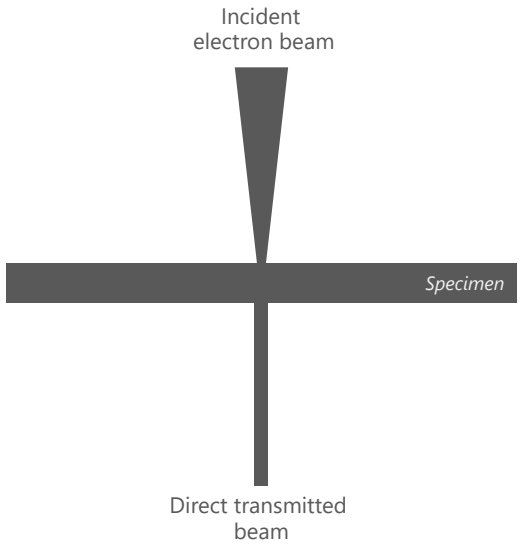
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In other directions

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

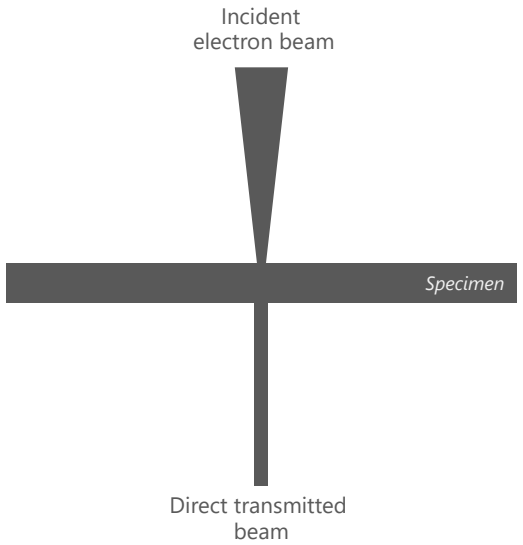
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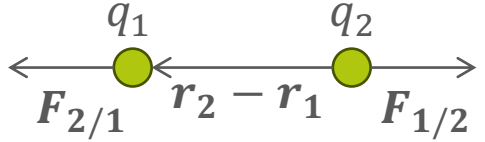
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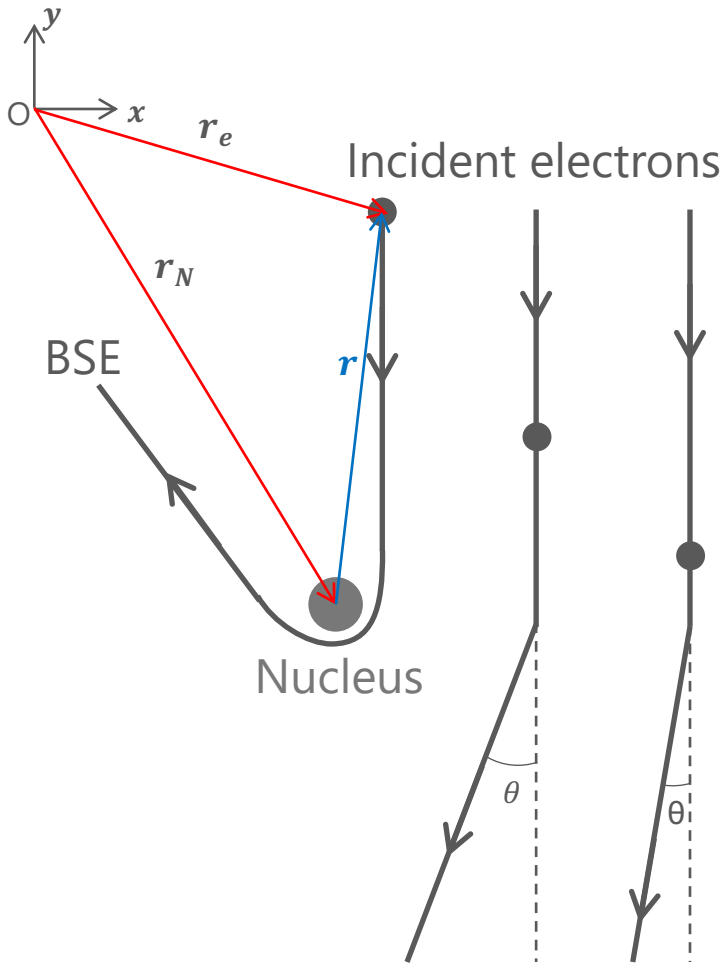
❖ The electrons are deflected from their path by Coulomb interactions with the positive potential of the atomic nuclei, screened by the surrounding electron cloud.



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

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

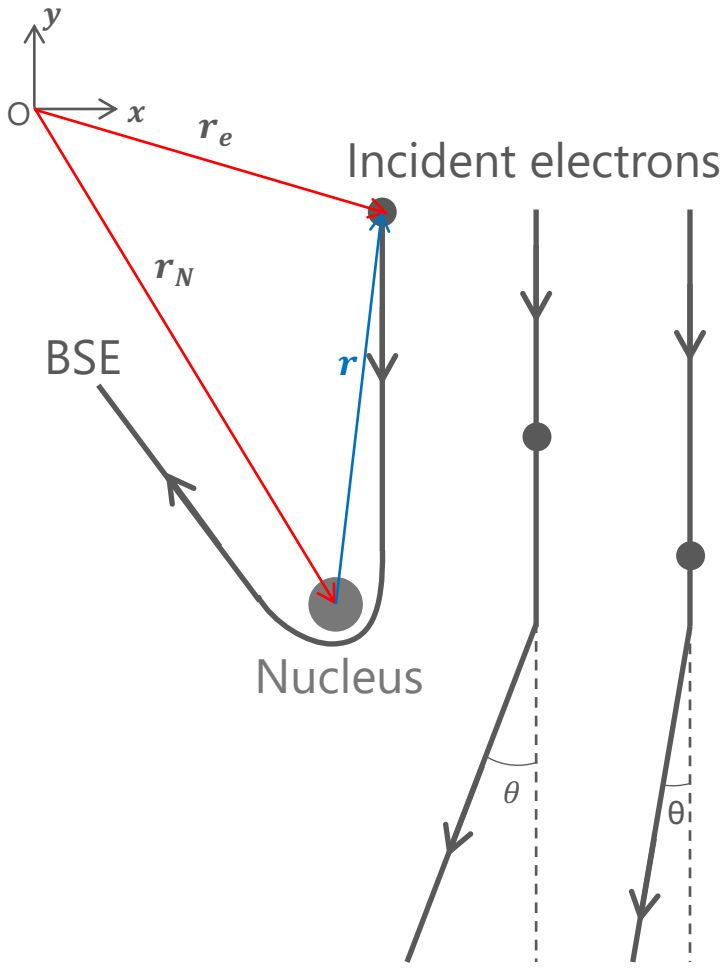
Elastically scattered electrons



- 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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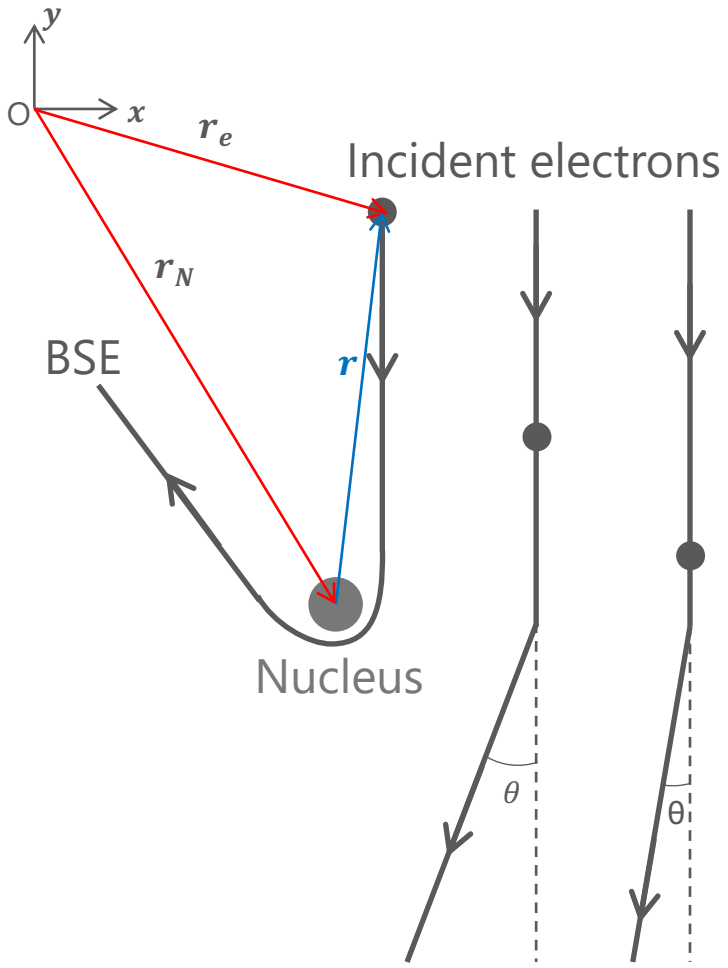
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  - Scattered angle θ

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

If $\theta > 90°$
⇒ BackScatterred electrons (BSE)

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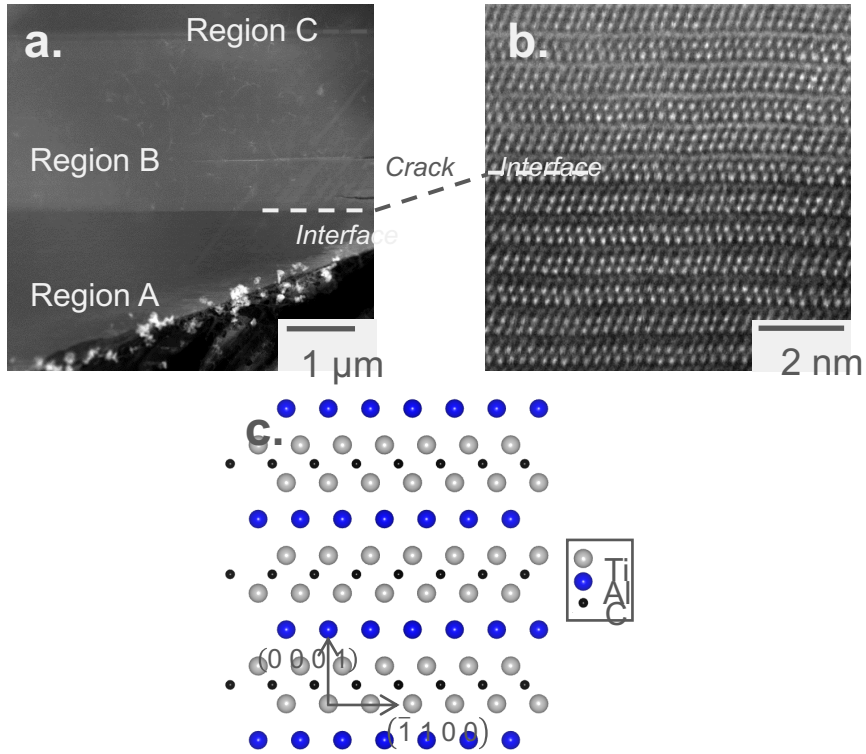
  - Influence of Z:

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

⇒ High Z produces higher intensity at large θ

Intensity at large θ

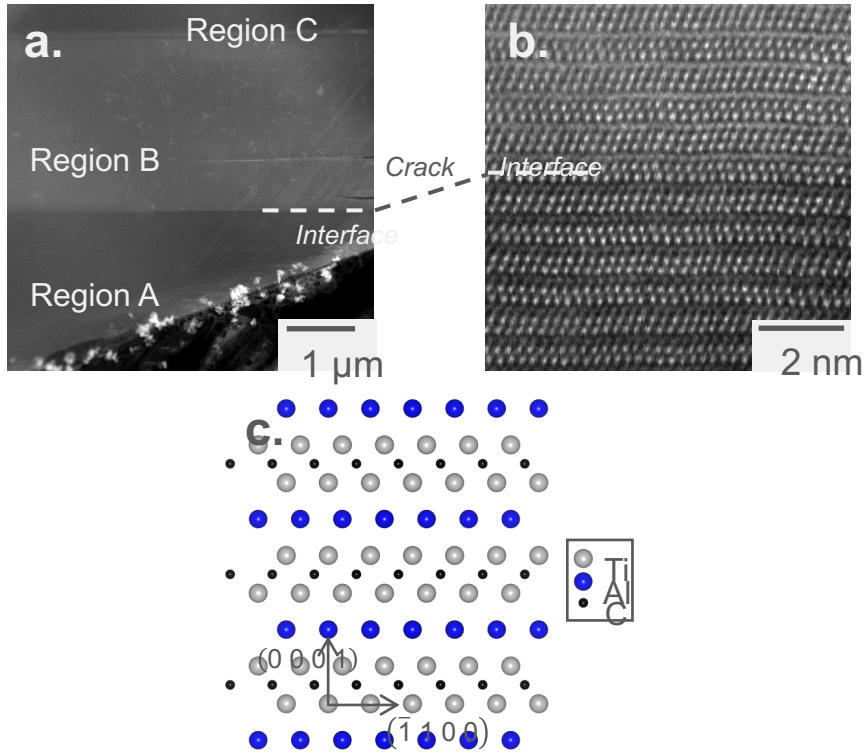
Inside the (S)TEM



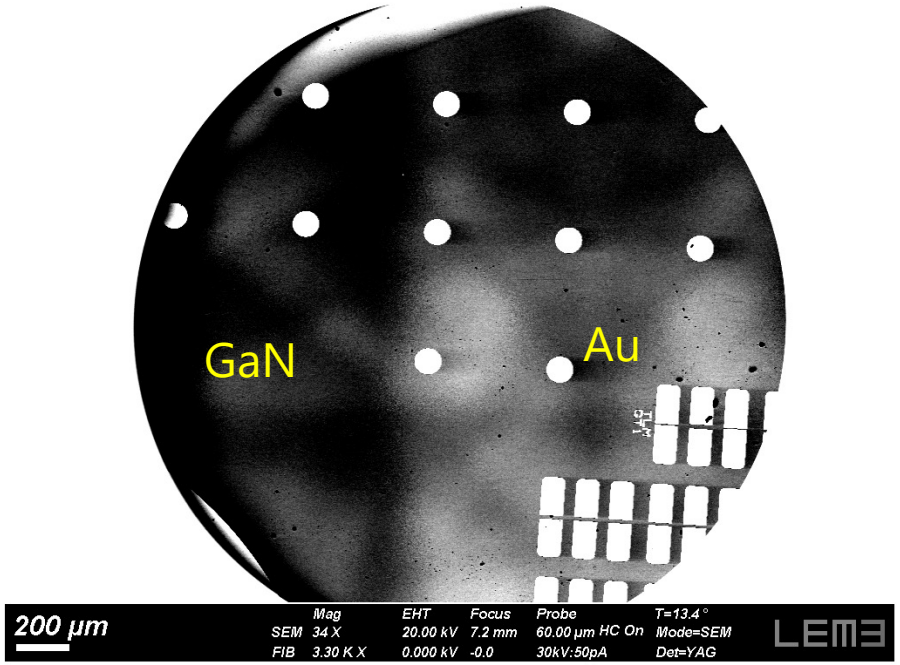
W. Yu, et al, *Scripta Mater.*. (2021)

Intensity at large θ

Inside the (S)TEM



Inside the SEM

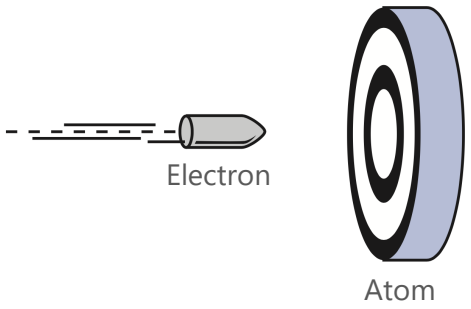


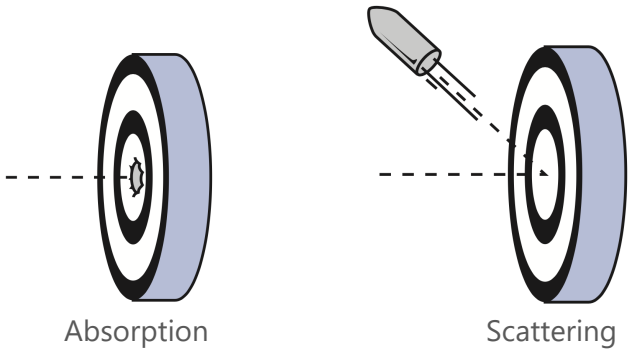
↳ Bright contrast ⇒ high Z; Dark contrast ⇒ low Z

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

The cross section of interaction

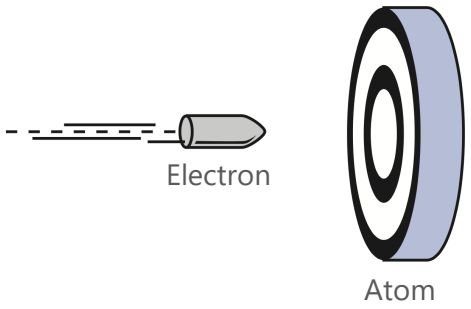
Cross section the probability that a certain interaction will occur between two particles for a given set of parameters.

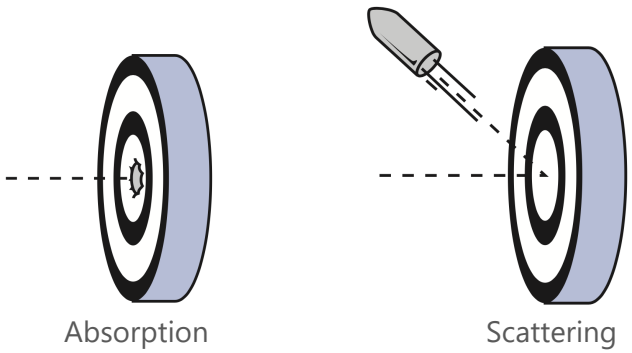




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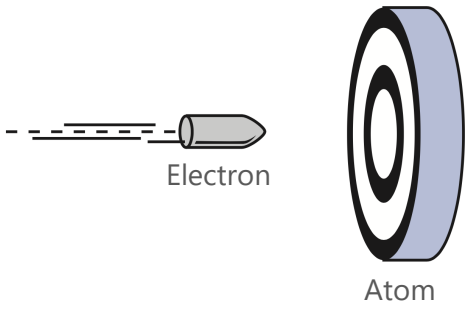


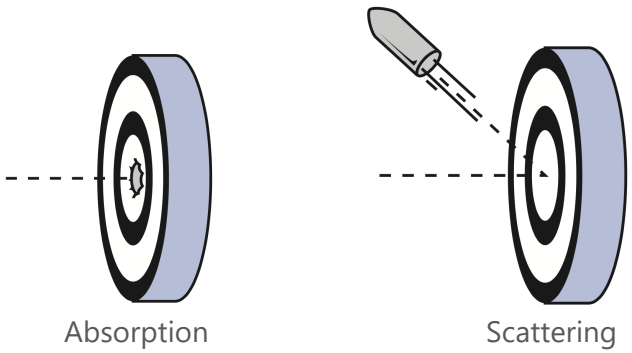


- **There are two kinds of cross-sections:**
  - Microscopic (σ): it refers to cross sections of electrons reacting with a specific atom
    $10^{-28}\text{cm}^2 = 1\ \text{b}$

The cross section of interaction

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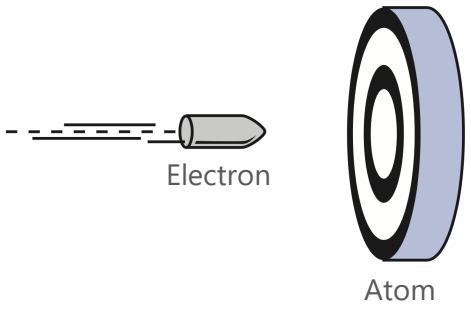


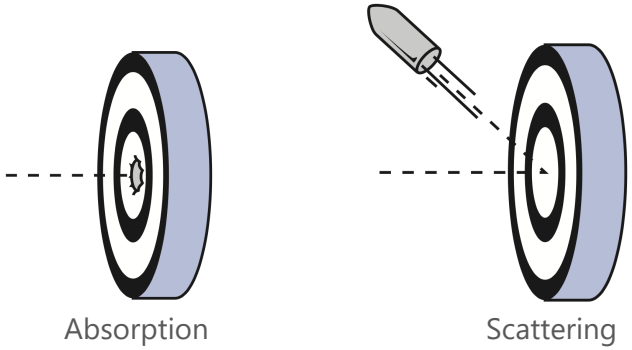
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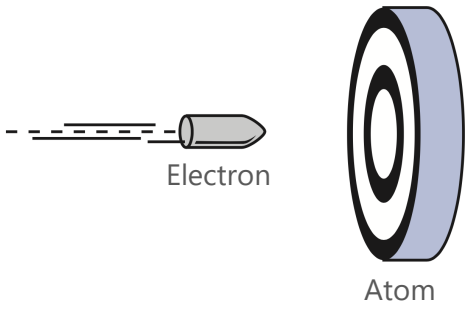
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  - ✓ Pure material

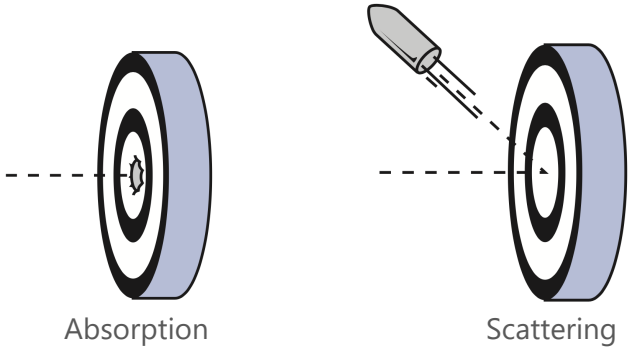
$$\Sigma[\text{m}^{-1}] = n\sigma = \frac{N_a \rho}{M}\sigma$$

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

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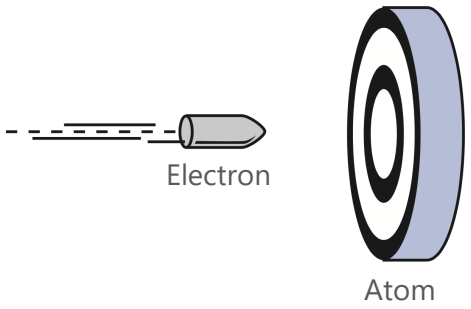
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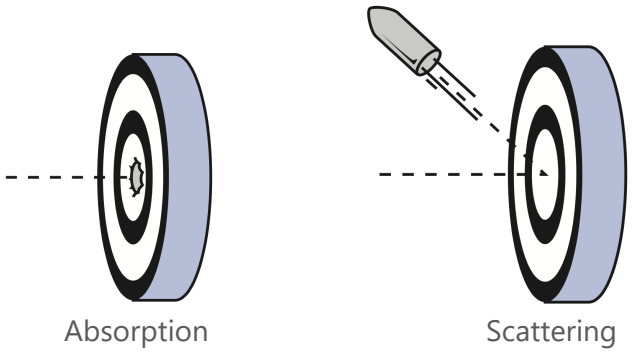
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  - ✓ Composite (with i components)

    $$\Sigma[\text{m}^{-1}] = \sum_i n_i \sigma_i$$

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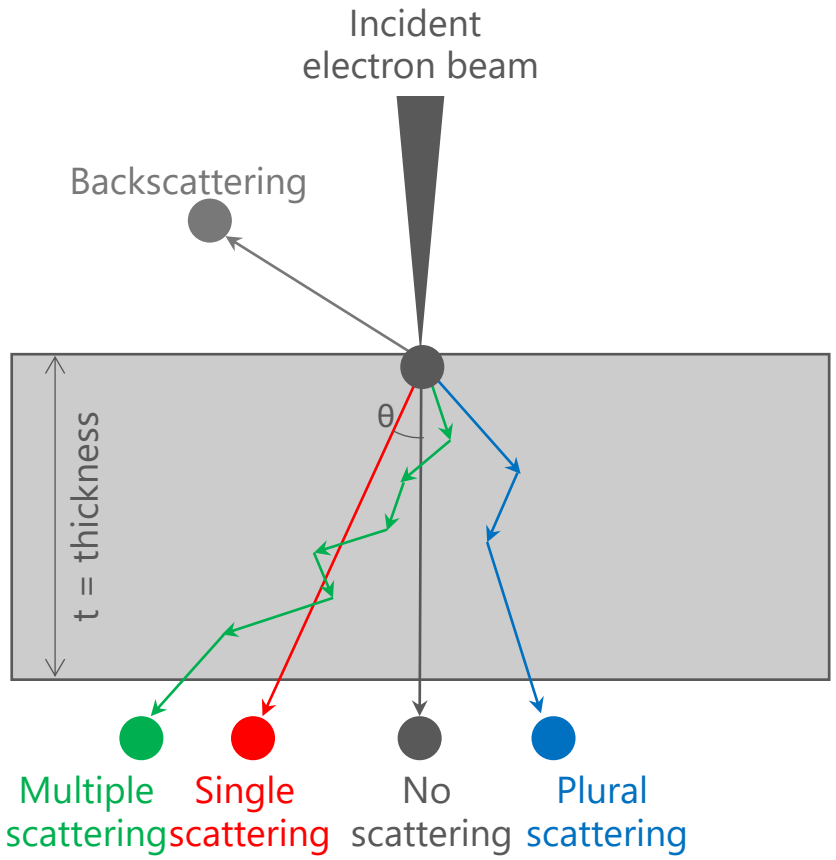
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❖ Mean free path:

- How far on average an electron will travel before it interacts with an atom

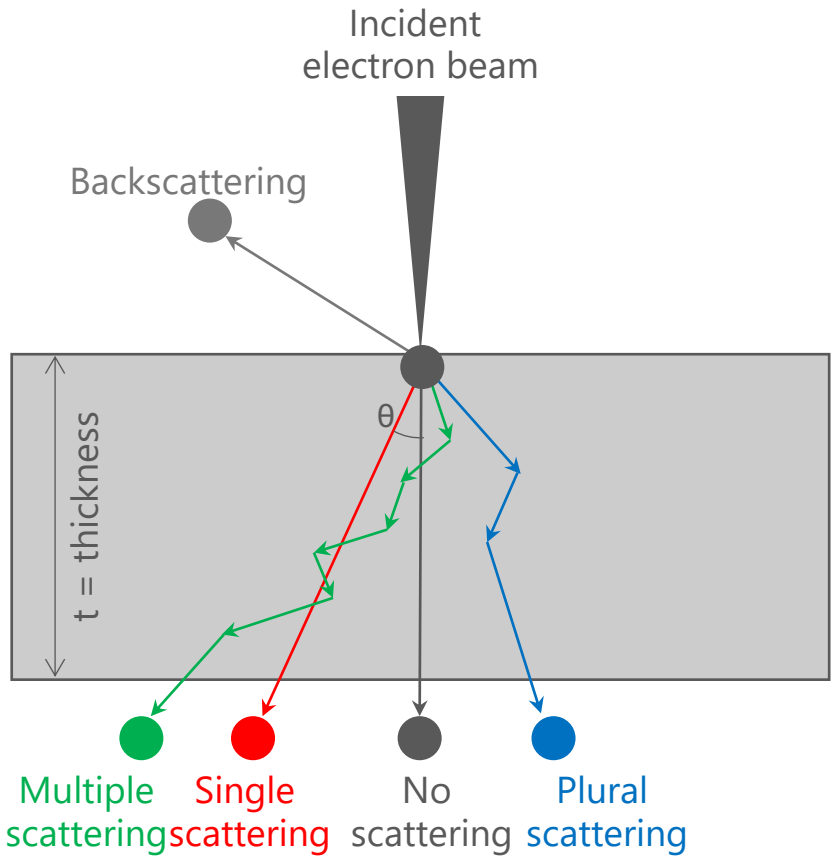
$$\bar{l}[\text{m}] = \frac{1}{\Sigma}$$

The cross section of interaction



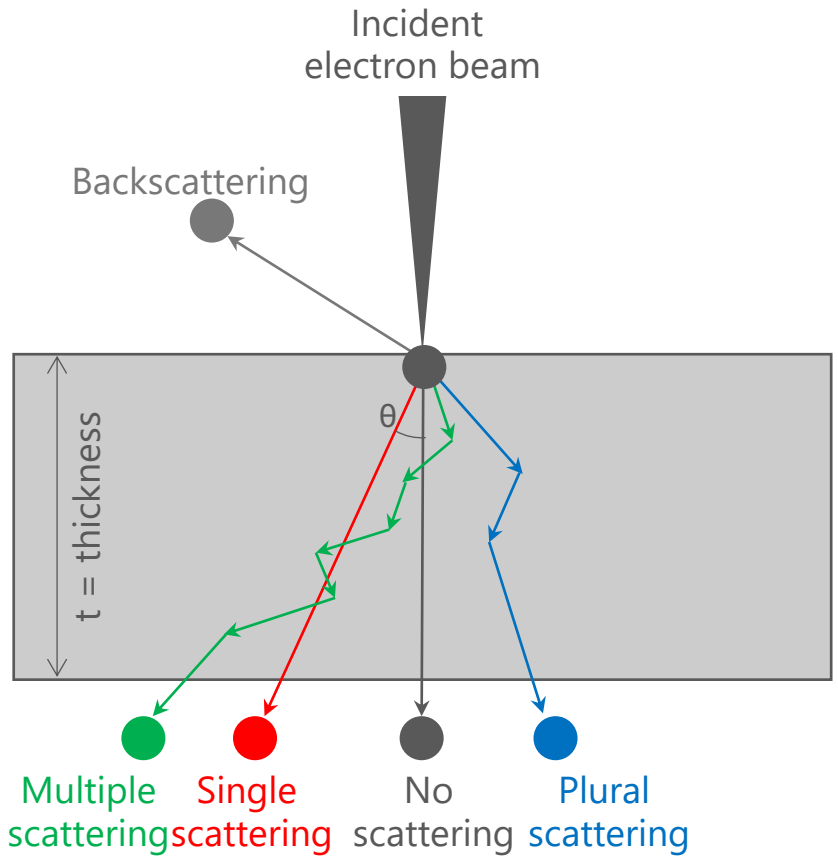
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The cross section of interaction



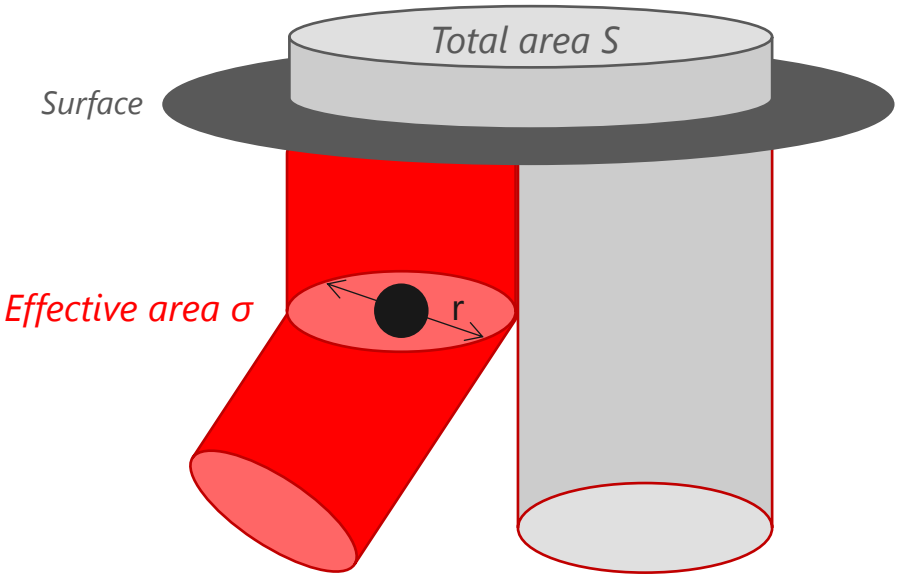
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  - Thick samples or in regions of slightly higher density.
- **Multiple scattering:**
  - Many scattering events.
  - The path becomes complex, and the original direction and energy of the electron are heavily modified.
  - Common in thick or dense 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} ↗ when σ_{tot} ↗



The cross section of interaction

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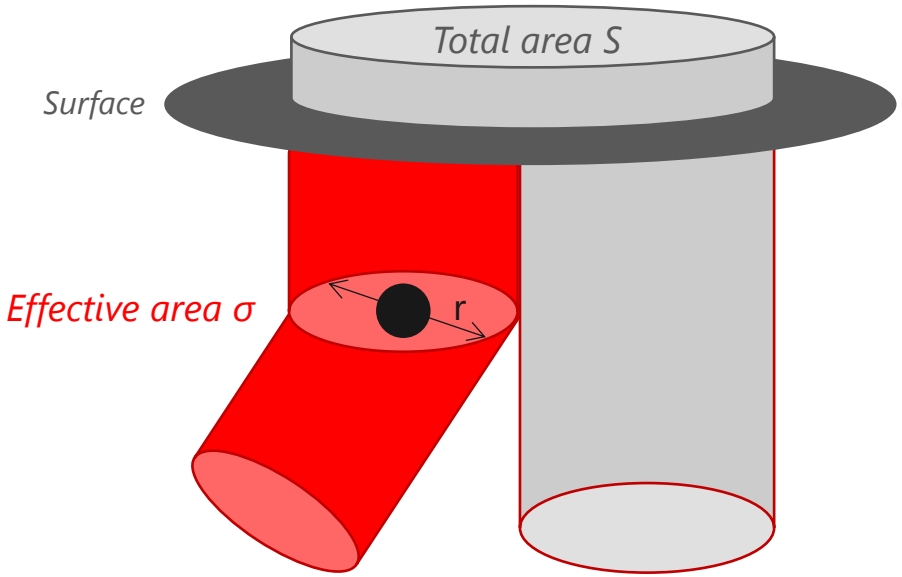
$$\sigma_{tot} = \sigma_{elastic} + \sigma_{inelastic}$$

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

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

$\Rightarrow P_{scat}$ ↗ when Z ↗

θ ↗ $\Rightarrow P_{scat}$ ↘



The cross section of interaction

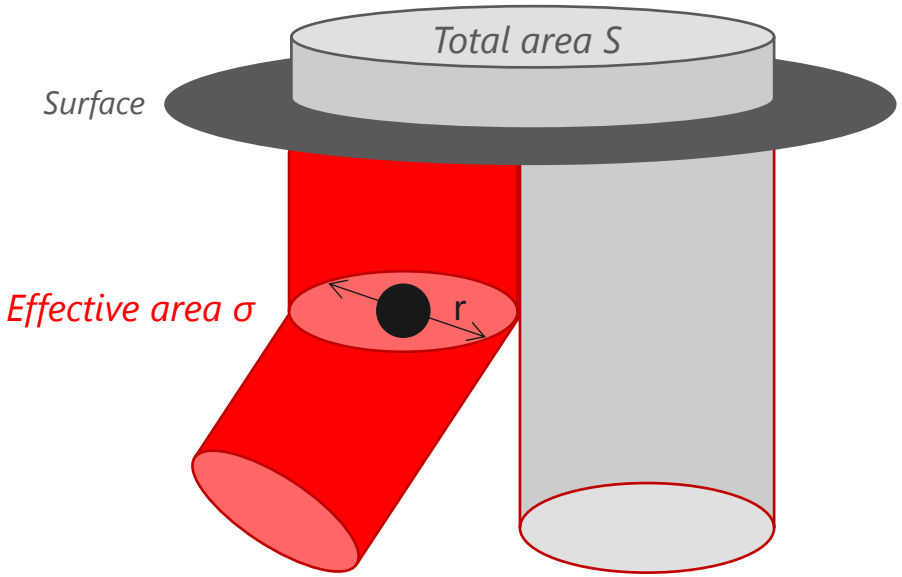
❖ **Cross section for a specimen:**

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

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

(t: thickness)

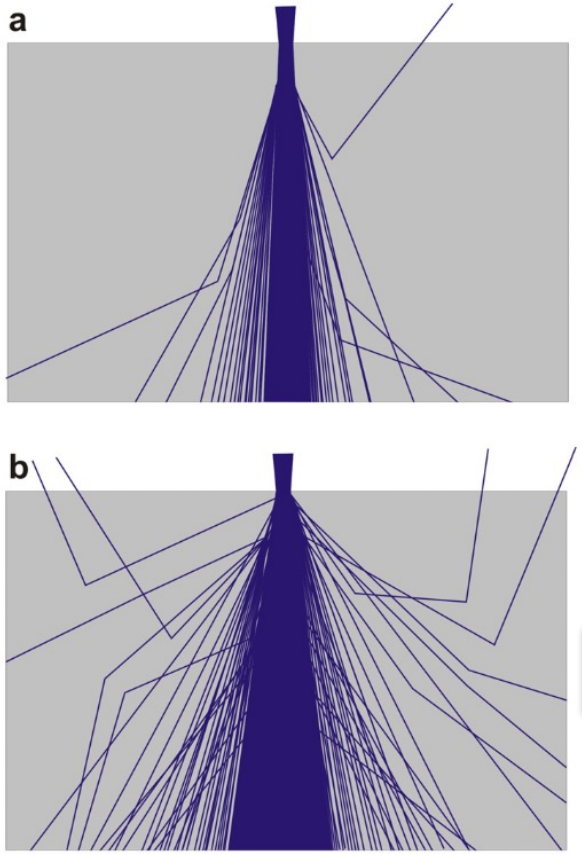
$\Rightarrow P_{scat} \nearrow$ when $t \nearrow$
$\Rightarrow P_{scat} \nearrow$ when $\rho \nearrow$



Interaction volume

Thin foil

Low Z

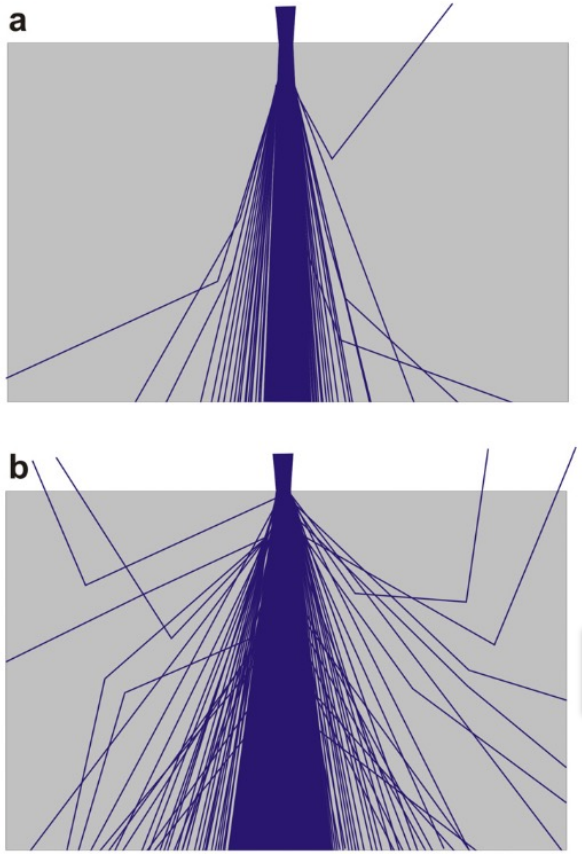


High Z

Interaction volume

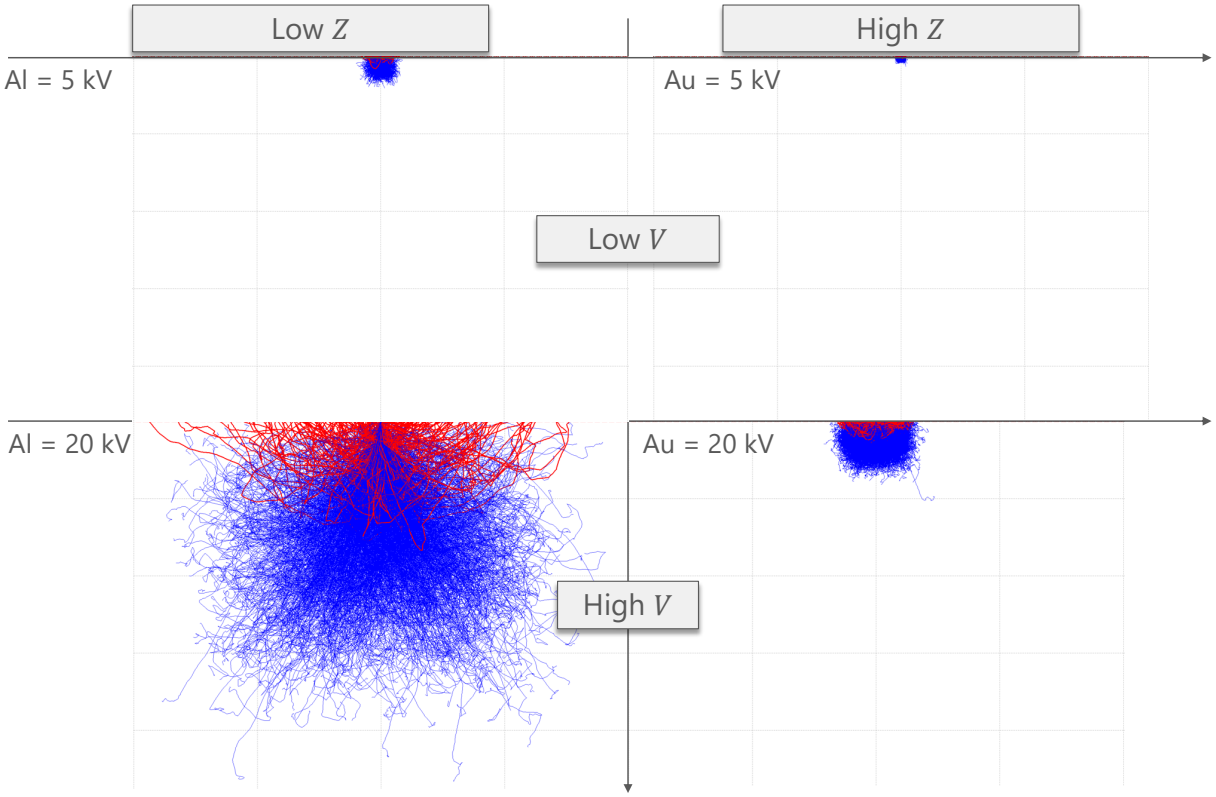
Thin foil

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)



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- Specimen : bulk
- Price: ~ 500 k€
- Comon in industry

↳Meso-/micro-/nano-metric scale

↳ The two microscopes are complementary.

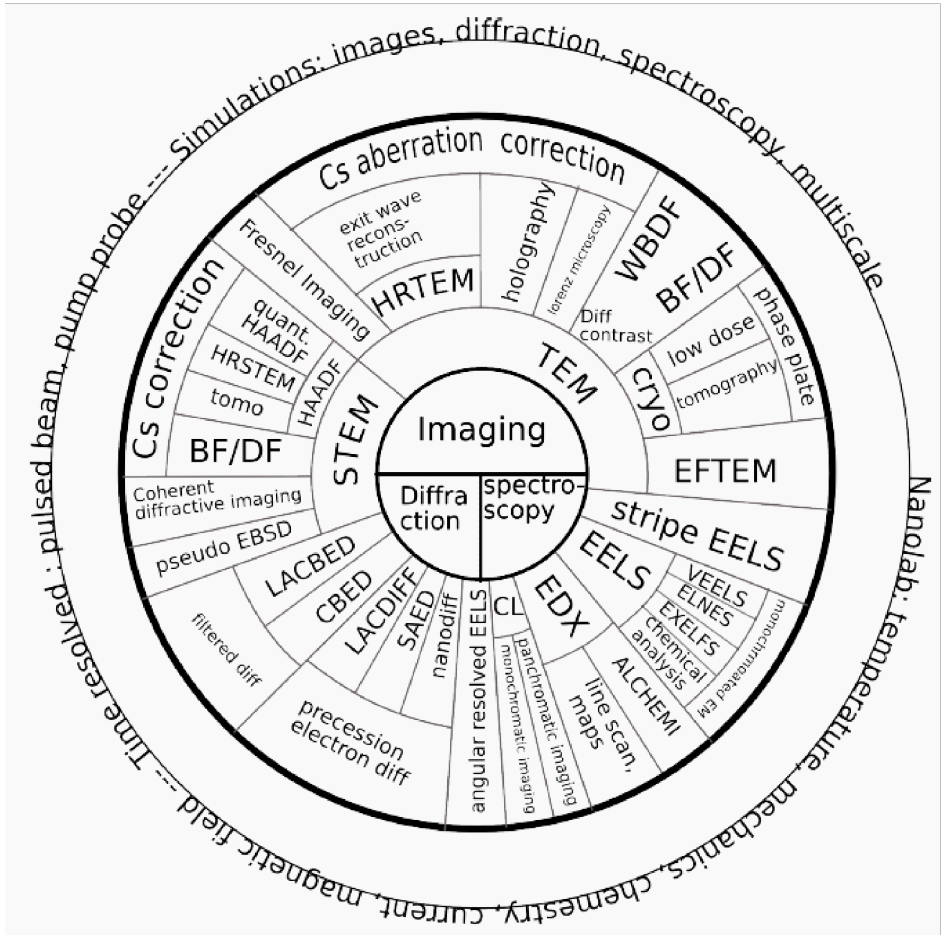
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Basics of TEM contrast

Probability of scattering event:

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Mass contrast:

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

You shall not study W by TEM…

High ρ

Low ρ

Basics of TEM contrast

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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* $t \nearrow$

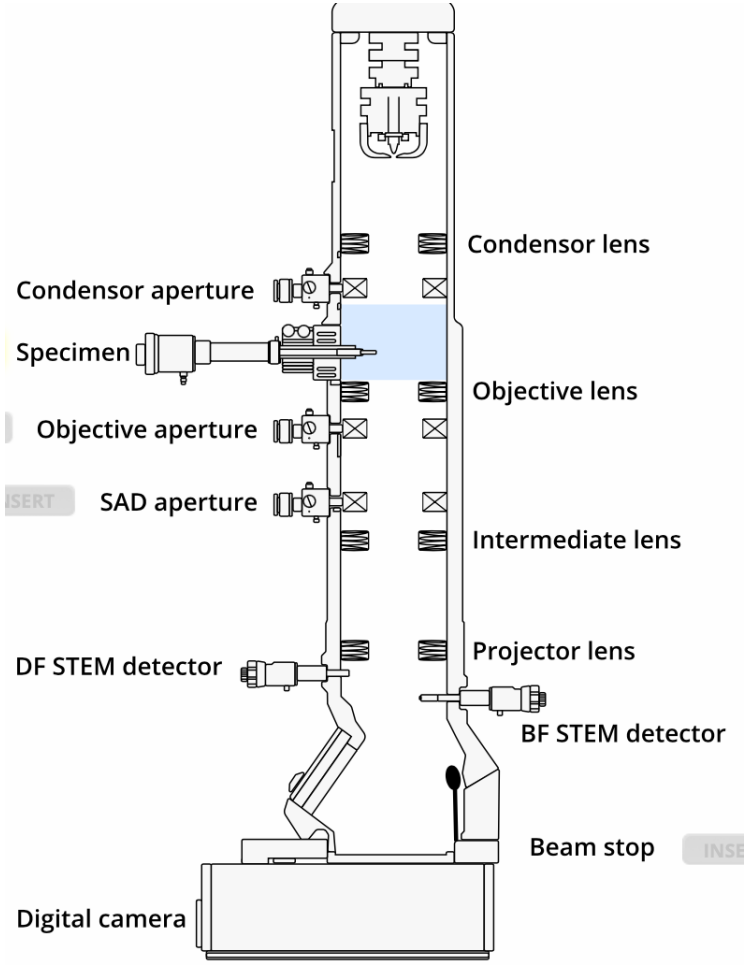
High t

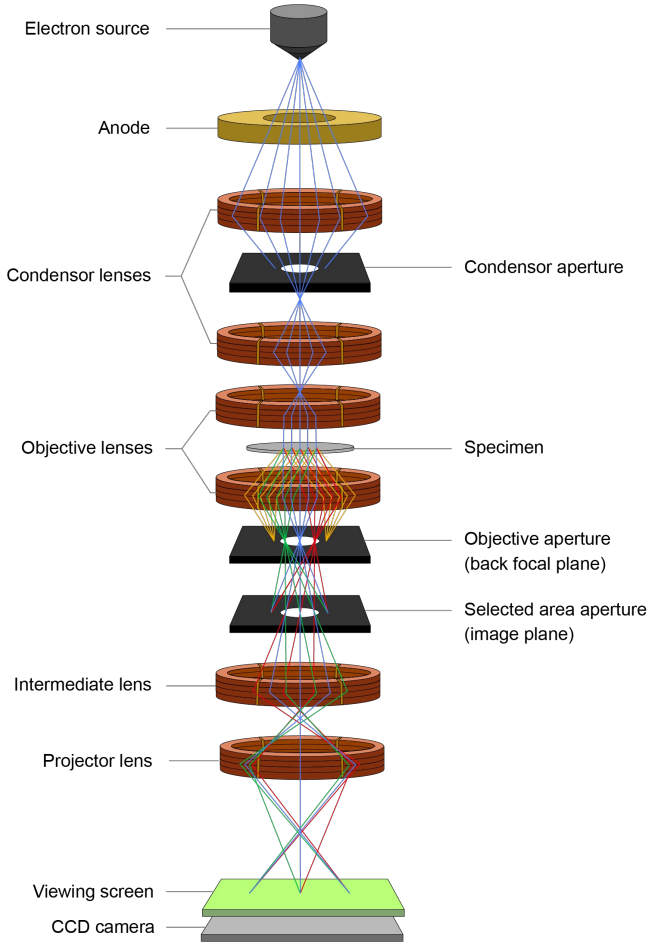
Low t

You shall not study thick TEM foils…

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

Basics of TEM

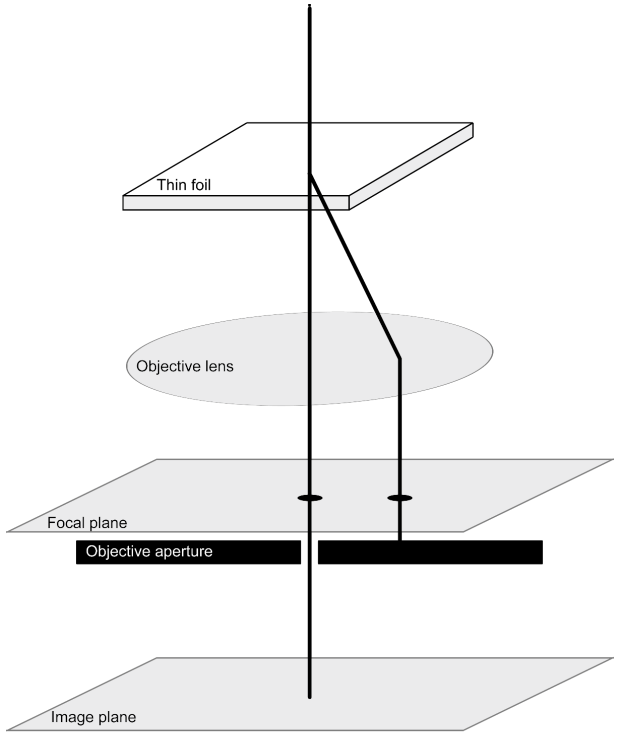


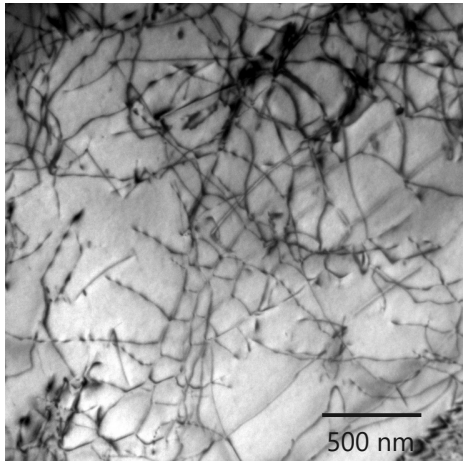


J. Aubourg, *PhD Thesis (2024)*

Basics of TEM

Bright field

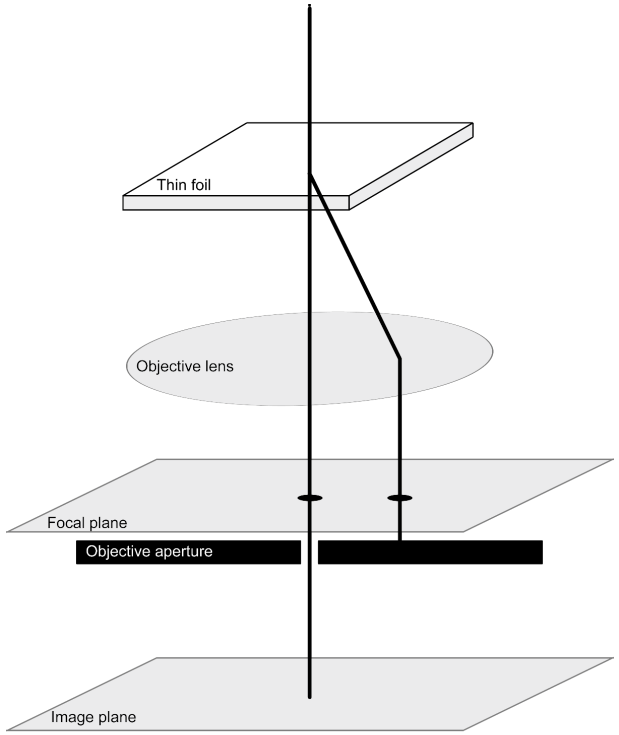


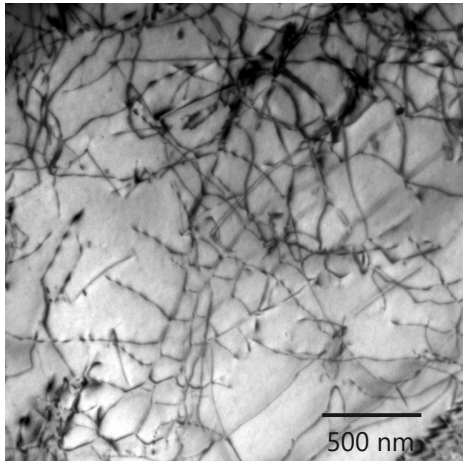


↳Dark contrasts on a bright background

Basics of TEM

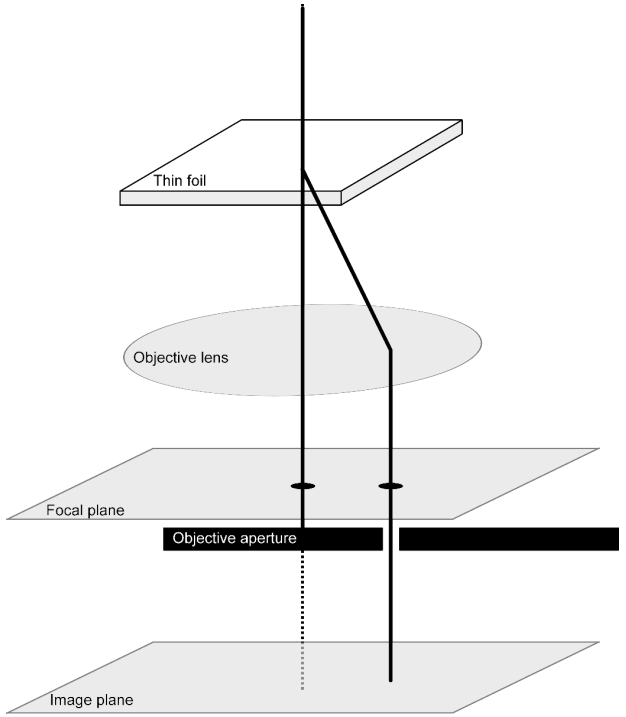
Bright field

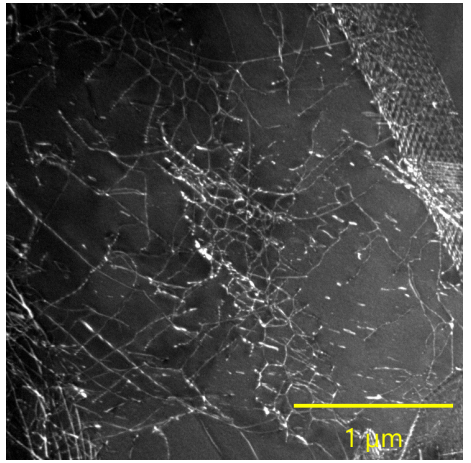




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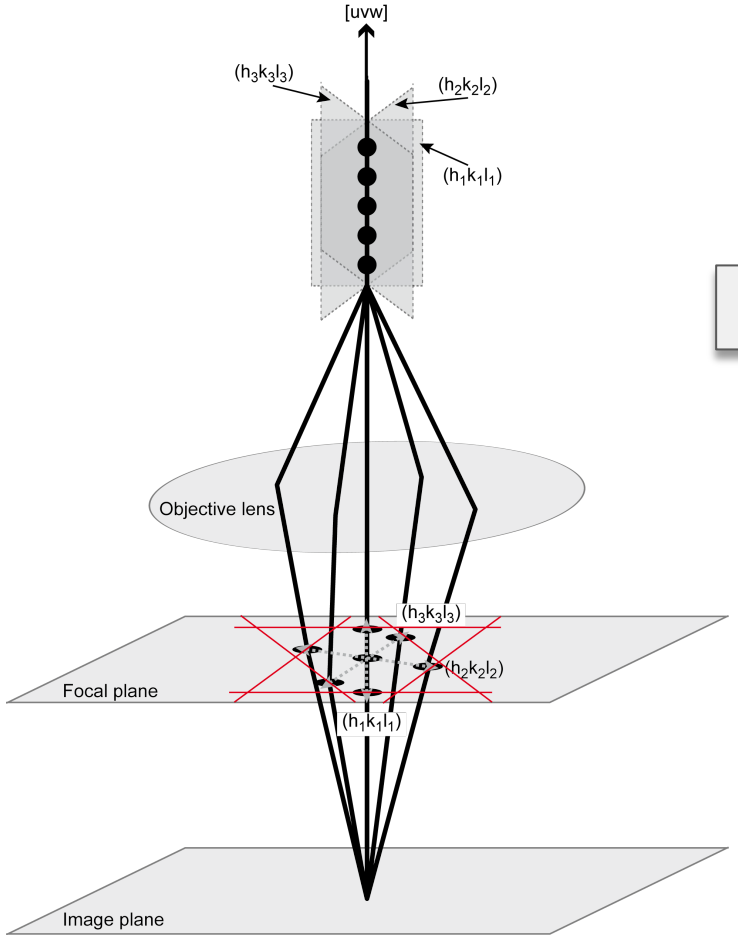
Dark field





↳Bright contrasts on a dark background

Basics of TEM contrast

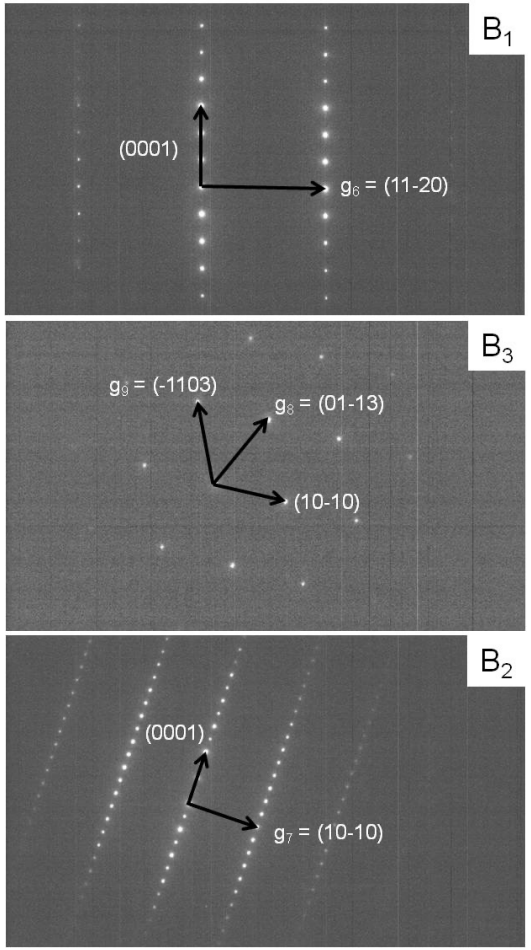


Beams are not convergent in the reality…

Diffraction mode

Spot pattern

Information on the crystallographic structure

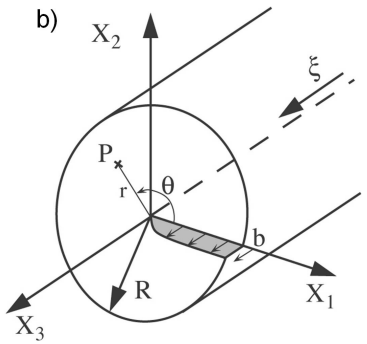


A. Guitton, *PhD Thesis* (2013)

Elastic theory of dislocations

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

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

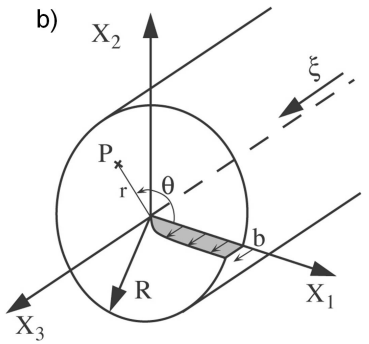


P.M. Anderson, J.P. Hirth, & J. Lothe, *Cambridge University Press* (2017)

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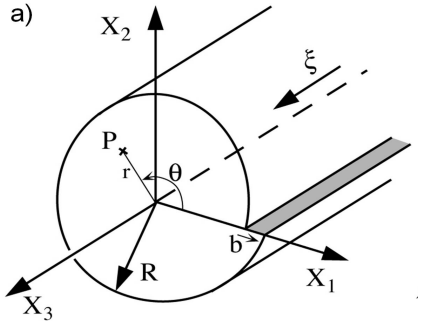
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$$\boldsymbol{R_e} = \underbrace{\frac{\boldsymbol{b}\theta}{2\pi}\left[\theta + \frac{\sin 2\theta}{2(1-\nu)}\right]}_{\parallel b} + \underbrace{\frac{\boldsymbol{b}\times\boldsymbol{\xi}}{2\pi}\left[\frac{1-2\nu}{2(1-\nu)}\ln r + \frac{\cos 2\theta}{4(1-\nu)}\right]}_{\perp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

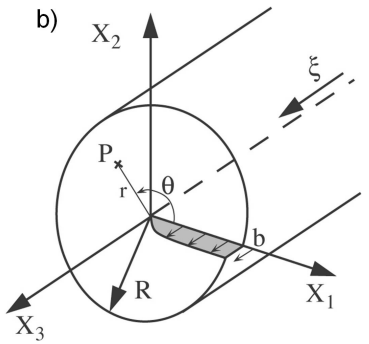


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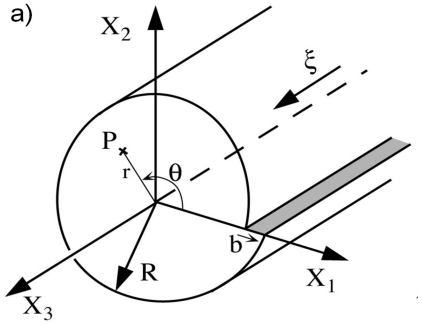
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  - Mixed dislocation ($\boldsymbol{b} \angle \boldsymbol{\xi}$)

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 $\boldsymbol{r_j}$

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

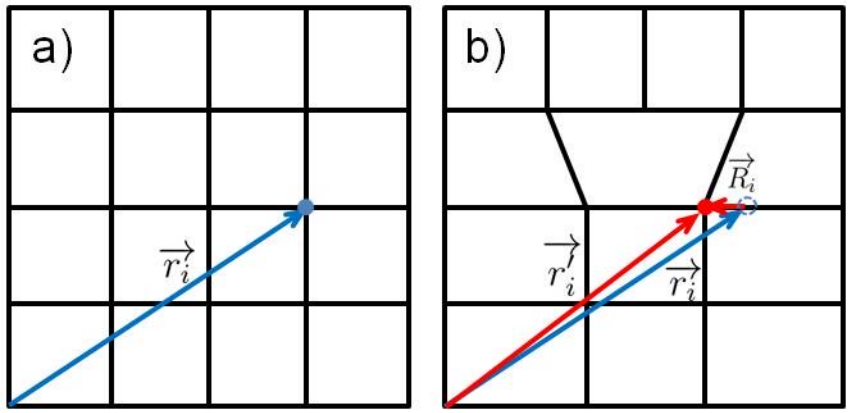
  - The total diffracted wave:

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

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

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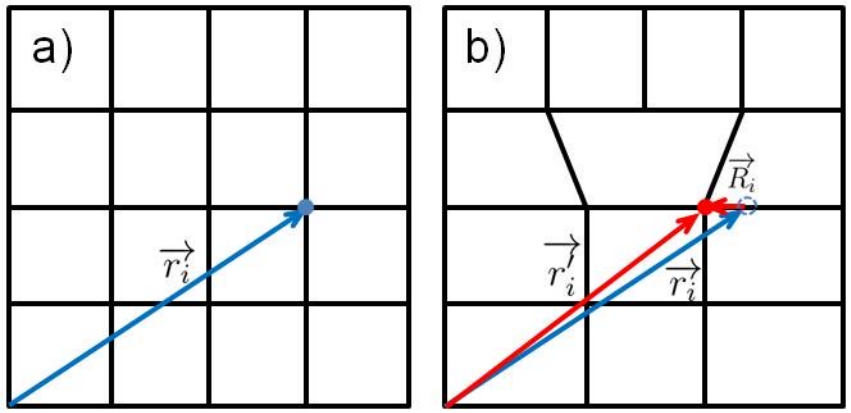
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❖ Unperfect crystal

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

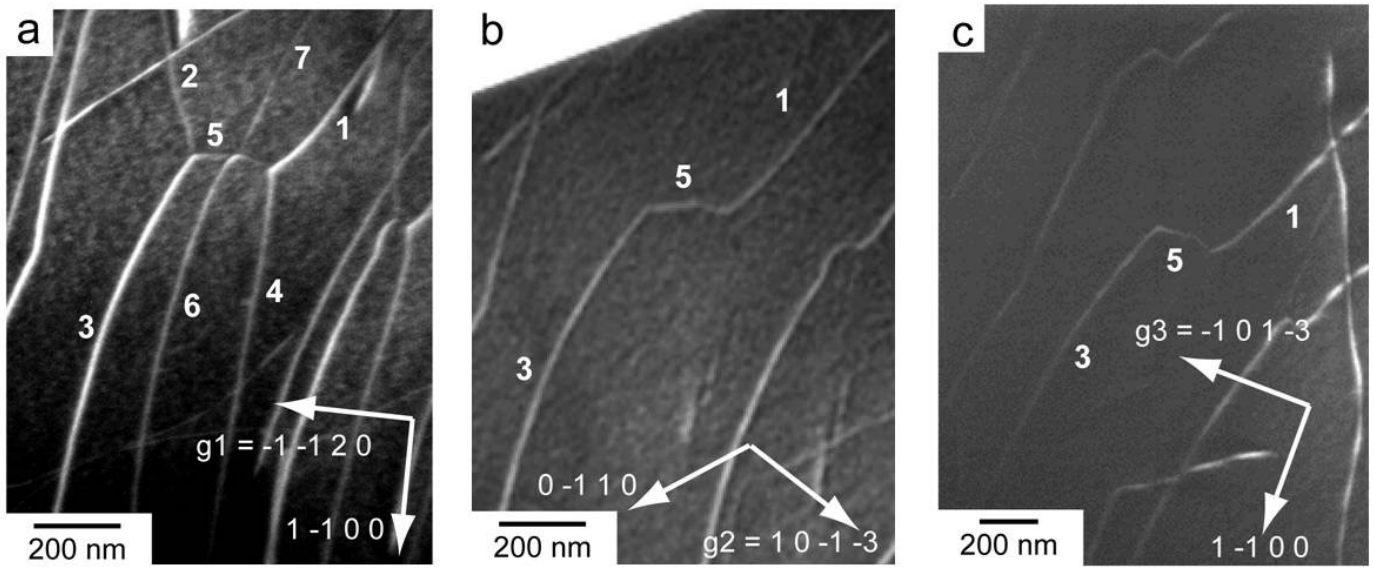
$$\psi'_d(\boldsymbol{r}, t) \propto \sum_j f_j \exp\left[2\pi i \boldsymbol{G} \cdot (\boldsymbol{r_j} + \boldsymbol{R_j})\right] = \sum_j f_j \exp\left[2\pi i \boldsymbol{G} \cdot \boldsymbol{r_j}\right] \times \sum_j f_j \exp\left[2\pi i \boldsymbol{G} \cdot \boldsymbol{R_j}\right] = \sum_j f_j \exp\left[2\pi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

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

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

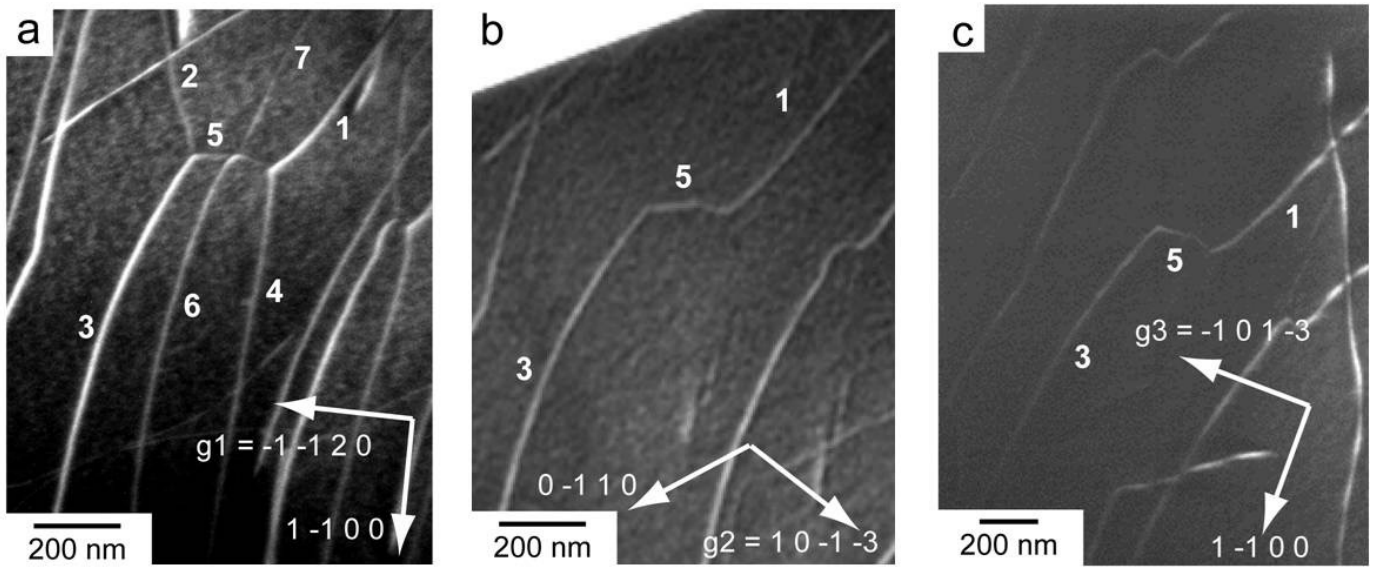
↳ If $\boldsymbol{G} \cdot \boldsymbol{R_j} = 0$ the defect will be out of contrast

Example of extinction condition



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

Example of extinction condition

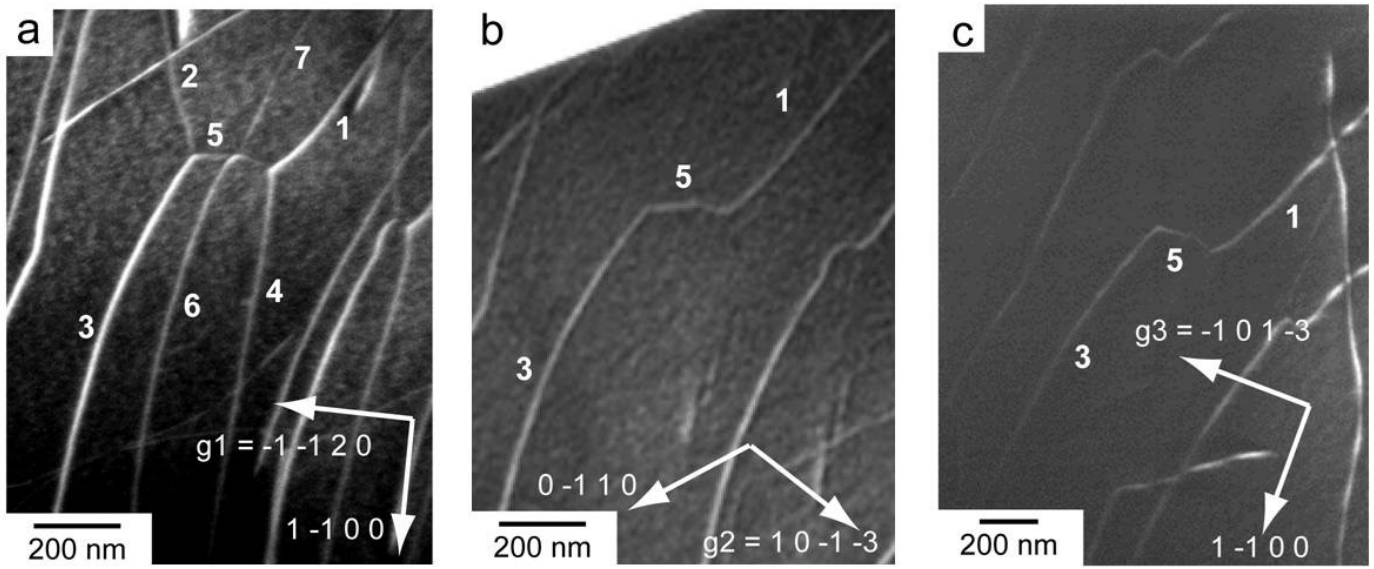


❖ The dislocation 2 is obviously out of contrast for both $g_2 = (1\ 0\ \bar{1}\ \bar{3})$ and $g_3 = (\bar{1}\ 0\ 1\ \bar{3})$

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

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

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

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

The 2 types of electron microscopes

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- Invention: 1931 by Ernst RUSKA et Max KNOL
- Specimen : thin foils (~ 100 nm)
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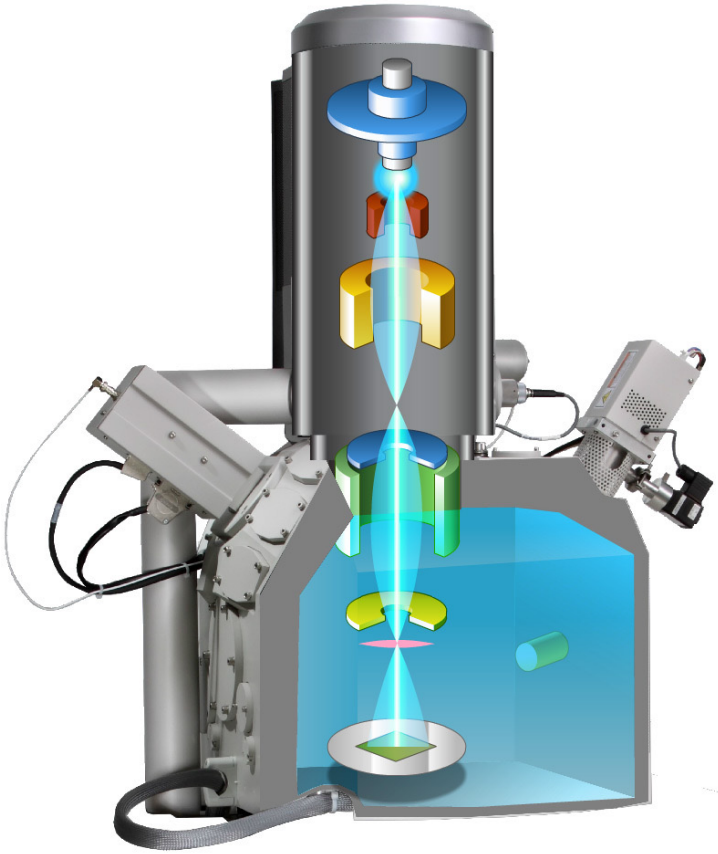


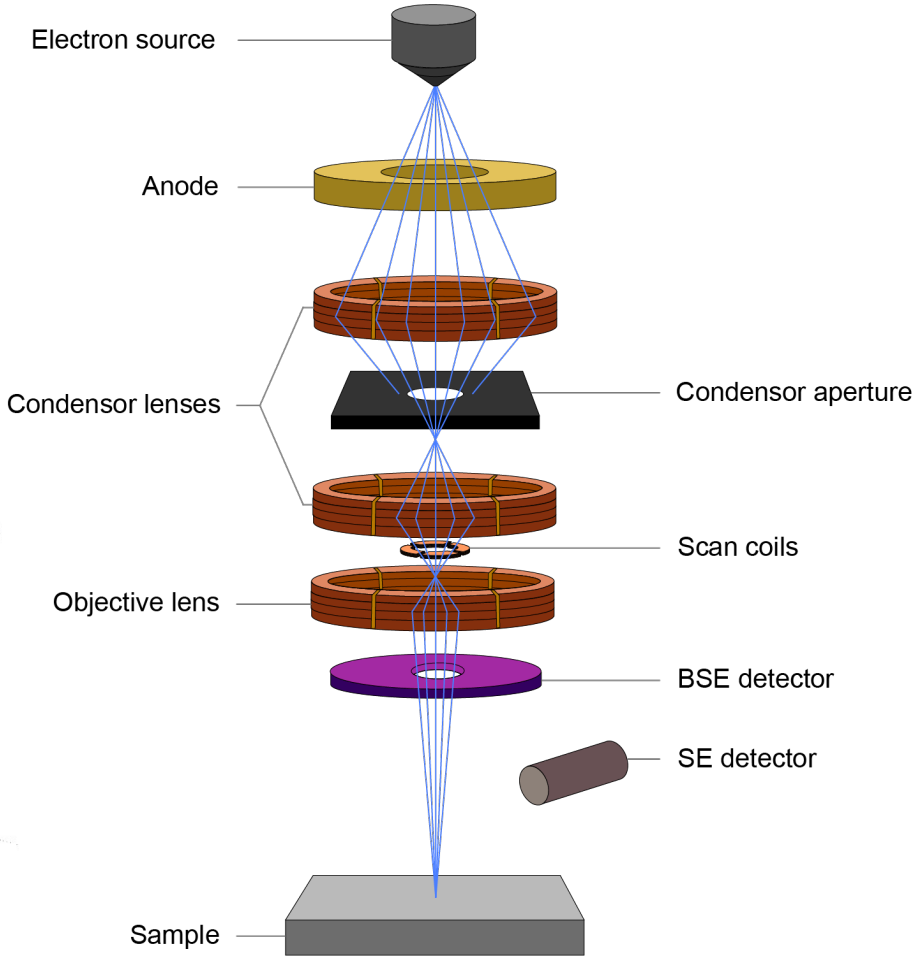
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Basics of SEM

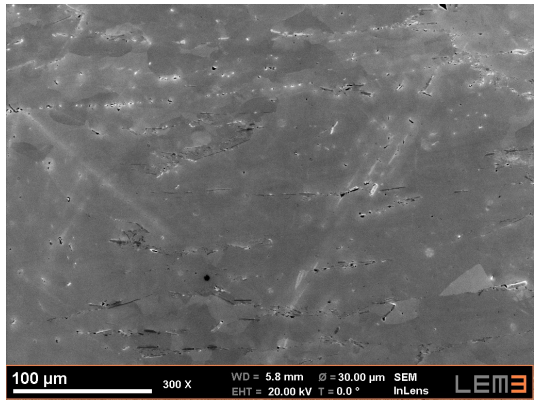




J. Aubourg, *PhD Thesis (2024)*

SEM imaging

Secondary electrons (SE)



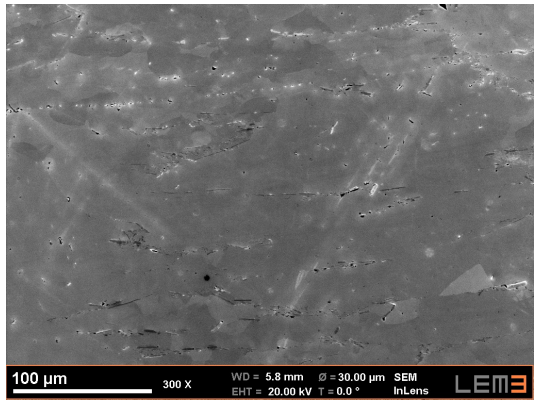
- Inelastic scattering between incident electrons and outer-shell electrons of sample atoms ⇒ emission of SE
- Inelastic scattering involving inner-shell electrons, followed by electronic transitions → emission of characteristic X-rays

⇒ EDS

↳Topographic contrast

SEM imaging

Secondary electrons (SE)



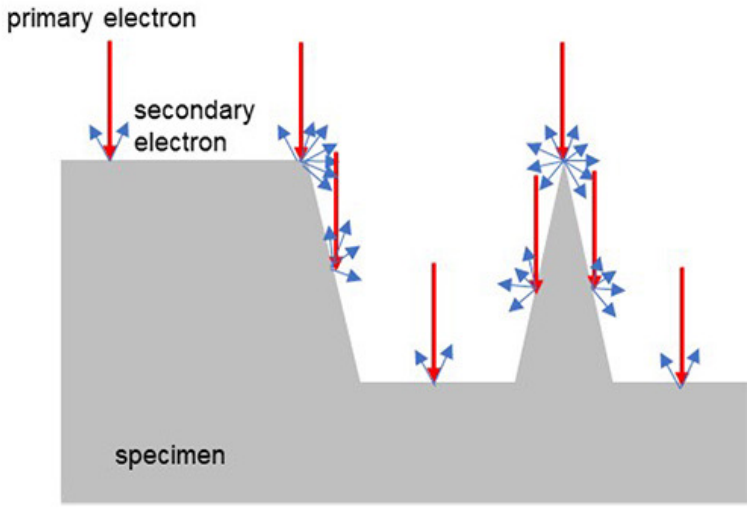
- Inelastic scattering between incident electrons and outer-shell electrons of sample atoms ⇒ emission of SE
- Inelastic scattering involving inner-shell electrons, followed by electronic transitions → emission of characteristic X-rays

⇒ EDS

↳Topographic contrast

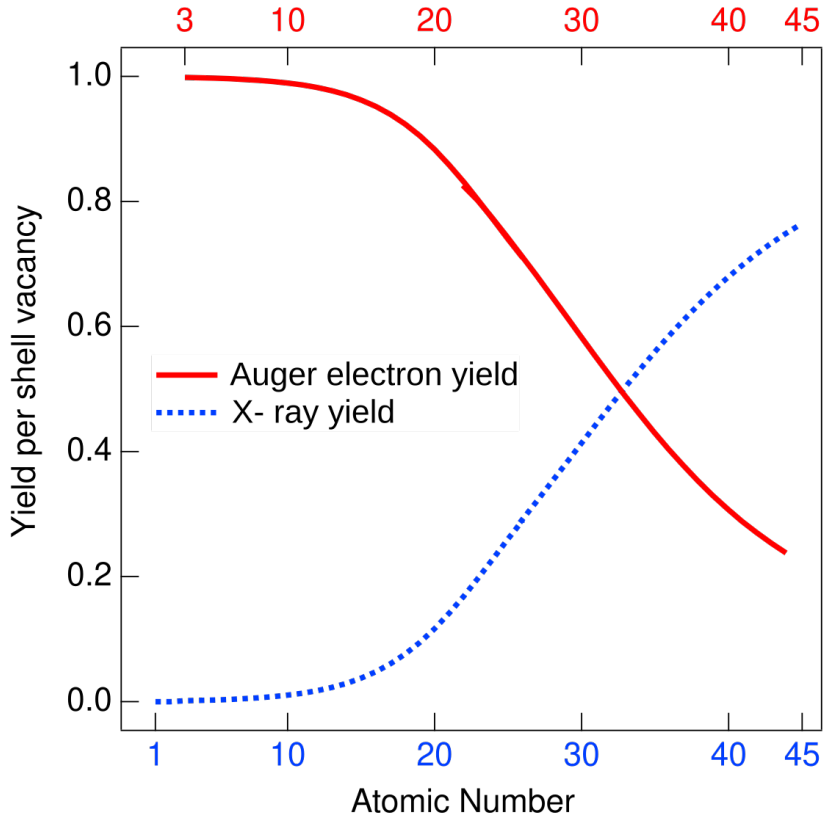
The edge effect

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



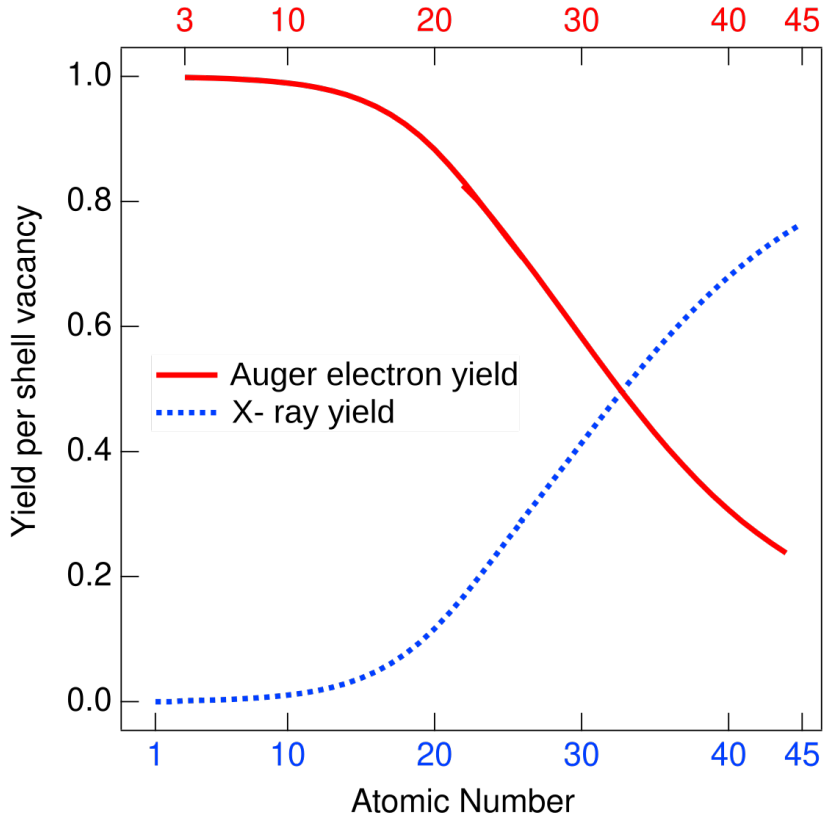
↳SEM is not 3D, but it gives a strong 3D impression.

Energy Dispersive X-ray Spectroscopy (EDS)



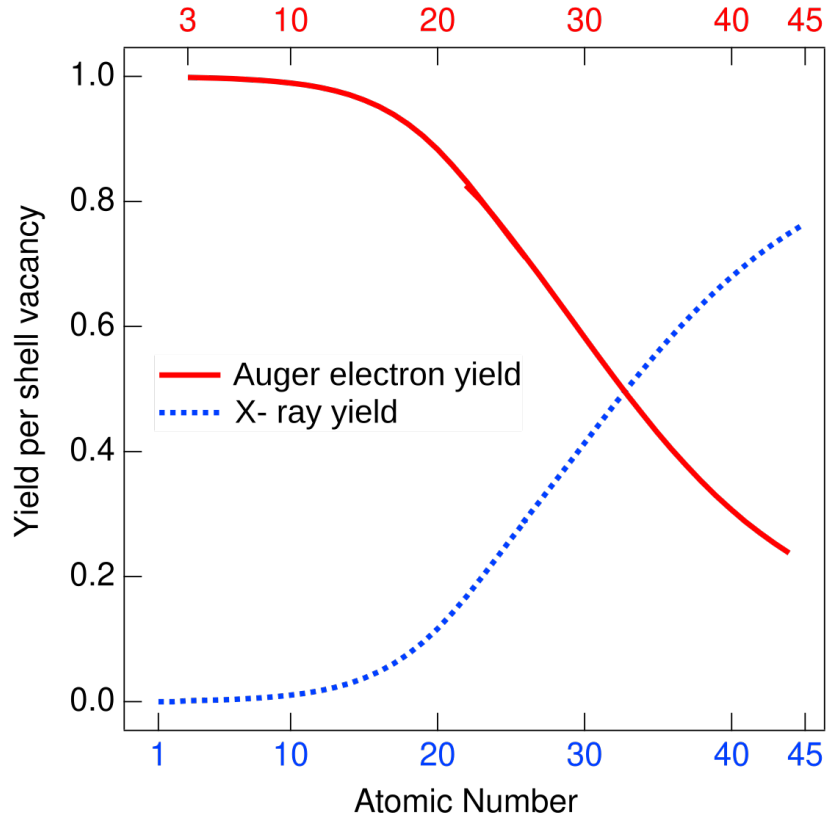
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  - When the high-energy electron beam hits the sample, it can eject inner-shell electrons (usually from K or L shells) in the atoms.

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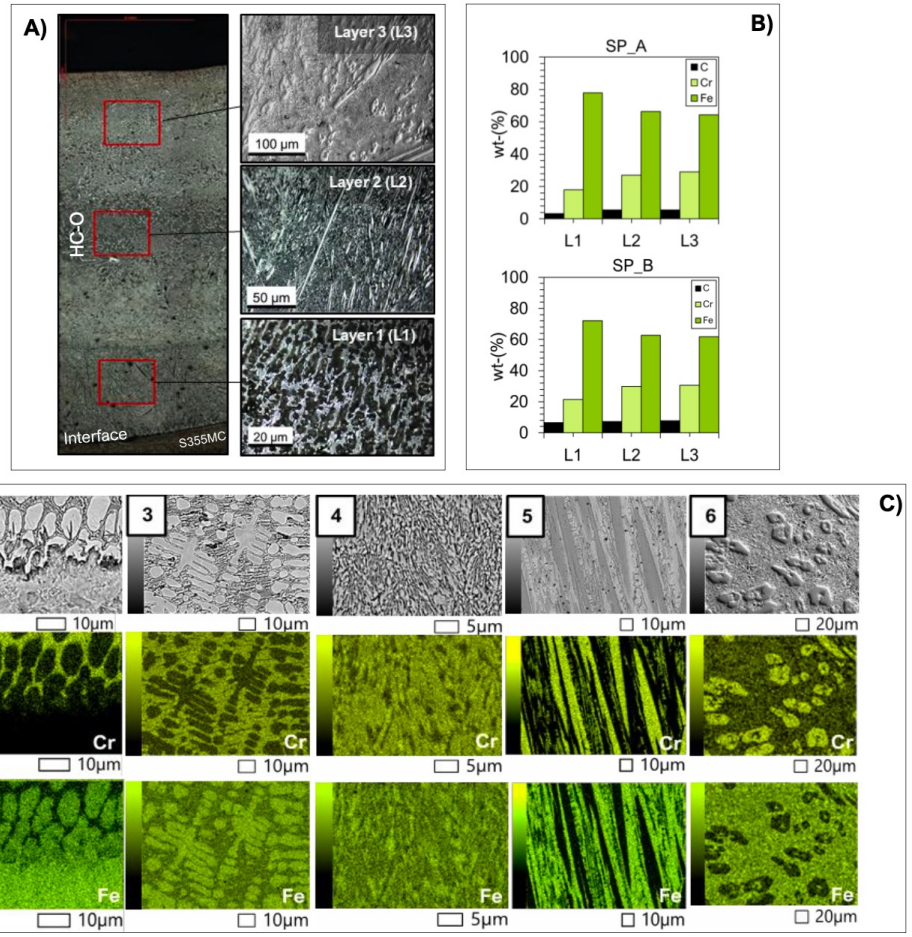
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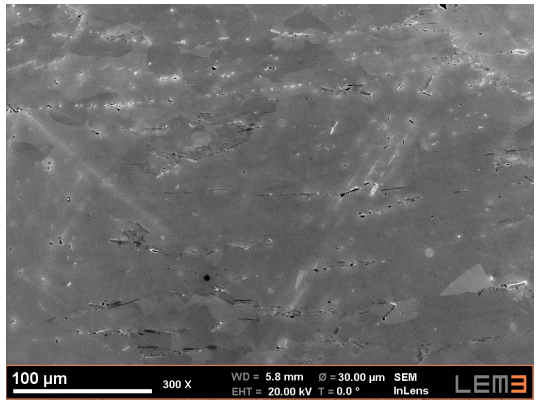


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- **Detection:**
  - An EDS detector measures the energy of these X-rays, allowing us to:
    - ✓ Identify the elements present (qualitative analysis)
    - ✓ Estimate their relative amounts (semi-quantitative analysis but not accurate for low Z elements)

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

SEM imaging

Secondary electrons (SE)

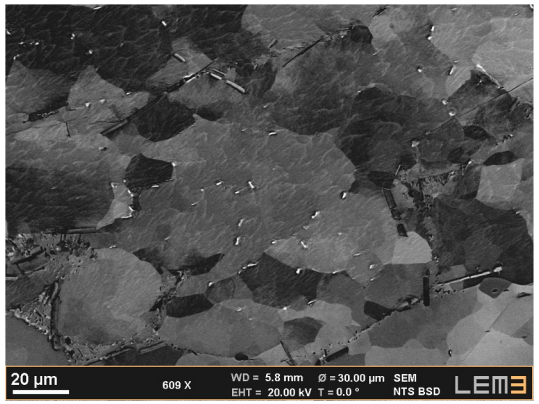


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↳Topographic contrast

BackScattered Electrons (BSE)



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

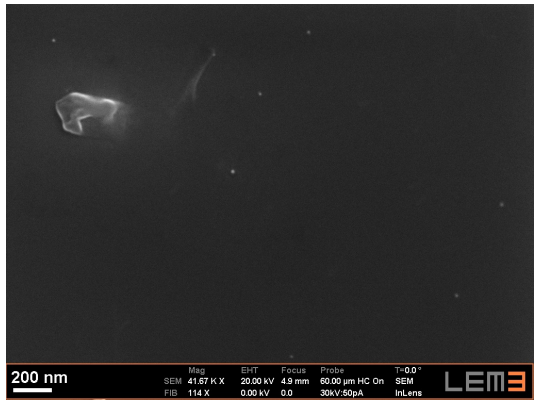
⇒ EBSD, ECCI

↳Topographic contrast
↳Chemistry contrast
↳Orientation contrast

↳ The two imaging modes are complementary.

SEM imaging

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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)

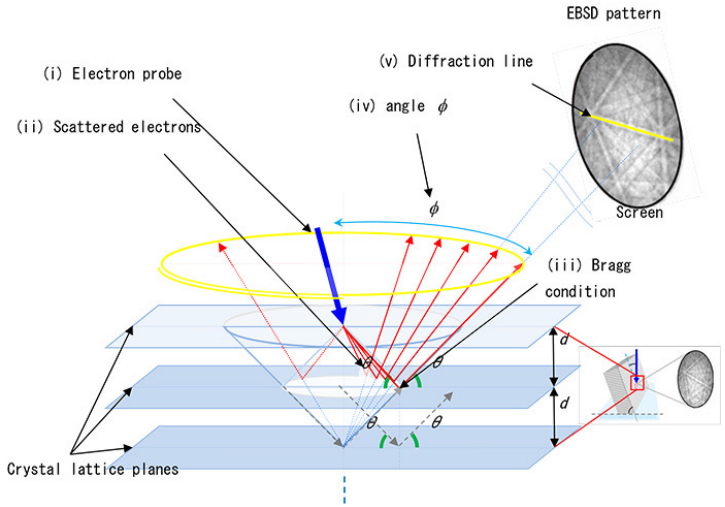
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- **Relative contributions within the BSE signal**
  - Orientation contrast (electron channeling): ~2–5% of the BSE contrast
  - Atomic number contrast (Z-contrast)
  - ~10–30% of the BSE contrast
  - Topographic contrast: ~65–90% of the BSE contrast

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

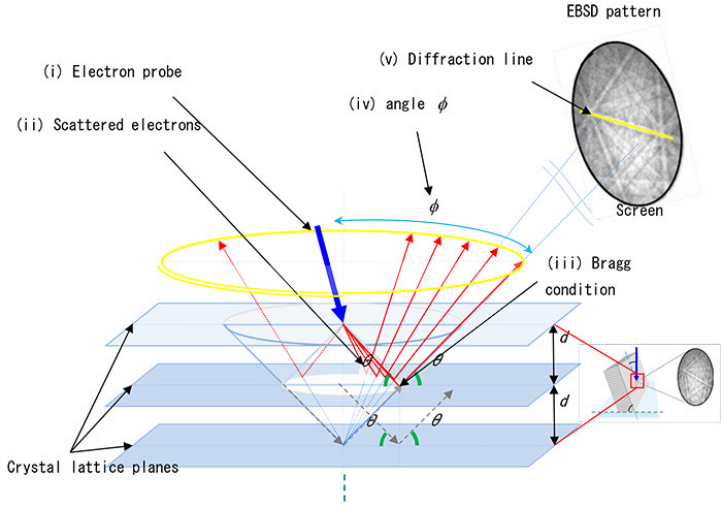
Basics of the Electron BackScattered Diffraction (EBSD)



❖ 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.

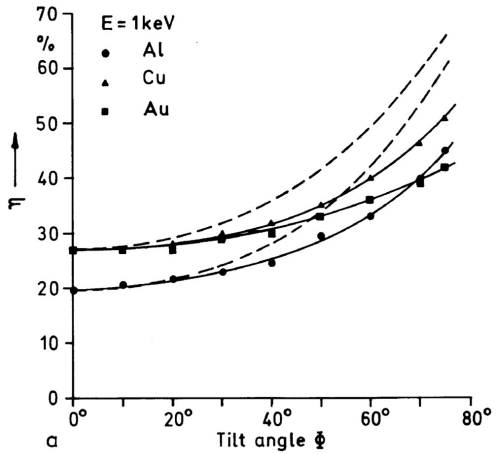
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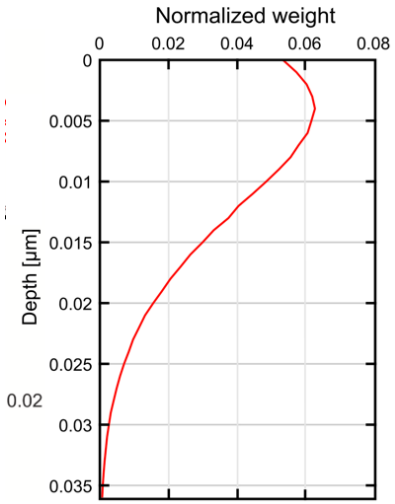


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↳The BSE yield increases with increasing tilt angle



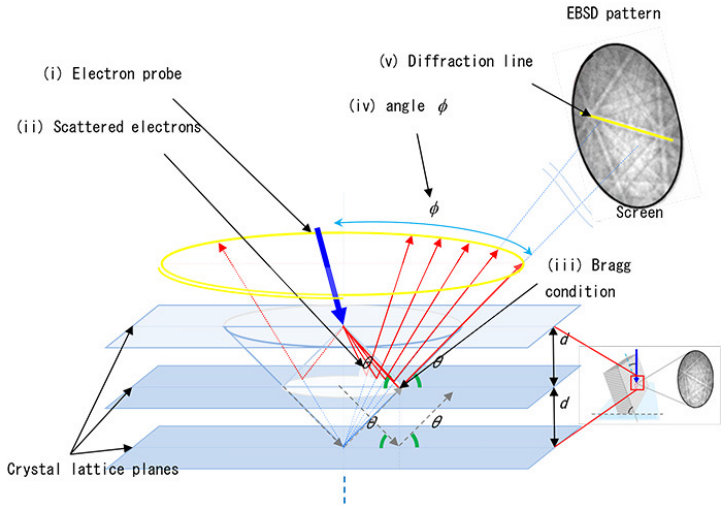


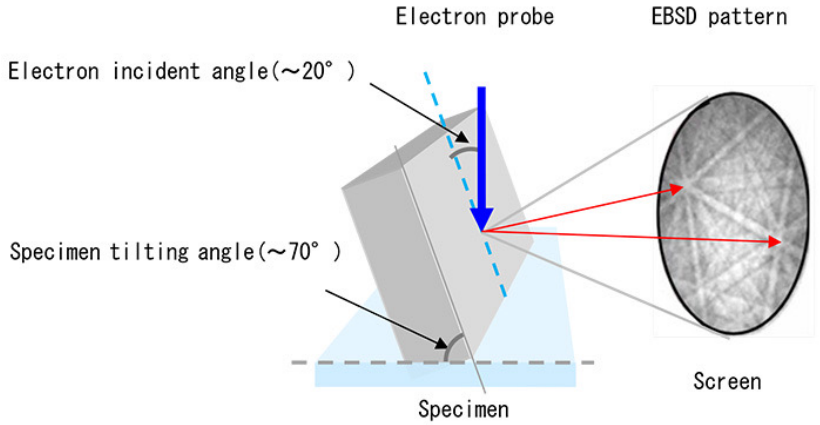
↳The probing depth in GaN at 15 keV is ~35 nm below the surface.

www.jeol.co.jp

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

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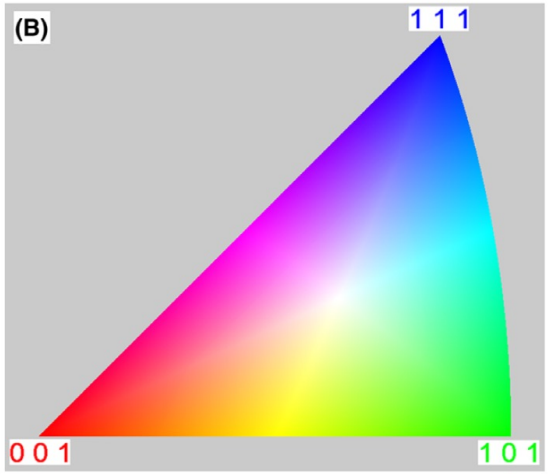
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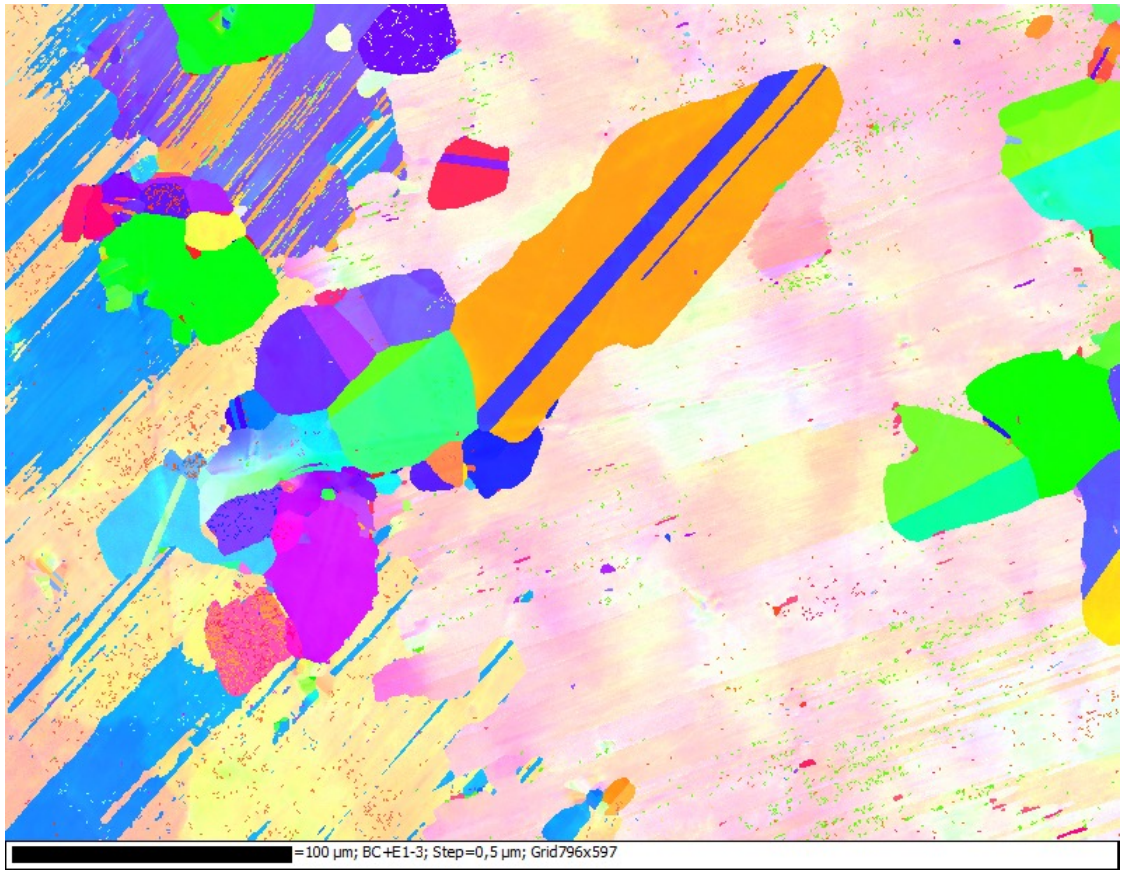
❖ Basics of the EBSD:

- 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.

Example of extinction condition

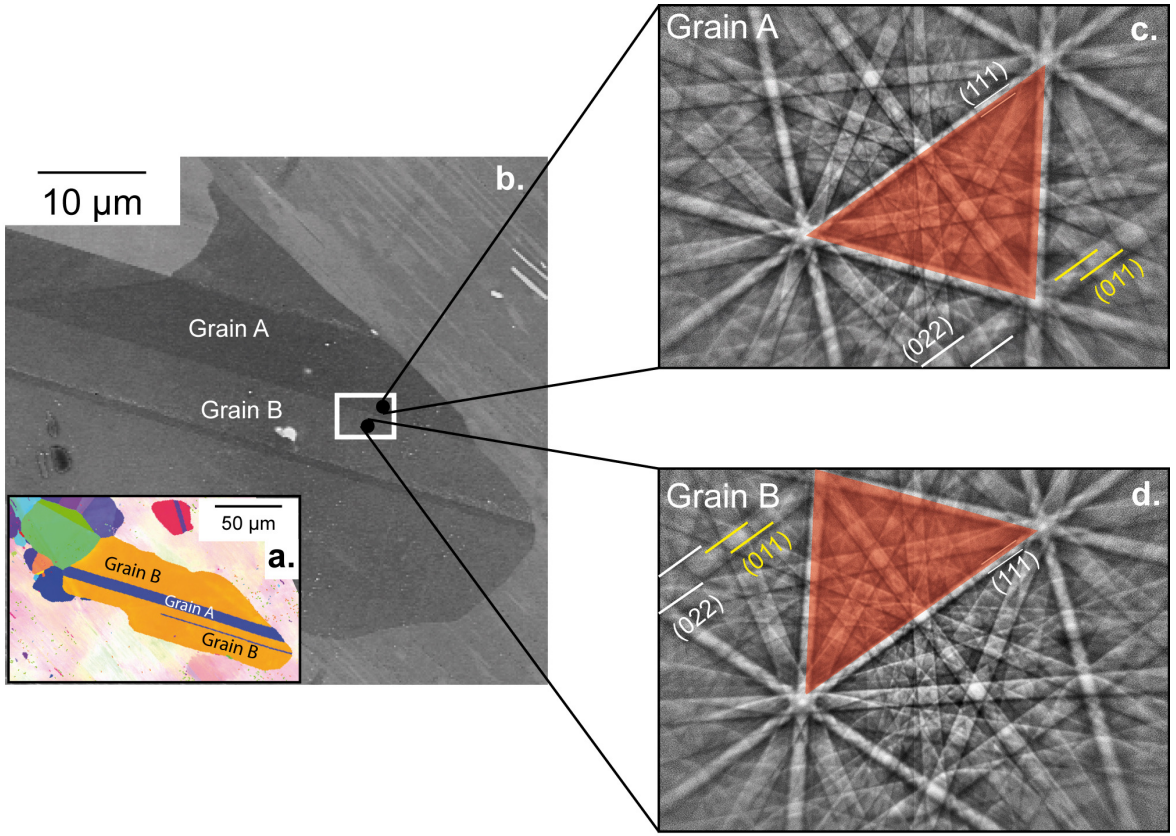
- **Capabilities of EBSD:**
  - Orientation measurements
  - Local misorientation measurements
  - Grain identification
  - Phase identification
  - Statistics on the microstructure





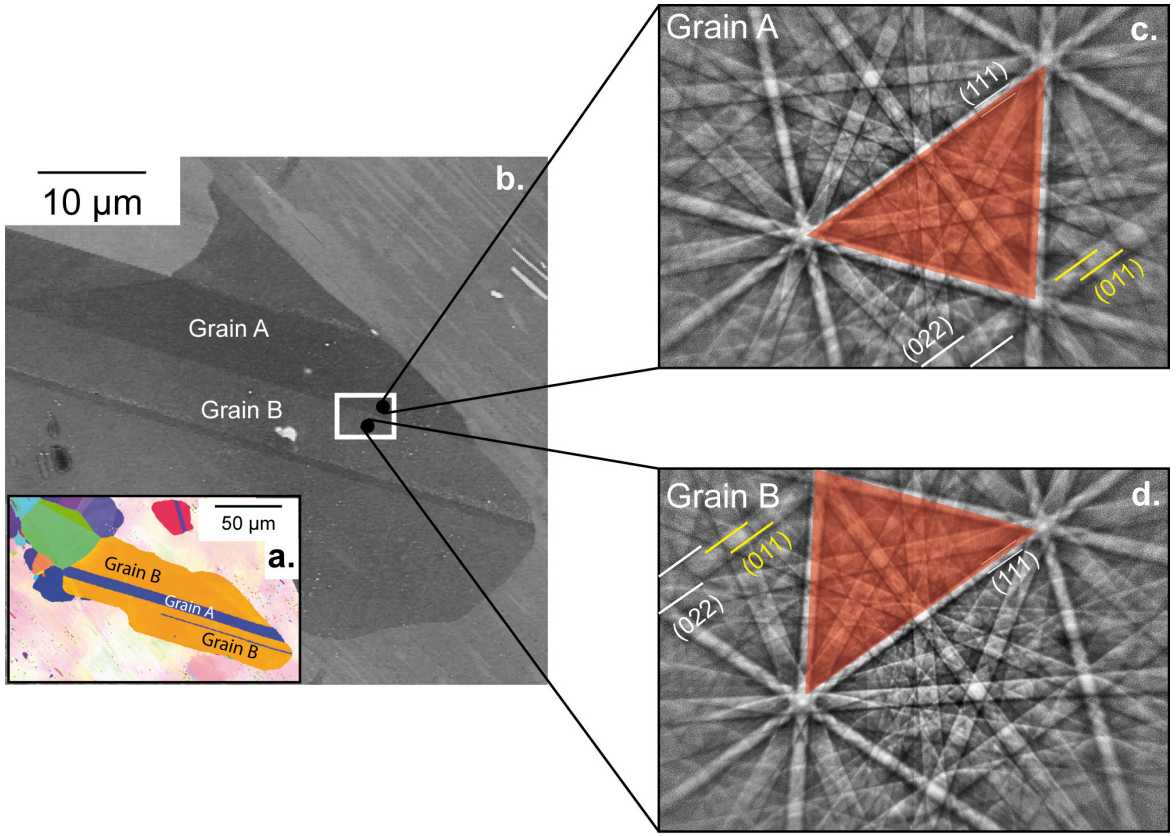
A. Guitton et al., *Materials (2018)*

What is the nature of the boundary?



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

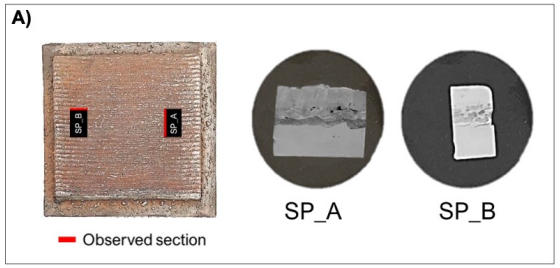
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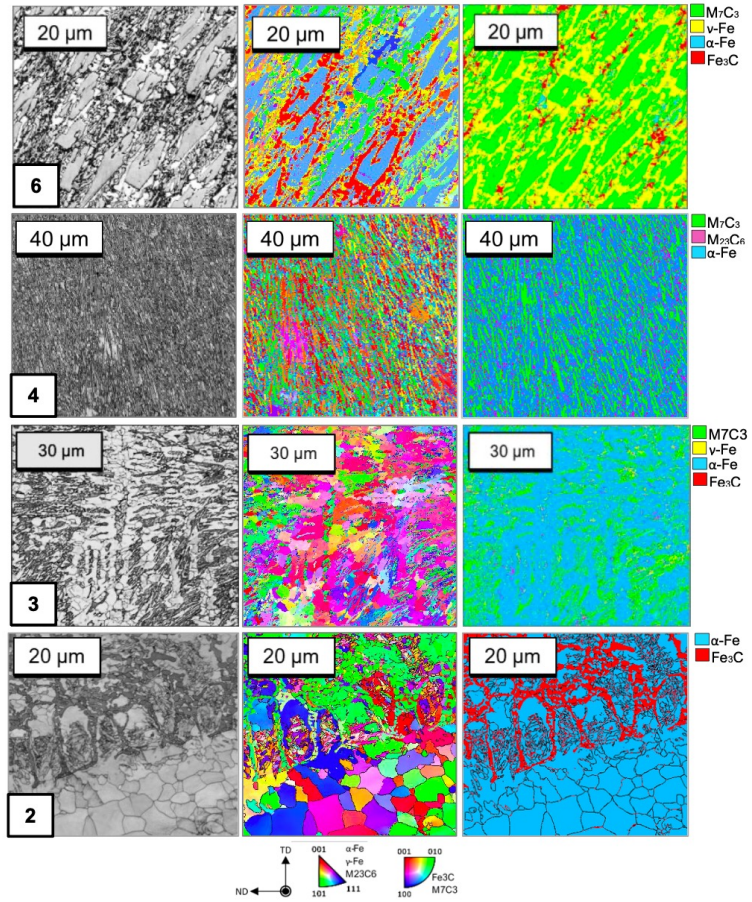


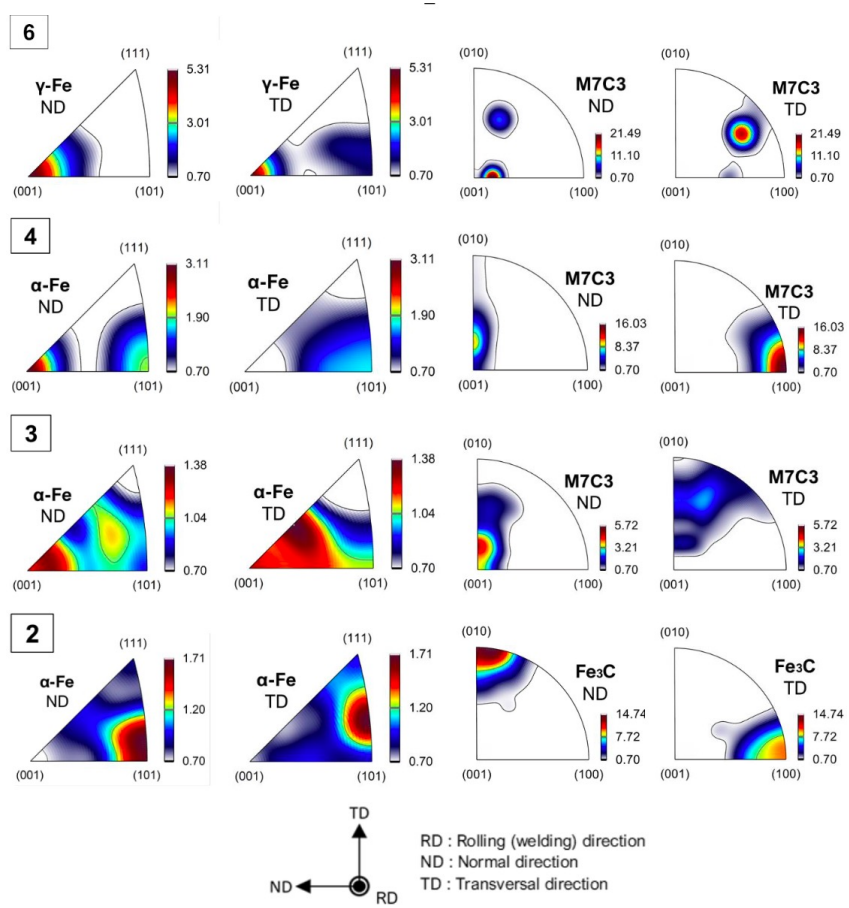
↳ **This is a $(1\,1\,1)$ twin boundary.**

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

Texture measurements by EBSD



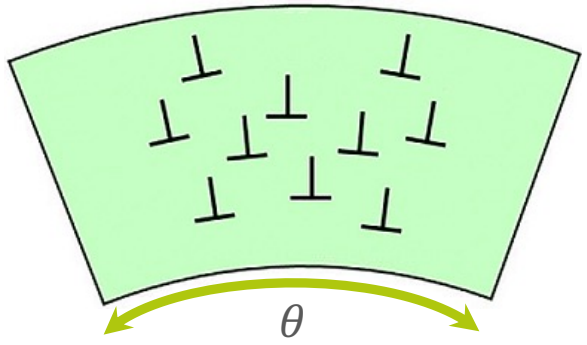




A. Monnet et al., *Mater. Charac.* (2026)

Different types of dislocation densities

Geometrically necessary dislocations (GND)



⇒ Lattice curvature at long range

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

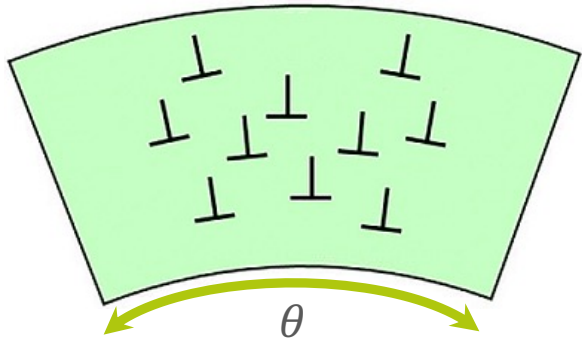
$$\boldsymbol{\alpha} = curl\ \boldsymbol{\varepsilon} + \mathrm{tr}(\boldsymbol{\kappa_e}) \cdot \mathbb{I} - \boldsymbol{\kappa_e^T}$$

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

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

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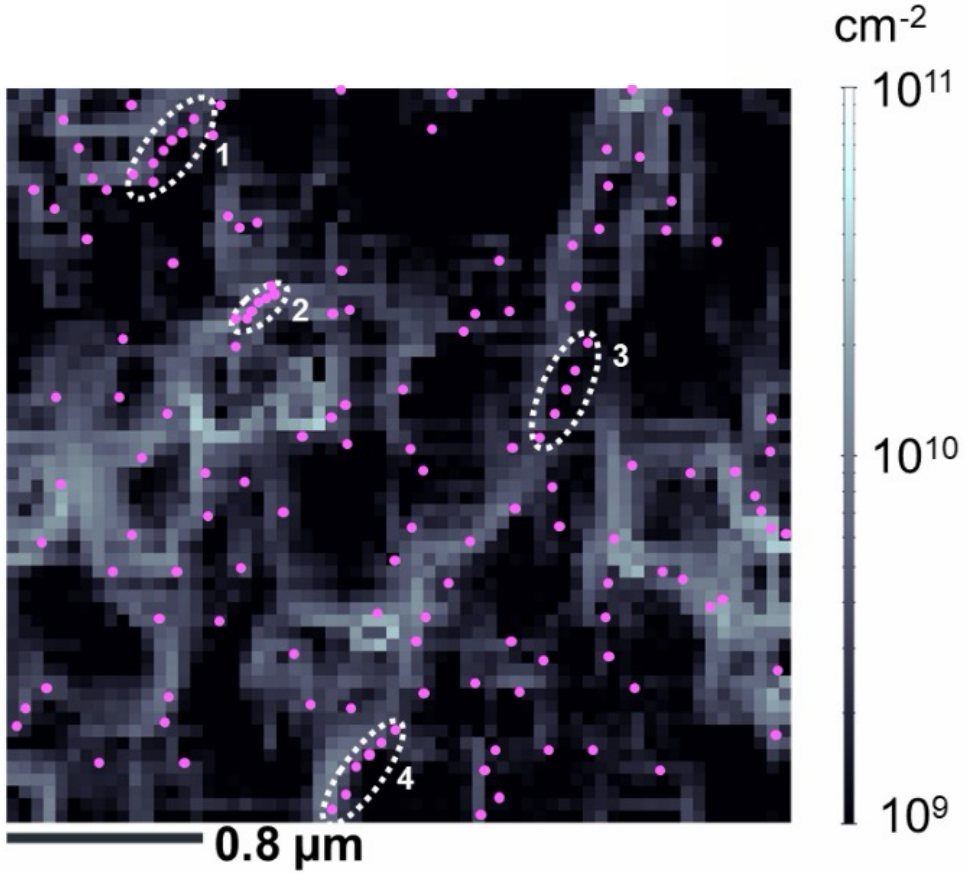
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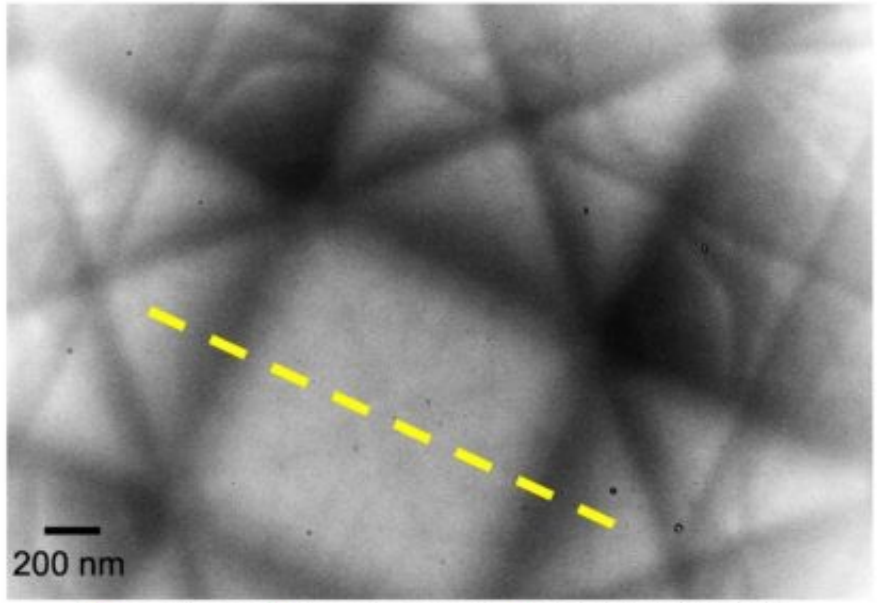
Norm of the Nve tensor ($\|\alpha\|$)



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.

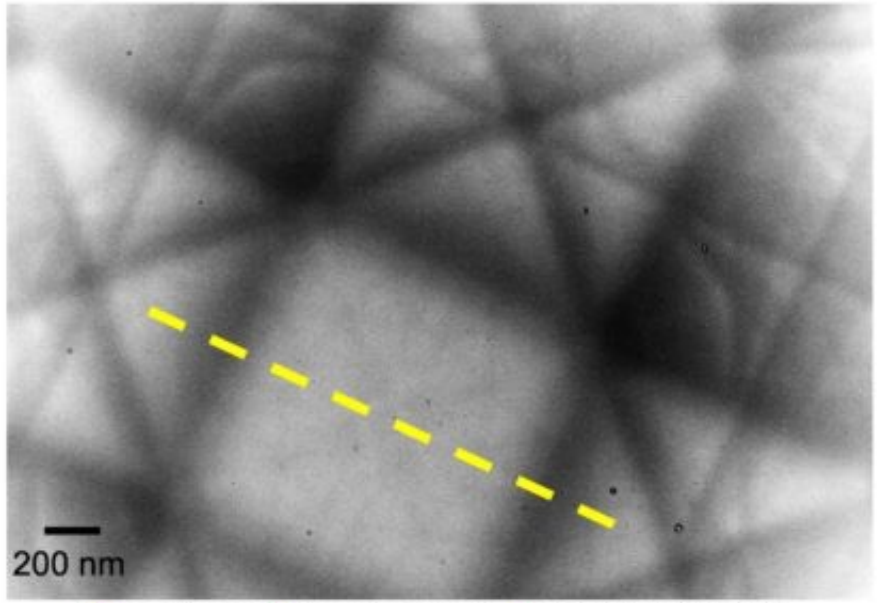


Electron Channeling Pattern "ECP" obtained from Si single crystal.

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

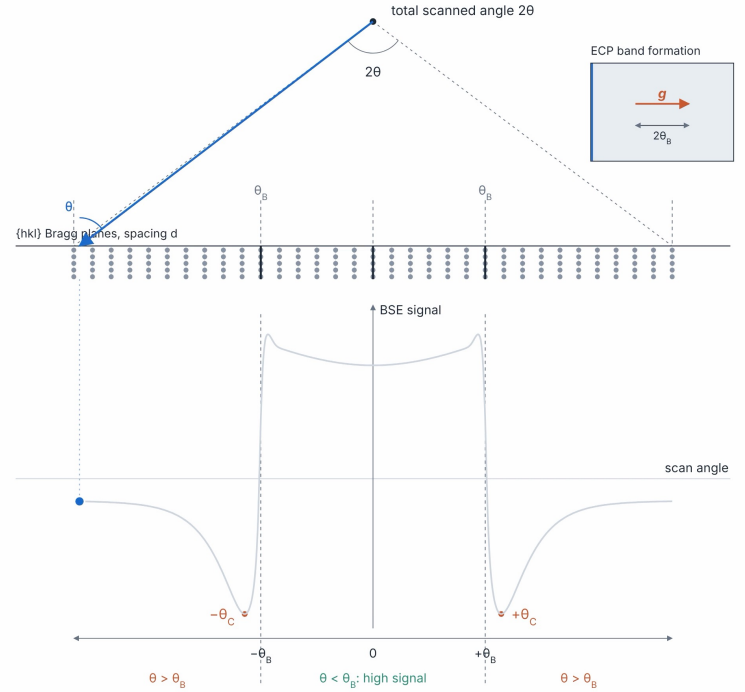
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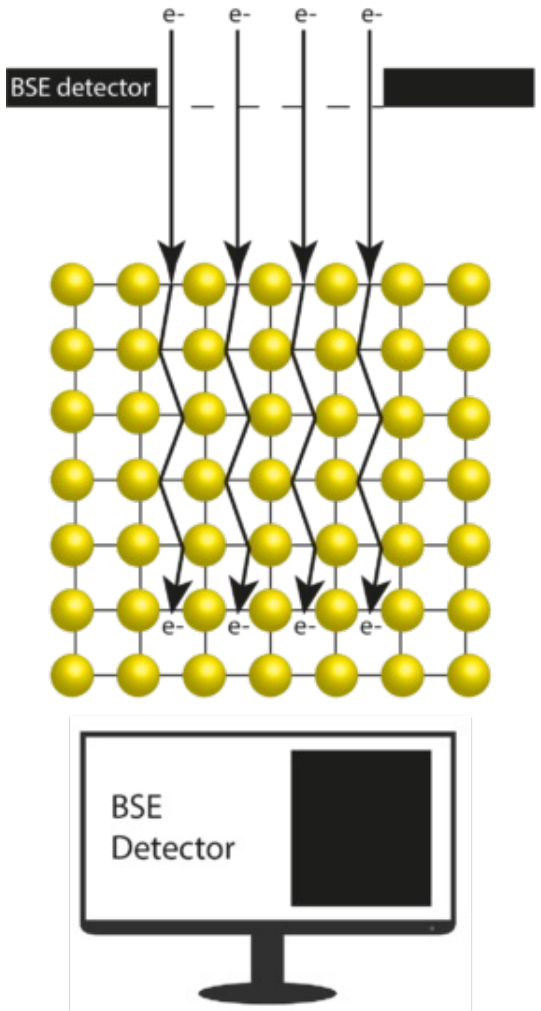
- BSE yield depends on the specimen orientation relative to the incident beam.



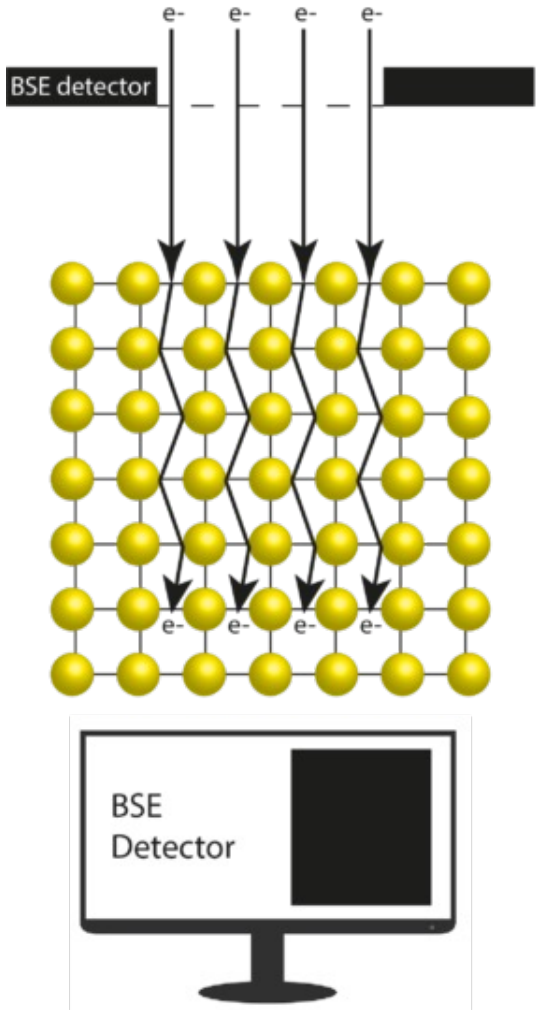
Channeling condition ≠ Bragg condition

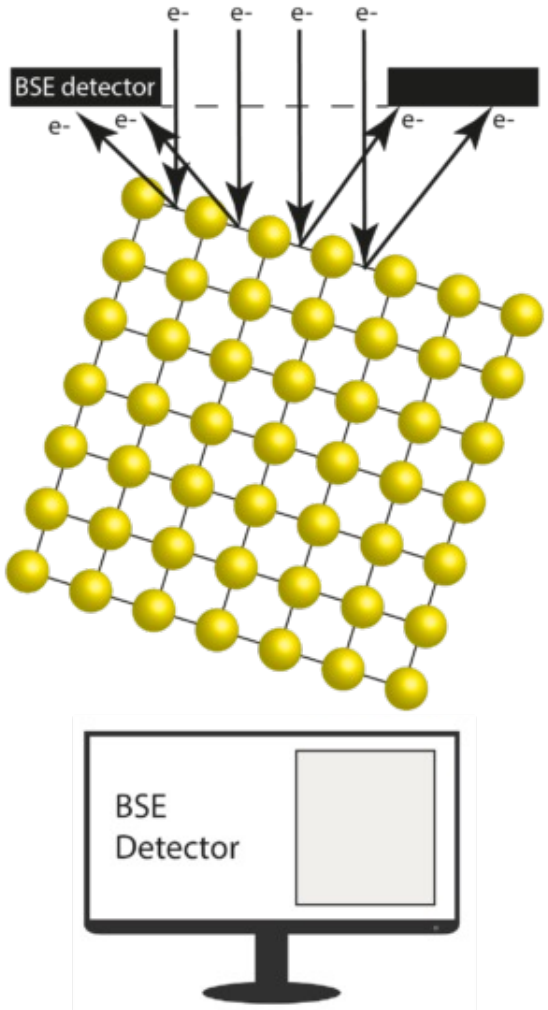
D. G. Coates, *Philos. Mag.* (1967); G.R. Booker et al., *Philos. Mag.* (1967)

What is the Electron Channeling Contrast Imaging?



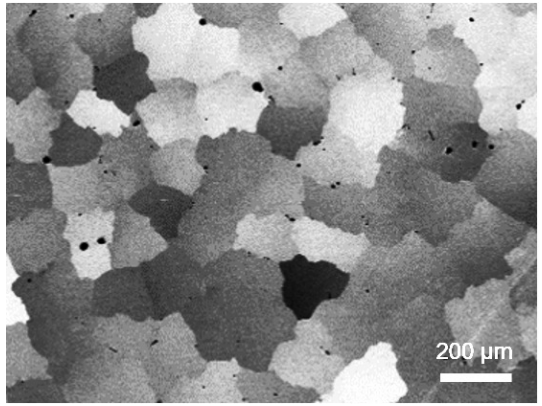
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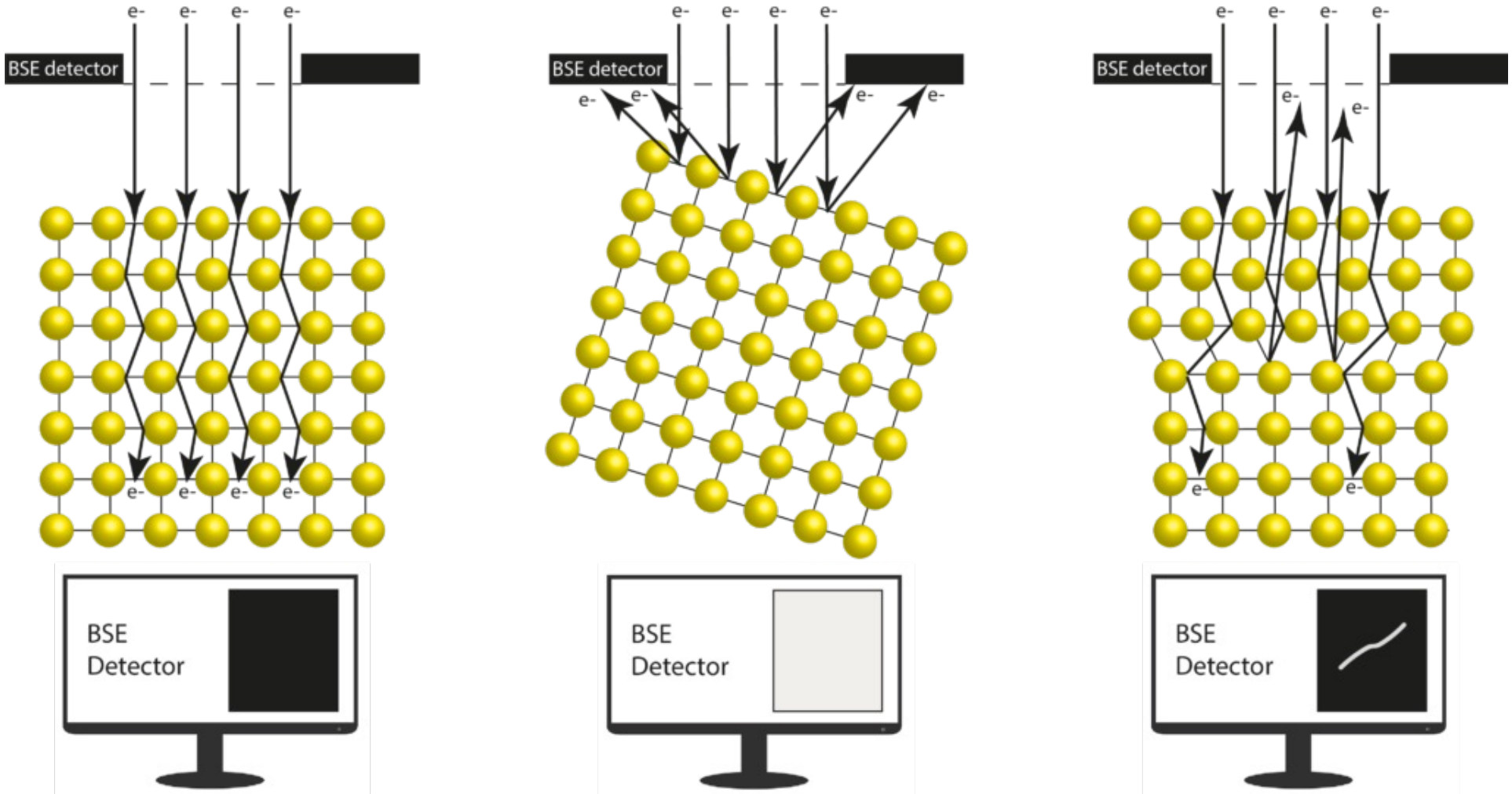
This is not a polycrystal!

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

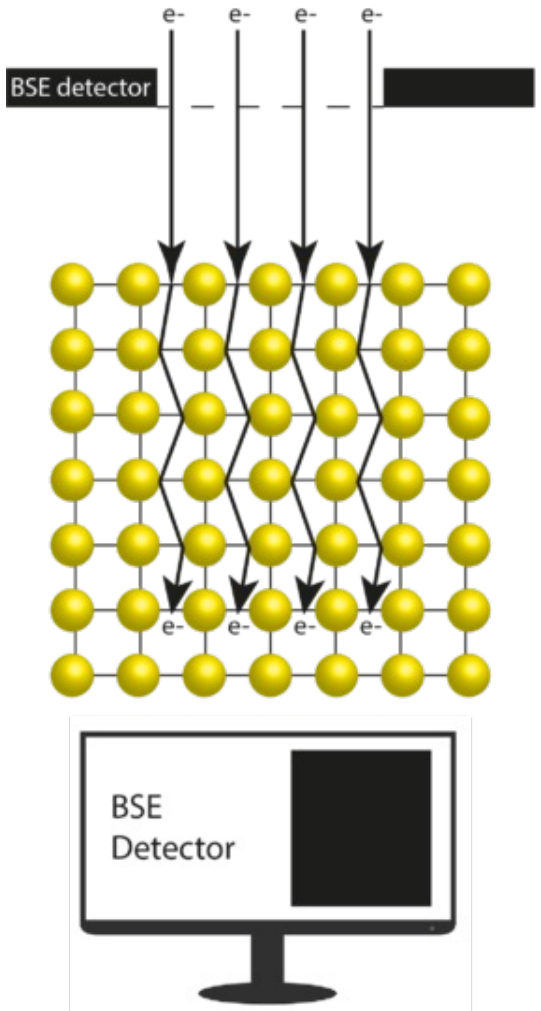


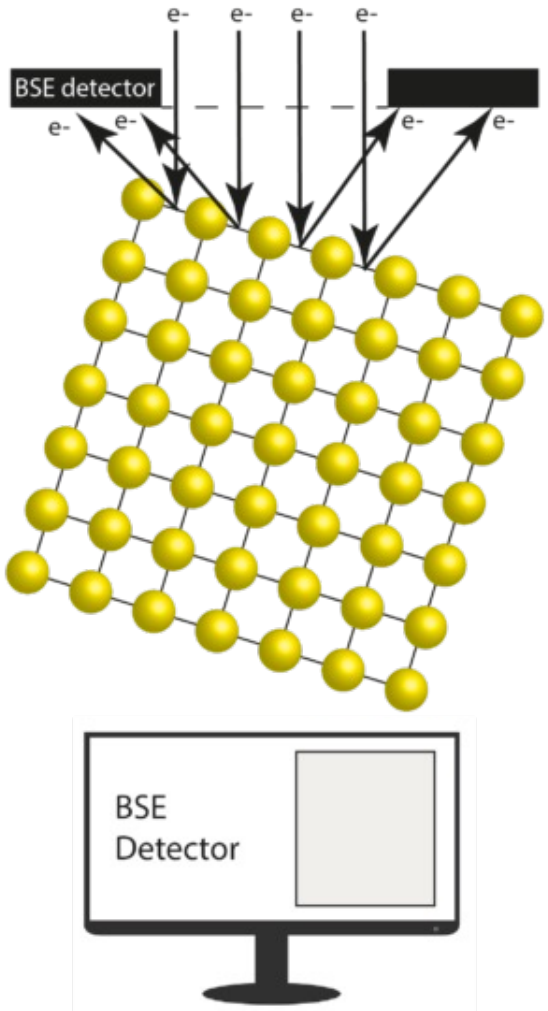
Single crystal of CrCoNi with dendrites
(mean misorientation: <1°)

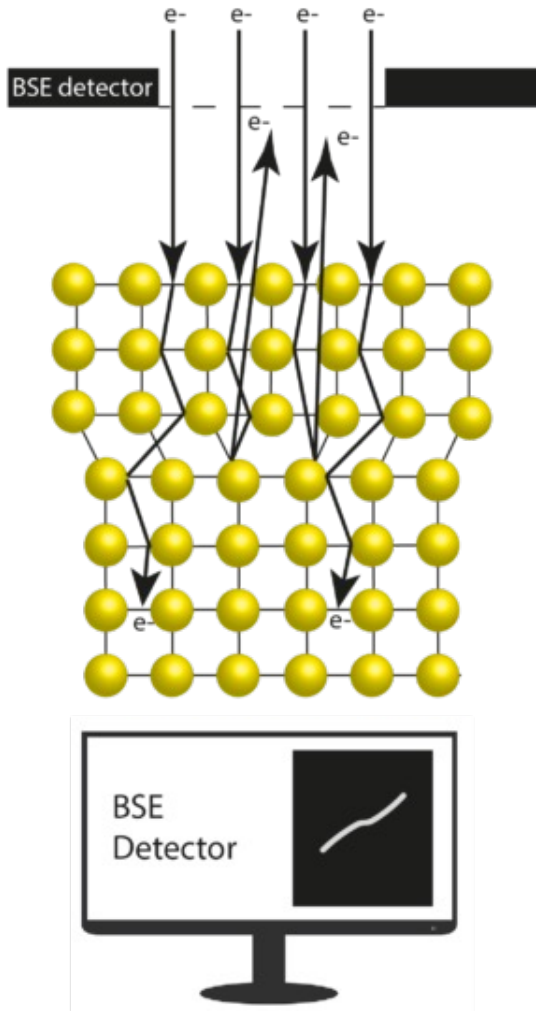
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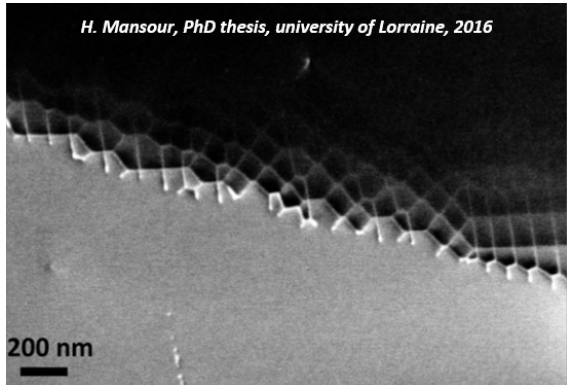


Dislocation $\Rightarrow \begin{cases} \boldsymbol{g} \cdot \boldsymbol{b} = 0 \\ \boldsymbol{g} \cdot \boldsymbol{b} \times \boldsymbol{\xi} = 0 \end{cases}$

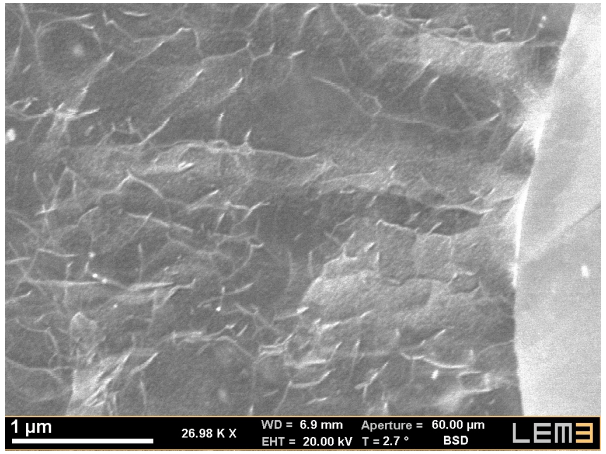
Stacking fault $\Rightarrow \boldsymbol{g} \cdot \boldsymbol{R} = 0$

Nature of the crystalline defects

Detailed characterization of crystalline defects

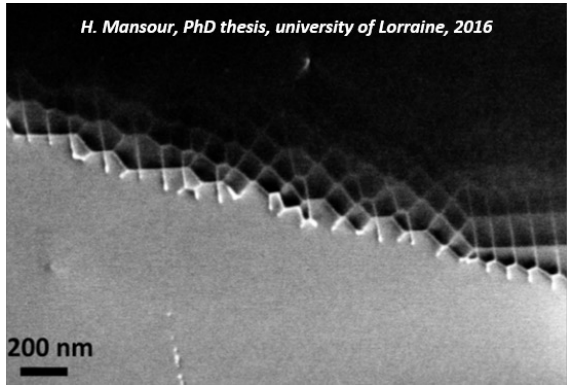


Ceramics (UO_2)

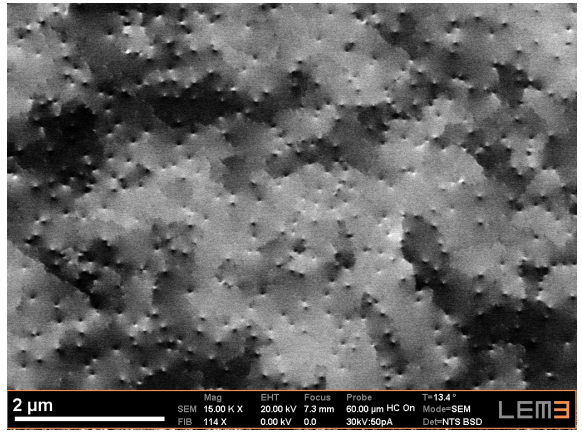


Ceramics (MAX phase)

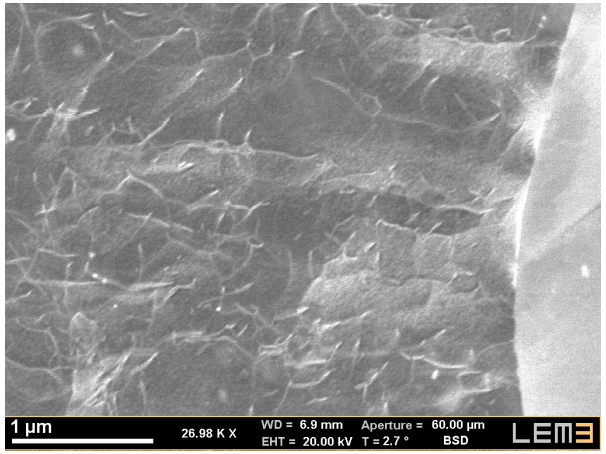
Detailed characterization of crystalline defects



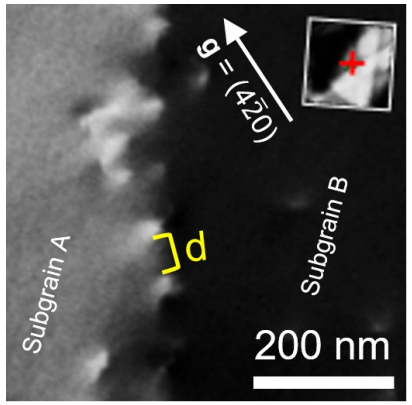
Ceramics (UO_2)



Semi-conductors (GaN)

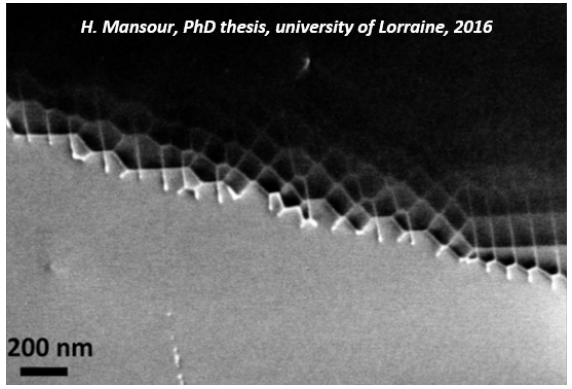


Ceramics (MAX phase)

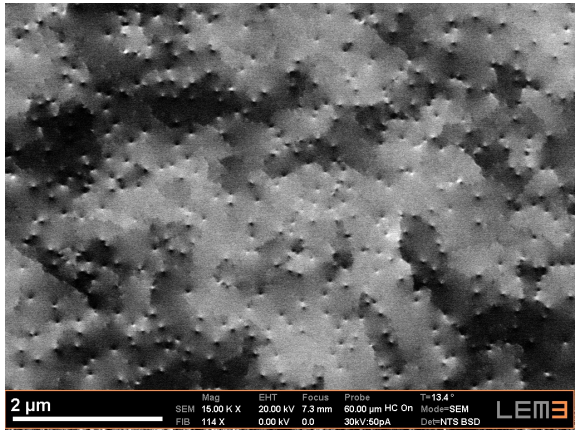


Metals (CrCoNi)

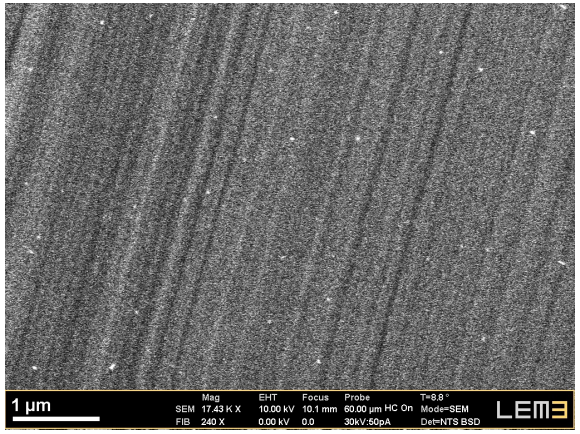
Detailed characterization of crystalline defects



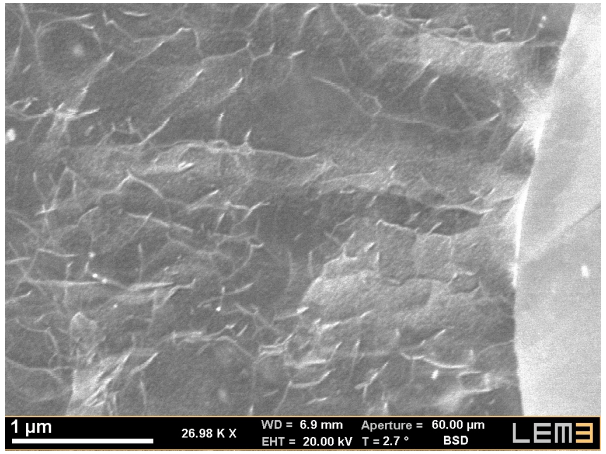
Ceramics (UO_2)



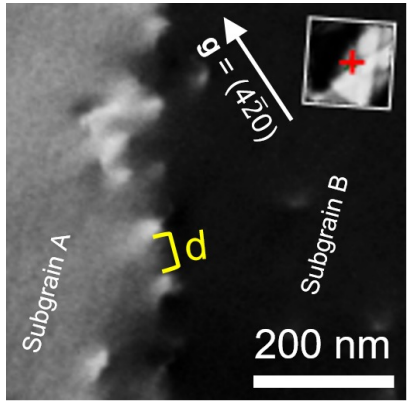
Semi-conductors (GaN)



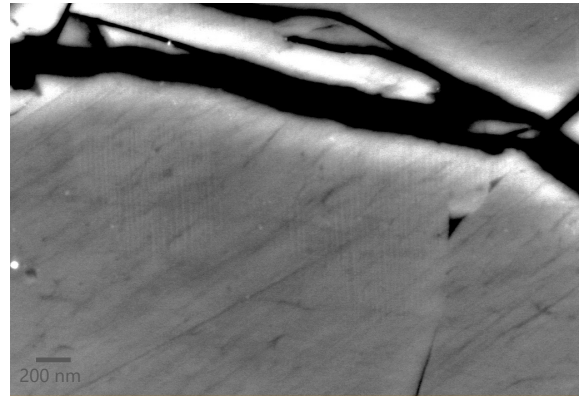
Diamond



Ceramics (MAX phase)

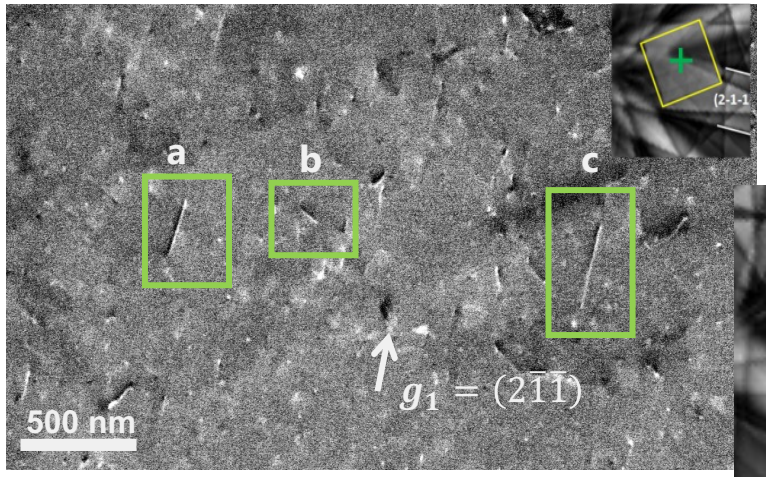


Metals (CrCoNi)



Rock (meteorite)

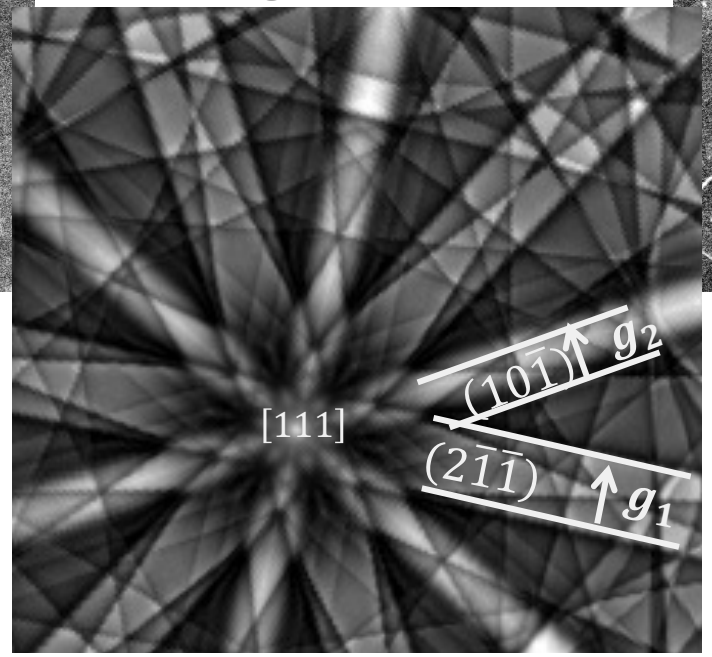
How to characterize dislocations by ECCI?

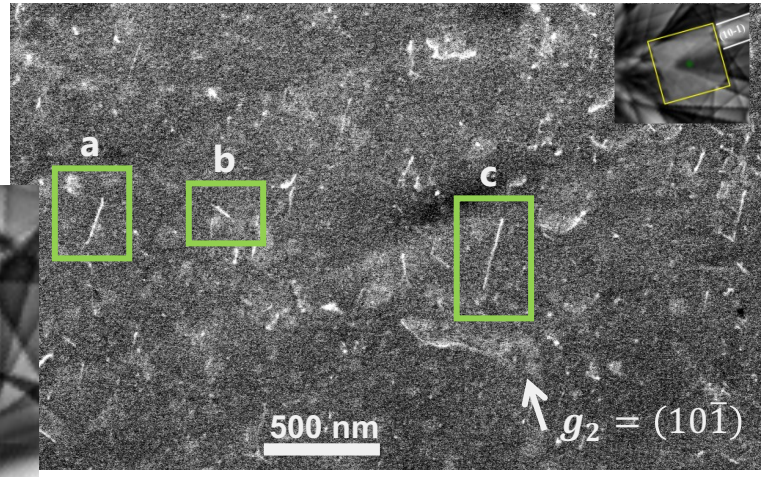


$\boldsymbol{g}$: diffraction vector

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

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





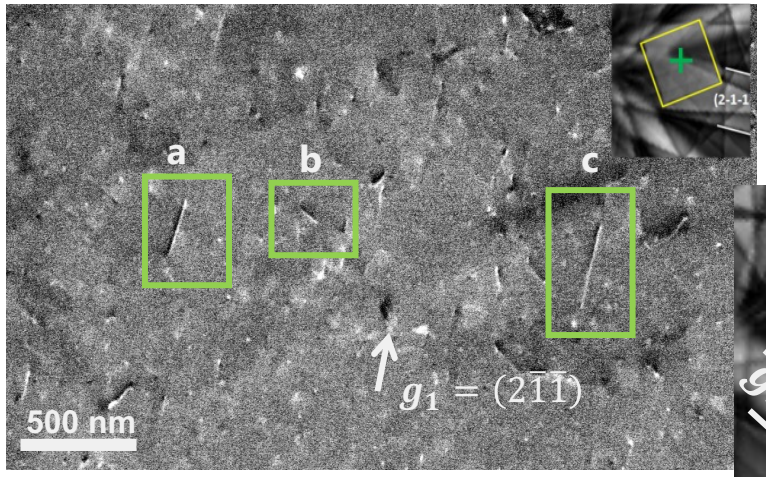
$\boldsymbol{b}$: Burgers vector

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

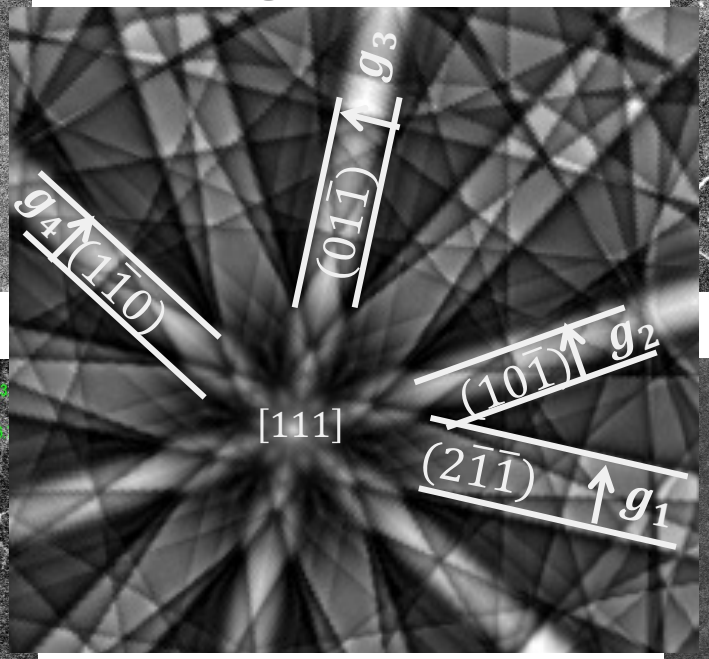
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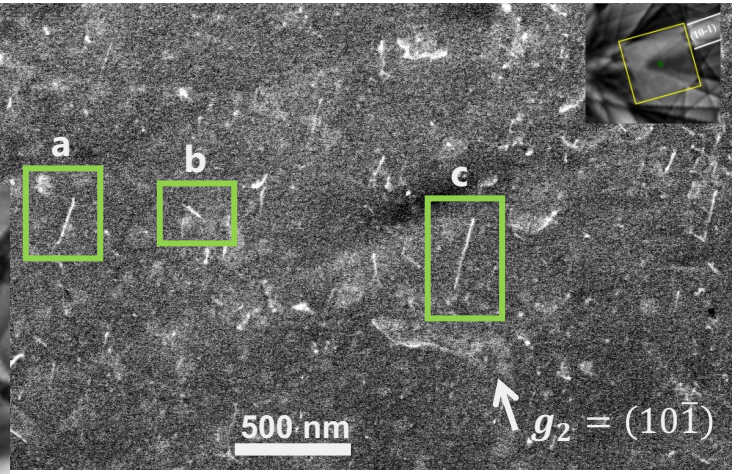
$$\boldsymbol{b} = \frac{1}{2}\langle 111 \rangle$$

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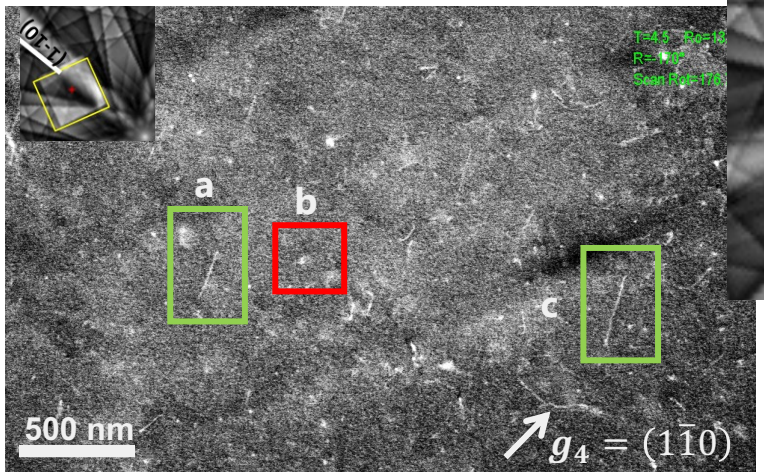
$\boldsymbol{g}$: diffraction vector

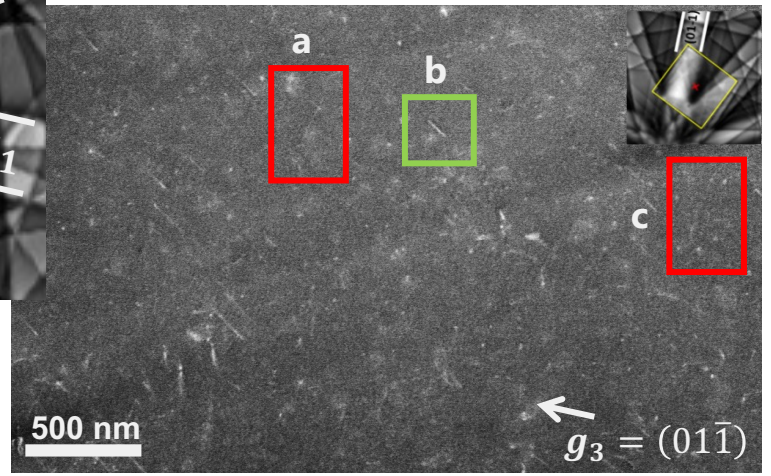




$\boldsymbol{b}$: Burgers vector

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





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

How to characterize dislocations by ECCI?

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

	$\boldsymbol{g_1 = (2\bar{1}\bar{1})}$	$\boldsymbol{g_2 = (10\bar{1})}$	$\boldsymbol{g_3 = (01\bar{1})}$	$\boldsymbol{g_4 = (1\bar{1}0)}$	$\boldsymbol{g_5 = (11\bar{2})}$	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)

How to characterize dislocations by ECCI?

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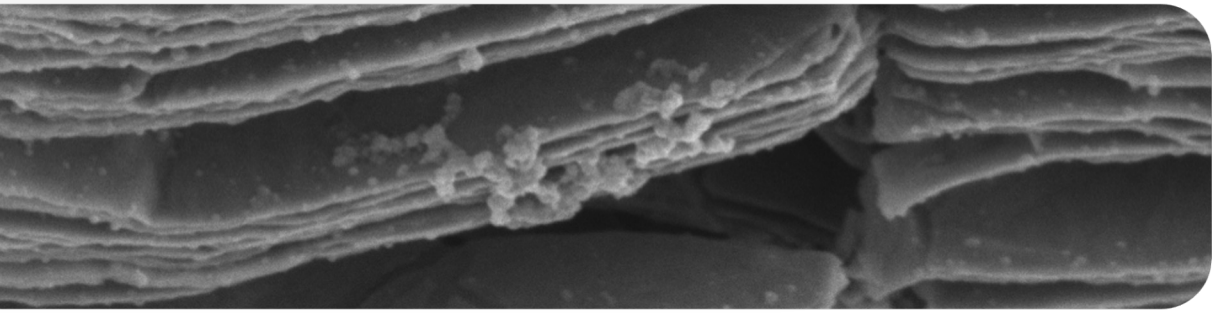
Note: ✓: visibility; ✗: invisibility, α: angle between $\boldsymbol{b}$ and $\boldsymbol{\xi}$

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)

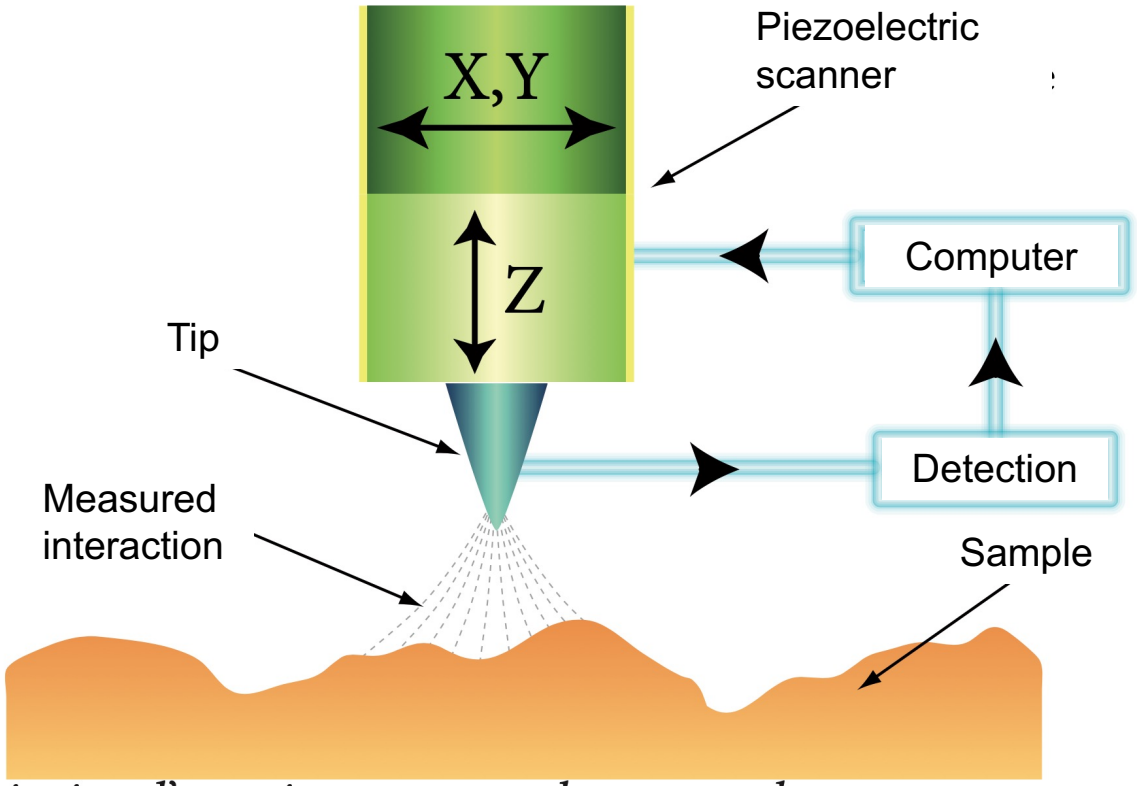


Near-field scanning microscopy

Nanometer scale

www.antoine-guitton.fr

Basics

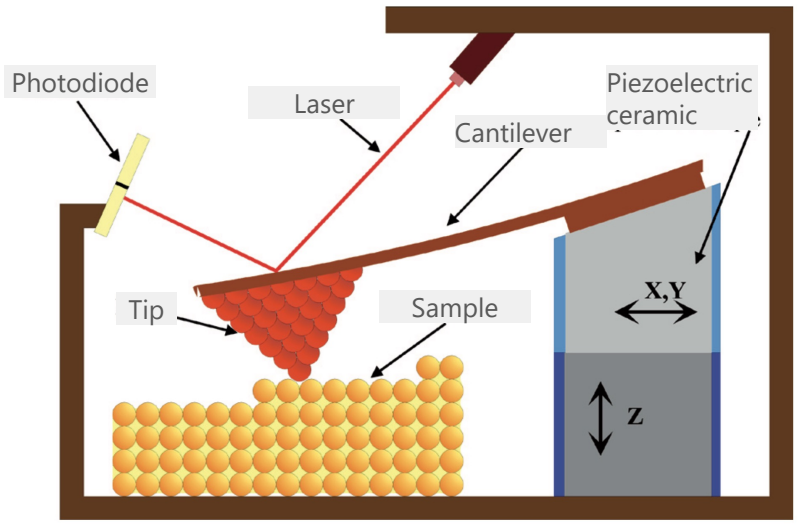


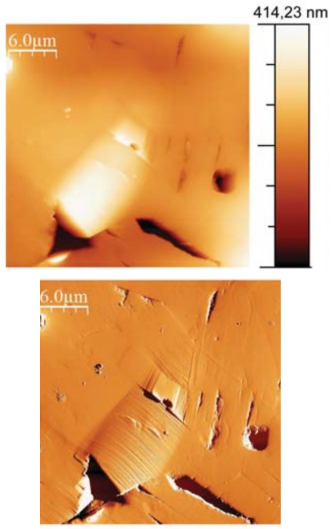
- Bypass the diffraction limit of wave microscopy
- Rely on evanescent waves captured in the near-field (within ~20–100 nm of the sample surface)
- Use a sharp tip to scan very close to the sample
- Topographic information
- Enable imaging of non-conductive or biological samples
- Require precise tip-sample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

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

Two types of near-field microscopes

Atomic force Microscope (AFM)



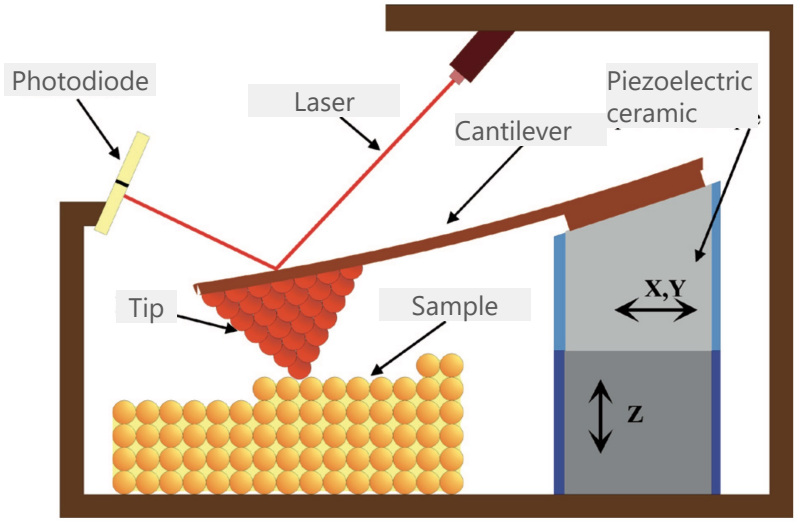


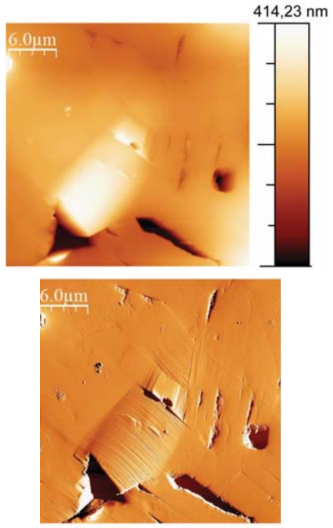
↳Interaction forces between the tip and the surface
↳ All materials
↳ nm resolution
↳Quantification in z

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

Two types of near-field microscopes

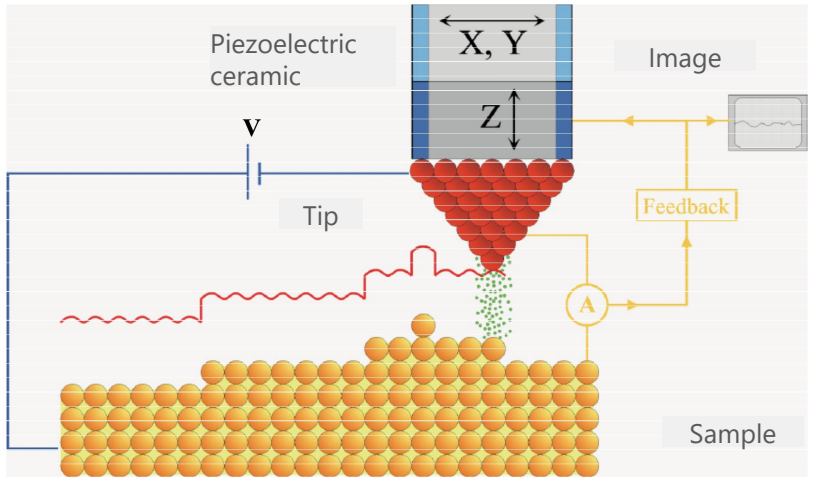
Atomic force Microscope (AFM)

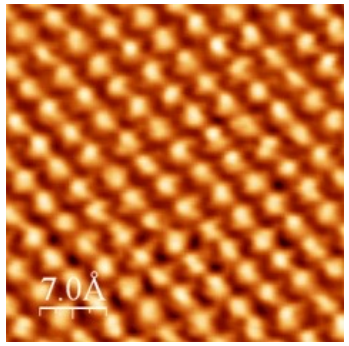




↳Interaction forces between the tip and the surface
↳ All materials
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↳Quantification in z

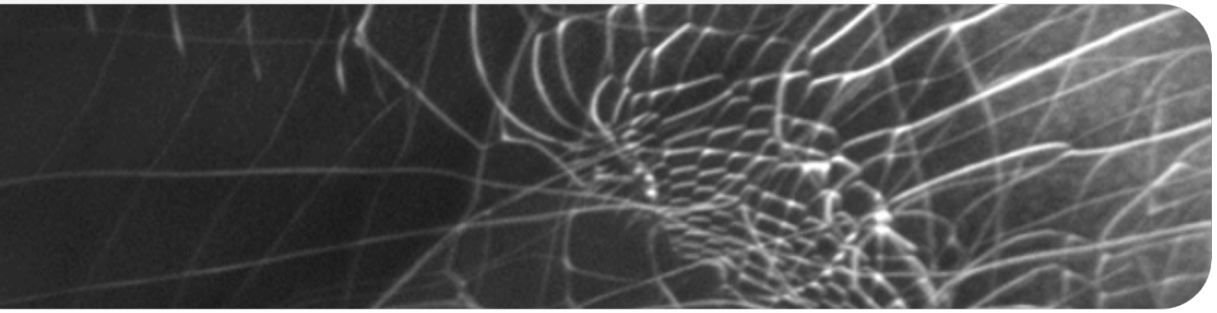
Scanning Tunneling Microscope (STM)





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

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

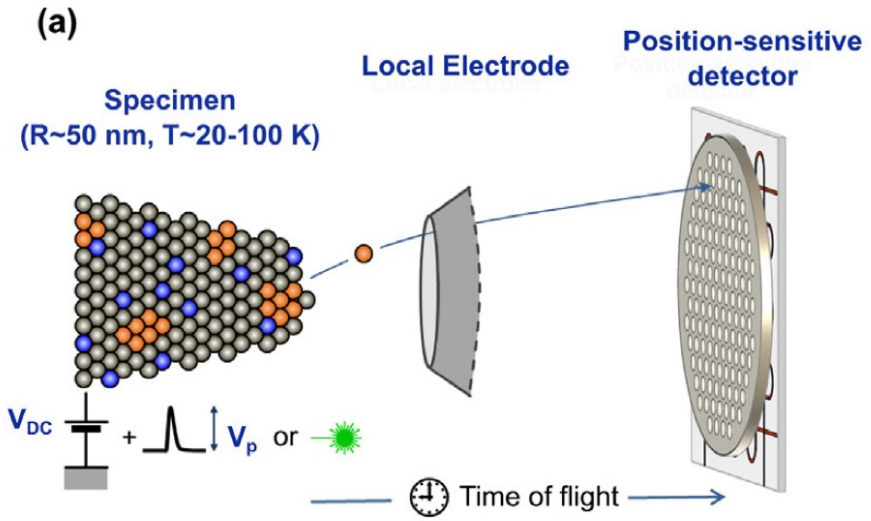


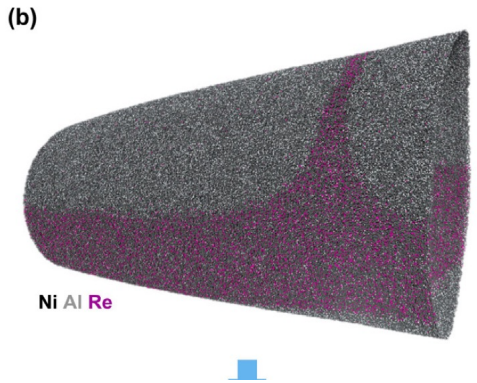
Atom Probe Tomography (APT)

Atomic scale

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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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LEM3 LABORATOIRE D'ÉTUDE DES MICROSTRUCTURES ET DE MÉCANIQUE DES MATÉRIAUX

UNIVERSITÉ DE LORRAINE

enim École Nationale d'Ingénieurs de Metz