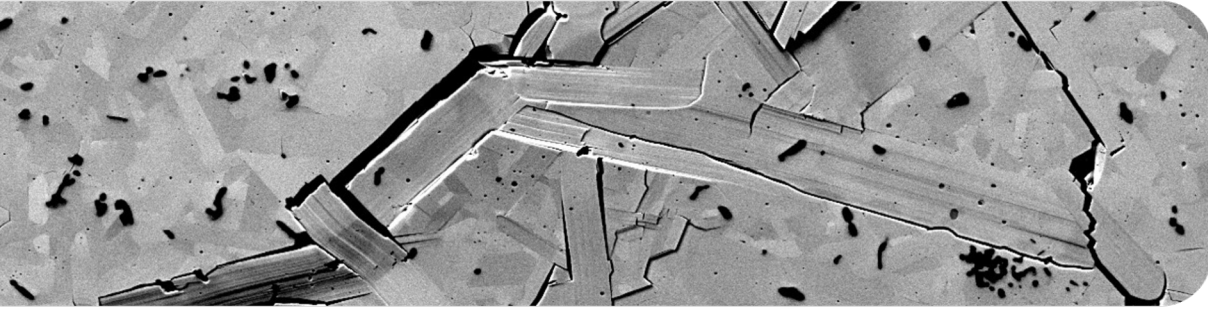




Characterization of micro-/nano-structures for radiation-induced damage in materials

Prof Antoine GUITTON

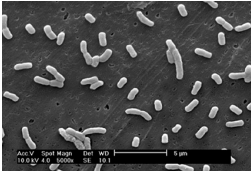
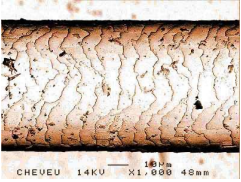
Université de Lorraine, CNRS, Arts et Métiers Institute of Technology, LEM3, F-57000 Metz,
antoine.guitton@univ-Lorraine.fr



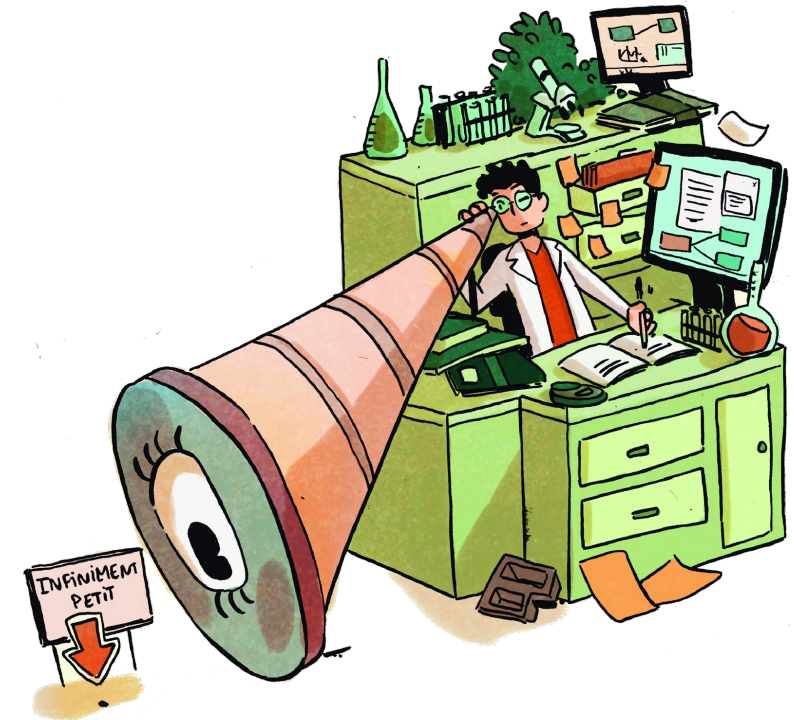
Some micro-/nano-structural features

Some orders of magnitude

❖ Micrometer ($1\ \mu\text{m} = 0.000001\ \text{m} = 10^{-6}\ \text{m}$)

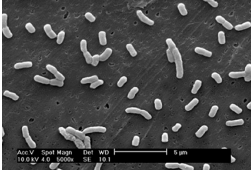
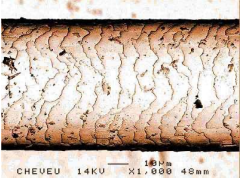


- Hair diameter = $50\text{-}100\ \mu\text{m}$
- Size of a bacteria = $0.1\text{-}10\ \mu\text{m}$



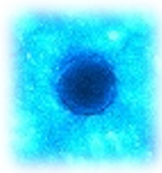
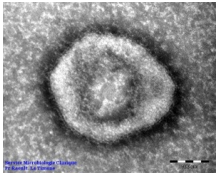
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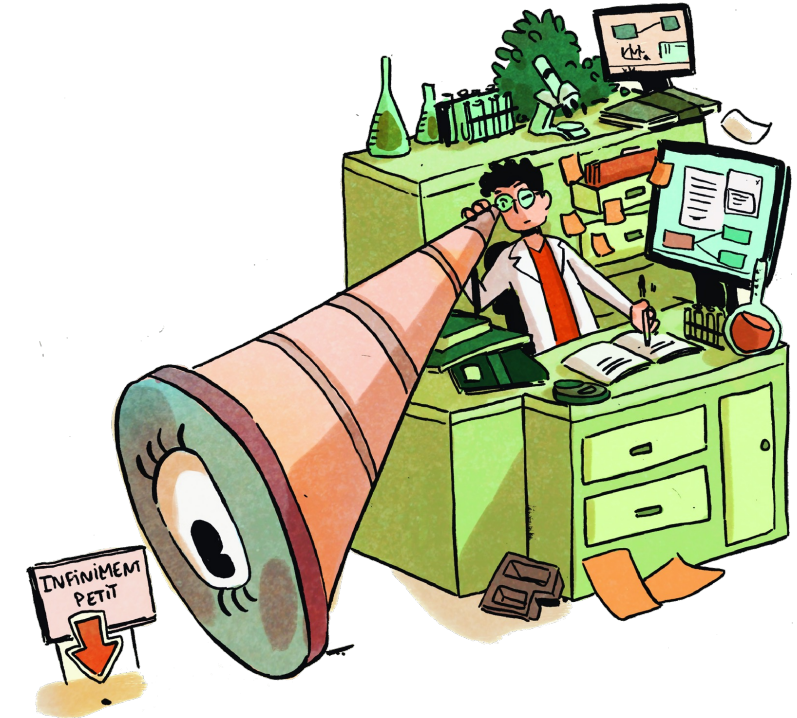


- Hair diameter = $50\text{-}100 \mu\text{m}$
- Size of a bacteria = $0.1\text{-}10 \mu\text{m}$

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

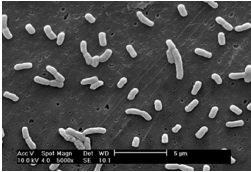
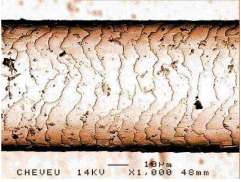


- Size of a virus = $20\text{-}450 \text{ nm}$



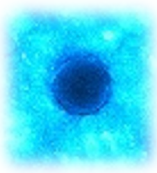
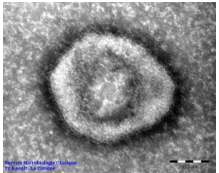
Some orders of magnitude

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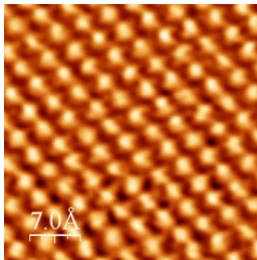
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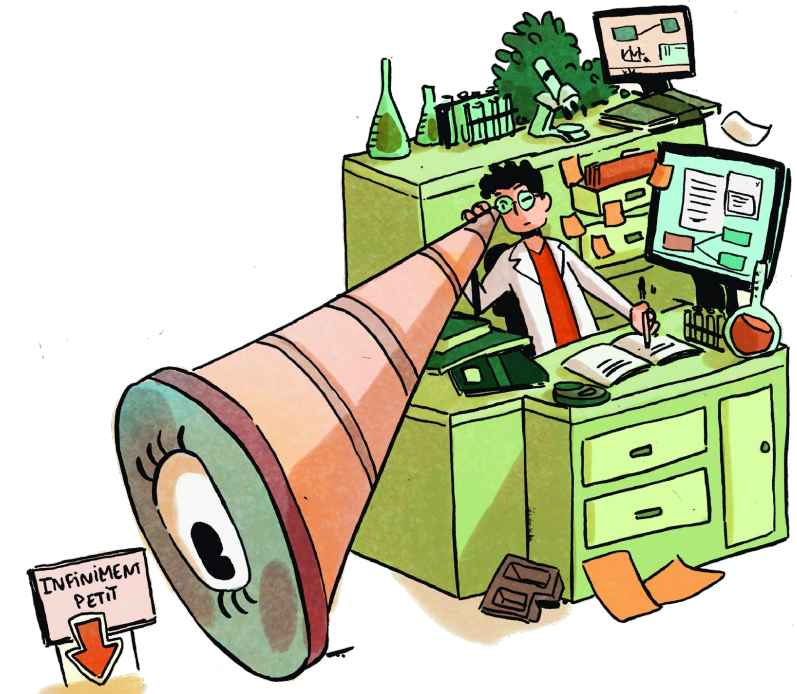


- Size of a virus = $20\text{-}450 \text{ nm}$

❖ Atomic scale ($1 \text{ \AA} = 0.0000000001 \text{ m} = 10^{-10} \text{ m}$)

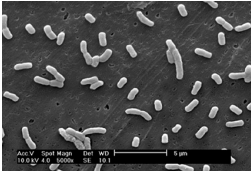
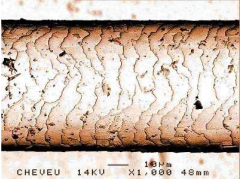


- Distance between atoms = $0.2 \text{ nm} = 2 \text{ \AA}$
- Atom radius = $0.1 \text{ nm} = 1 \text{ \AA}$



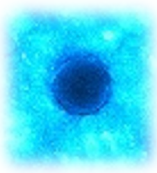
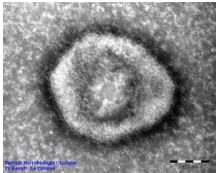
Some orders of magnitude

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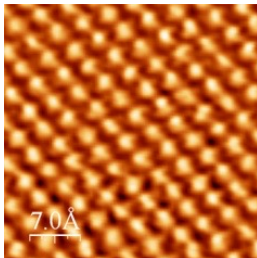
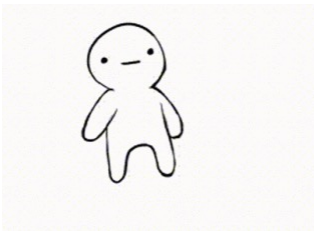
- Hair diameter = 50-100 μm
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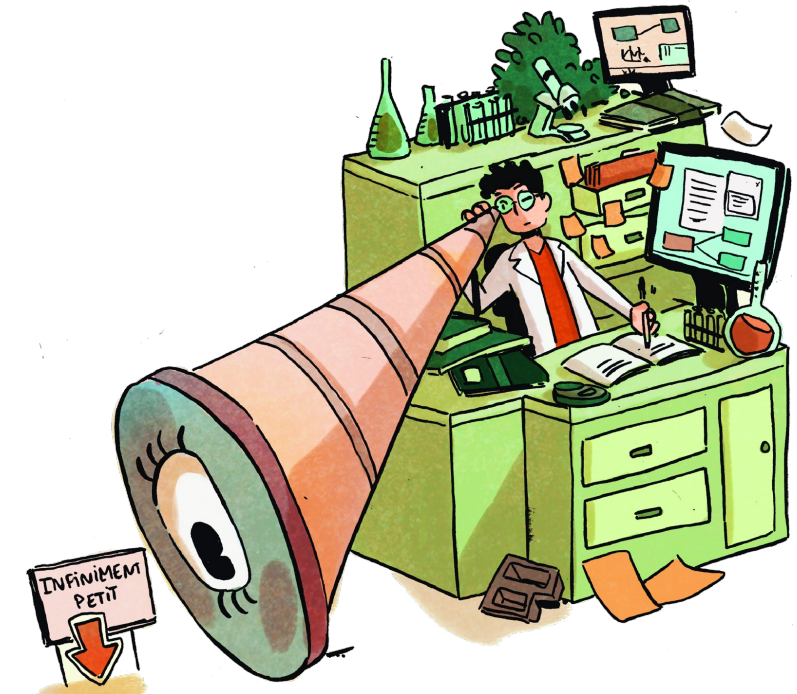
- Size of a virus = 20-450 nm

❖ Atomic scale ($1 \text{ \AA} = 0.0000000001 \text{ m} = 10^{-10} \text{ m}$)



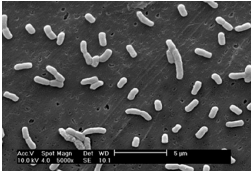
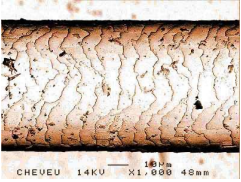
- Distance between atoms = 0.2 nm = 2 $\text{\AA}$
- Atom radius = 0.1 nm = 1 $\text{\AA}$

This is neither copper nor gold!!!



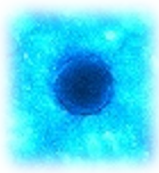
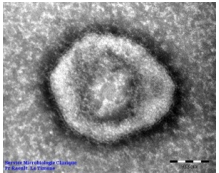
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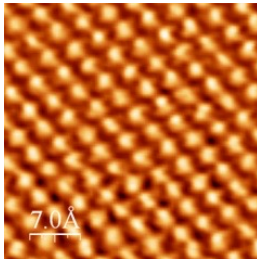
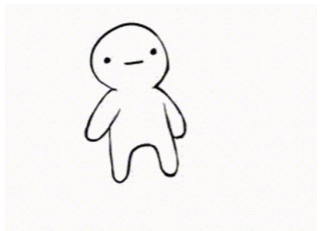
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- Size of a virus = 20-450 nm

❖ Atomic scale ($1 \text{ \AA} = 0.0000000001 \text{ m} = 10^{-10} \text{ m}$)

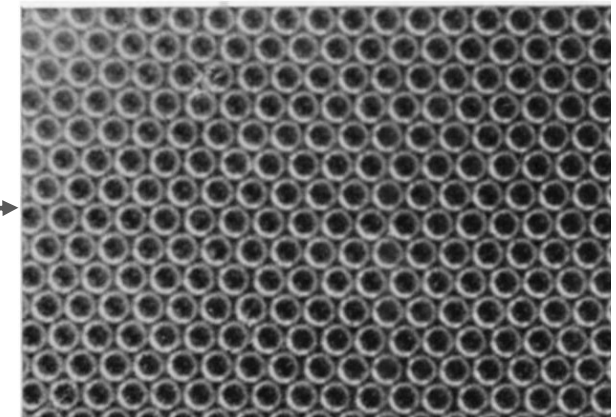


- Distance between atoms = 0.2 nm = 2 $\text{\AA}$
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This is neither copper nor gold!!!

Stacking of spheres

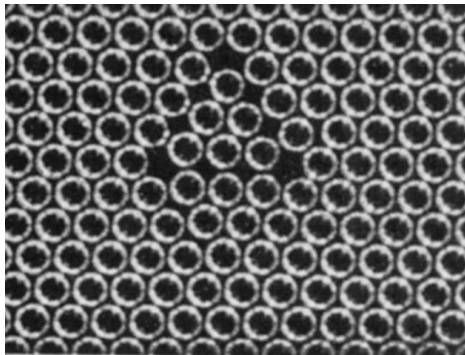
L. Bragg, J.F. Nye, *Proc. R. Soc. Lond.*, 1947



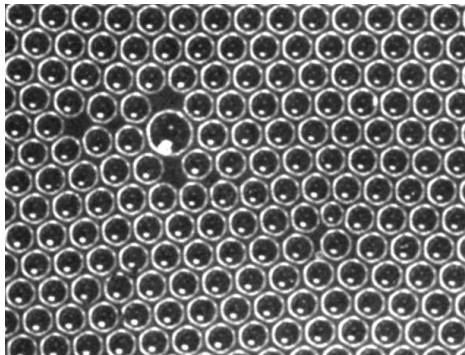
Defects in crystalline materials

0 dimension

Vacancy



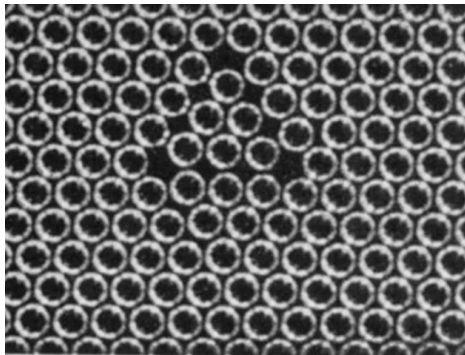
Interstitial



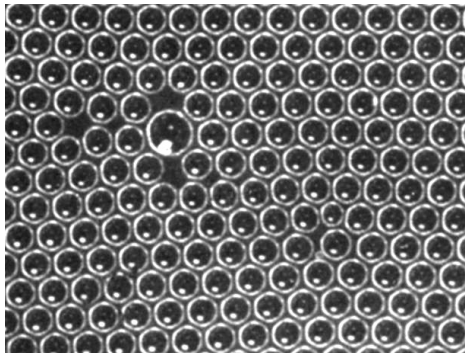
Defects in crystalline materials

0 dimension

Vacancy

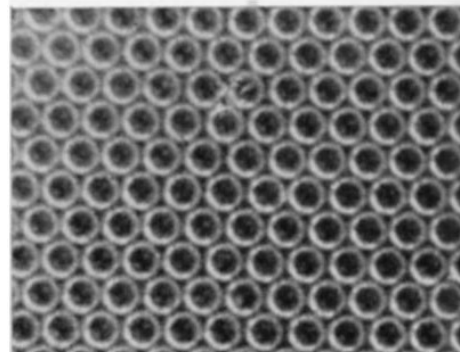


Interstitial



1 dimension

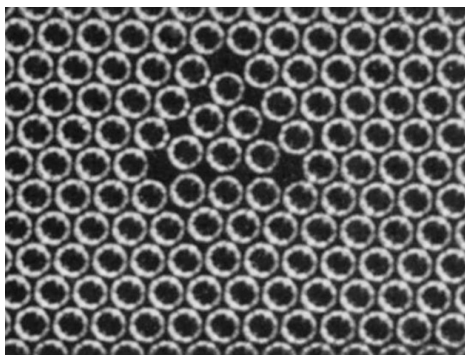
Dislocation



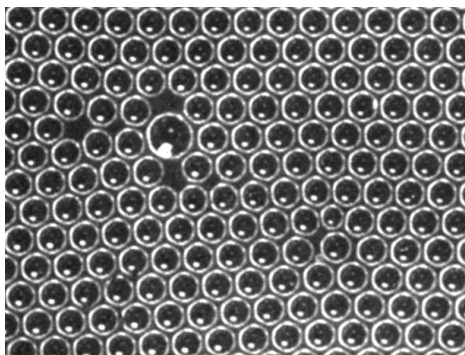
Defects in crystalline materials

0 dimension

Vacancy

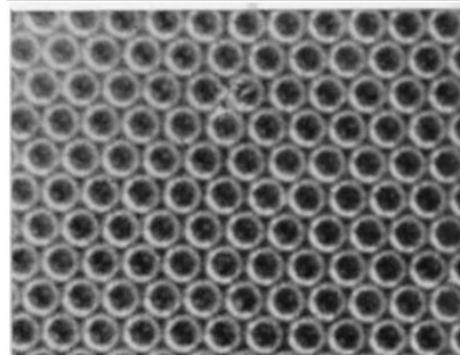


Interstitial



1 dimension

Dislocation

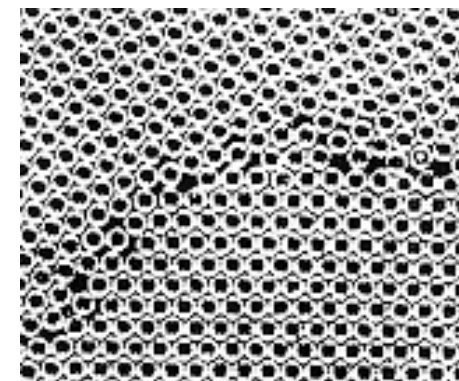


2 dimensions

Stacking fault



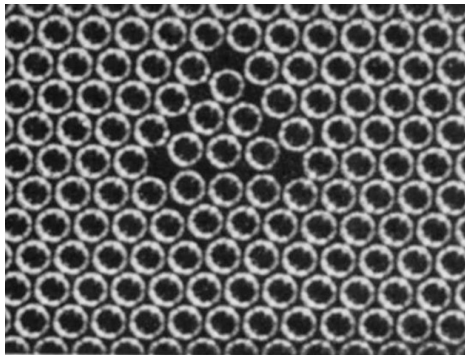
Grain boundaries



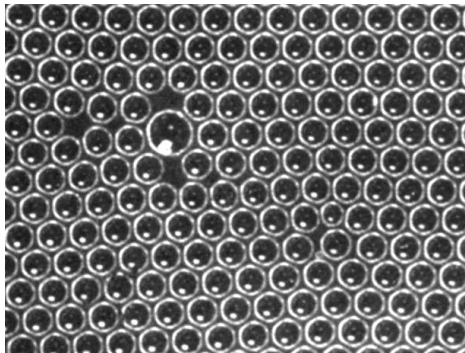
Defects in crystalline materials

0 dimension

Vacancy

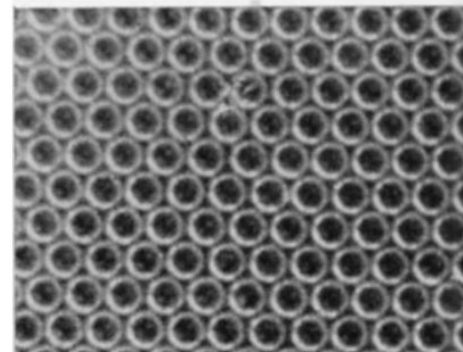


Interstitial



1 dimension

Dislocation



3 dimensions

Inclusions, precipitates...

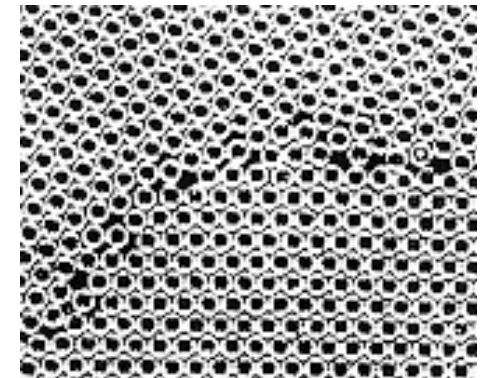
"Crystals are like people; it is the defects in them which tend to make them interesting!"
(Colin J. Humphreys)

2 dimensions

Stacking fault

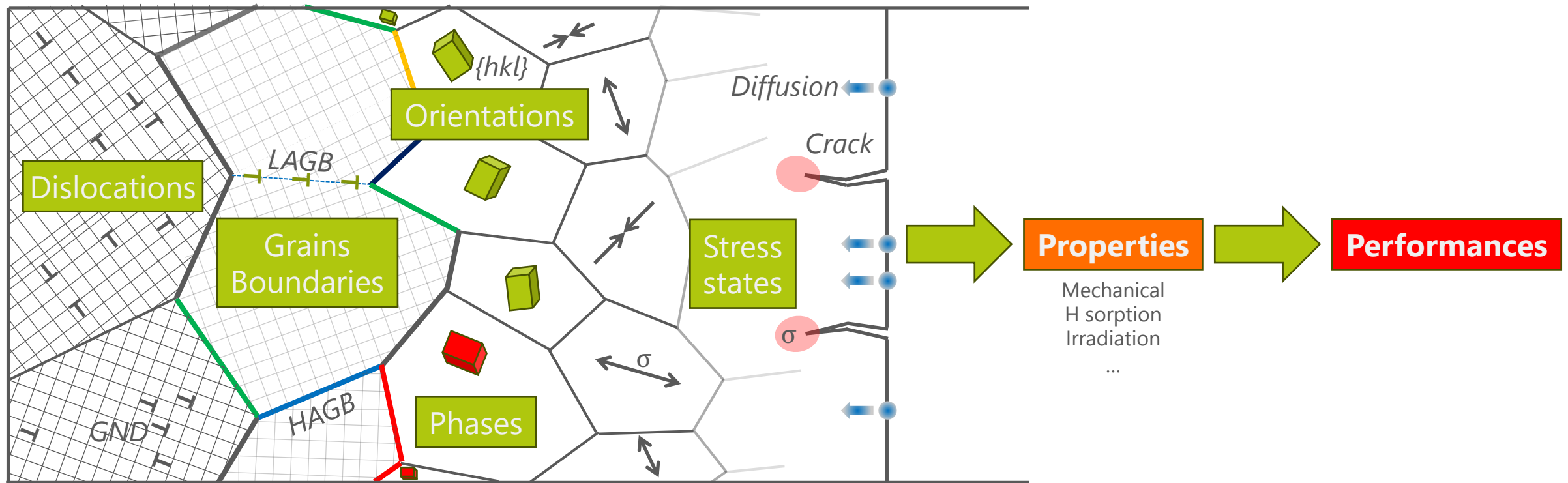


Grain boundaries



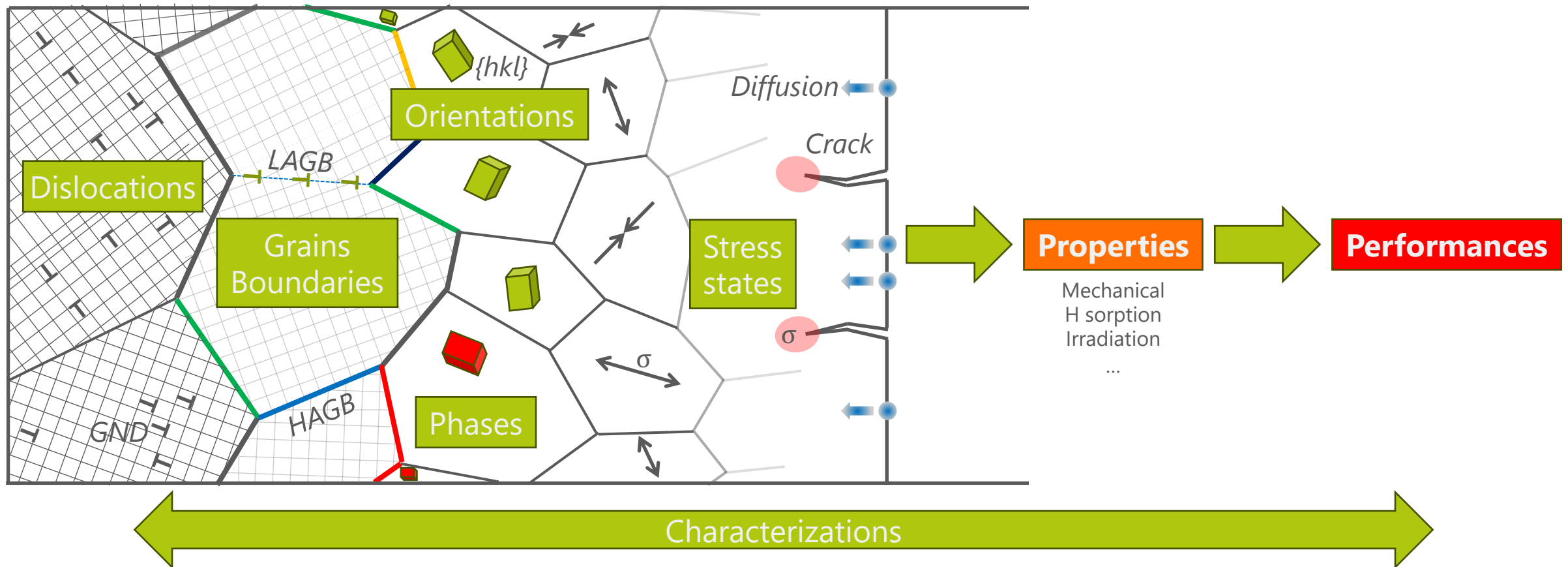
Micro-/nano-structures

Microstructure ($\sim 1 \mu\text{m} \rightarrow 1 \text{mm}$) **and nanostructure** ($\sim 1 \text{nm} \rightarrow 100 \text{nm}$) **refer to the internal features of a material, at the micrometer and nanometer scales respectively, influencing its properties**



Micro-/nano-structures

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Phases

FORM & MELTING POINT		DESCRIPTION & PROPERTIES
I	17.3°C	BOTH SOFT AND CRUMBLY WITH NOTICEABLE BLOOMING Form I is produced by cooling melted chocolate rapidly (e.g. by putting it in the freezer). Form II is produced by cooling melted chocolate at 2°C per minute. Form I crystals also gradually become Form II after a short time of freezing temperature storage.
II	23.3°C	
III	25.5°C	BOTH FIRM, BUT DON'T GIVE A GOOD 'SNAP', AND SHOW SOME BLOOMING Form III is produced by cooling at 5-10°C. Form II becomes Form III after storage at low temperatures above freezing. Form IV is produced by allowing melted chocolate to cool at room temperature; Form III also becomes Form IV after storage at room temperature for some time.
IV	27.3°C	
V	33.8°C	SHINY, SMOOTH TEXTURE, GOOD 'SNAP', AND MELTS IN THE MOUTH Formed by tempering chocolate slowly at room temperature. Most desirable!
VI	36.3°C	HARD AND MELTS SLOWLY IN THE MOUTH, SHOWS SOME BLOOMING Can't be formed from melted chocolate - can only be formed after solid, tempered chocolate has rested for at least 4 months.

INCREASED STABILITY & DENSITY

❖ 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

❖ 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:

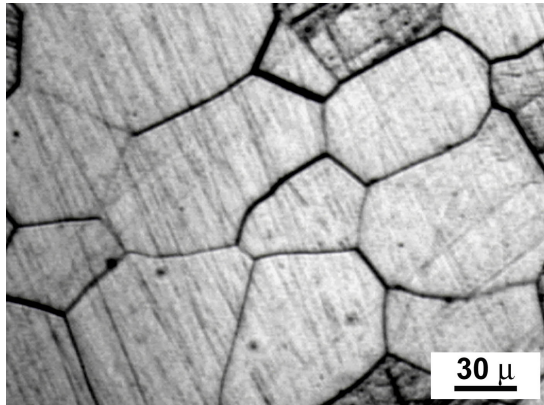
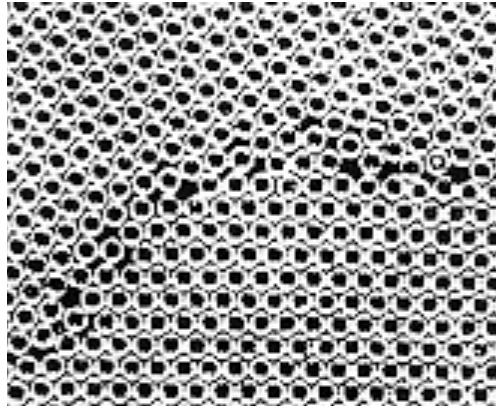
- Used in two-phase regions to determine phase fractions; applies to both metals and chocolate crystallization.

❖ Importance in processing:

- Helps control microstructure, texture, and mechanical properties in both food and engineering materials.

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:

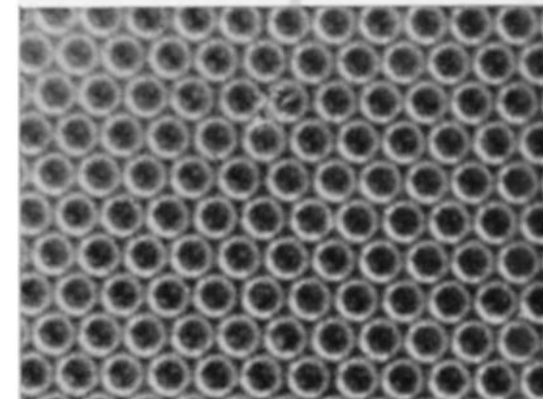
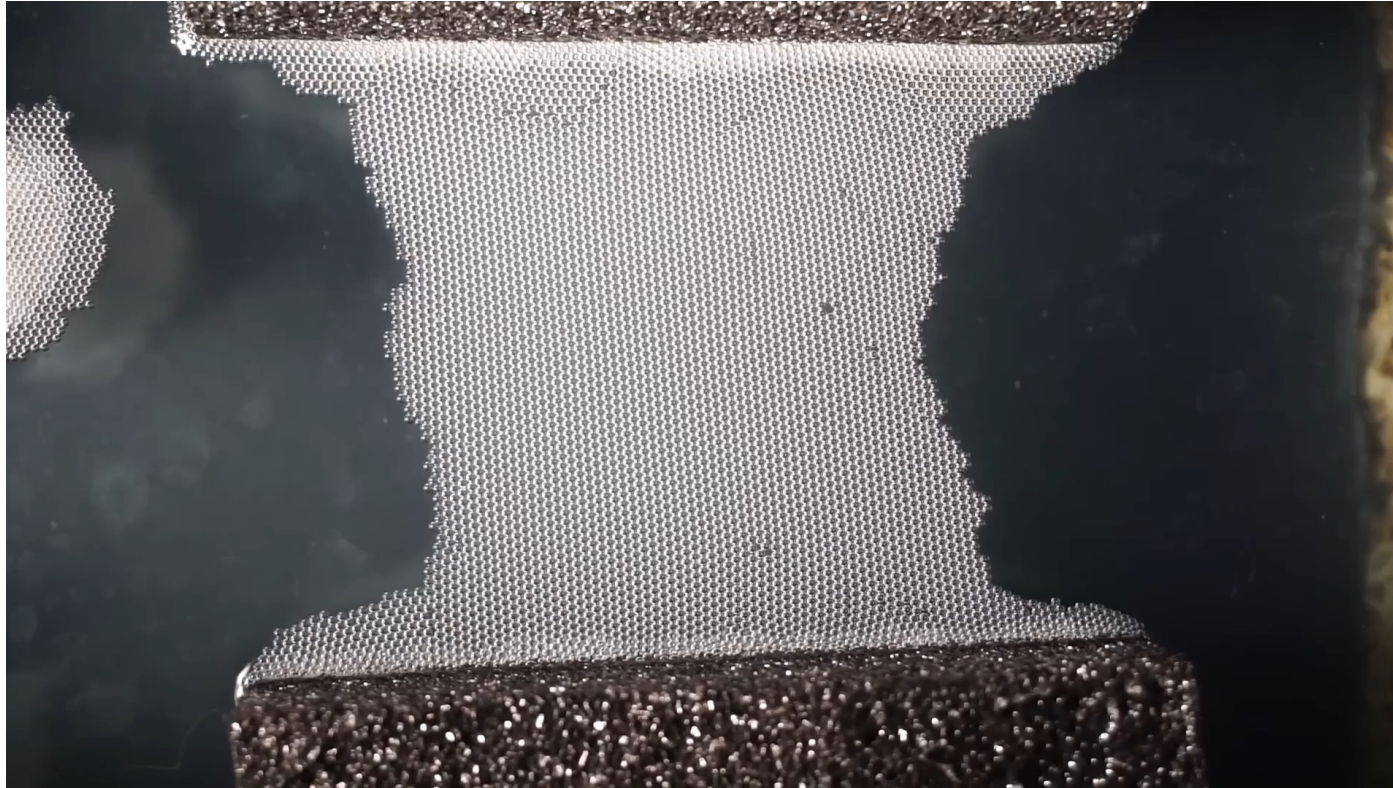
- Shape
- Size

❖ Texture:

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

Dislocations

16

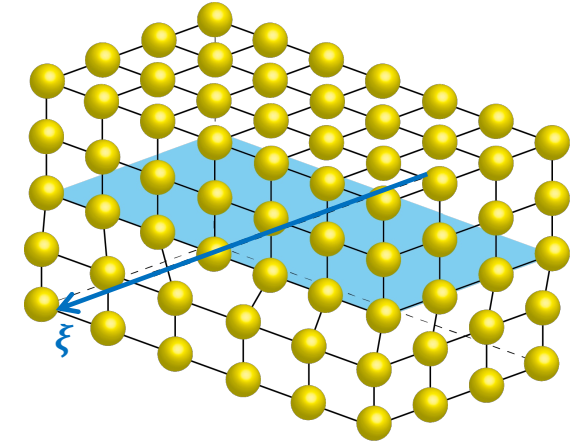
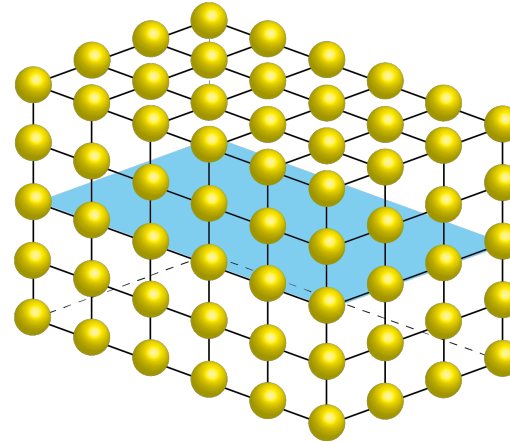
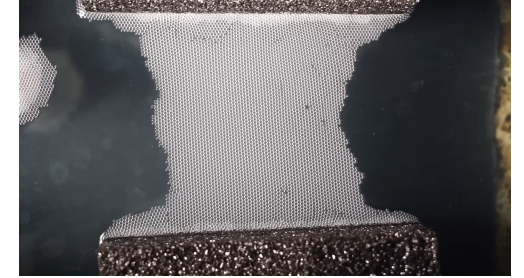
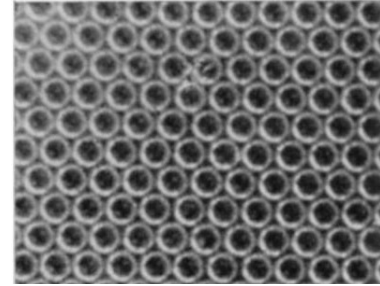


Dislocations

❖ Definition:

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

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

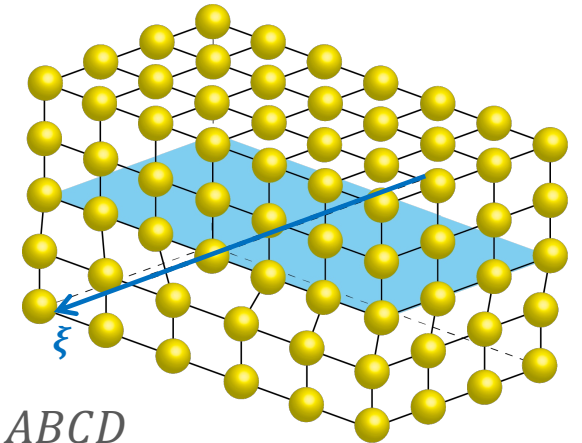
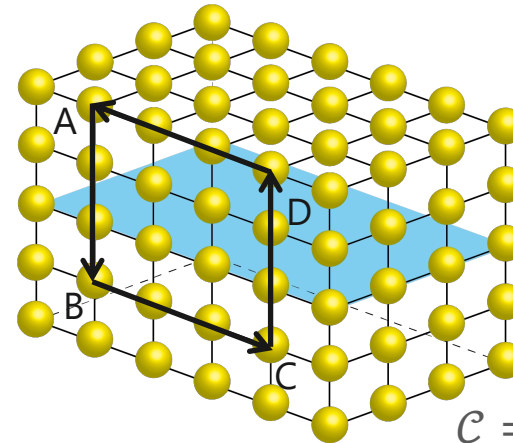
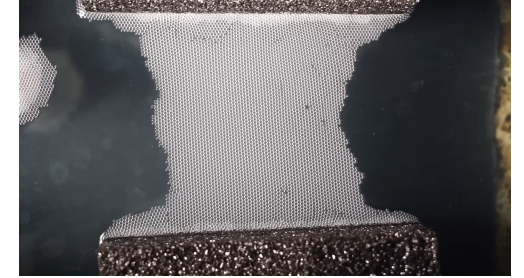
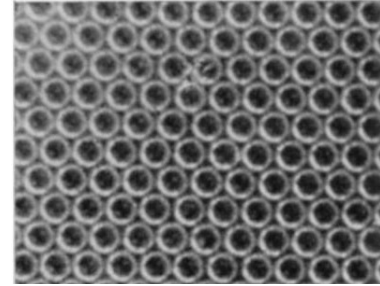


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Dislocations

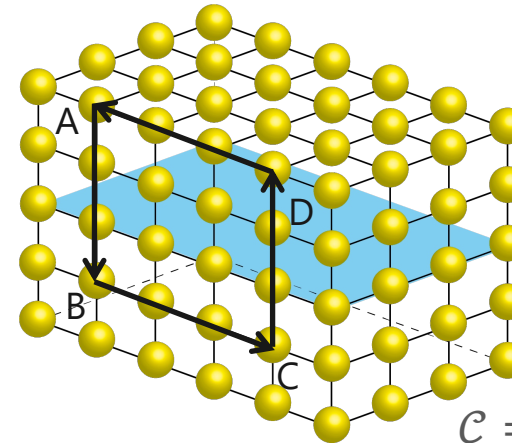
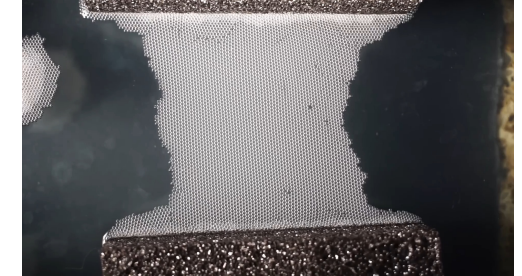
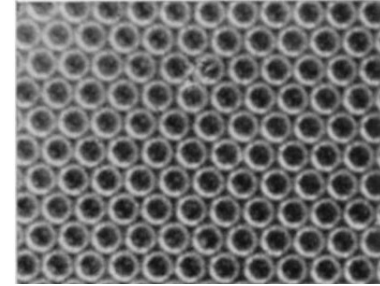
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❖ Types:

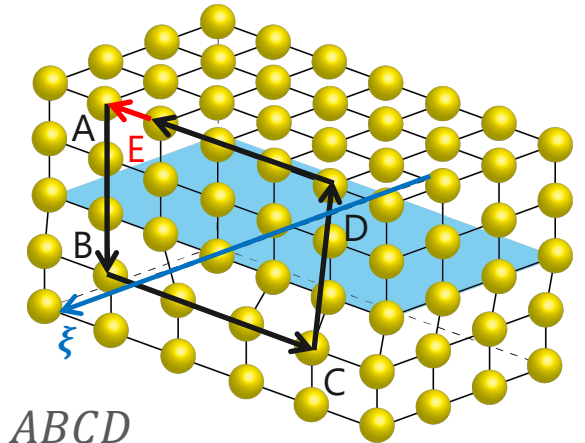
- *Edge dislocation*: characterized by an extra half-plane of atoms inserted into the lattice ($\mathbf{b} \perp \xi$).
- *Screw dislocation*: results from a shear distortion, forming a helical ramp in the crystal lattice ($\mathbf{b} \parallel \xi$).
- *Mixed dislocation*: a combination of edge and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

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



$$\mathcal{C} = ABCD$$

$$EA = \mathbf{b}$$



Punctual defects

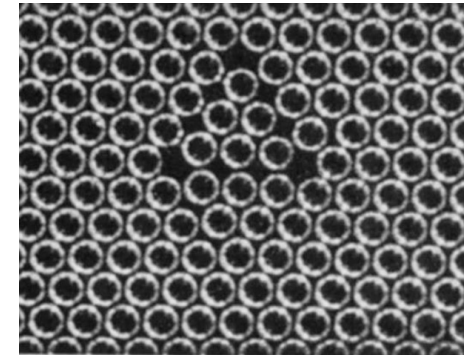
❖ Definition:

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

❖ Types:

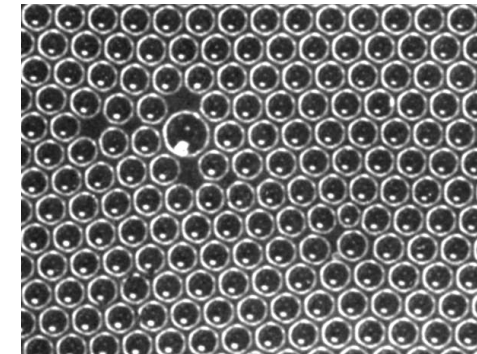
- *Vacancies*: Missing atoms in the lattice.
- *Interstitials*: Extra atoms positioned in the interstitial sites between the regular lattice points.
- *Substitutional defects*: Foreign atoms replacing host atoms in the lattice.

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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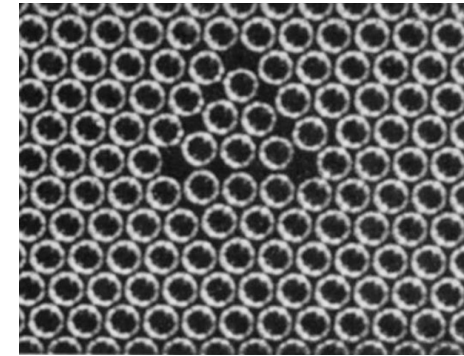
❖ Types:

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❖ Diffusion:

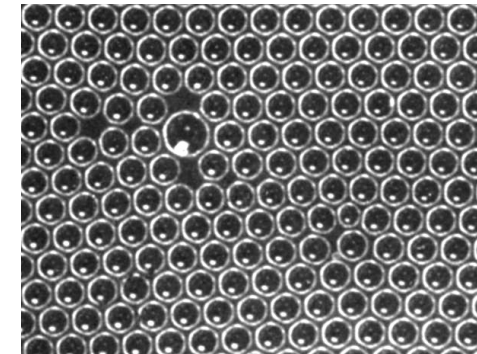
- They enable atomic diffusion, which is governed by Fick's Laws...

Vacancy



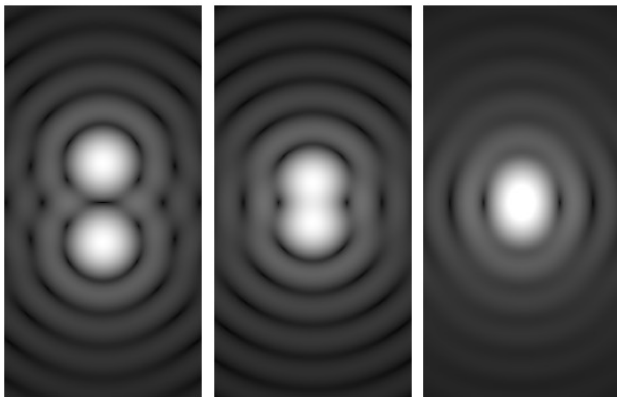
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Interstitial



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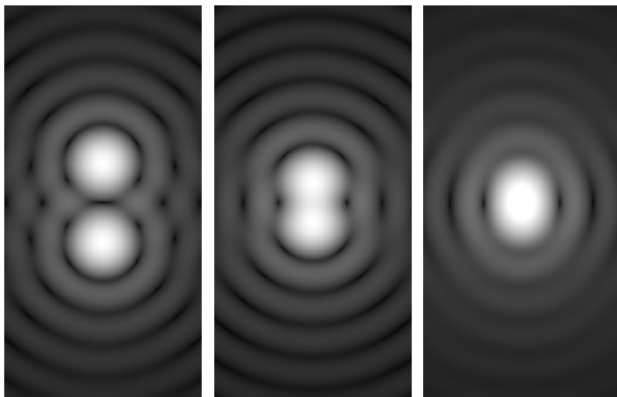
Limit of resolution



❖ Airy disk and Rayleigh criterion

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

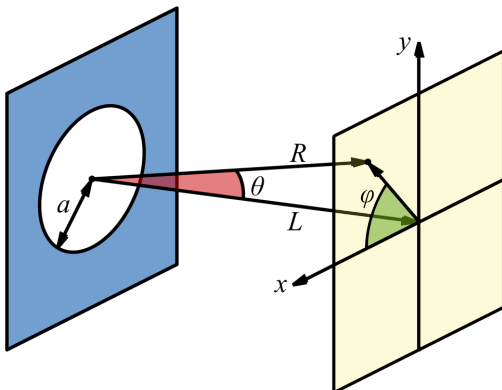
Limit of resolution



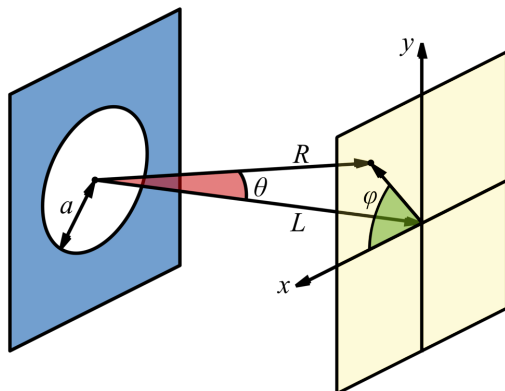
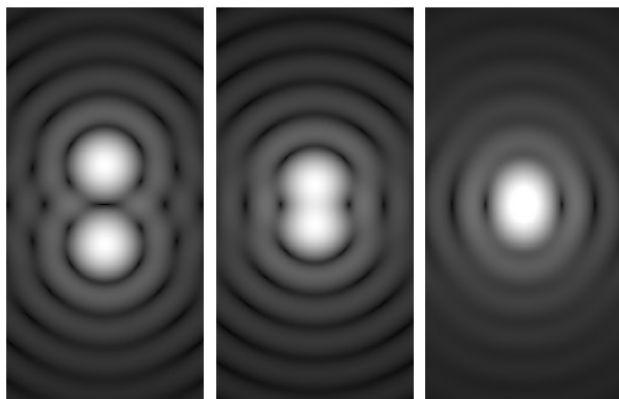
❖ Airy disk and Rayleigh criterion

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

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



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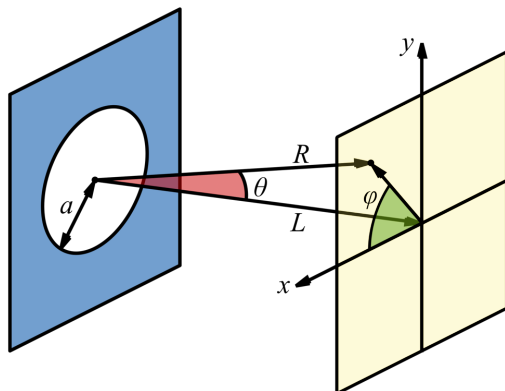
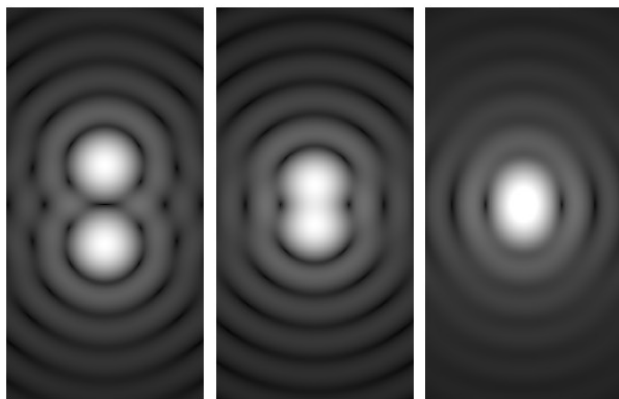
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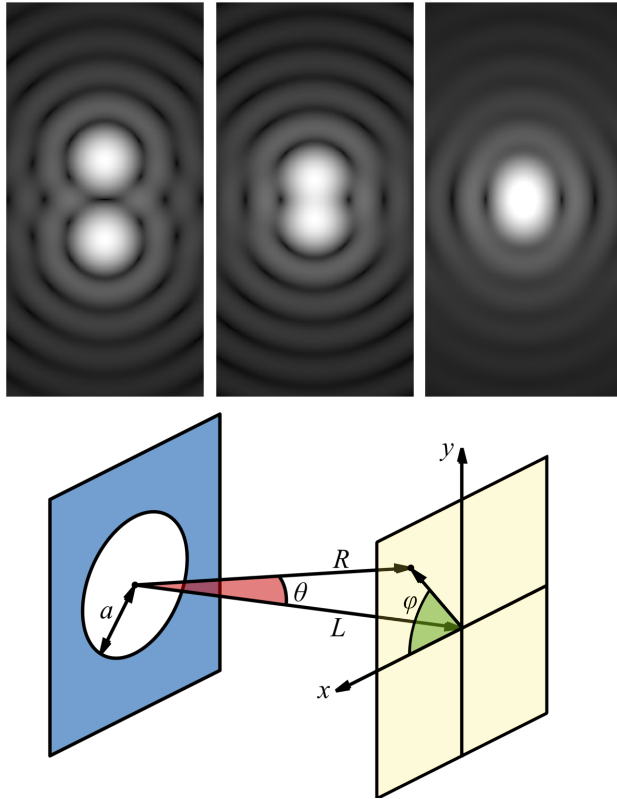
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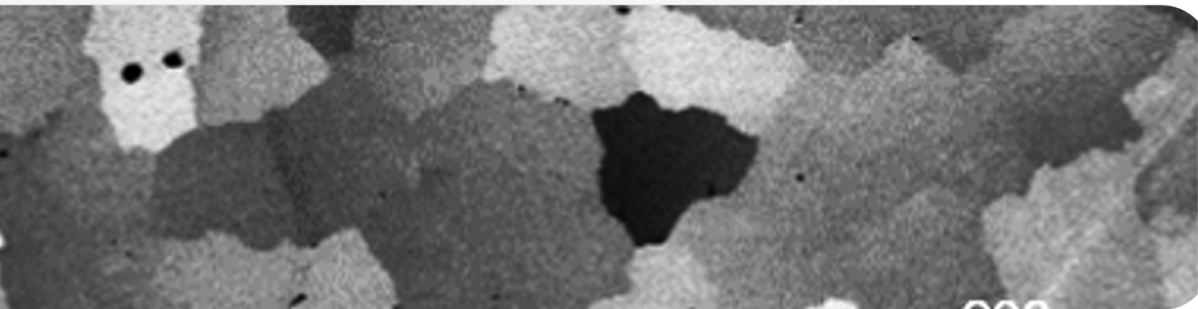
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↪ Two objects cannot be resolved if they are closer than one wavelength λ .



Characterization by X-rays diffraction

Meso- Micro-meter scale

“Radiations studied by M. Röntgen”

❖ 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) 🇩🇪
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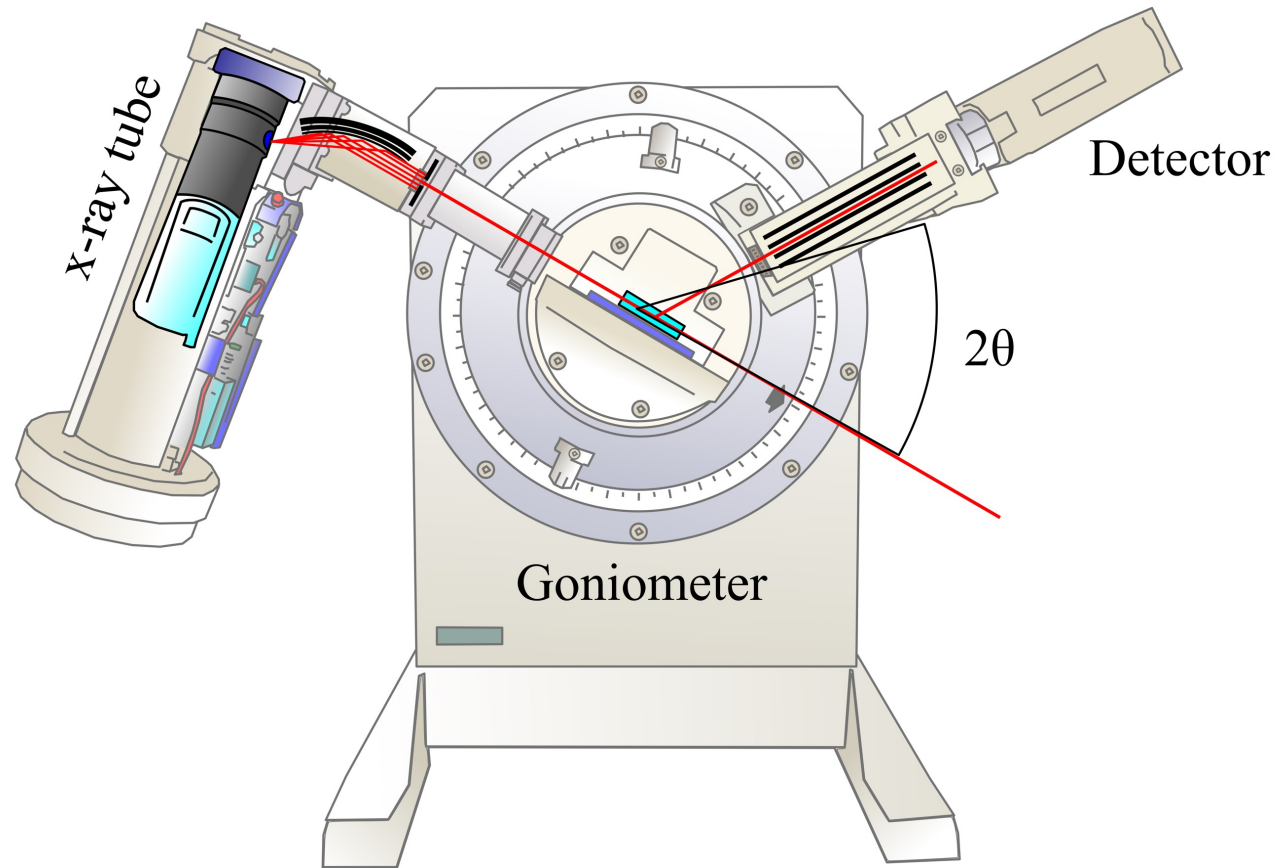
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↪ Since Röntgen did not understand their nature, he called these rays, X-rays!

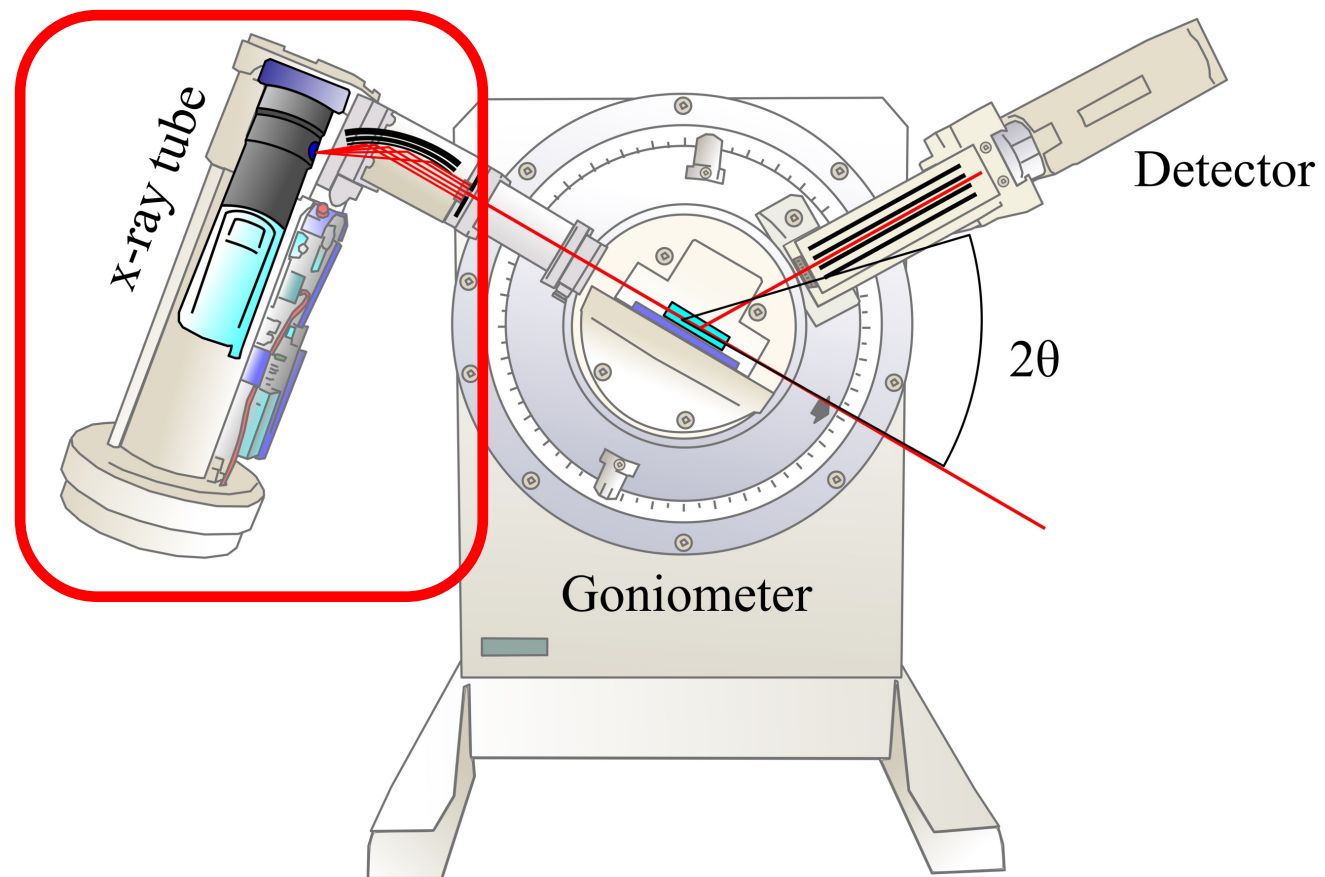
The experimental setup: the diffractometer

The diffractometer (Bragg-Brentano configuration)

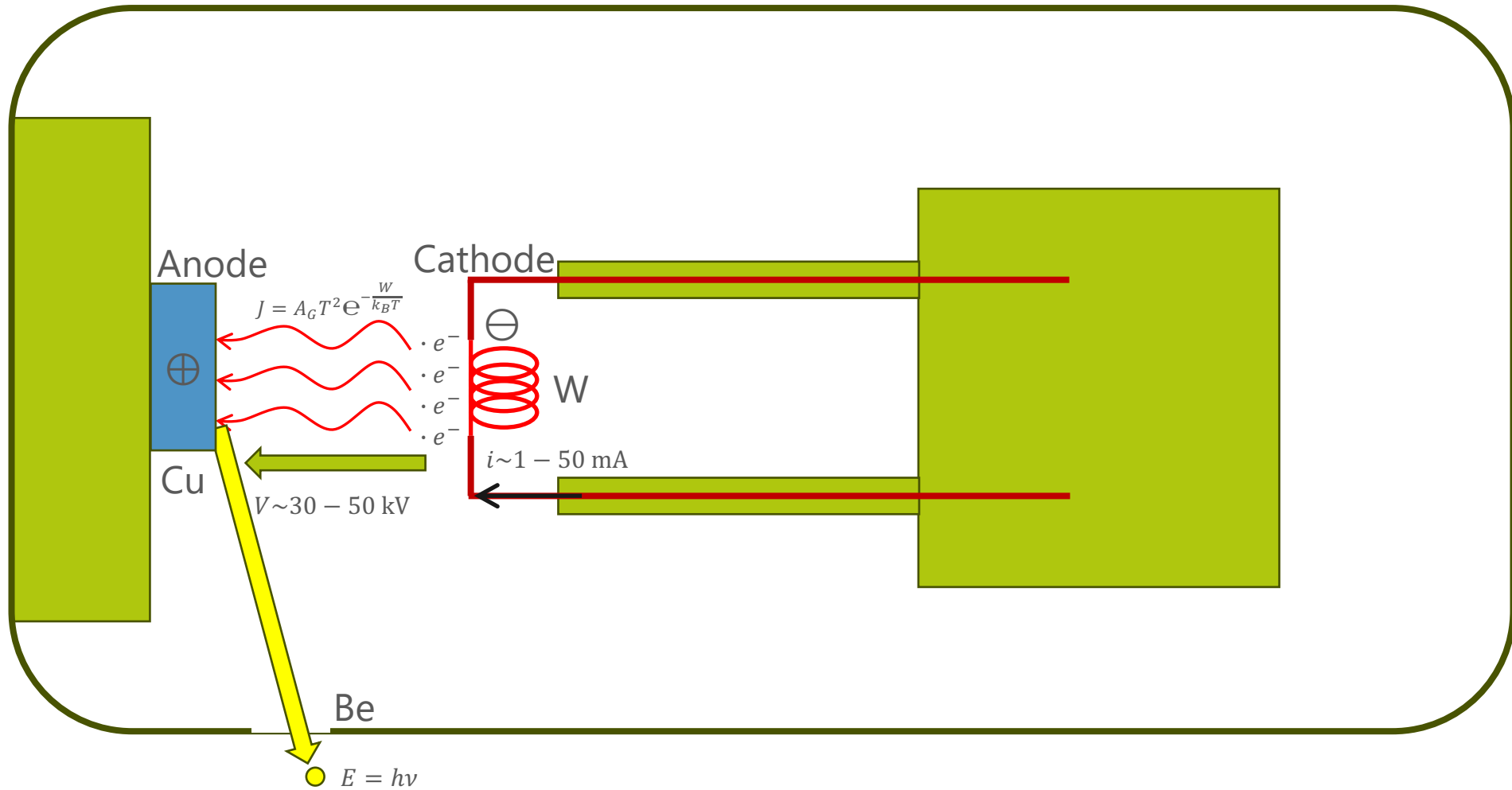


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X-ray production



X-ray production

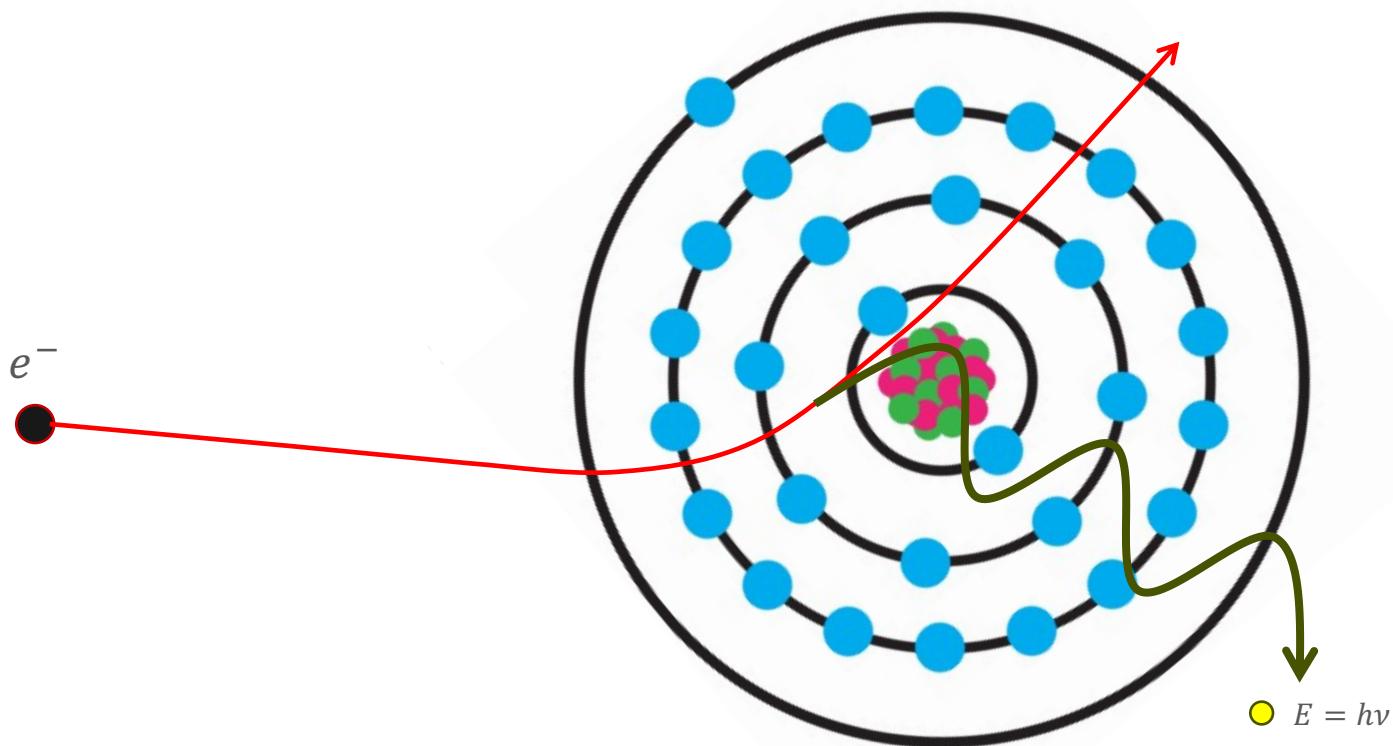
Bremsstrahlung radiation

❖ Definition:

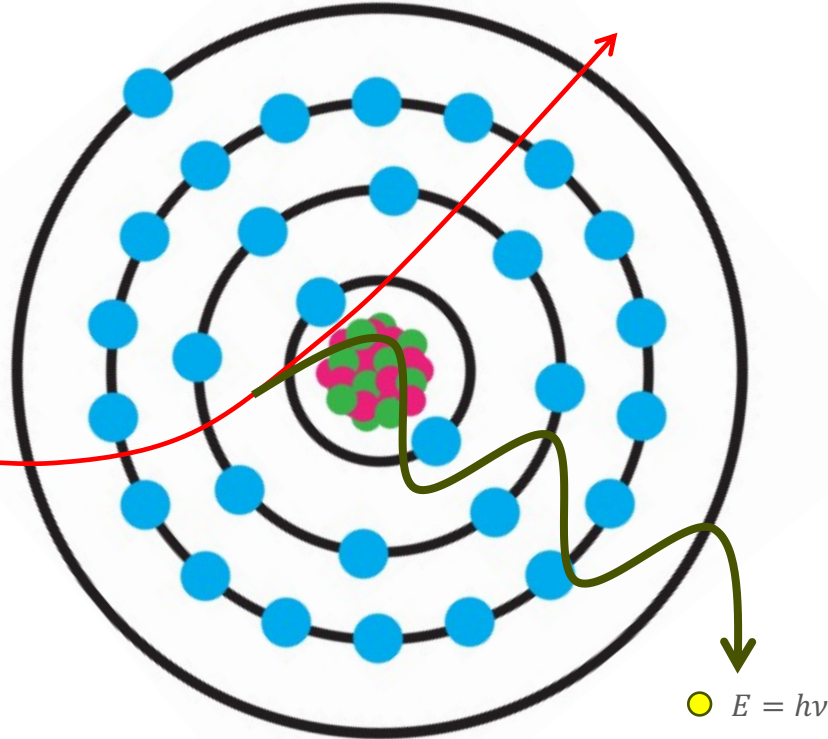
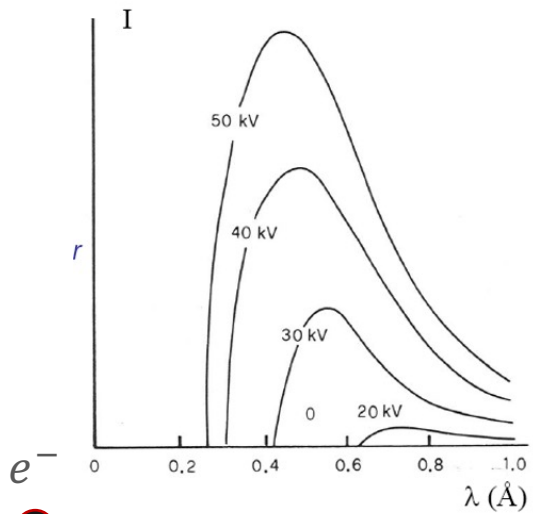
- X-ray radiation produced when an electron is decelerated or deflected by the nucleus.

❖ 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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❖ Mechanism:

- 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

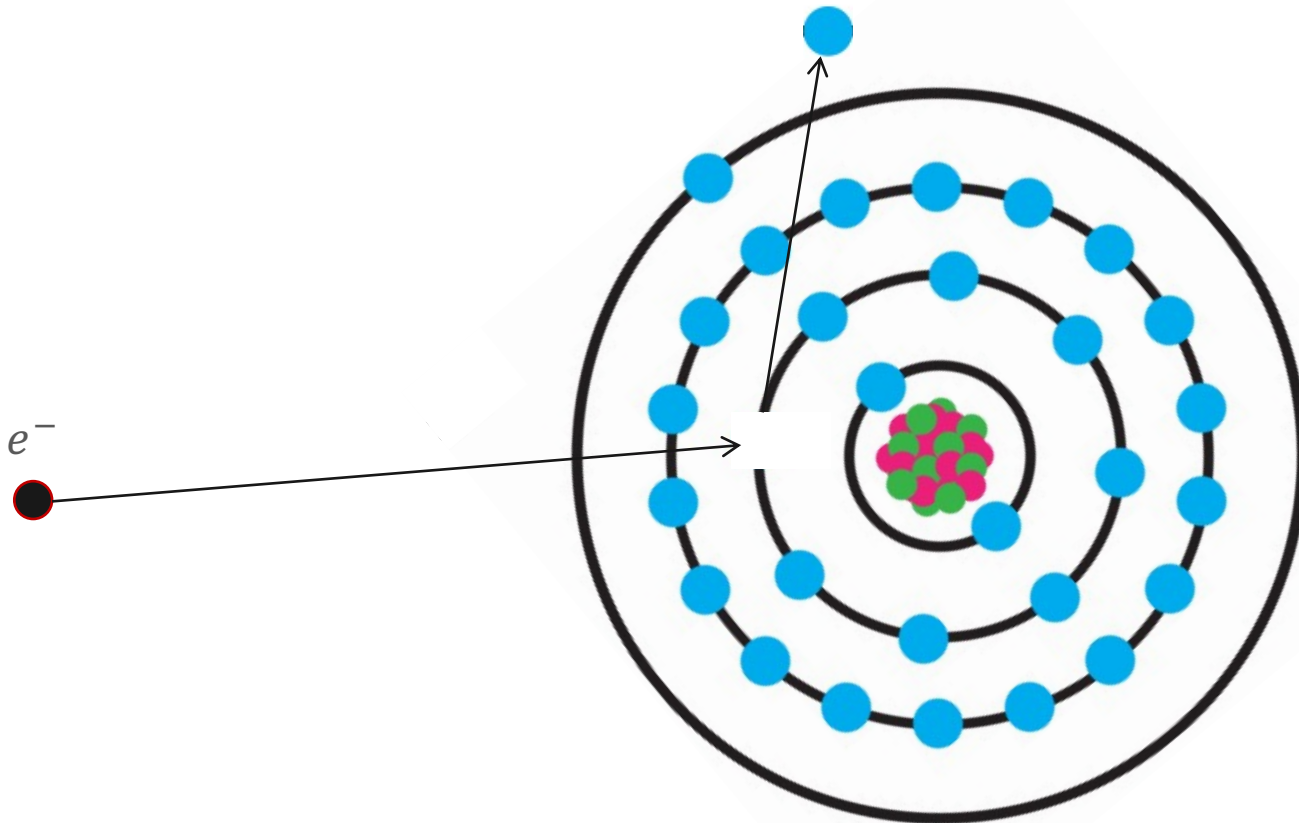
Characteristic radiation

❖ **Definition:**

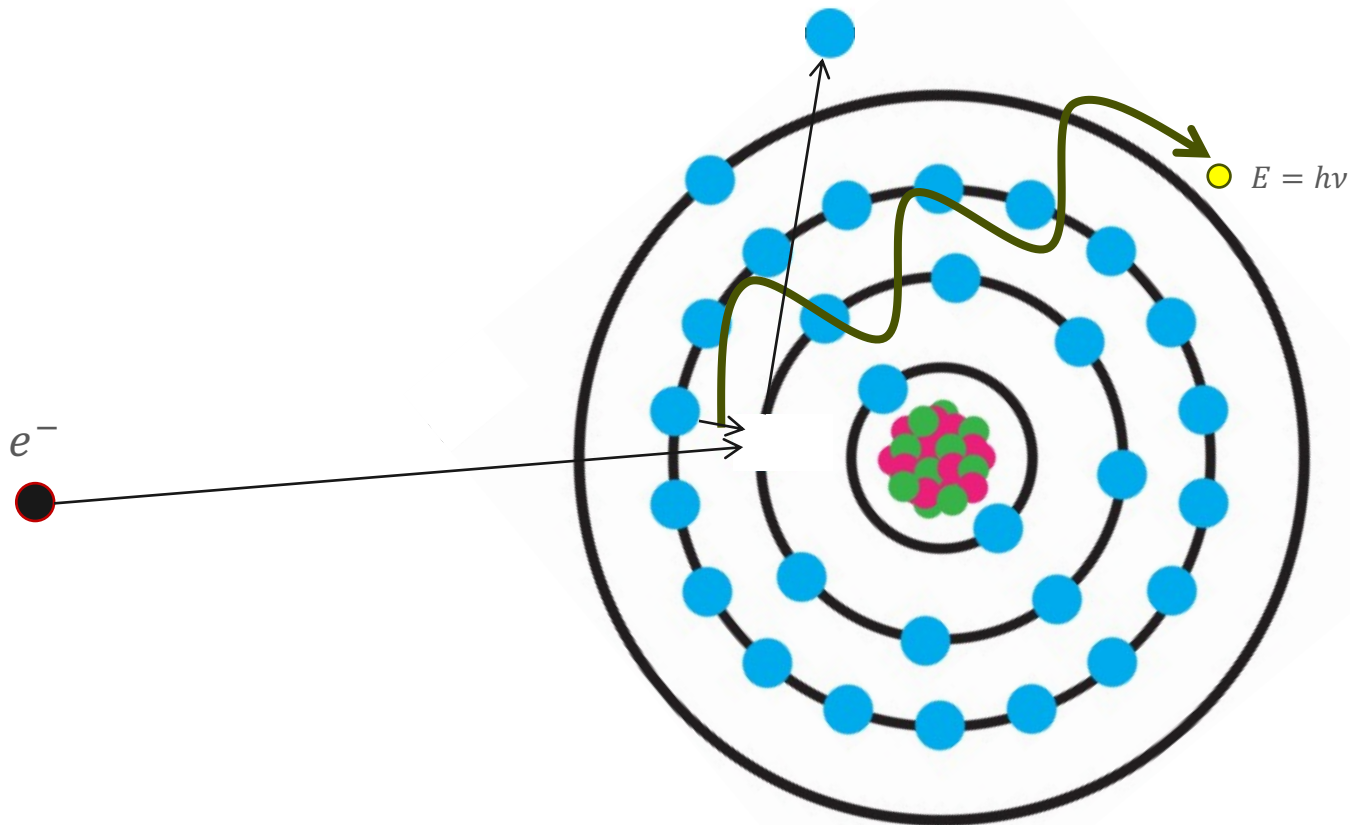
- X-rays emitted as an outer electron fills an inner-shell vacancy.

○ **Mechanism:**

- The incident electron removes an inner-shell electron.



X-ray production



Characteristic radiation

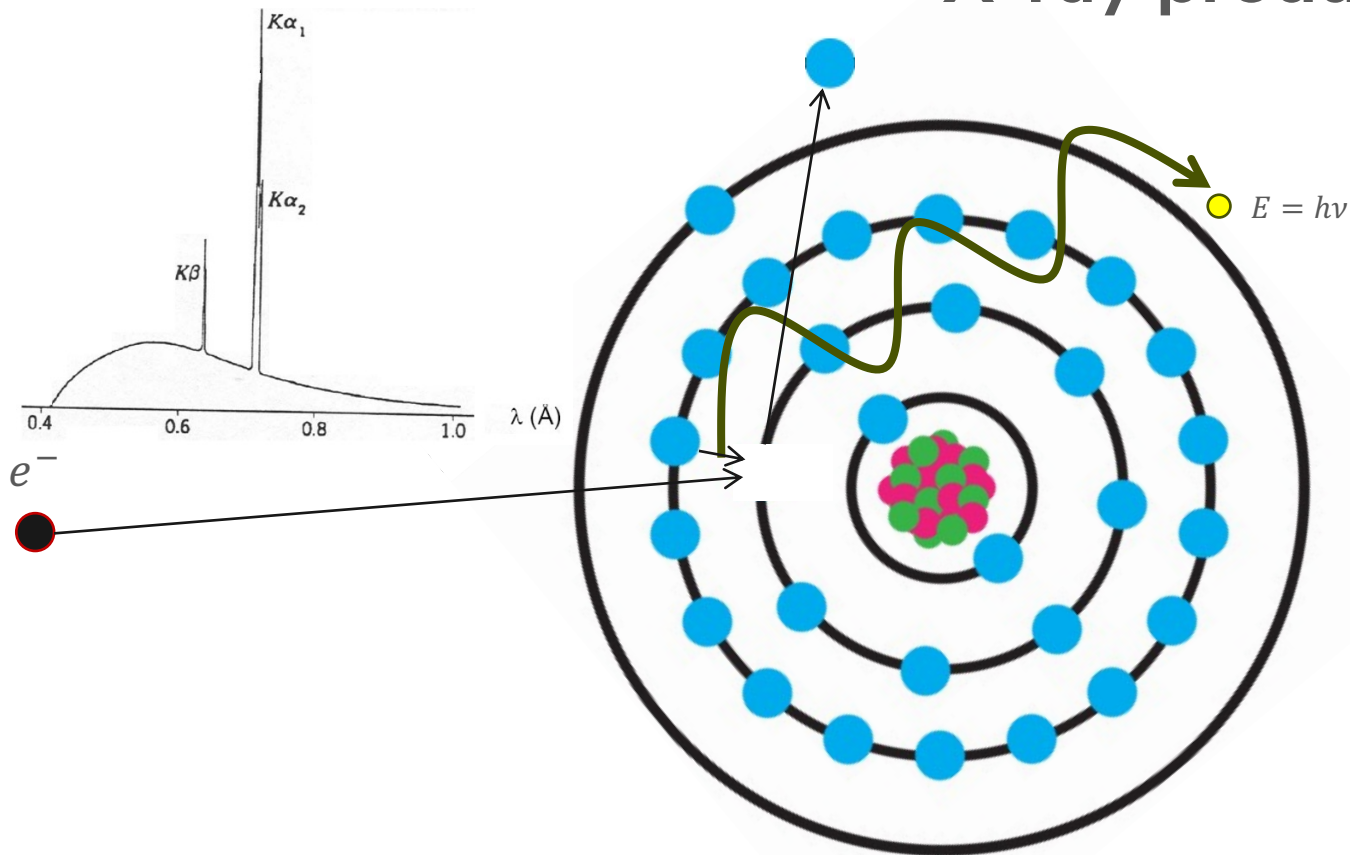
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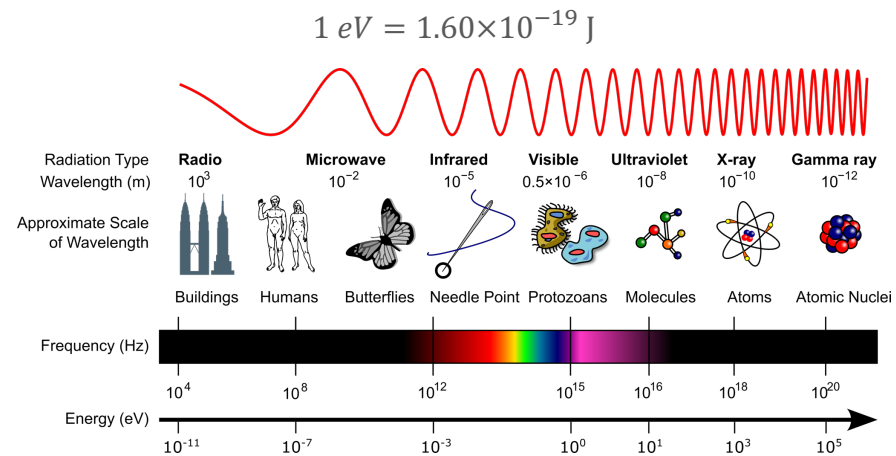
- Distinct peaks
- Element-specific

➡ 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 ($E_{rad} \sim 10 \text{ keV}$), some electrons can be removed from atoms or molecules, thereby ionizing them ($E_{ion} \sim 10 \text{ eV}$).



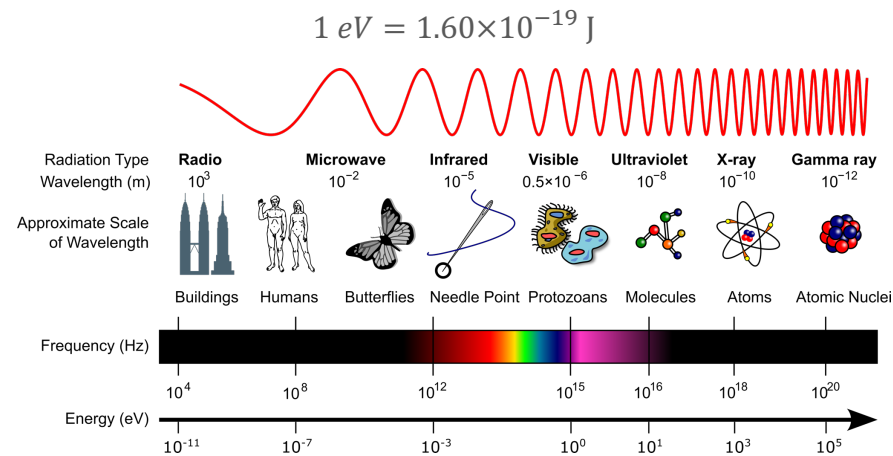
$$E = h\nu = \hbar\omega$$

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

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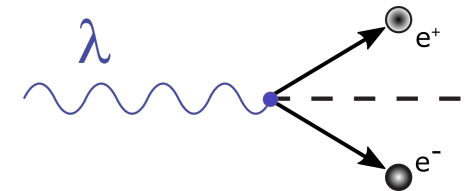
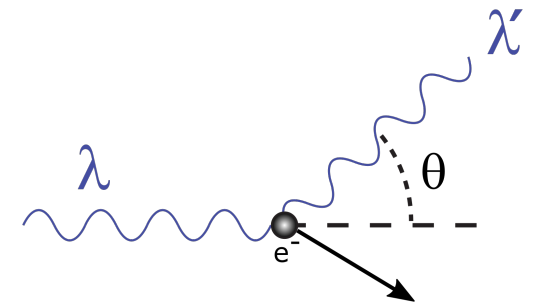
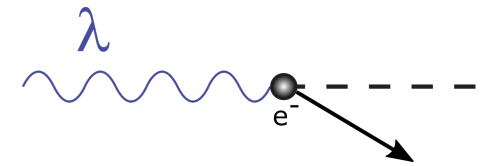


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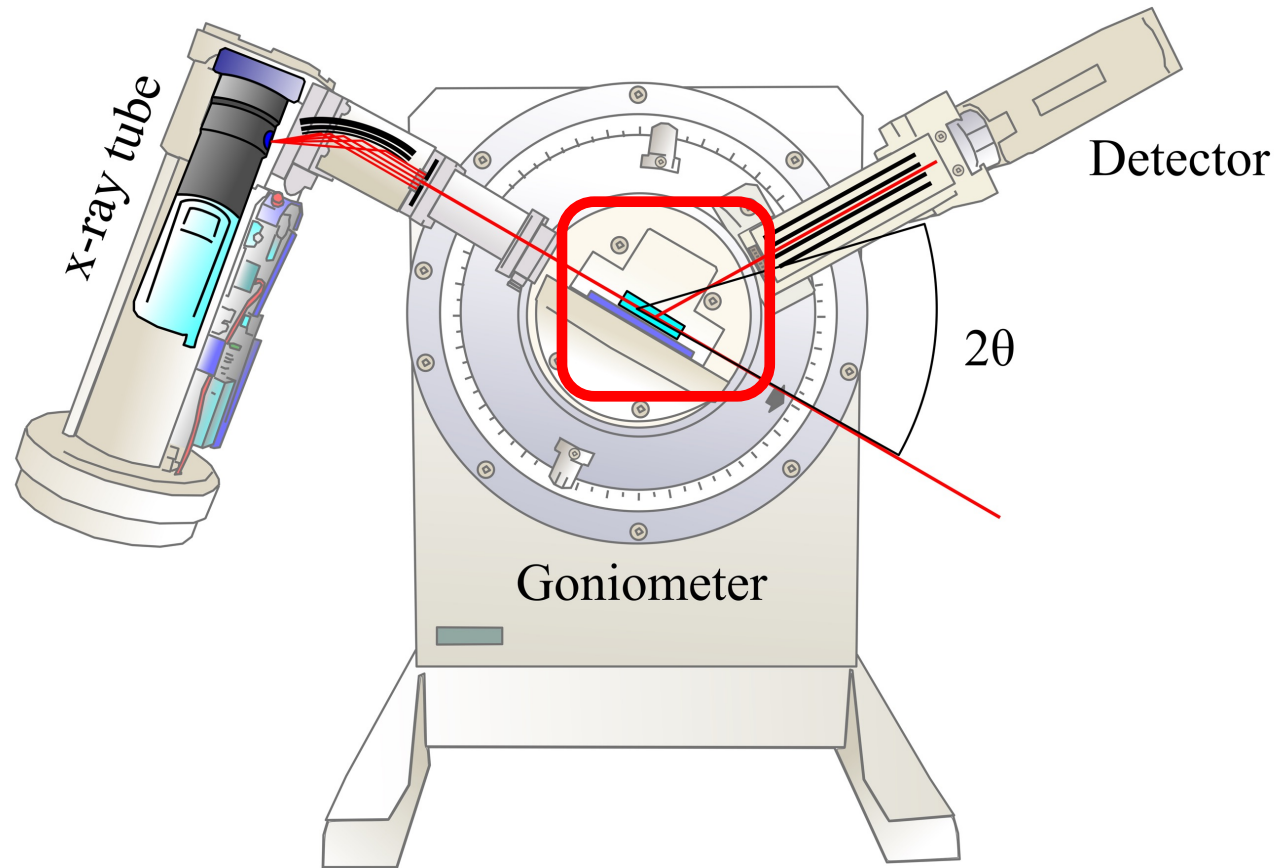
Photon-matter interaction

- ❖ **Photoabsorption effect:** A photon transfers all its energy to an electron, ejecting it if the energy exceeds the electron's binding energy.
- ❖ **Compton scattering:** 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:** A high-energy photon interacts with an atom's nucleus and converts into an electron-positron pair.

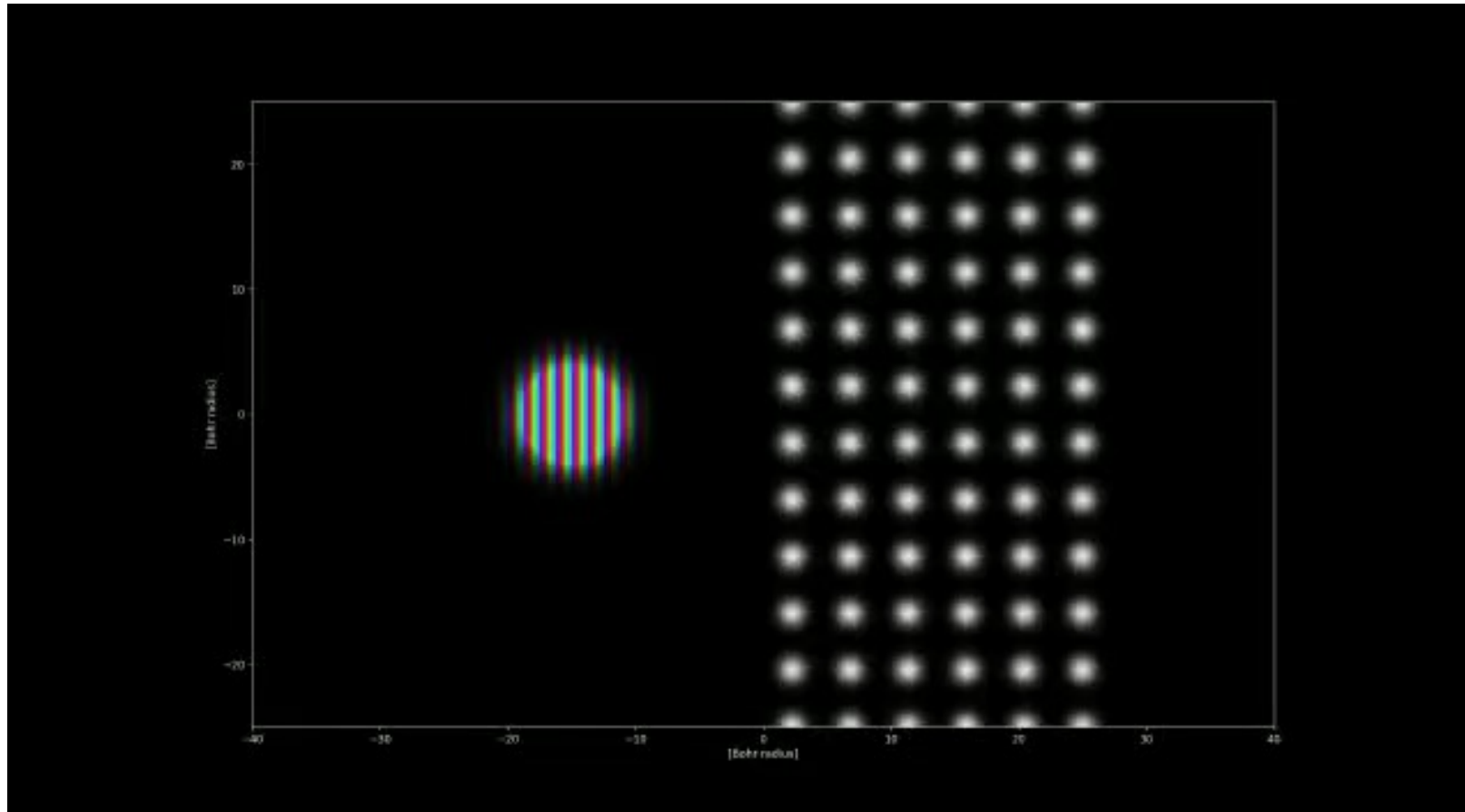


The experimental setup: the diffractometer

The diffractometer (Bragg-Brentano configuration)



The diffraction

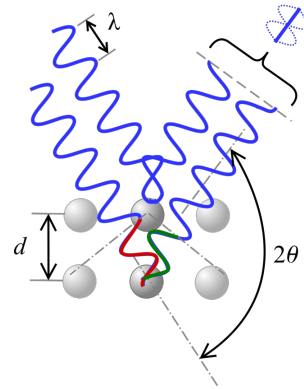
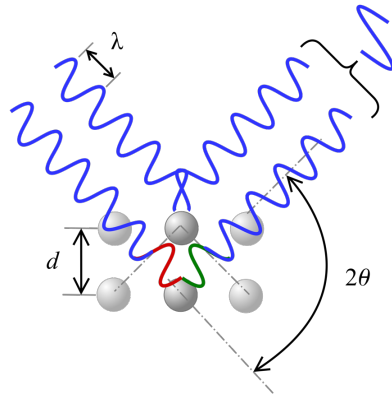


Interaction X-ray matter

❖ Liquids or amorphous materials:

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

Interaction X-ray matter



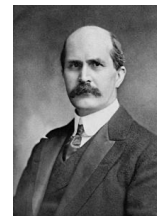
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❖ In crystalline materials:

- X-rays produce sharp and intense peaks of radiation at certain angles

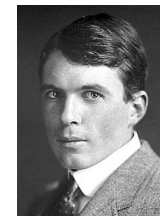
➡ This suggests that X-rays interact in a structured manner with the internal atomic planes of the crystal.



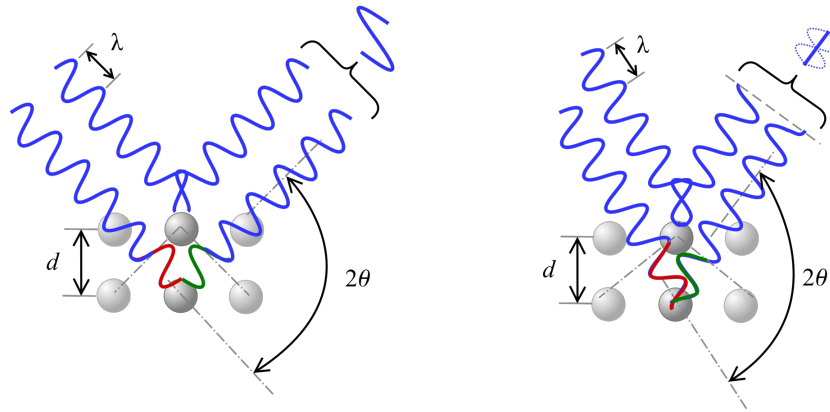
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(1862 – 1940) 

William Lawrence BRAGG
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2/2 Nobel Prize (1915)



Interaction X-ray matter



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

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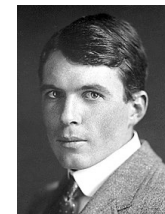
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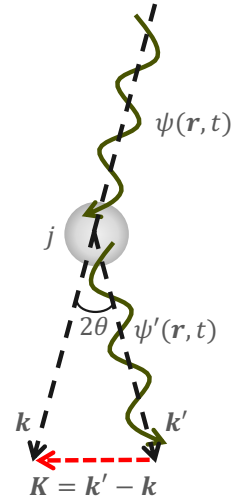


The Bragg's law

❖ From the Laue Conditions:

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

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



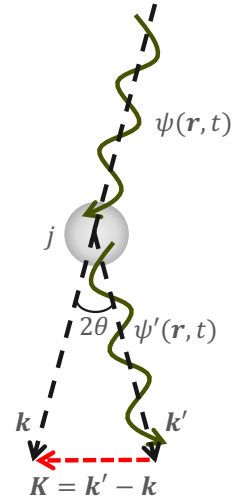
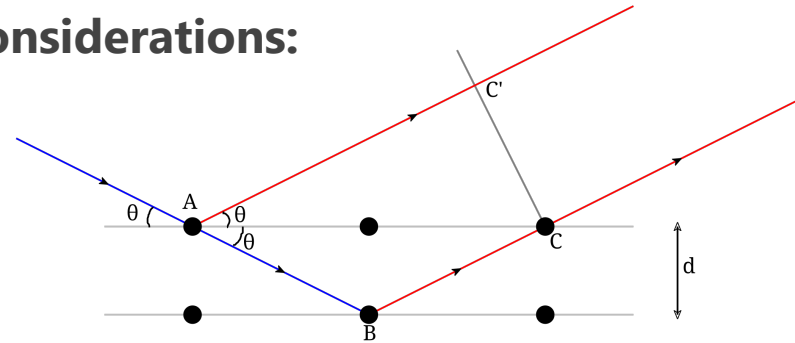
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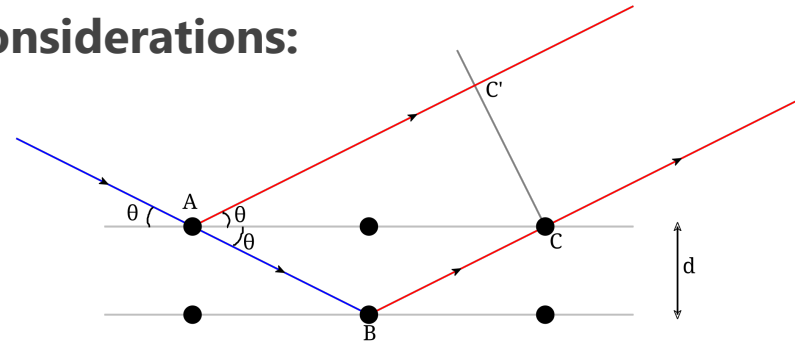
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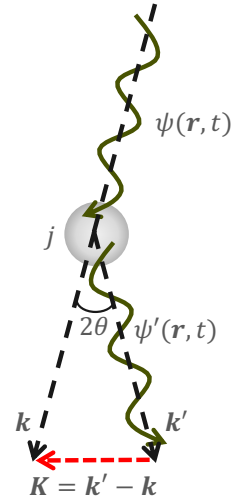
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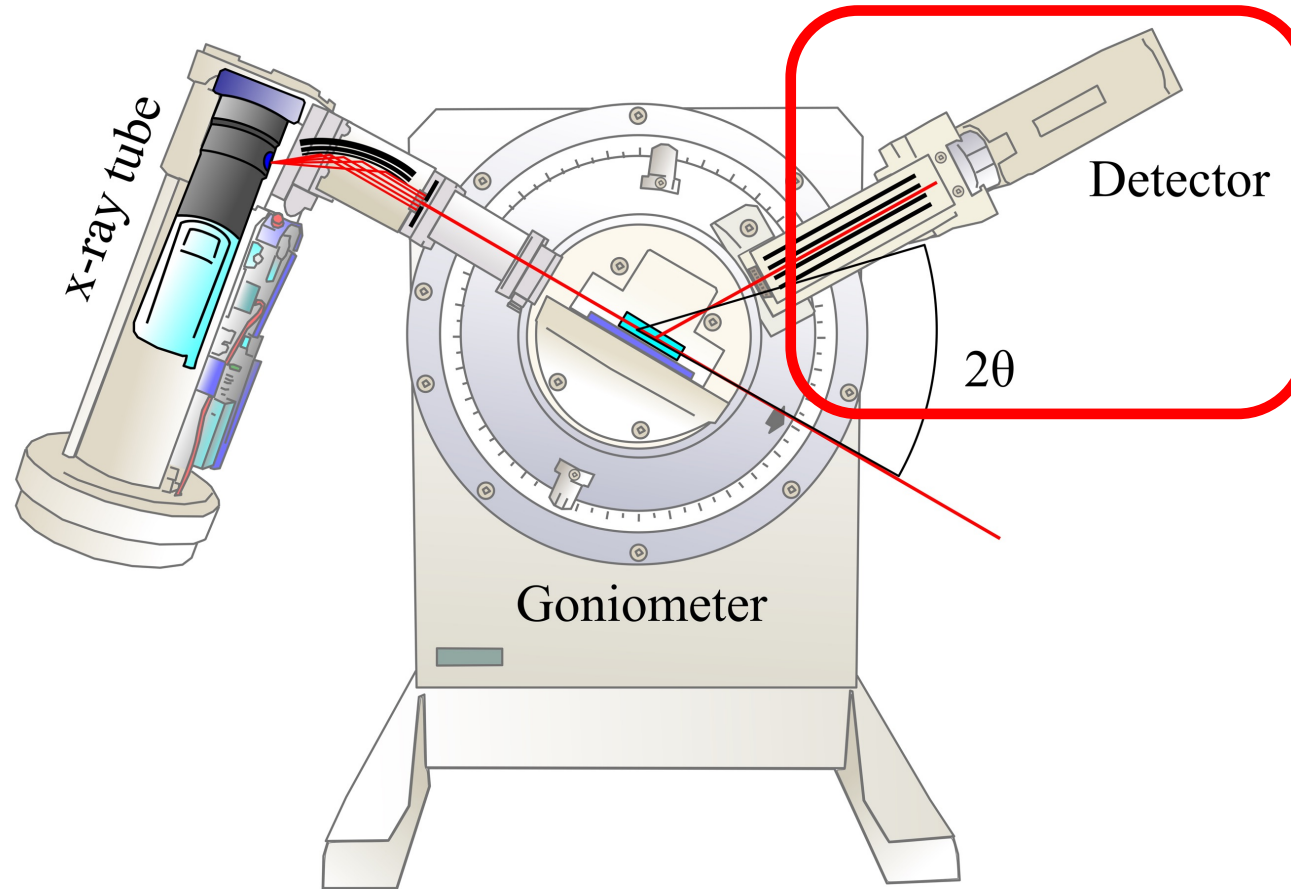
- The path difference $\Rightarrow (AB) - (BC) - (AC') = \frac{d}{\sin \theta} + \frac{d}{\sin \theta} - \frac{2d}{\tan \theta} \cos \theta$
- Constructive diffraction occurs when both waves are in phase $\Rightarrow n\lambda = (AB) - (BC) - (AC')$

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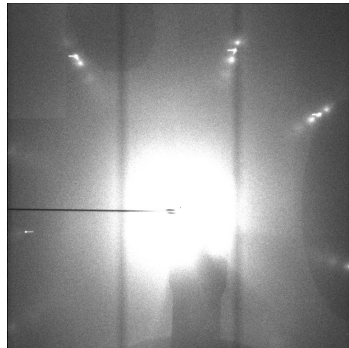


X-ray diffraction techniques

Polychromatic beam

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

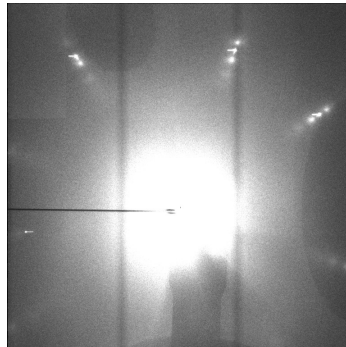
- ❖ Several wave lengths [$\lambda_1; \lambda_2$]
 - ❖ Sample: one monocrystal
 - ❖ In the Bragg's law:
 - Fixed: d, θ
 - Varied: λ
- ⇒ **Isolated spots corresponding to the $\{\lambda, \theta, d\}$ combinations**



X-ray diffraction techniques

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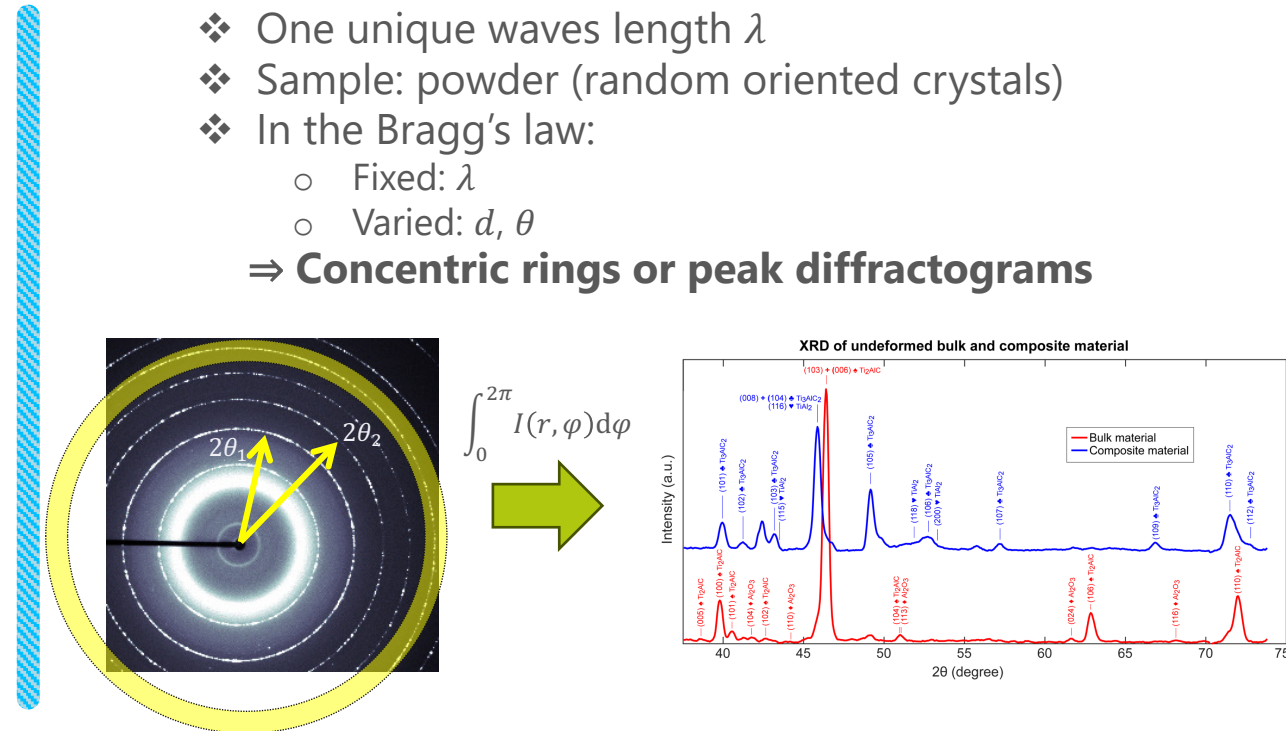
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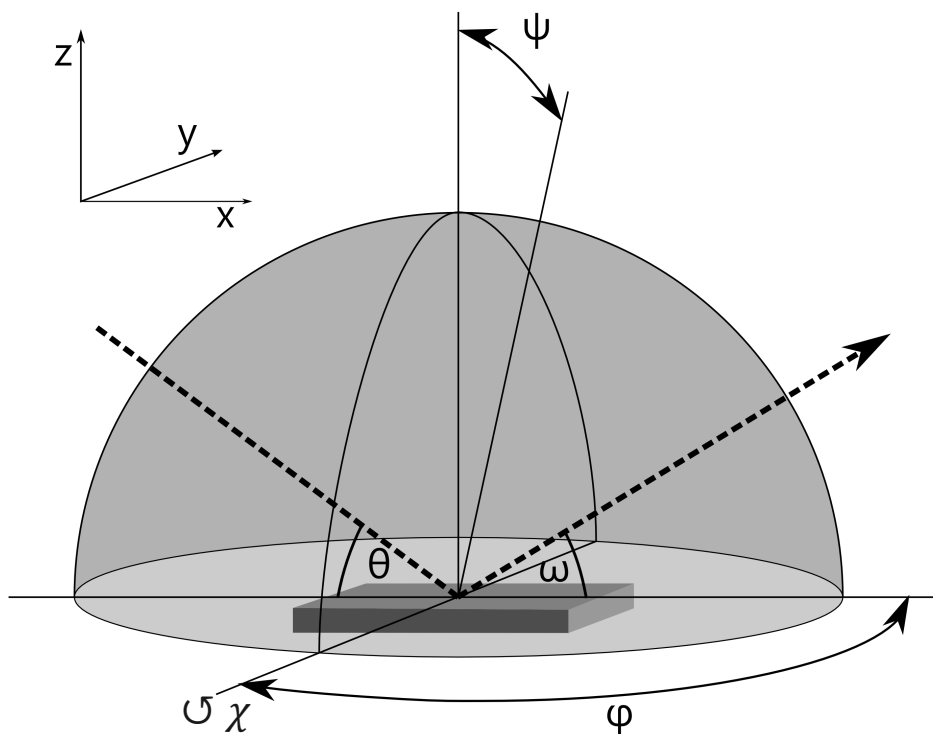
$$n\lambda = 2d \sin \theta$$

Monochromatic beam

- ❖ One unique waves length λ
 - ❖ Sample: powder (random oriented crystals)
 - ❖ In the Bragg's law:
 - Fixed: λ
 - Varied: d, θ
- ⇒ **Concentric rings or peak diffractograms**

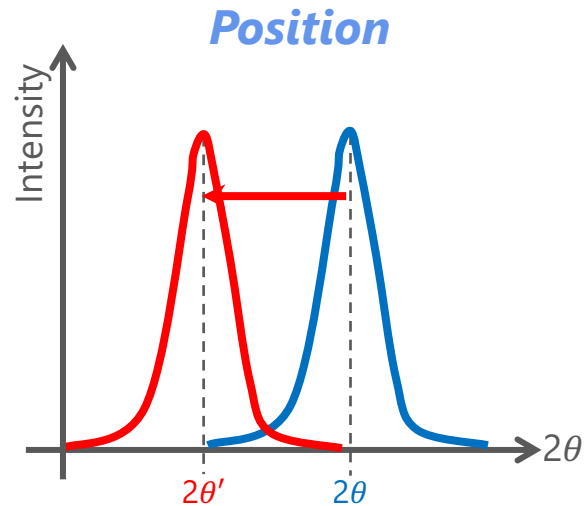


Angle definitions for powder diffraction



- ❖ 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



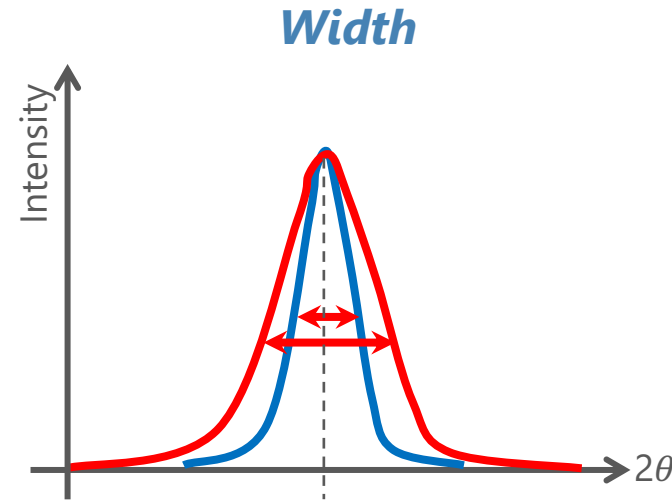
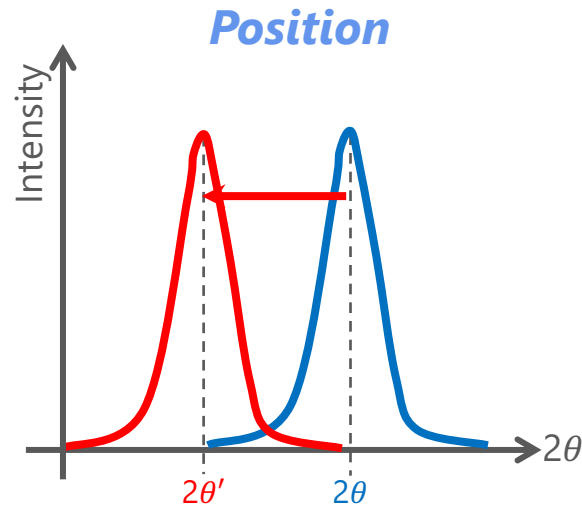
❖ Peak shift:

- $n\lambda = 2d_{hkl} \sin \theta$
 - ✓ $2\theta \searrow \Rightarrow \sin \theta \searrow \Rightarrow d_{hkl} \nearrow \Rightarrow$ macrocompression
 - ✓ $2\theta \nearrow \Rightarrow \sin \theta \nearrow \Rightarrow d_{hkl} \searrow \Rightarrow$ macrotraction

❖ Average elastic strain in the direction normal to the $(h\ k\ l)$ planes:

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

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❖ Peak width:

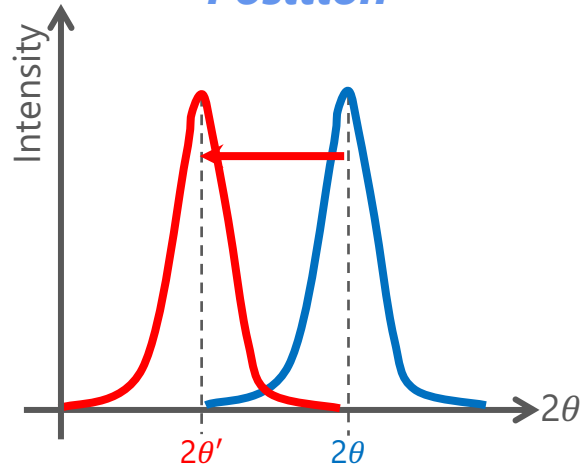
- Crystallite size (L)

$$FWHM \propto \frac{\lambda}{L \cos \theta}$$
- Lattice strain heterogeneities
 - ✓ Dislocation density (ρ)

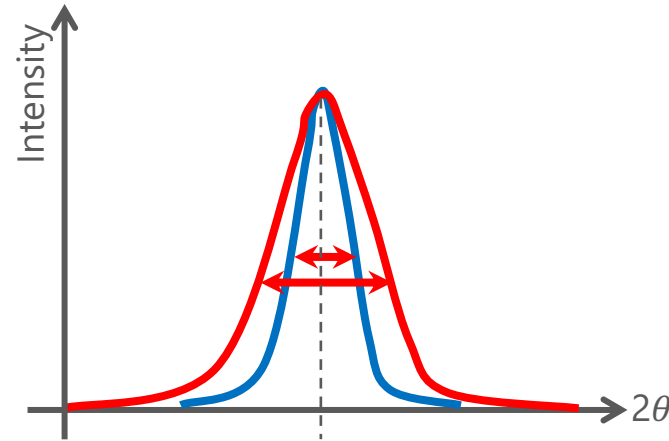
$$\rho \approx \frac{FWHM \cos \theta}{2b^2}$$
 - ✓ Microstrain

Shape of the diffracted peaks

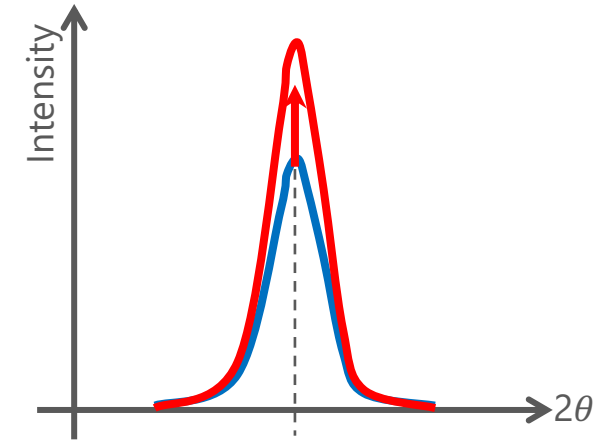
Position



Width



Height



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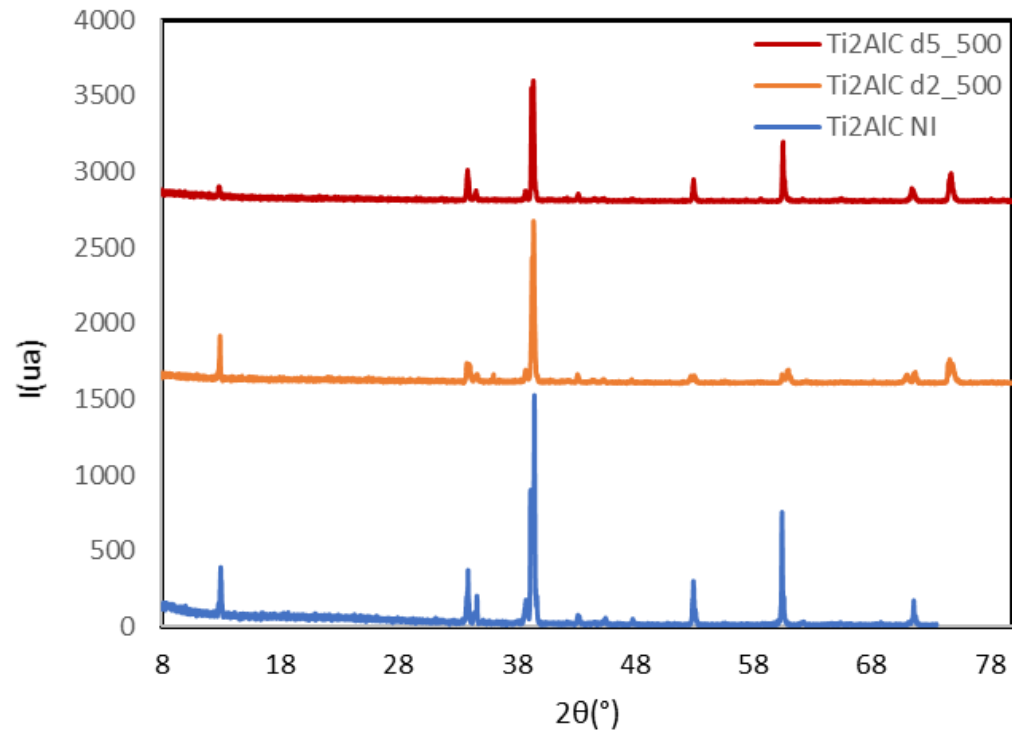
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❖ Peak height:

- Structure factor (F)
- Phases
- Texture

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

Irradiation Ni^{3+} (@500°C)	
Fluence 1	$2 \cdot 10^{15} \text{ cm}^{-2}$
Fluence 2	$5 \cdot 10^{15} \text{ cm}^{-2}$

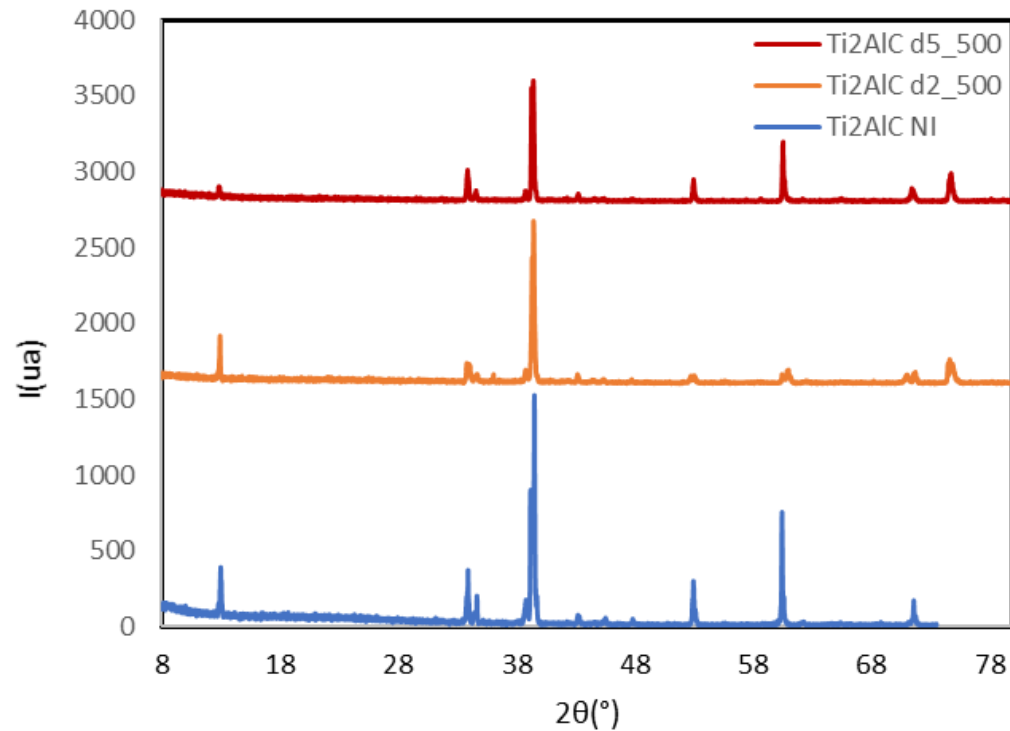


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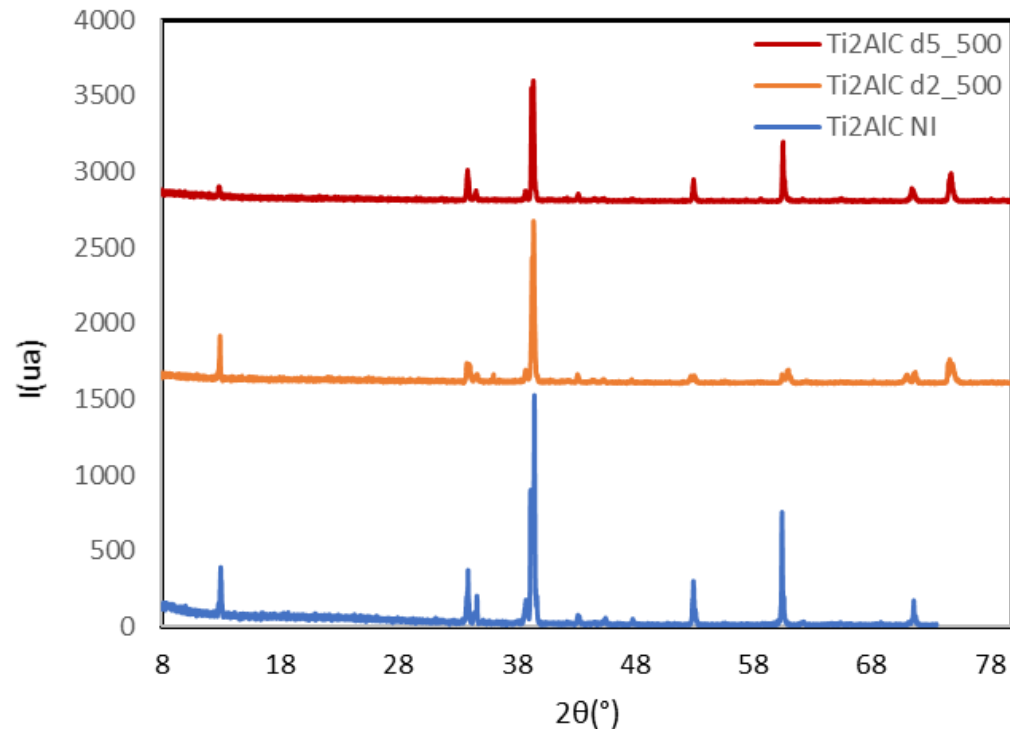
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- All three diffractograms show sharp peaks typical of crystalline Ti_2AlC .



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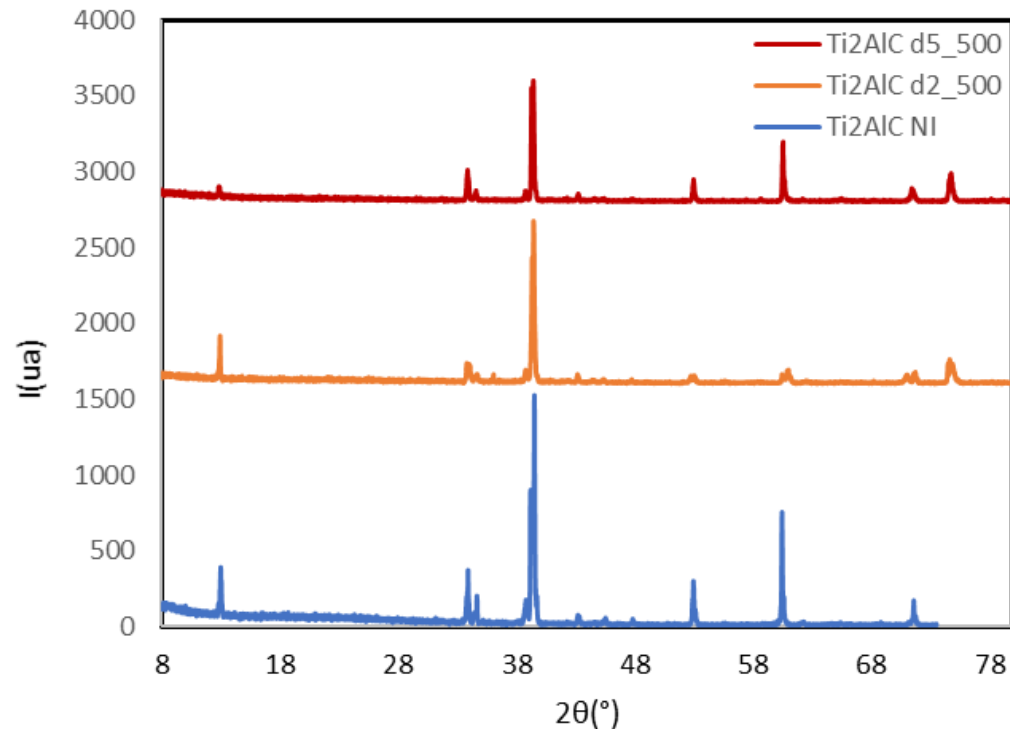
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❖ Comparison of intensities

- The as-received / non-irradiated sample shows strong and well-defined peaks.
- The irradiated samples present similar peak positions but with slightly reduced peak intensities, suggesting a change in crystallinity.

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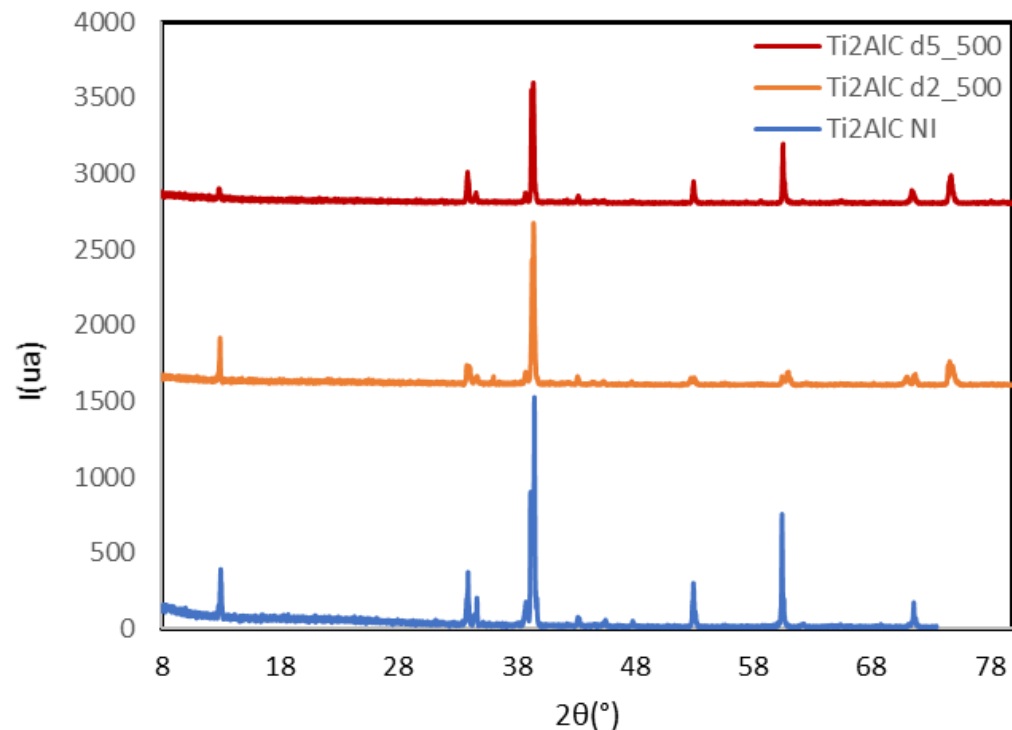
- The as-received / non-irradiated sample shows strong and well-defined peaks.
- The irradiated samples present similar peak positions but with slightly reduced peak intensities, suggesting a change in crystallinity.

❖ Peak broadening

- Peaks in the irradiated samples appear slightly broader than in the non-irradiated one.
- This broadening is consistent with irradiation-induced defects, microstrain, or reduced coherent domain size.

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

Irradiation Ni^{3+} (@500°C)	
Fluence 1	$2 \cdot 10^{15} \text{ cm}^{-2}$
Fluence 2	$5 \cdot 10^{15} \text{ cm}^{-2}$



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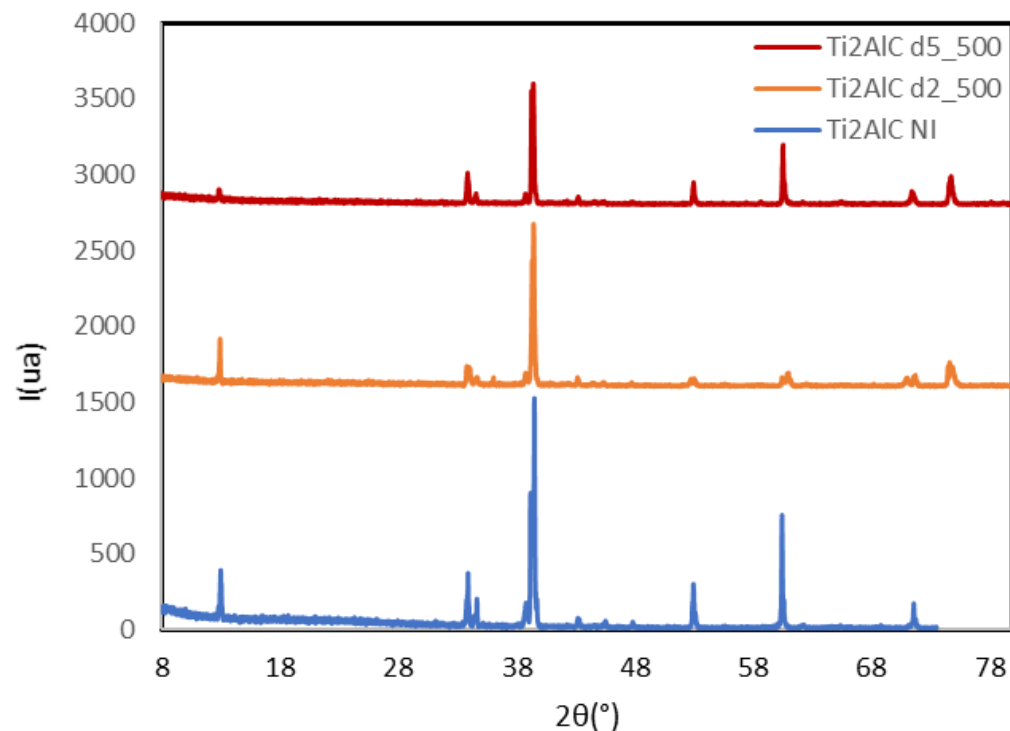
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↪ The structure of Ti_2AlC seems preserved after irradiation, which is significant for radiation tolerance.

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

Where $\sigma_\varphi = \sigma_1 \cos^2 \varphi + \sigma_2 \sin^2 \varphi$

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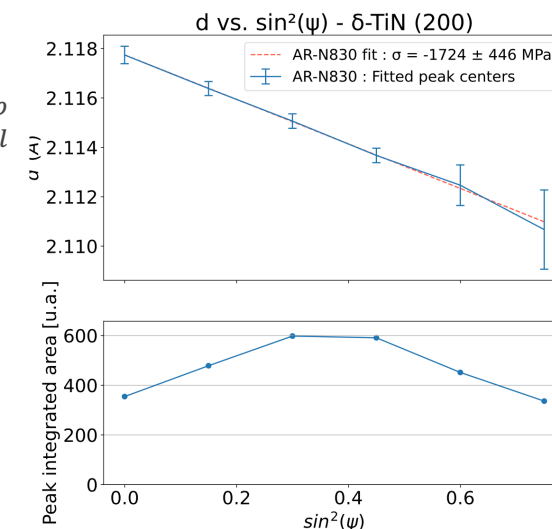
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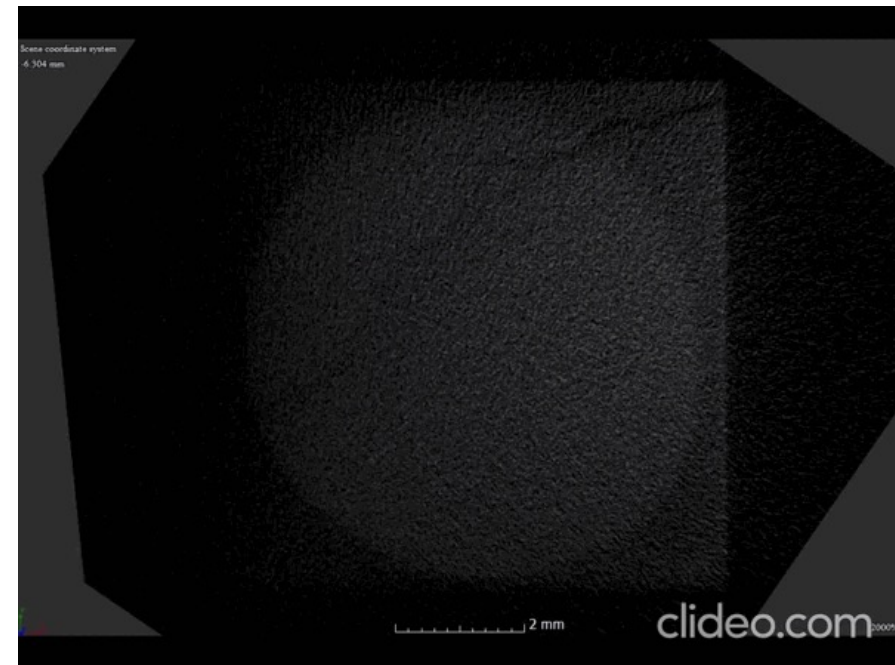
Plotting $\varepsilon_{hkl}^{\psi\varphi} = f(\sin^2 \psi)$ yields a straight line; making residual stress measurable through its slope.

X-ray tomography

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

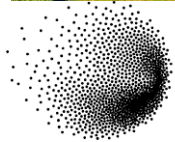
$$N = N_0 \exp(-\mu_{\rho} \rho t)$$

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

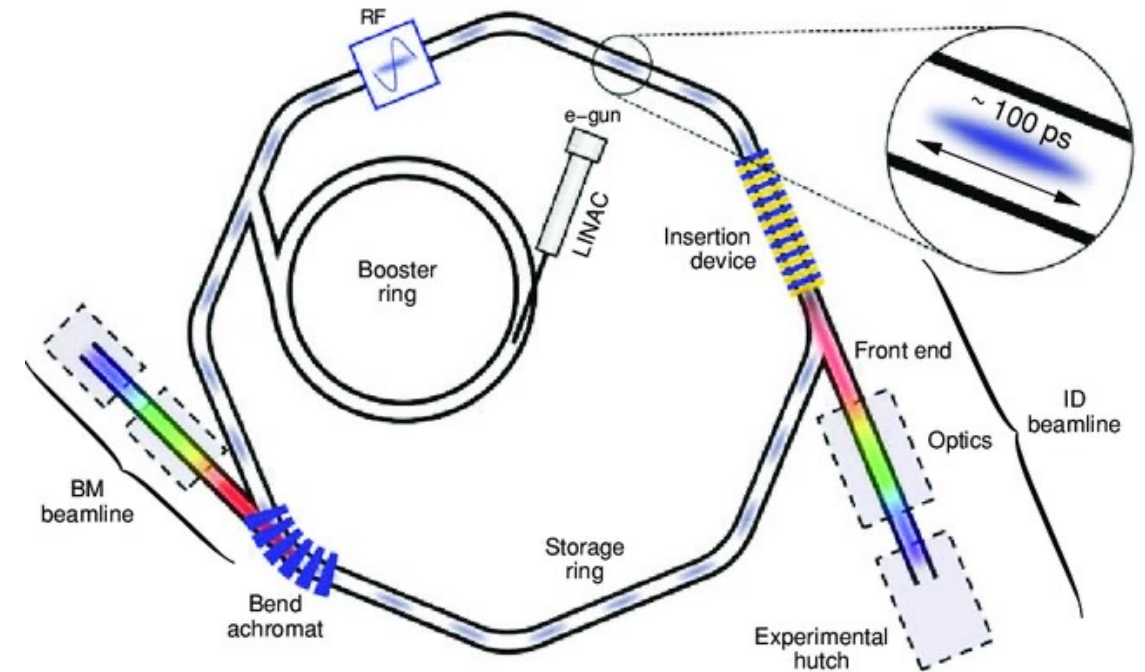


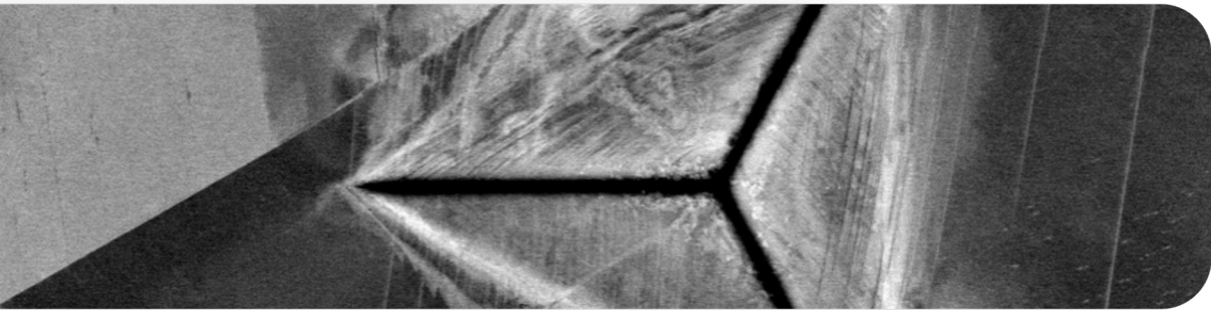
Large x-ray facilities

69



PSI





Electron microscopy

Micro- Nano-meter scale

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

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Scanning electron microscope (SEM)



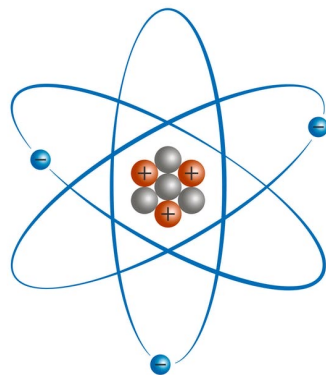
- ❖ Invention: 1937 by Manfred VON ARDENNE but developed in the 1960s
- ❖ Specimen : bulk
- ❖ Price: ~ 500 k€
- ❖ Comon in industry

↳ Meso-/micro-/nano-metric scale

↳ The two microscopes are complementary.

Focus on the electron

Particle



Atom structure

- ⊕ Proton
- Neutron
- ⊖ Electron

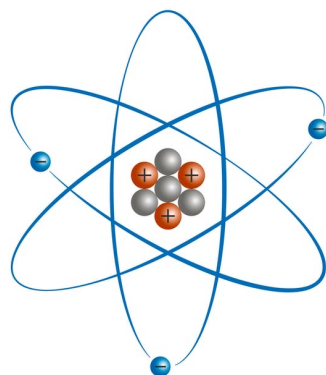


Joseph John THOMSON
(1856–1940) 

$$\begin{aligned} \diamond m_e &= 9.11 \times 10^{-31} \text{ kg} \\ \diamond e &= -1.60 \times 10^{-19} \text{ C} \end{aligned}$$

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Wave



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

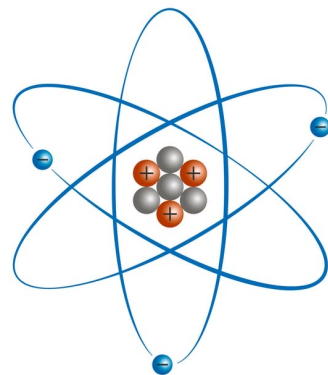
- ❖ All moving particle has wave properties with the wavelength λ being related to the momentum p by:

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

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

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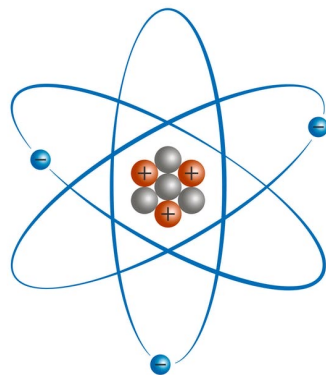
- ❖ Energy of an electron in an electric field of a voltage V :

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

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

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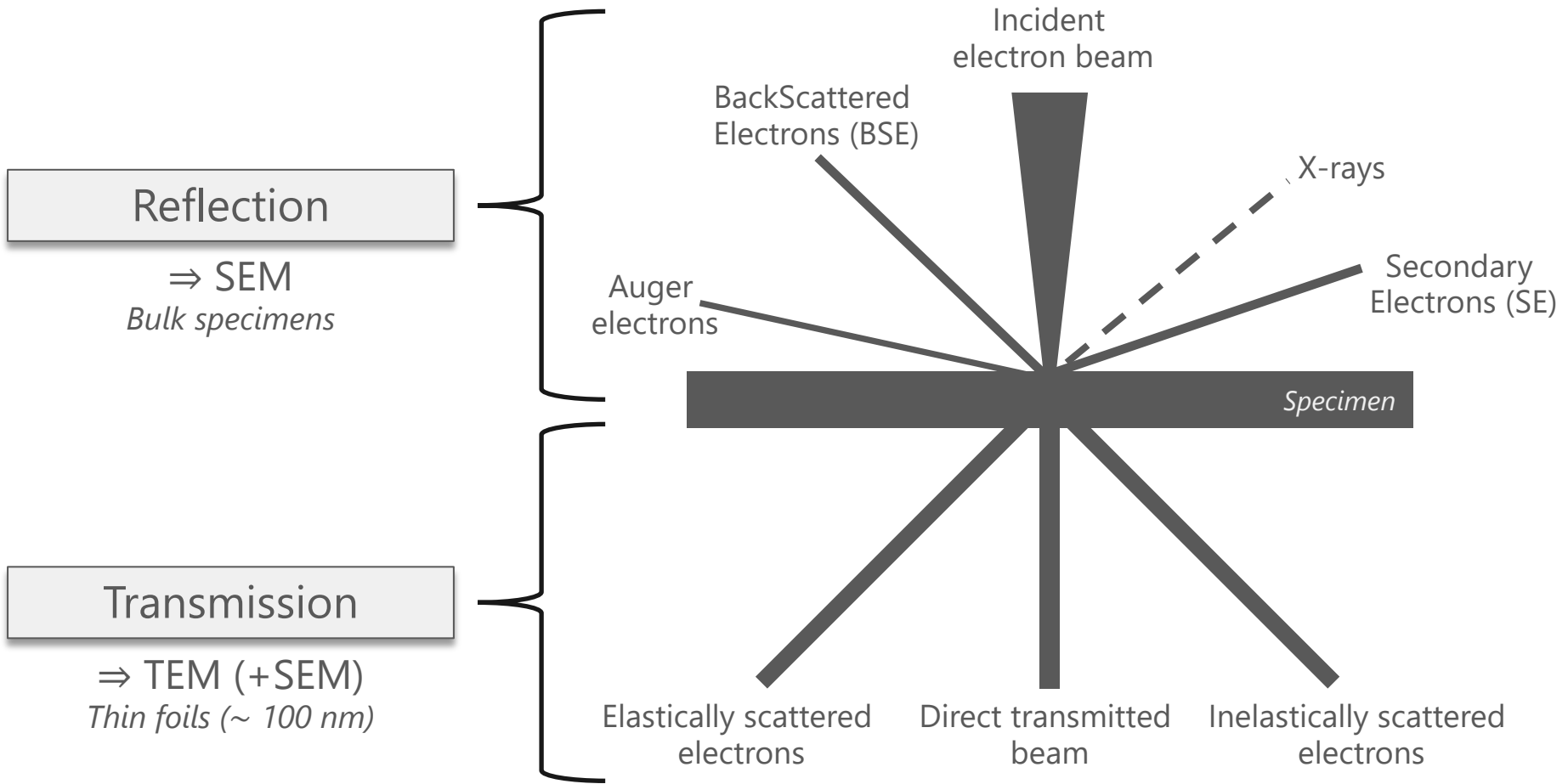
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$$\begin{aligned} \lambda(30 \text{ kV}) &= 7 \text{ pm} \\ \lambda(200 \text{ kV}) &= 2.5 \text{ pm} \end{aligned}$$

Electron-matter interaction



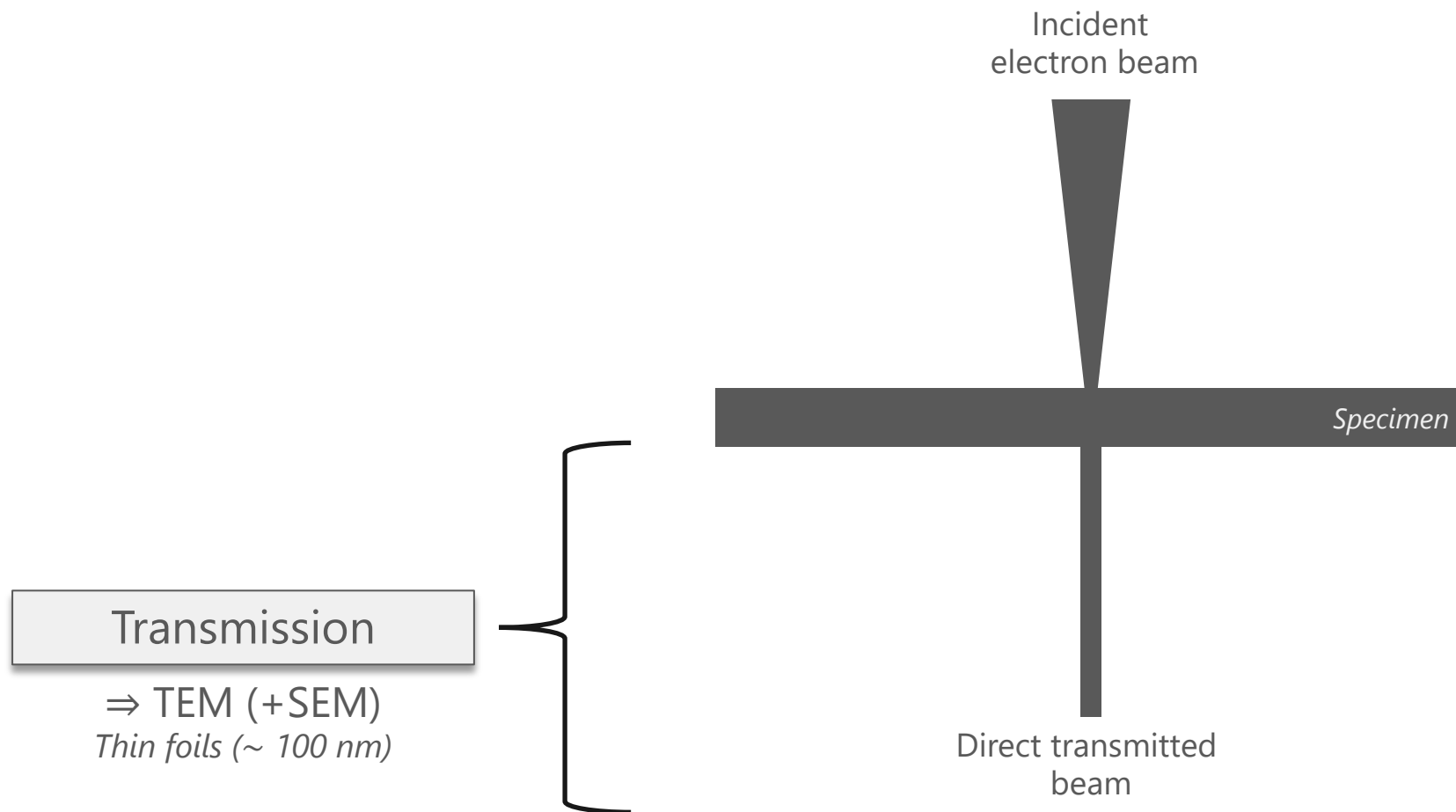
Reflection

⇒ SEM
Bulk specimens

Transmission

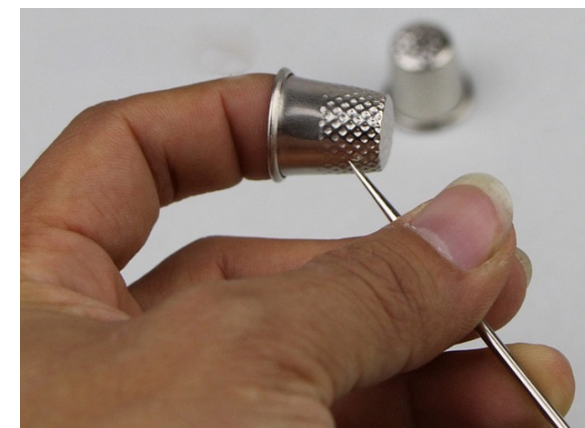
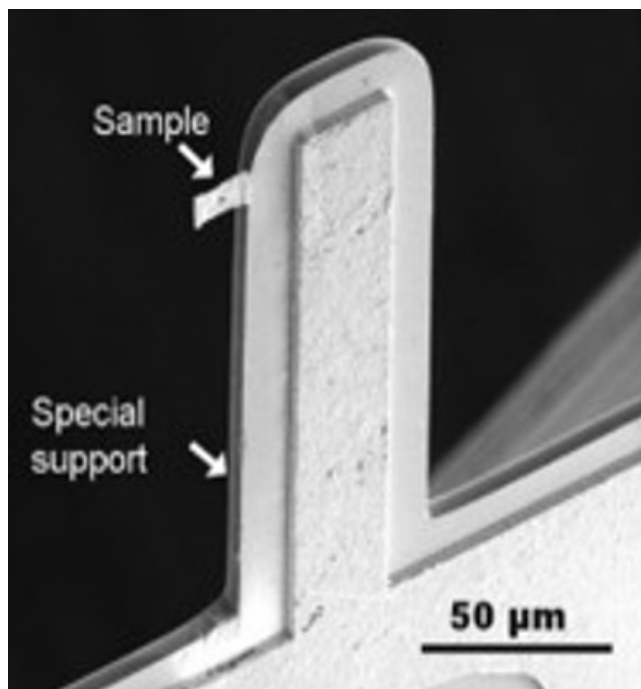
⇒ TEM (+SEM)
Thin foils (~ 100 nm)

Electron-matter interaction



Specimen thickness

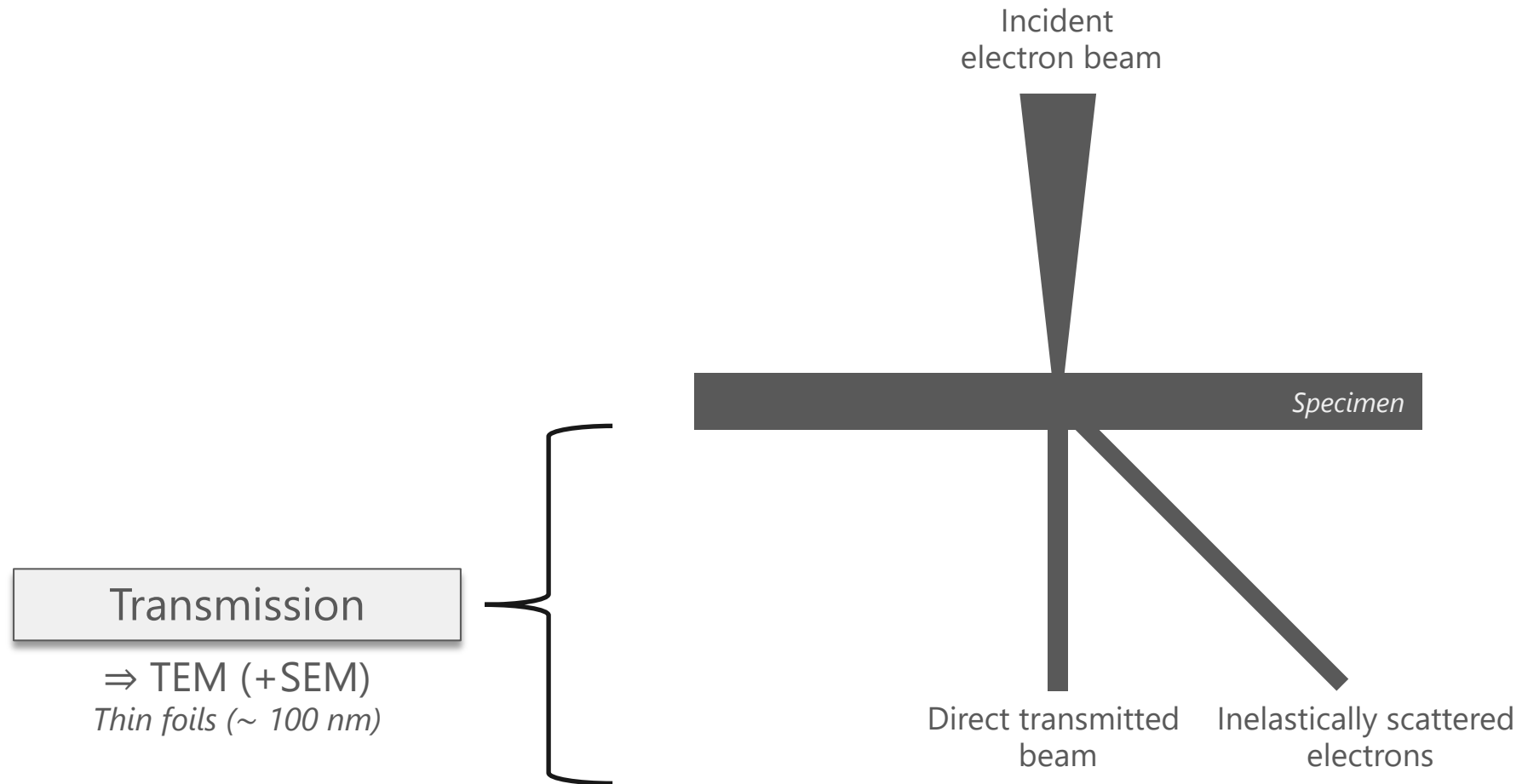
Thickness ≈ 100 nm @ 200 kV (for conventional TEM)



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...

Electron-matter interaction



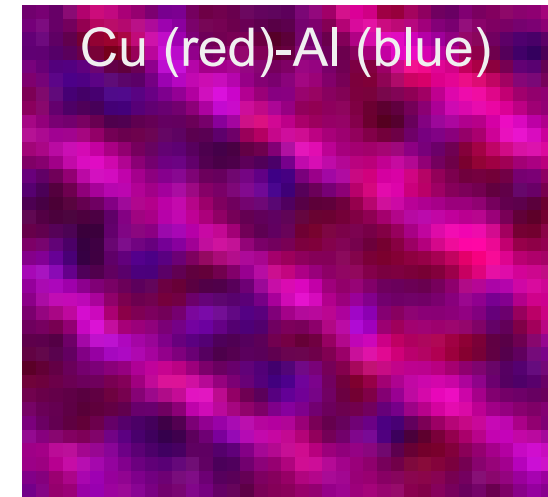
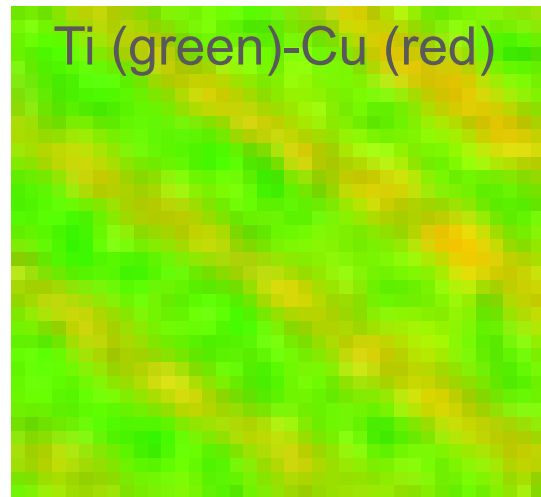
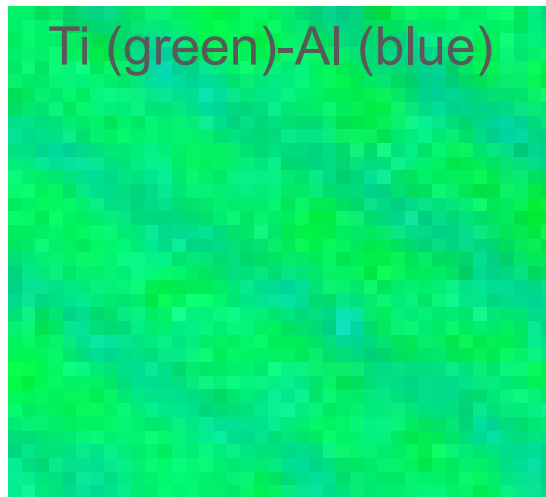
Inelastically scattered electrons

The energy is transferred from the electron to the sample.

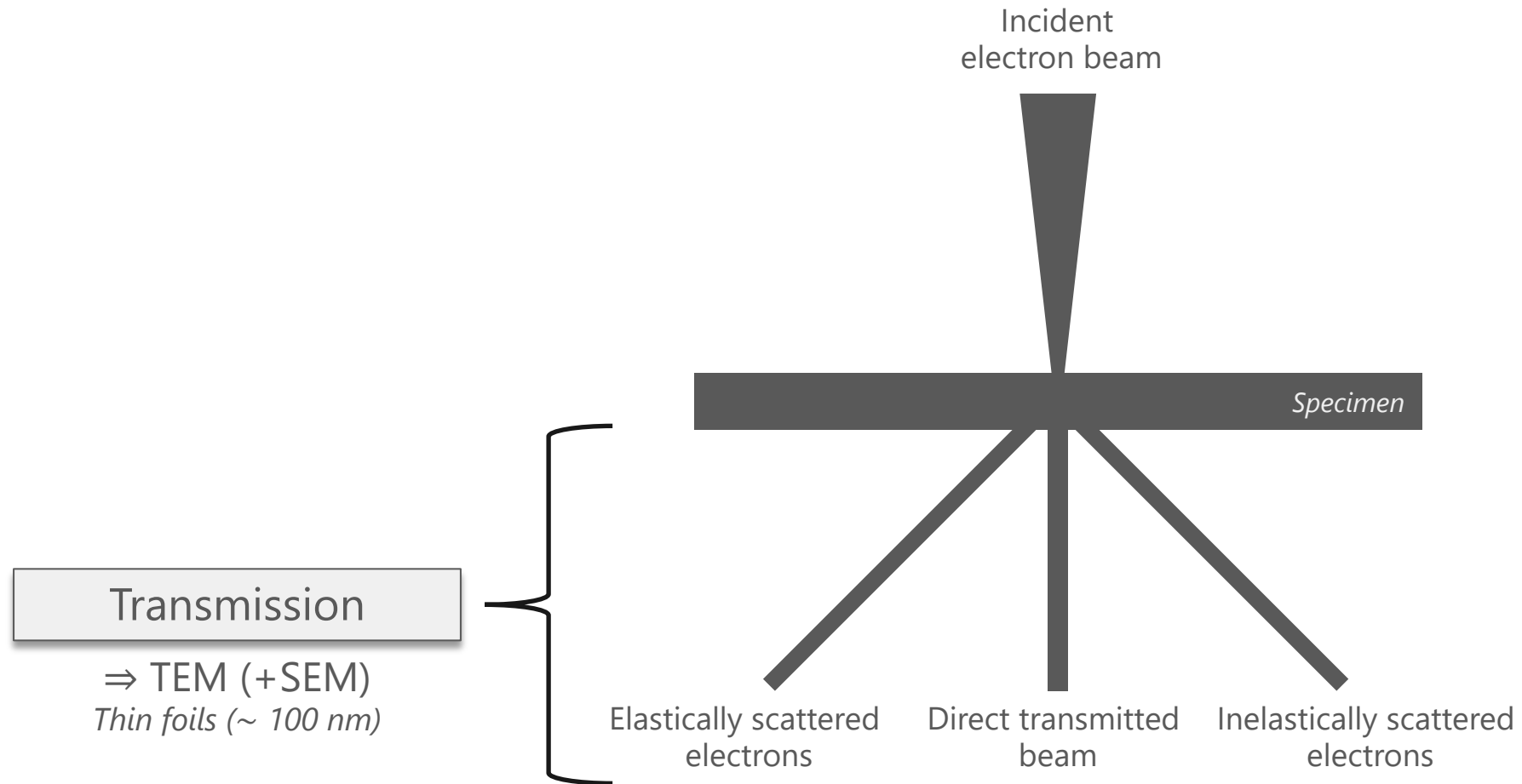
$$\Rightarrow E_0 > E_{e^-}$$

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

Electron Energy Loss Spectroscopy (EELS)



Electron-matter interaction



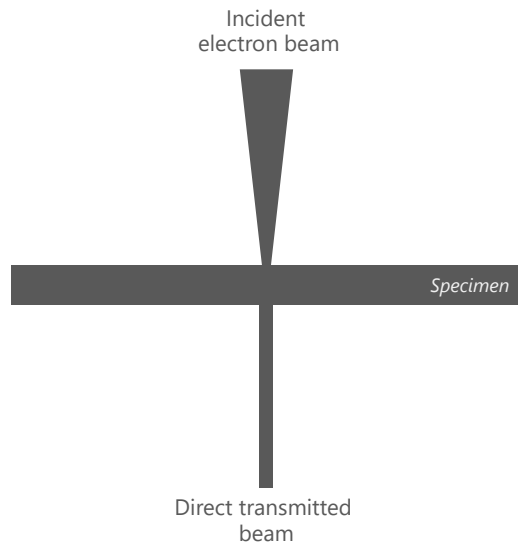
Elastically scattered electrons

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

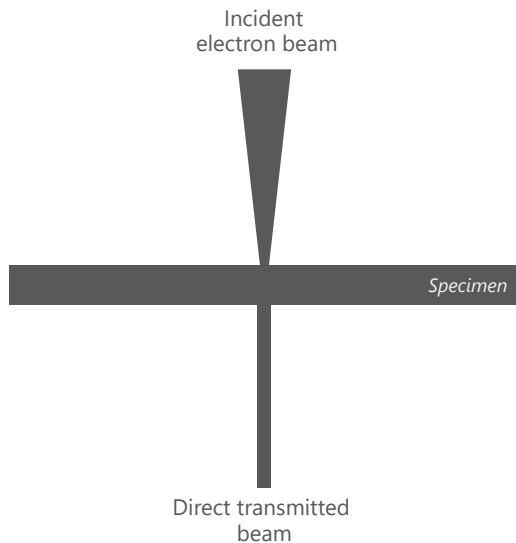
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In other directions

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

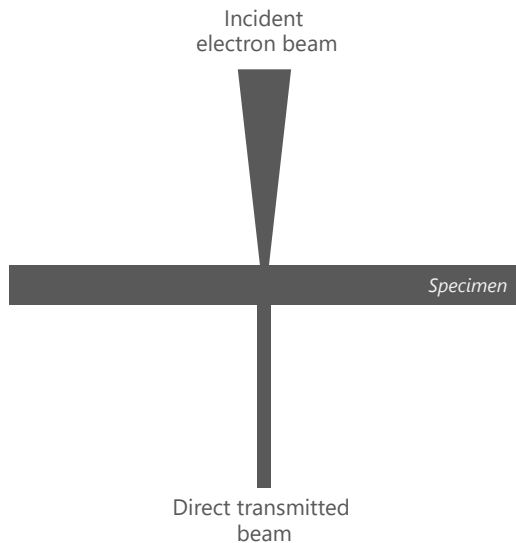
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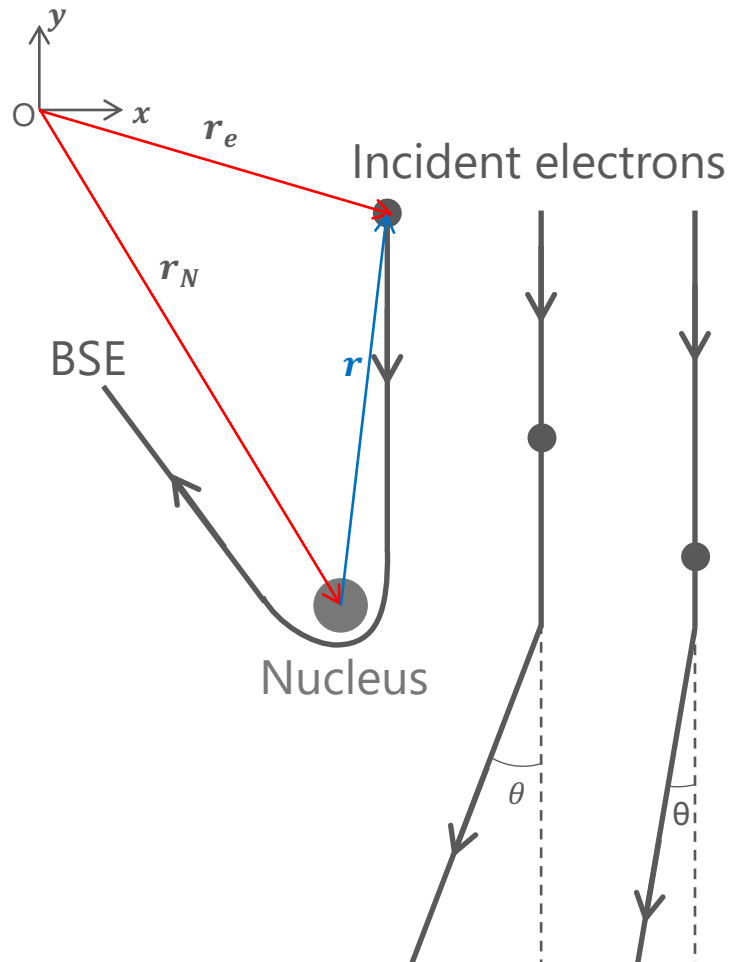
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The diagram shows two positive charges, q_1 and q_2 , represented by green circles. A horizontal line with arrows at both ends passes through them. The distance between the charges is labeled $r_2 - r_1$. The force exerted by q_1 on q_2 is labeled $F_{2/1}$, and the force exerted by q_2 on q_1 is labeled $F_{1/2}$.

$$F_{1/2} = \frac{1}{4\pi\epsilon_0} \frac{q_1 q_2 (r_2 - r_1)}{\|r_2 - r_1\|^3}$$

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

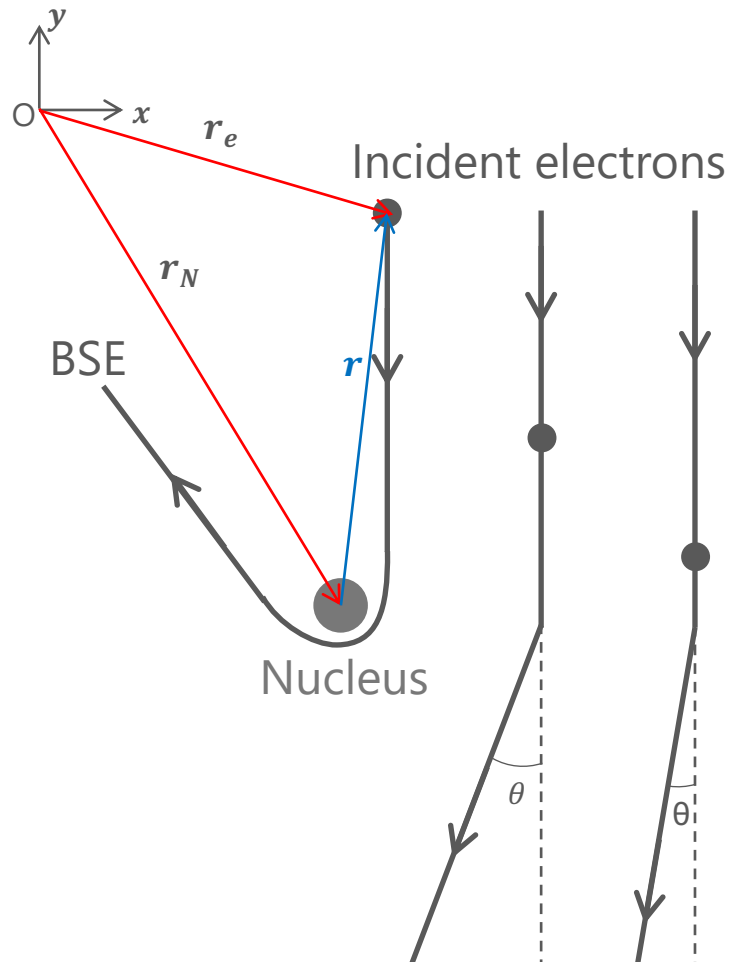
Elastically scattered electrons



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

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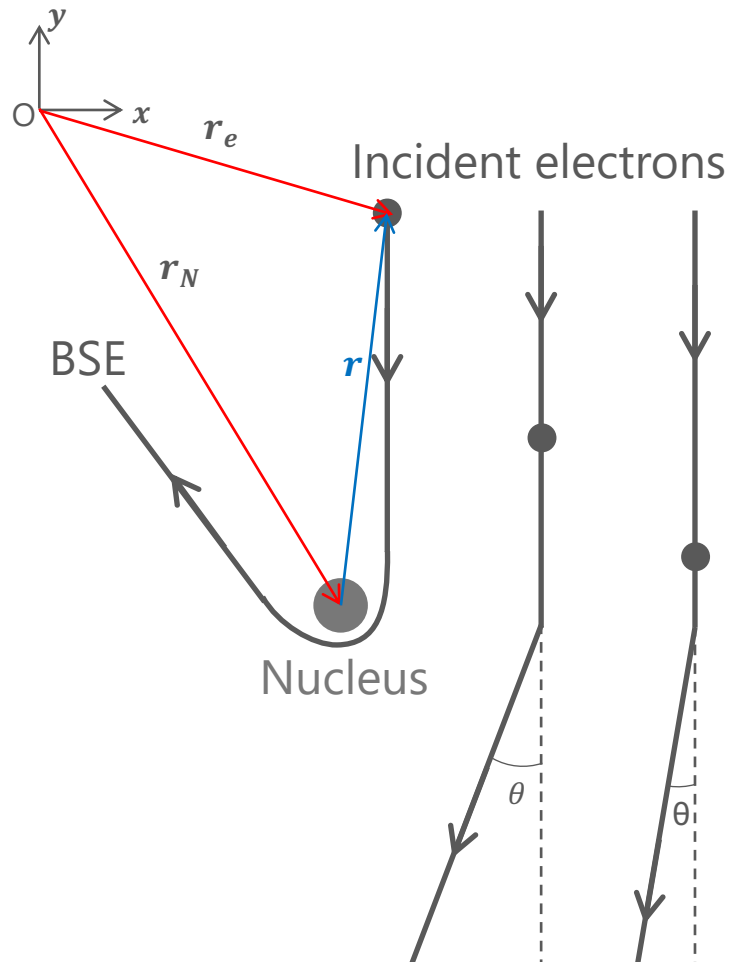
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- Scattered angle θ
 $\Rightarrow r \searrow \Rightarrow F_{N/e} \nearrow \Rightarrow \theta \nearrow$

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

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- Influence of Z :

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

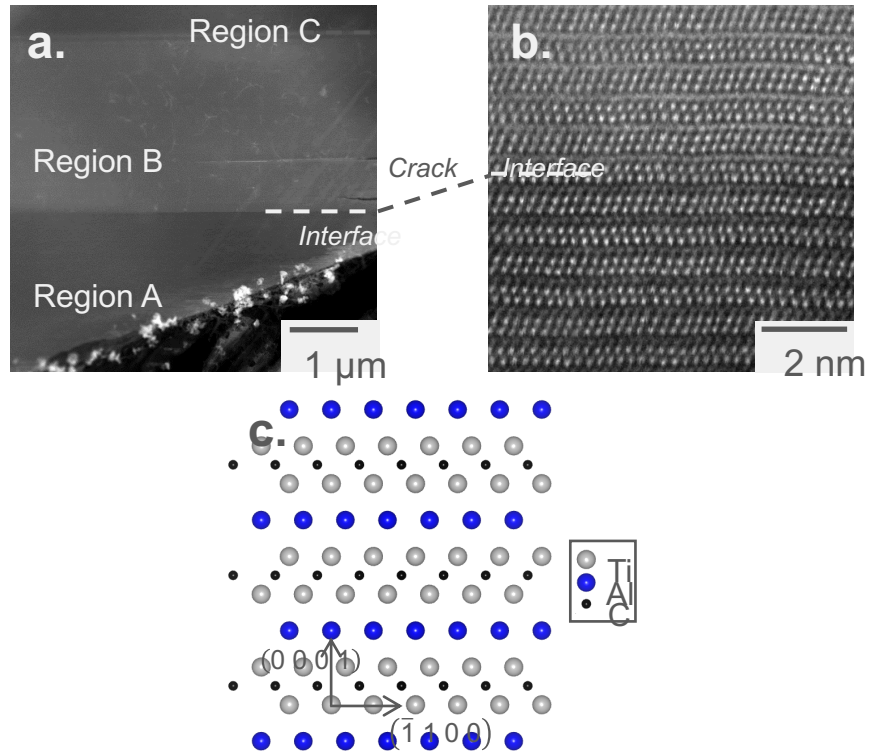
$$q_N \nearrow \Rightarrow Z \nearrow$$

$$Z \nearrow \Rightarrow \theta \nearrow$$

$\Rightarrow$ High Z produces higher intensity at large θ

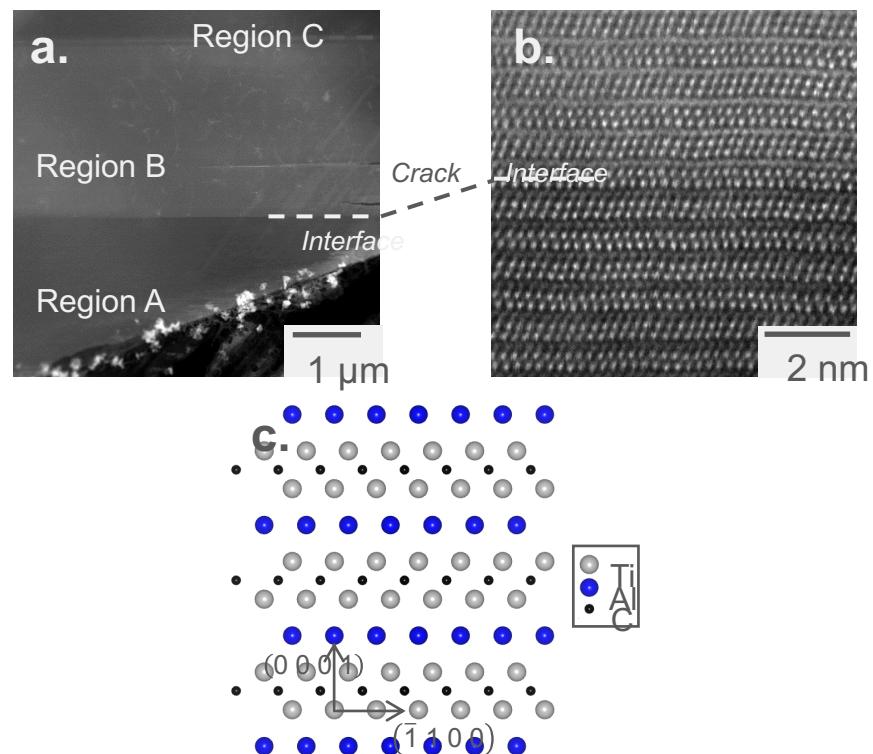
Intensity at large θ

Inside the (S)TEM

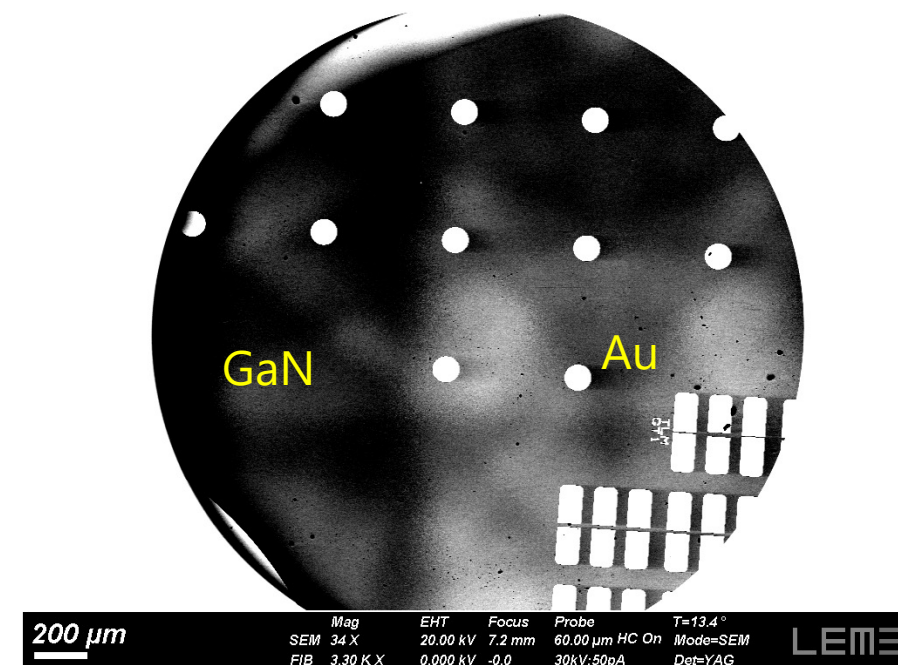


Intensity at large θ

Inside the (S)TEM



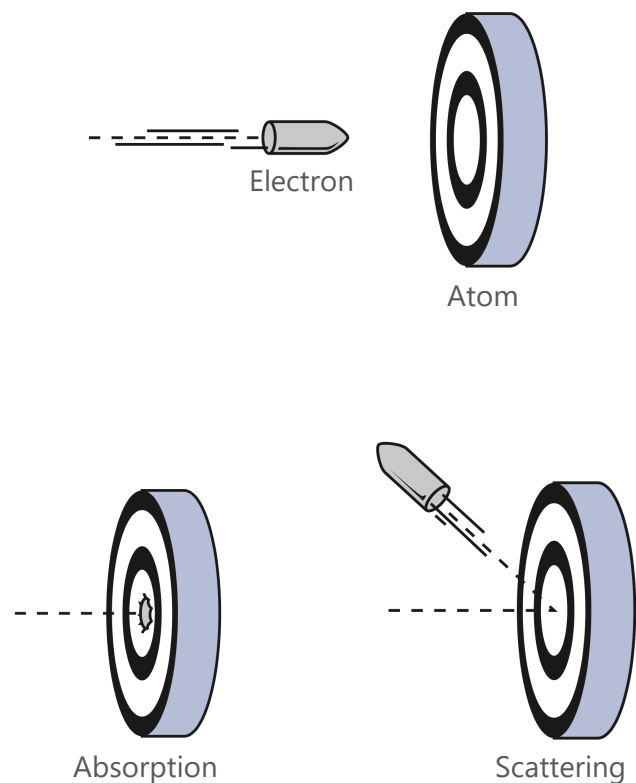
Inside the SEM



↘ Bright contrast $\Rightarrow$ high Z ; Dark contrast $\Rightarrow$ low Z

The cross section of interaction

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

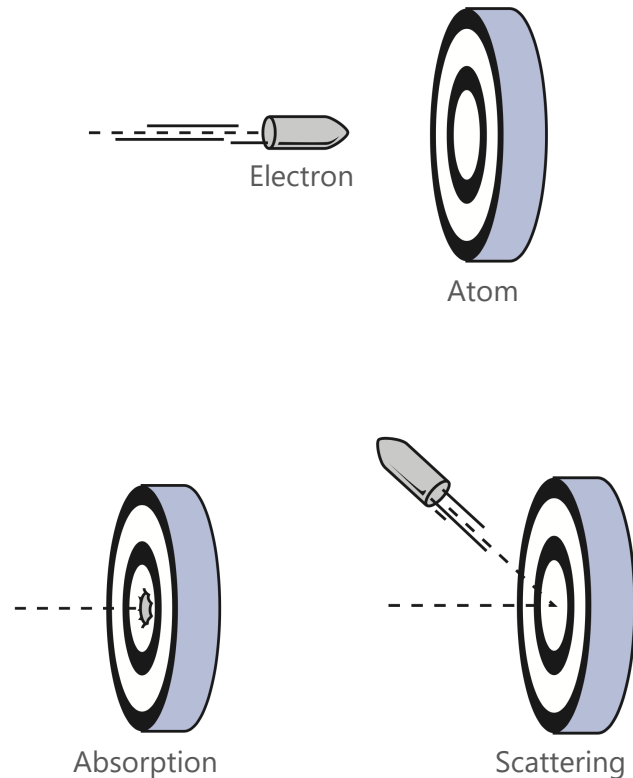


The cross section of interaction

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

❖ **There are two kinds of cross-sections:**

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

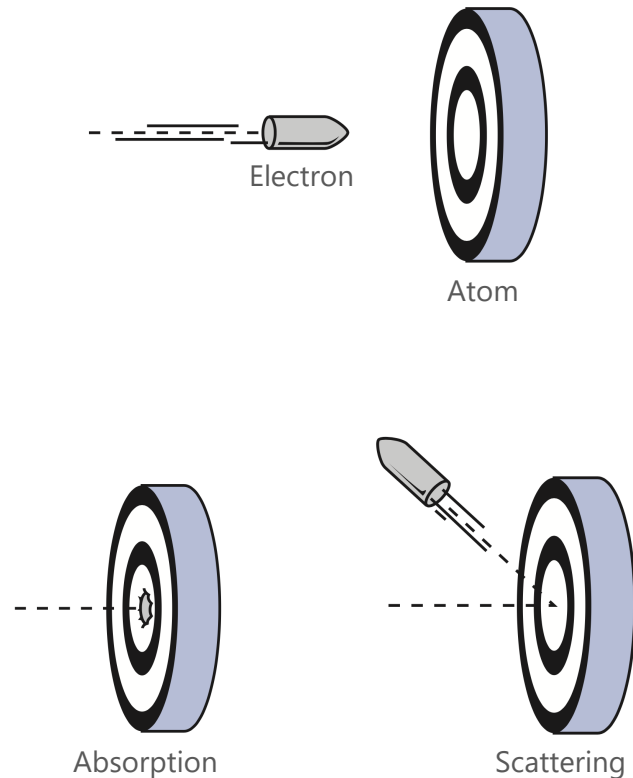


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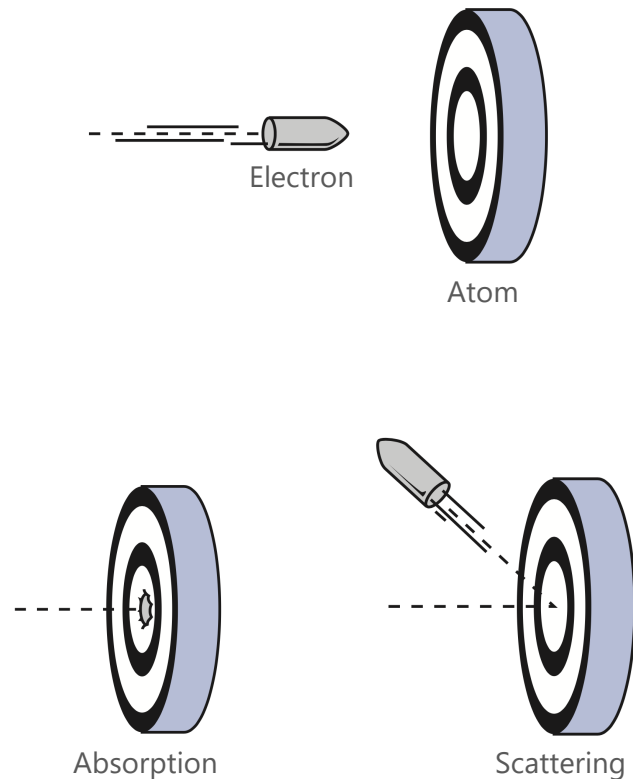
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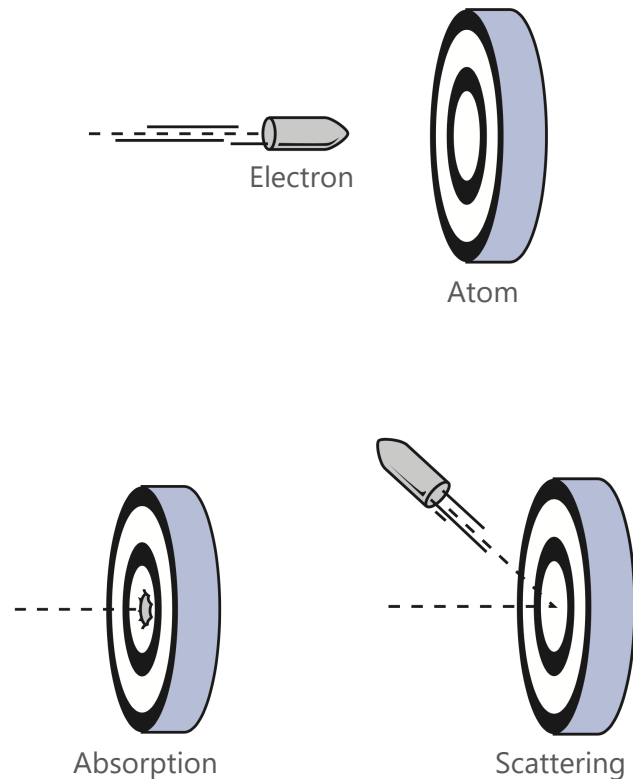
✓ Pure material

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

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

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- Microscopic (σ): it refers to cross sections of electrons reacting with a specific atom
 $10^{-28}\text{cm}^2 = 1 \text{ b}$
- Macroscopic (Σ): it gives the probability for an electron to interact with a target per unit length.

✓ Pure material

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

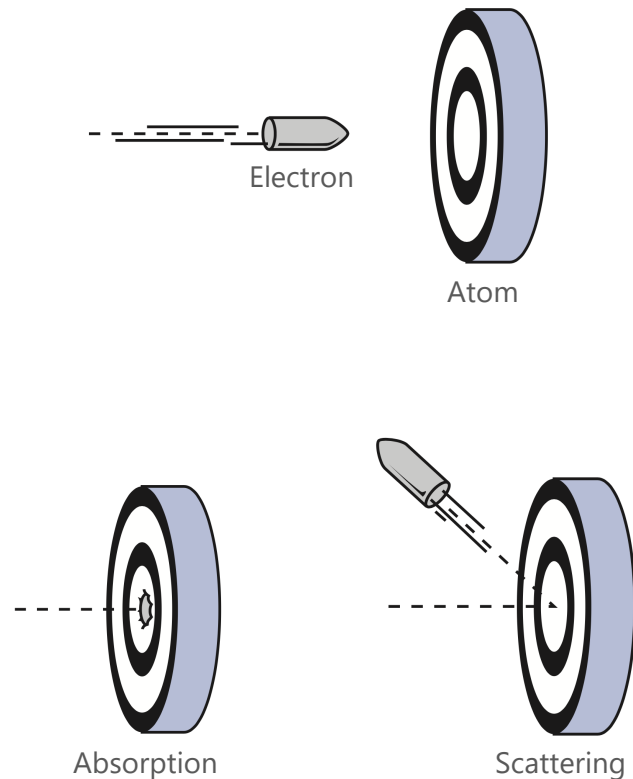
(n : number of nuclei per volume; M : molar mass; ρ : density; N_a : Avogadro's number)

✓ Composite (with i components)

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

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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❖ Mean free path:

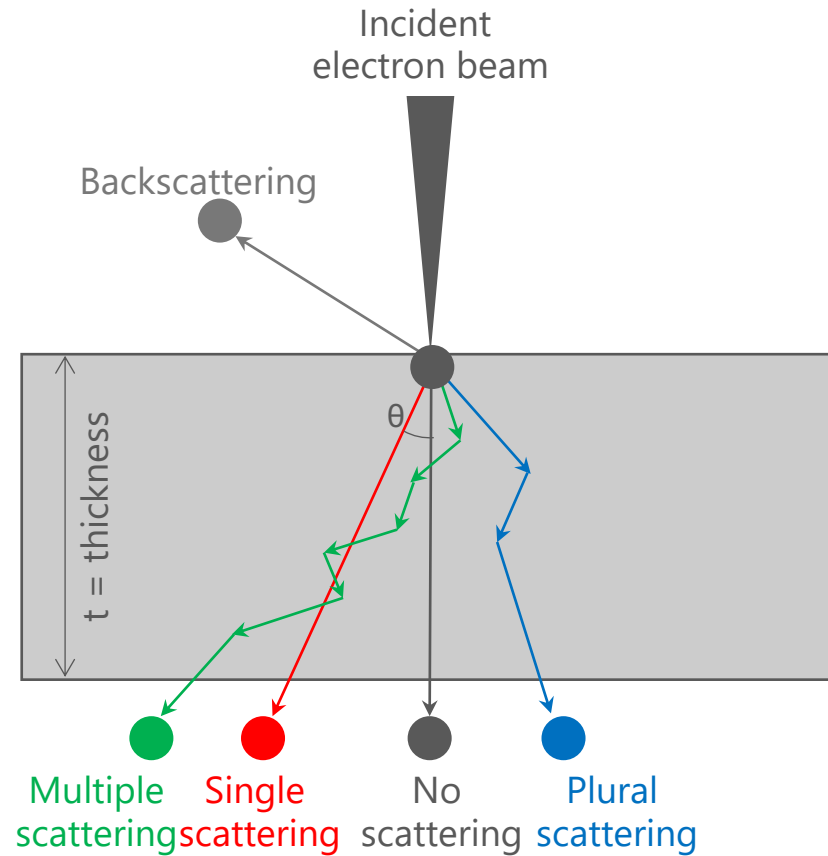
- How far on average an electron will travel before it interacts with an atom

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

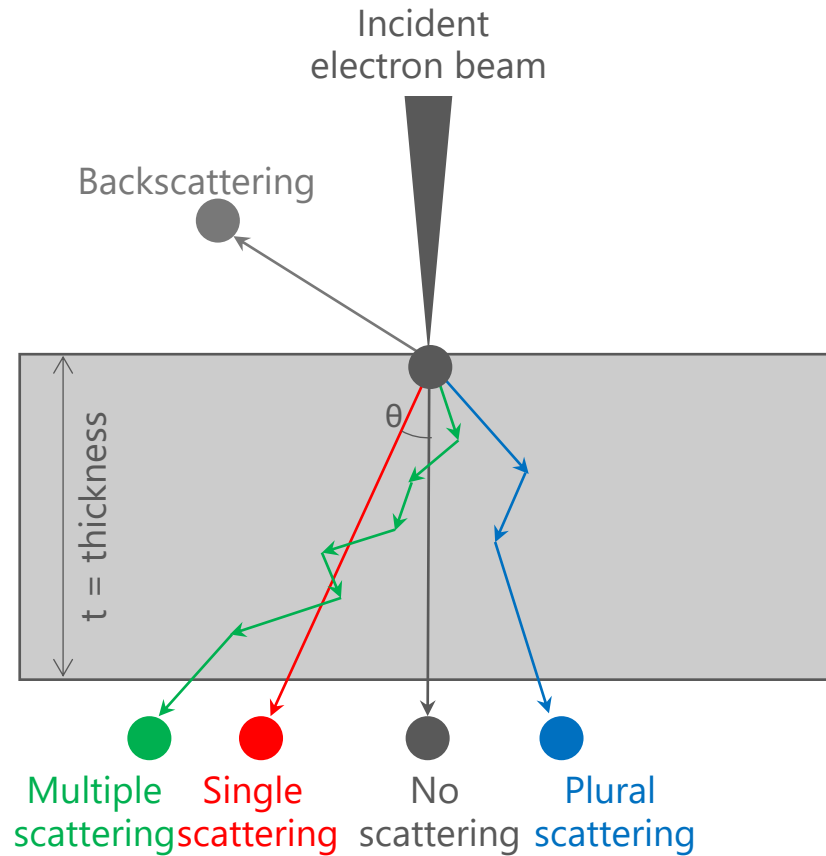
The cross section of interaction

❖ Single scattering:

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



The cross section of interaction



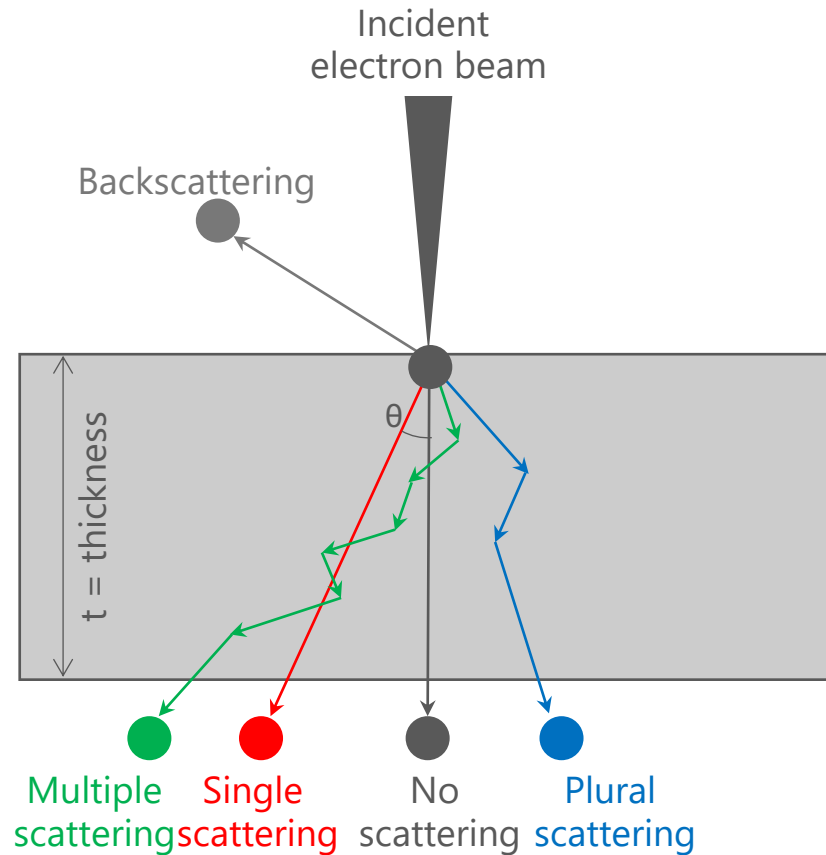
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❖ Multiple scattering:

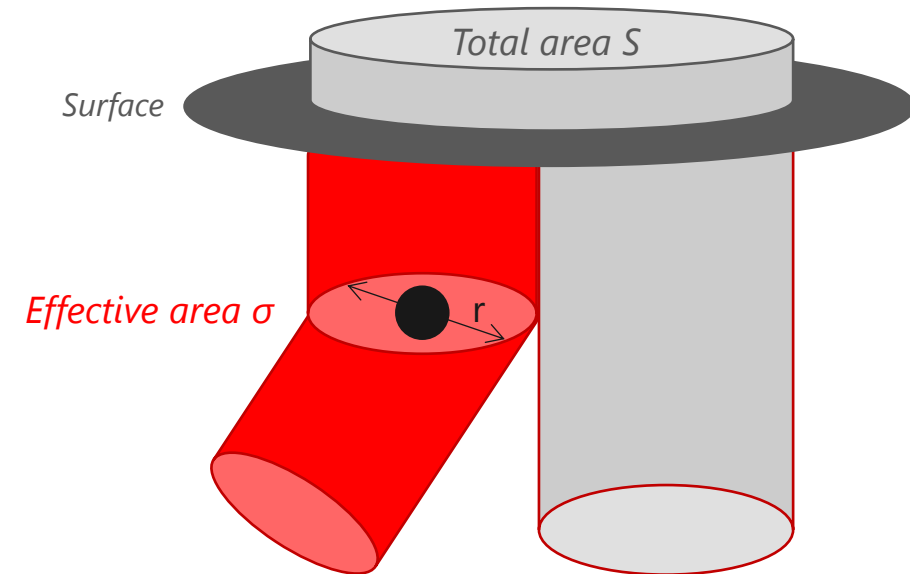
- Many scattering events.
- The path becomes complex, and the original direction and energy of the electron are heavily modified.
- Common in thick or dense samples, especially in SEM where BSE and SE often result from multiple scattering.
- Makes quantitative analysis more difficult, and signal interpretation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

The cross section of interaction

❖ Probability of scattering:

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

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



The cross section of interaction

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❖ Cross section:

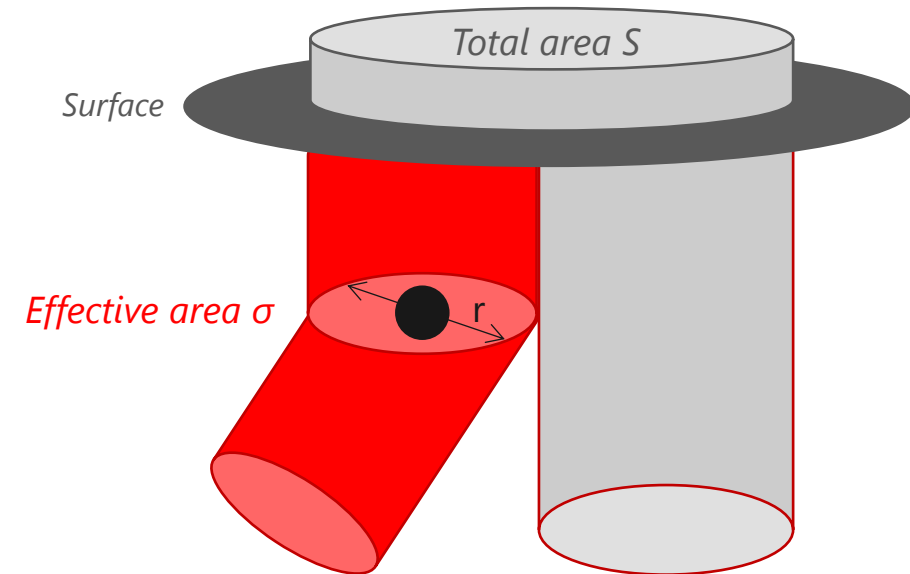
$$\sigma_{tot} = \sigma_{elastic} + \sigma_{inelastic}$$

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

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

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

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



The cross section of interaction

❖ Cross section for a specimen:

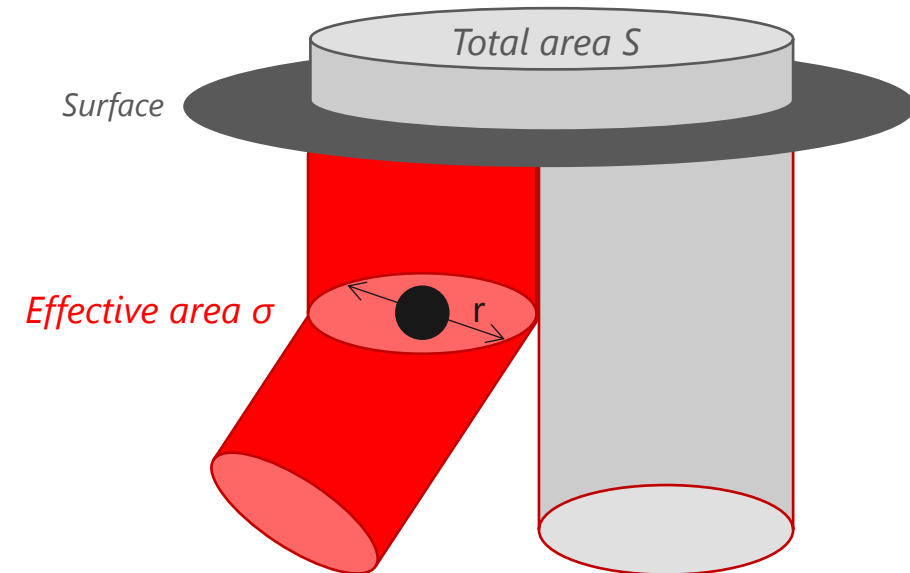
$$\Sigma = n\sigma = \frac{N_a \rho}{M} \sigma_{tot}$$

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

(t: thickness)

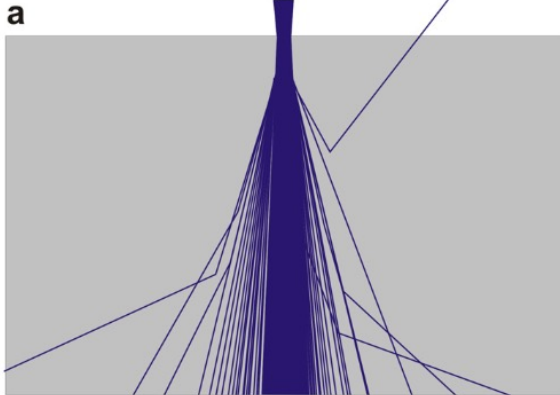
$$\Rightarrow P_{scat} \nearrow \text{ when } t \nearrow$$

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

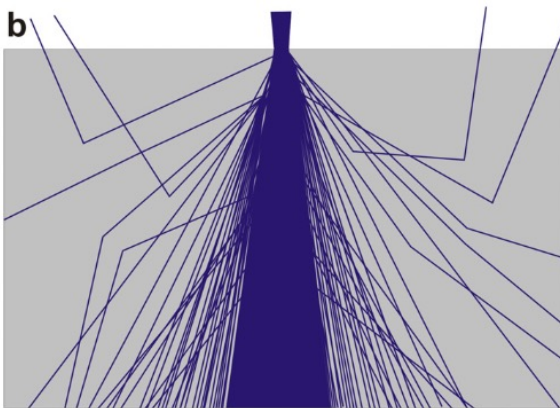


Interaction volume

Thin foil



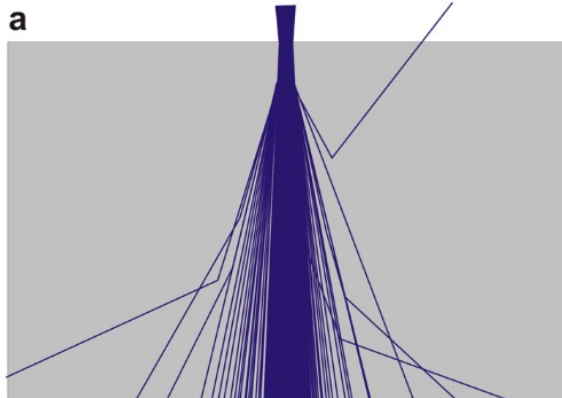
Low Z



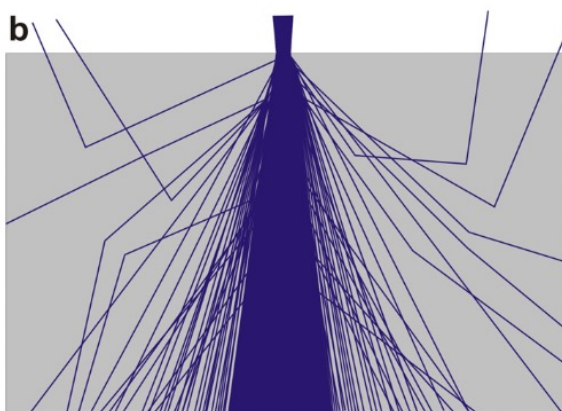
High Z

Interaction volume

Thin foil

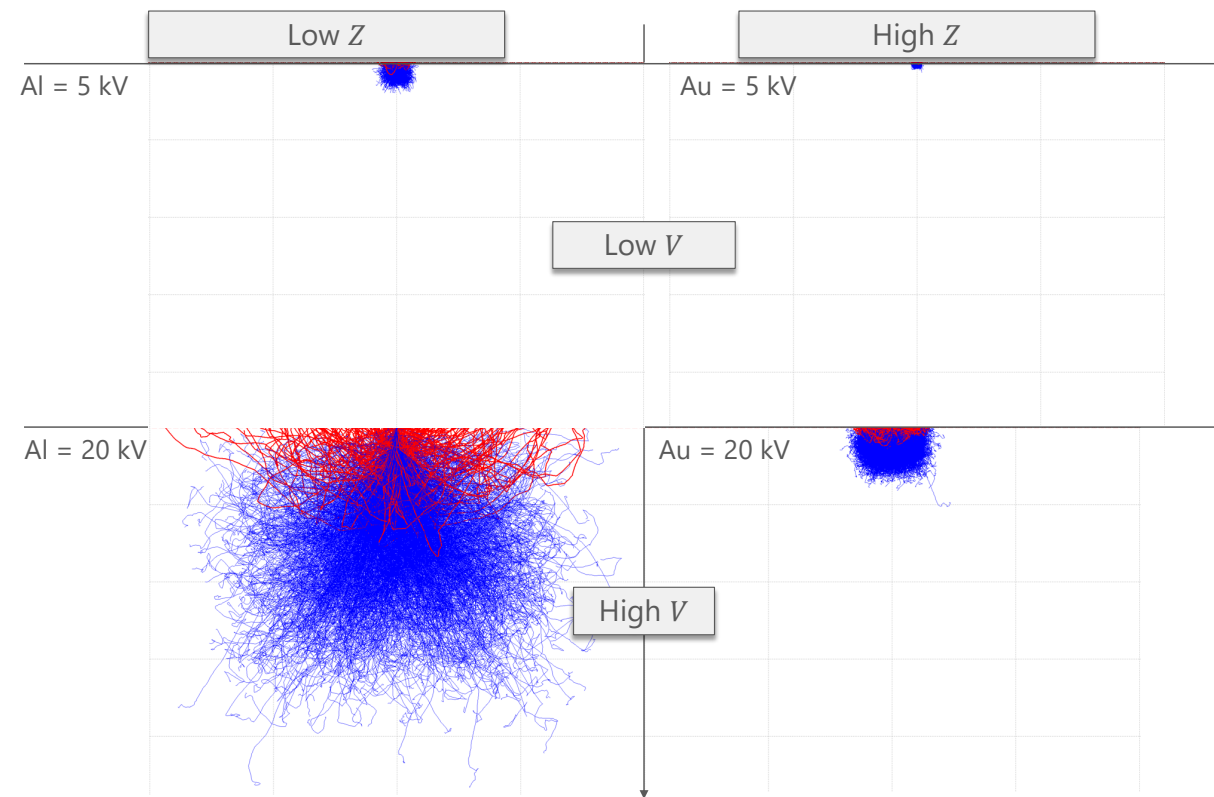


Low Z



High Z

Bulk specimen



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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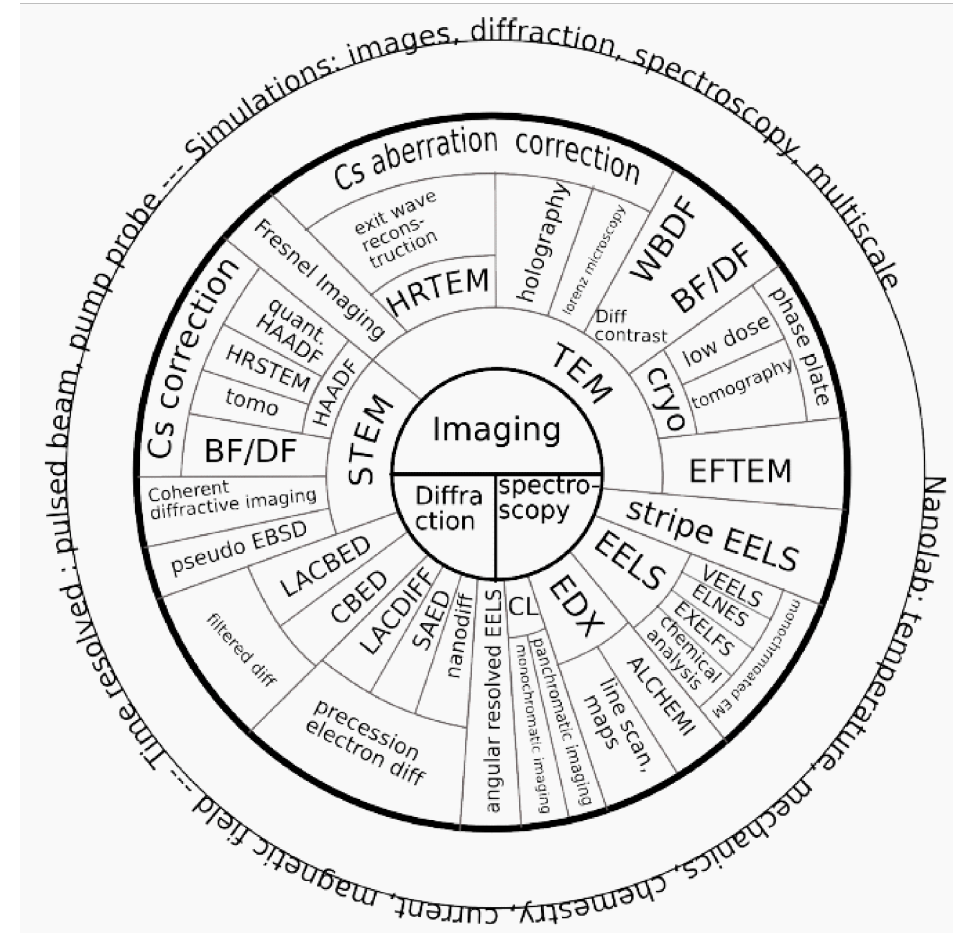
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Basics of TEM contrast

Probability of scattering event:

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

Basics of TEM contrast

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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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You shall not study
W by TEM...

High ρ

Low ρ

Thickness contrast:

$Q_T \nearrow$ when $t \nearrow$

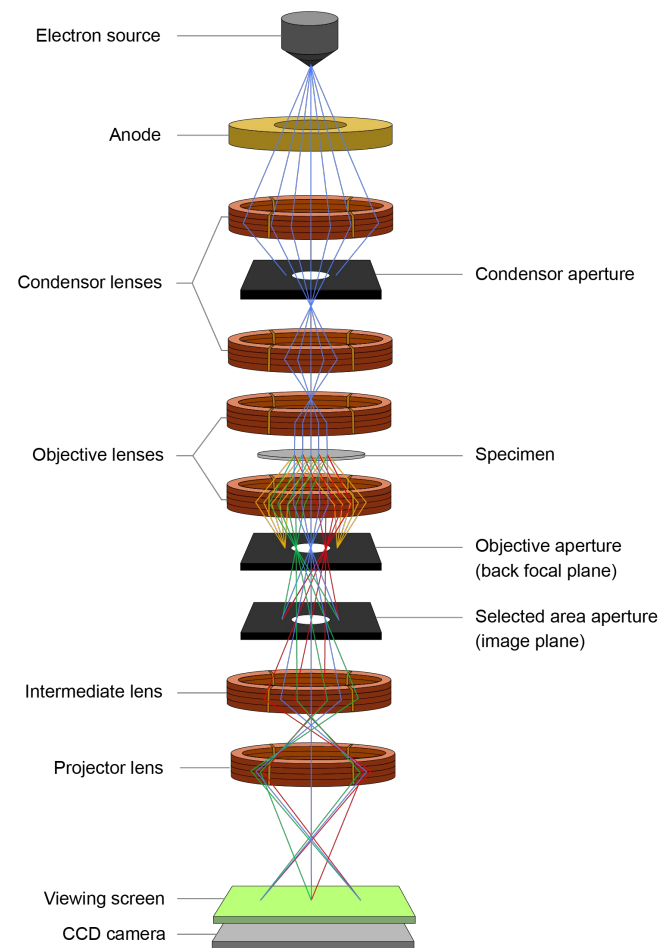
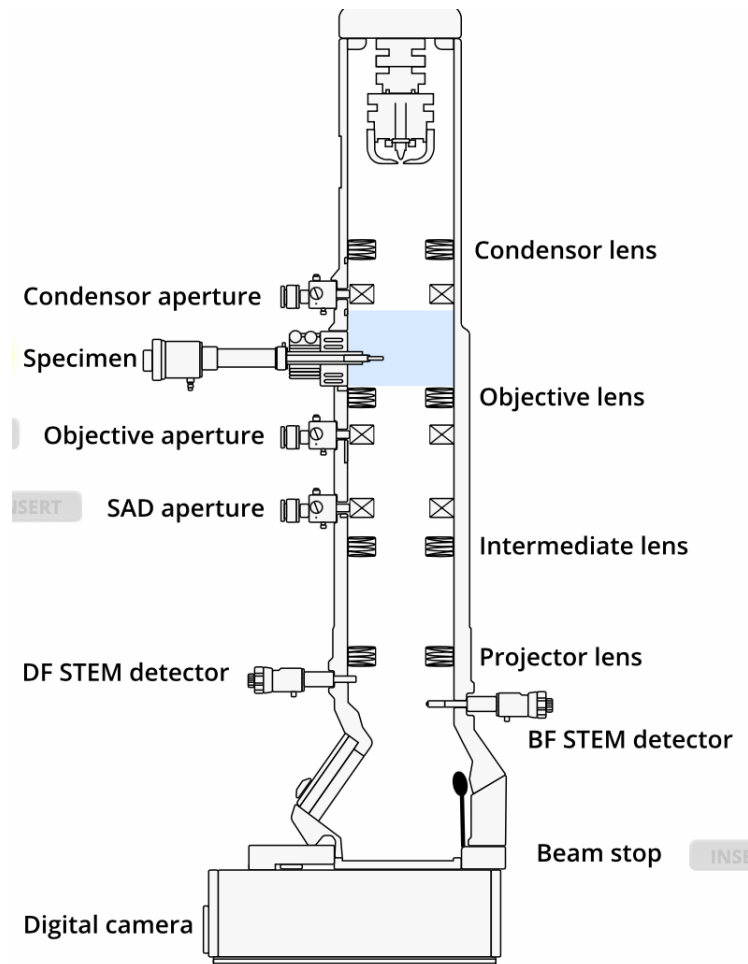
High t

Low t

You shall not study
thick TEM foils...

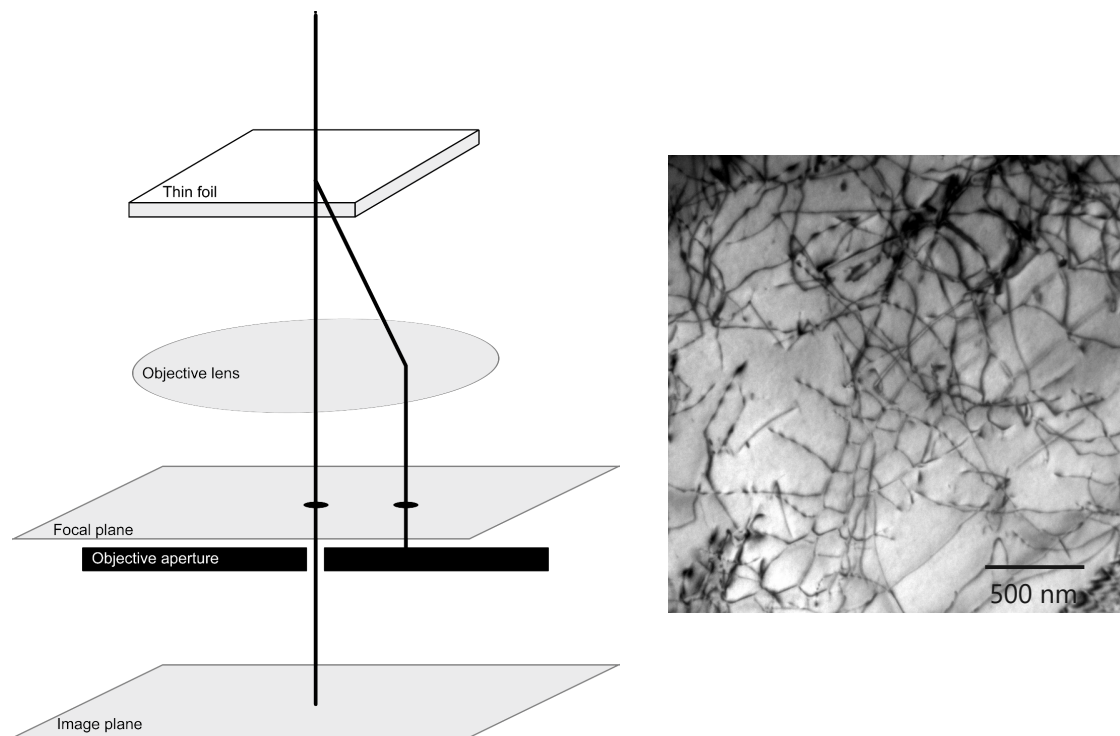
➡ It is the local scattering power that determines the contrast of TEM images

Basics of TEM



Basics of TEM

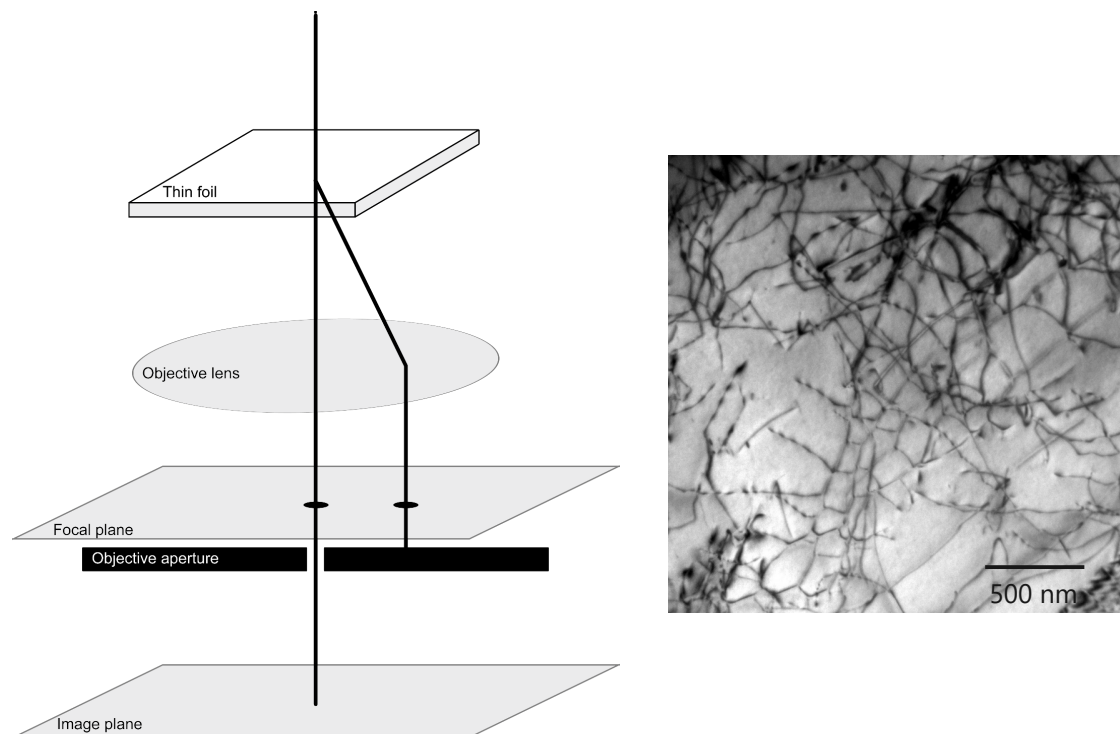
Bright field



➡ Dark contrasts on a bright background

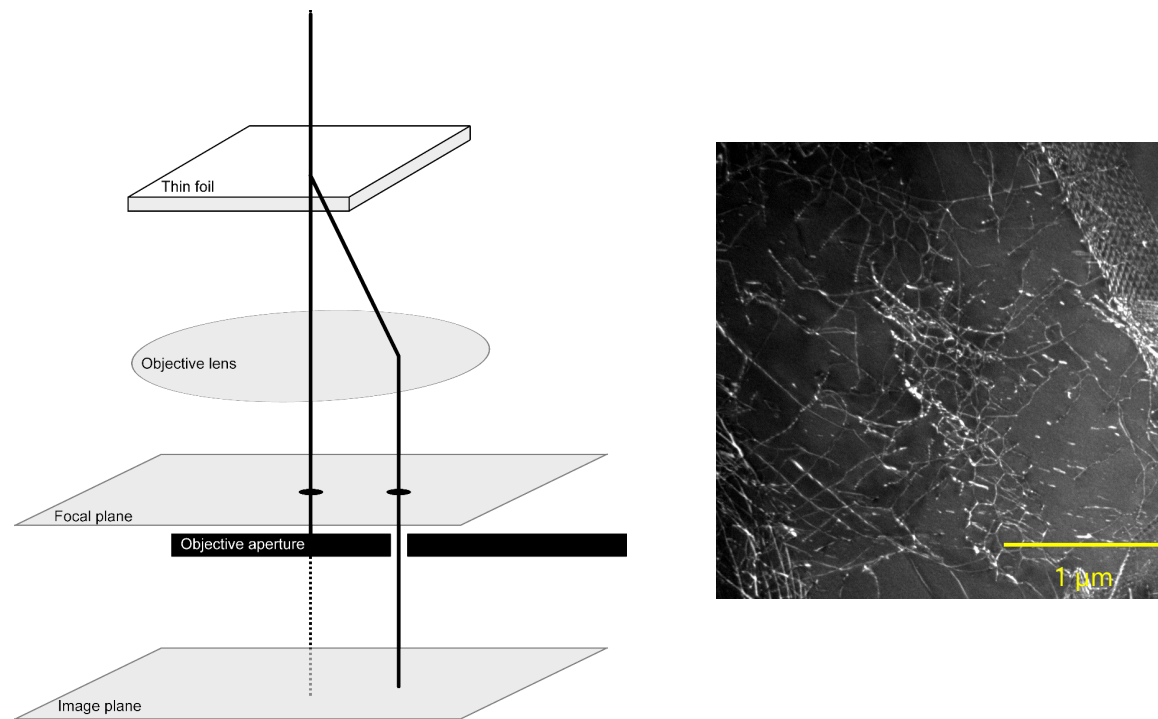
Basics of TEM

Bright field



↪ Dark contrasts on a bright background

Dark field



↪ Bright contrasts on a dark background

Formation of dislocation loops under irradiation

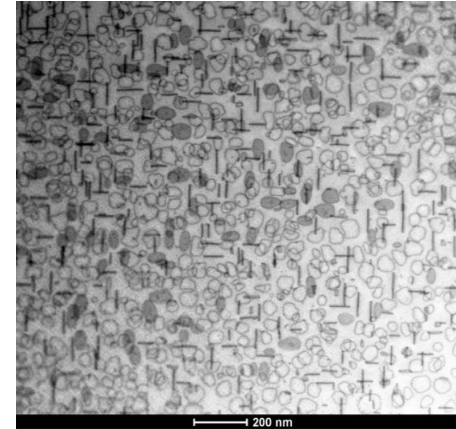
113

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Formation of dislocation loops under irradiation

114

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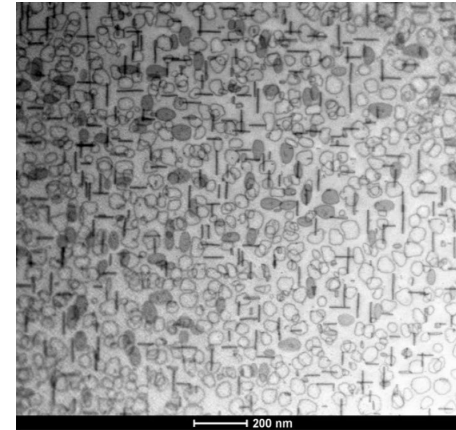


Dislocation loop microstructure in ion-irradiated CrFeMnNi concentrated solid-solution alloy (15Cr-46Fe-17Mn-22Ni at.%, STEM-BF, [001] zone axis, 6 MeV Ni²⁺, 823 K, 0.2 dpa).

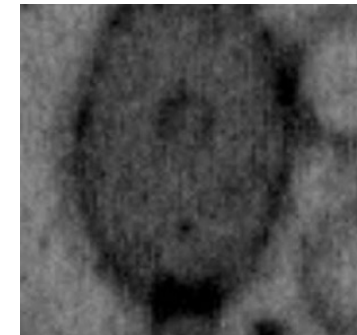
Formation of dislocation loops under irradiation

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- ❖ Two kinds of loops:
 - Faulted loop: the disc is bounded by a stacking fault (a local error in the stacking sequence of atomic planes); this loop cannot move (sessile).



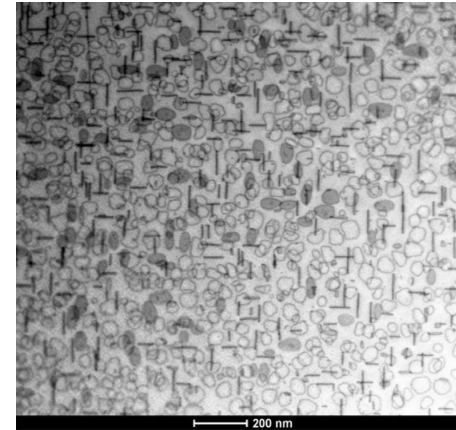
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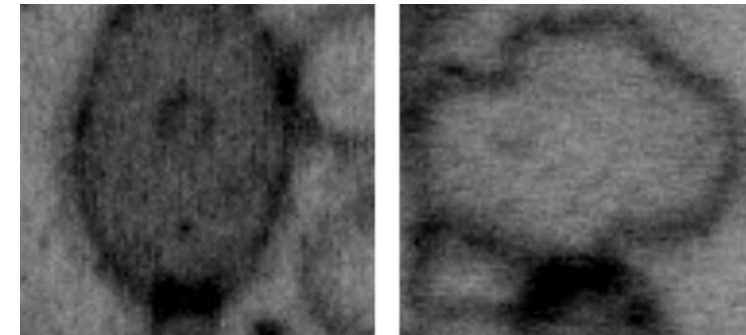
Formation of dislocation loops under irradiation

116

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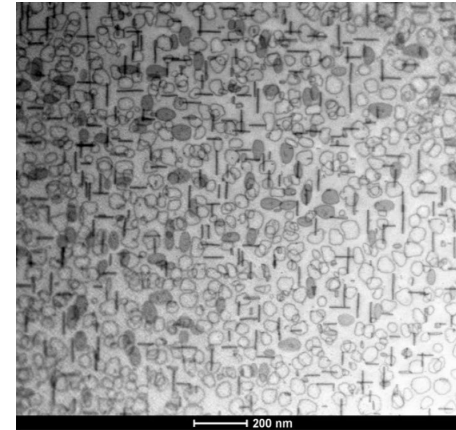
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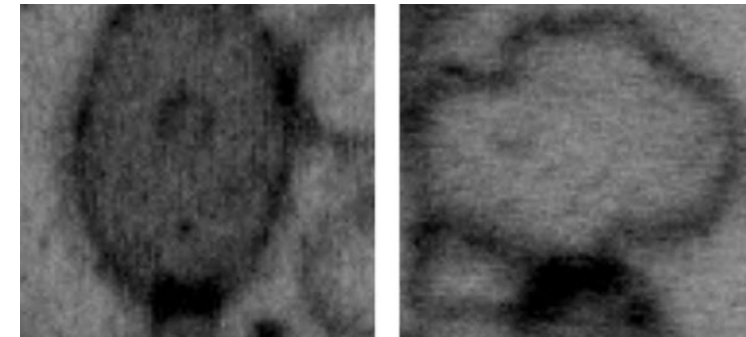
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→ TEM contrast remains the only technique resolving individual dislocation loops down to a few nanometers and identifying their nature.

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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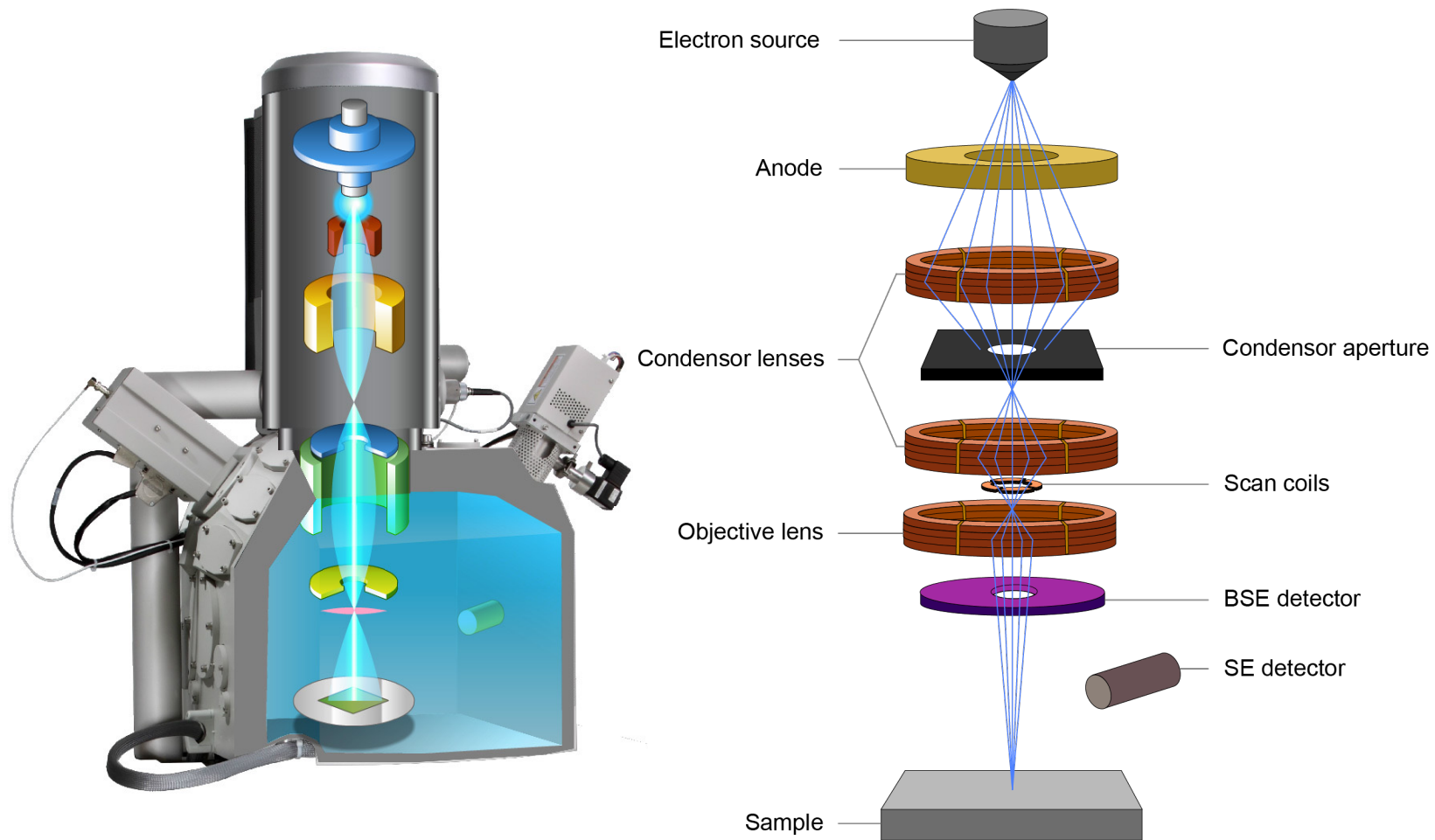


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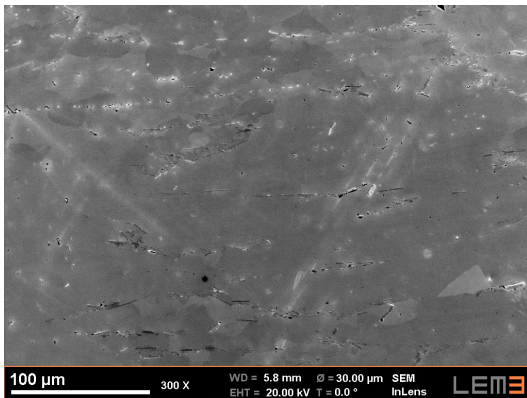
↳ The two microscopes are complementary.

Basics of SEM



SEM imaging

Secondary electrons (SE)



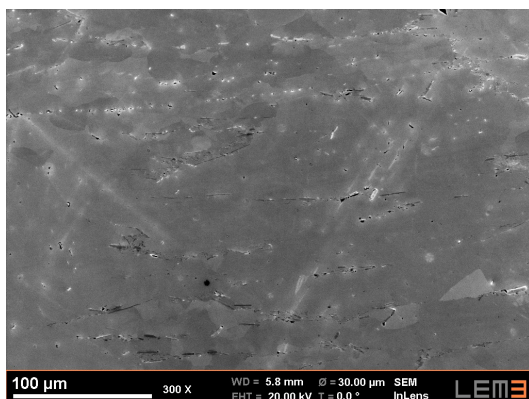
- ❖ Inelastic scattering between incident electrons and outer-shell electrons of sample atoms $\Rightarrow$ emission of SE
- ❖ Inelastic scattering involving inner-shell electrons, followed by electronic transitions $\rightarrow$ emission of characteristic X-rays

$\Rightarrow$ EDS

↪ Topographic contrast

SEM imaging

Secondary electrons (SE)



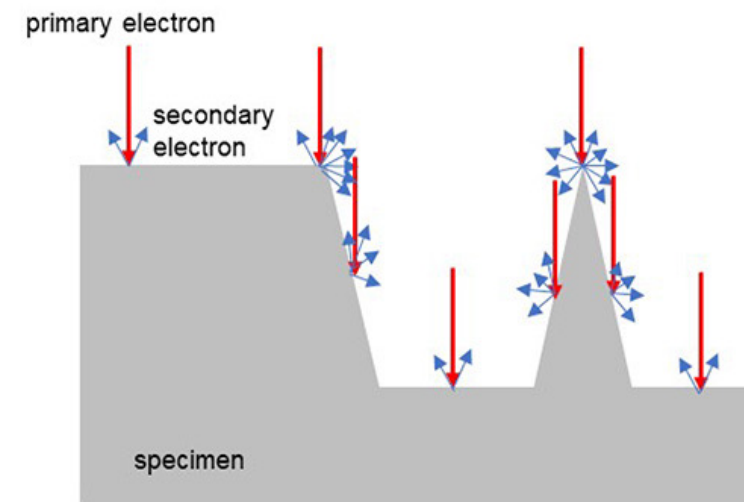
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 Topographic contrast

The edge effect

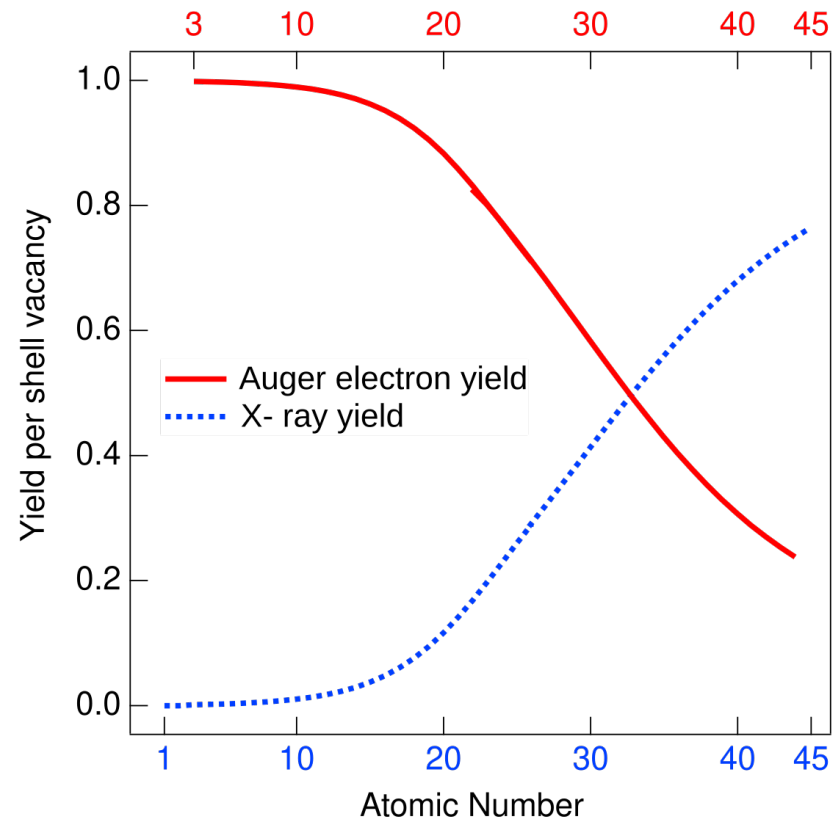
- ❖ Areas at edges and corners appear very bright in SE images due to increased SE emission.



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

Energy Dispersive X-ray Spectroscopy (EDS/EDX)

122

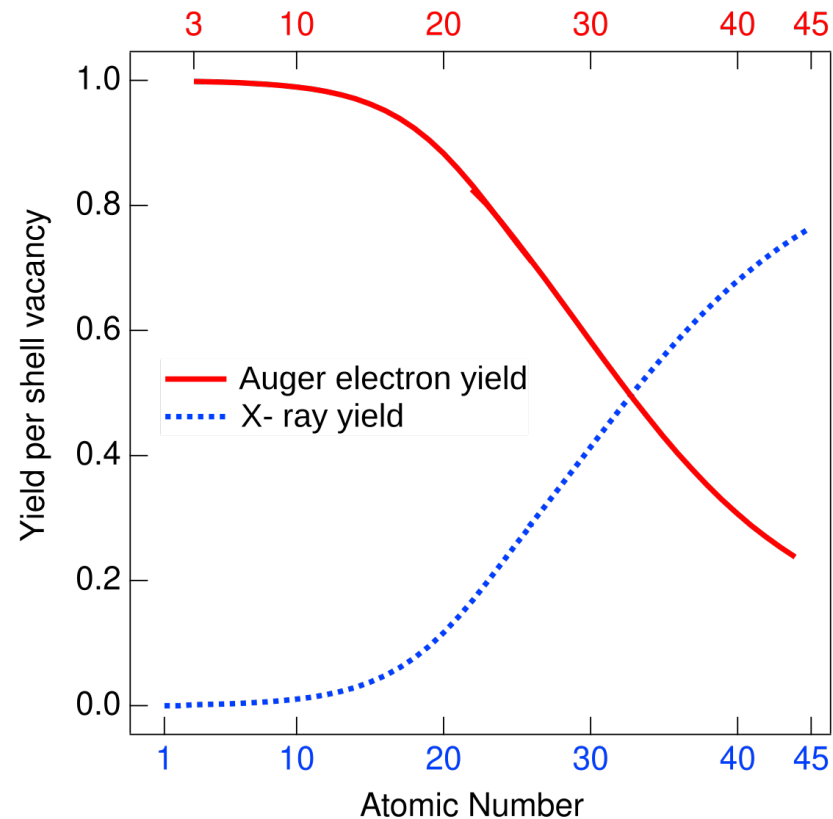


❖ Inner-shell ionization:

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

Energy Dispersive X-ray Spectroscopy (EDS/EDX)

123



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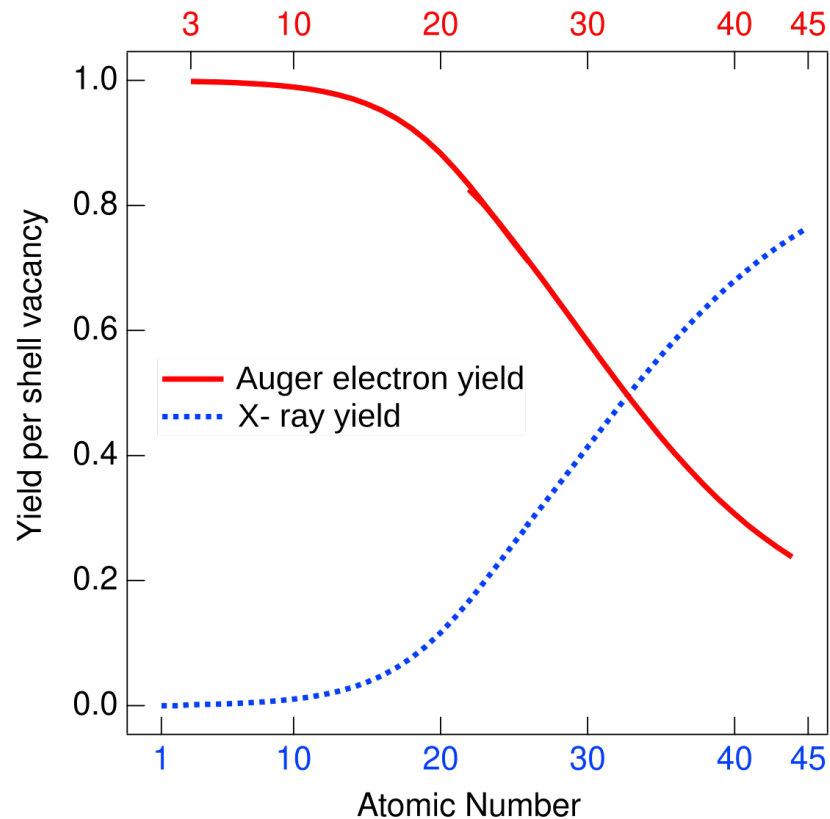
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❖ Electronic transition:

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

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124



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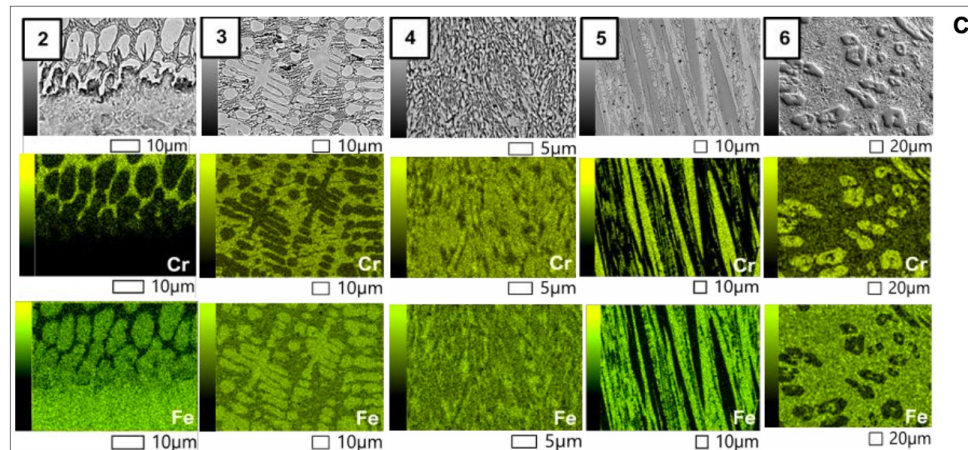
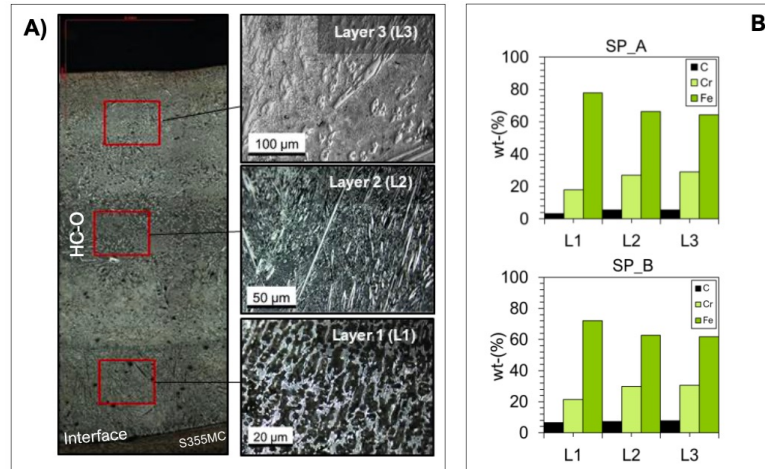
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❖ X-ray emission:

- The emitted characteristic X-ray has an energy equal to the difference between the two shells. This energy is unique to each element.

Energy Dispersive X-ray Spectroscopy (EDS/EDX)

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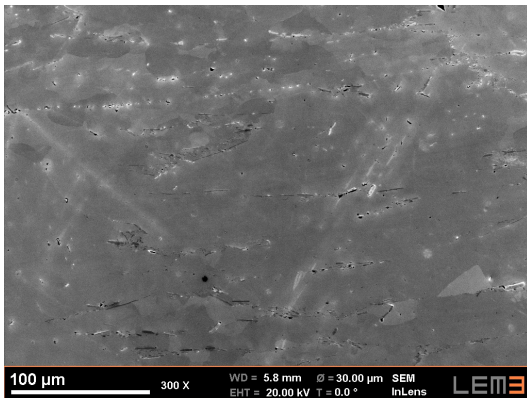
- The emitted characteristic X-ray has an energy equal to the difference between the two shells. This energy is unique to each element.

❖ 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)

SEM imaging

Secondary electrons (SE)

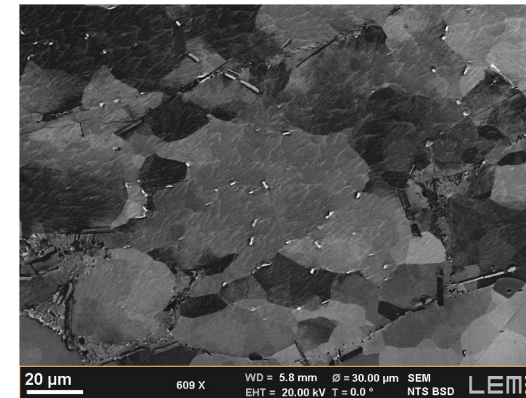


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$\Rightarrow$ EDS

↪ Topographic contrast

BackScattered Electrons (BSE)



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

$\Rightarrow$ EBSD, ECCI

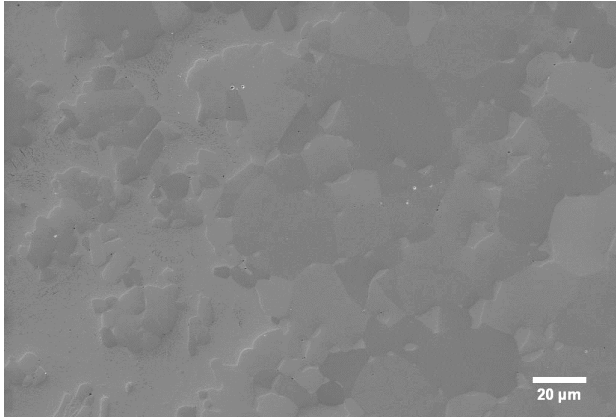
↪ Topographic contrast
↪ Chemistry contrast
↪ Orientation contrast

↪ The two imaging modes are complementary.

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

127

**Non irradiated Ti_2AlC
(BSE imaging)**



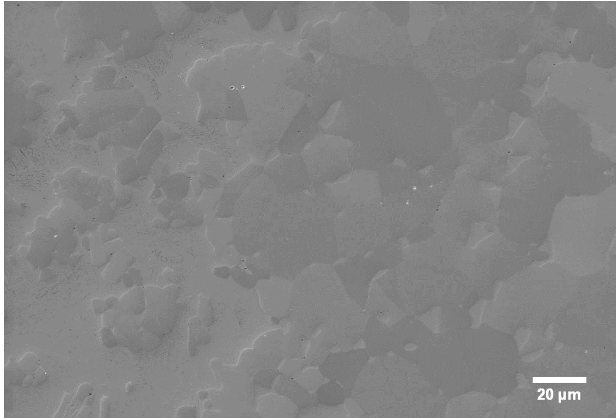
❖ Non-irradiated Ti_2AlC

- The surface appears relatively clean and smooth, with well-defined grains visible.
- Grain boundaries can be clearly distinguished, suggesting good crystalline integrity and limited damage.
- Few visible defects (no major cracks or voids), characteristic of a non-irradiated or less damaged state.

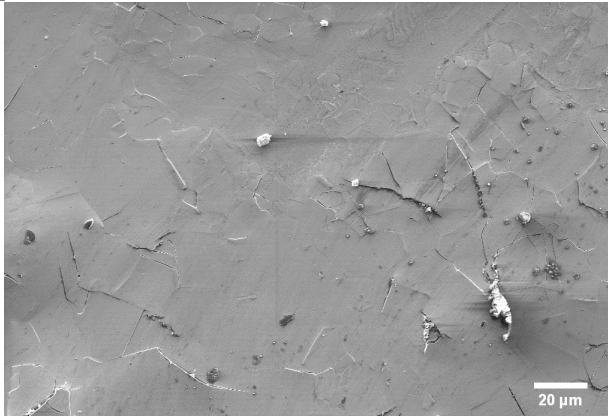
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128

**Non irradiated Ti_2AlC
(BSE imaging)**



**Irradiated Ti_2AlC with Ni^{3+}
 $5 \cdot 10^{15} \text{ cm}^{-2}$ @RT
(BSE imaging)**



❖ Non-irradiated Ti_2AlC

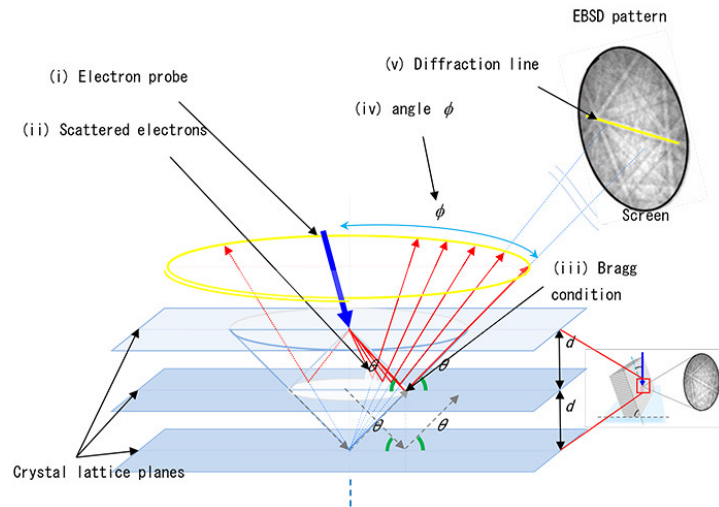
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❖ Irradiated Ti_2AlC

- The surface is noticeably more damaged and rough compared to the non-irradiated Ti_2AlC .
- Presence of microcracks and fissures, many of them following grain boundaries.
- Evidence of debris or redeposited material (bright particles) on the surface, possibly due to irradiation-induced sputtering or contamination.
- The network of cracks suggests stress accumulation and structural degradation under irradiation.
- No clear signs of surface swelling (blistering or bubble formation) are observed at this scale; the dominant effect appears to be cracking and surface roughening.

Basics of the Electron BackScattered Diffraction (EBSD)

129

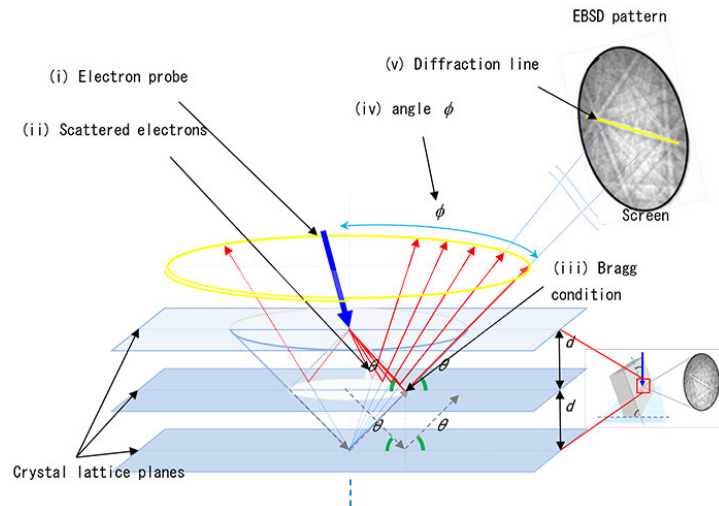


❖ Generation of Kikuchi patterns:

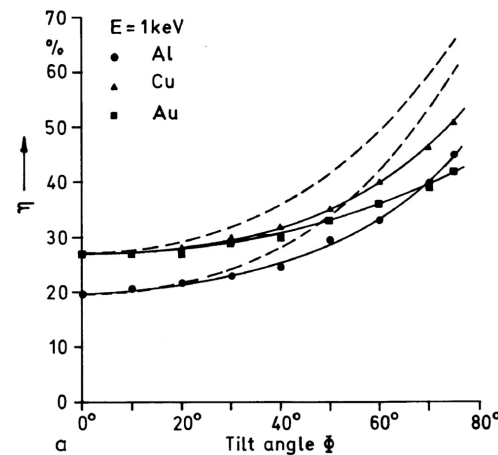
- The incident electron beam interacts with the crystal lattice, generating BSE through inelastic and elastic scattering.
- Some of these BSE undergo coherent elastic diffraction by the crystal planes, fulfilling the Bragg condition.
- This diffraction produces Kikuchi bands, which appear as pairs of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

Basics of the Electron BackScattered Diffraction (EBSD)

130

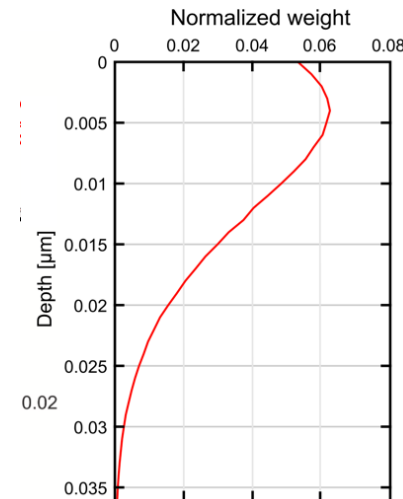


↗ The BSE yield increases with increasing tilt angle



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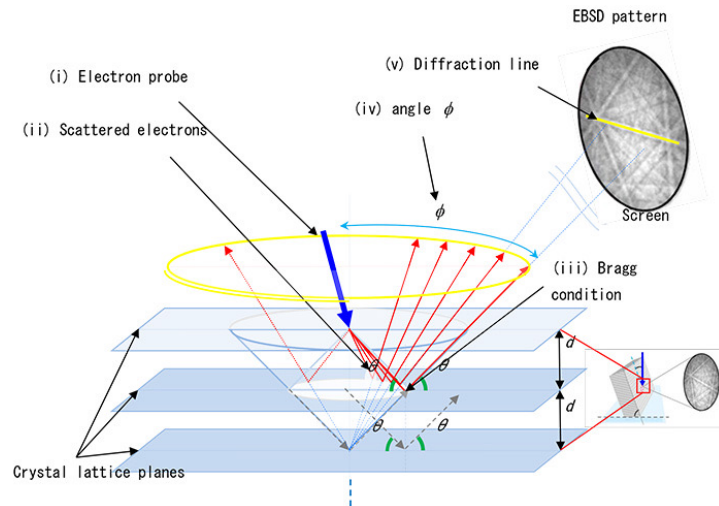
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↗ The probing depth in GaN at 15 keV is ~35 nm below the surface.

Basics of the Electron BackScattered Diffraction (EBSD)

131

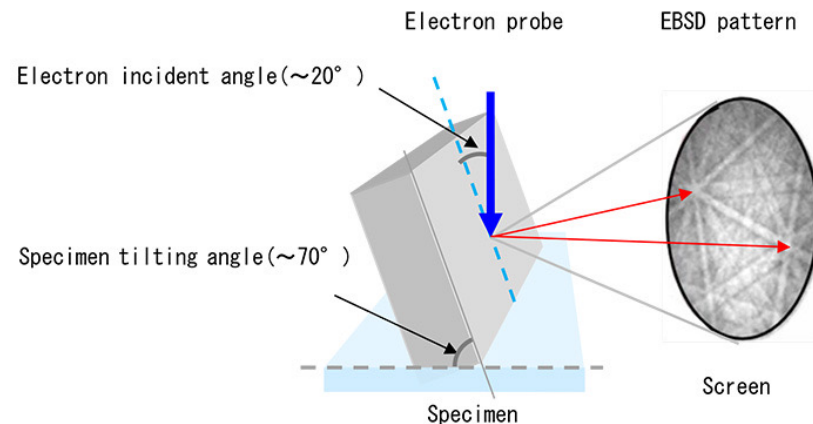


❖ Generation of Kikuchi patterns:

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

❖ Basics of the EBSD:

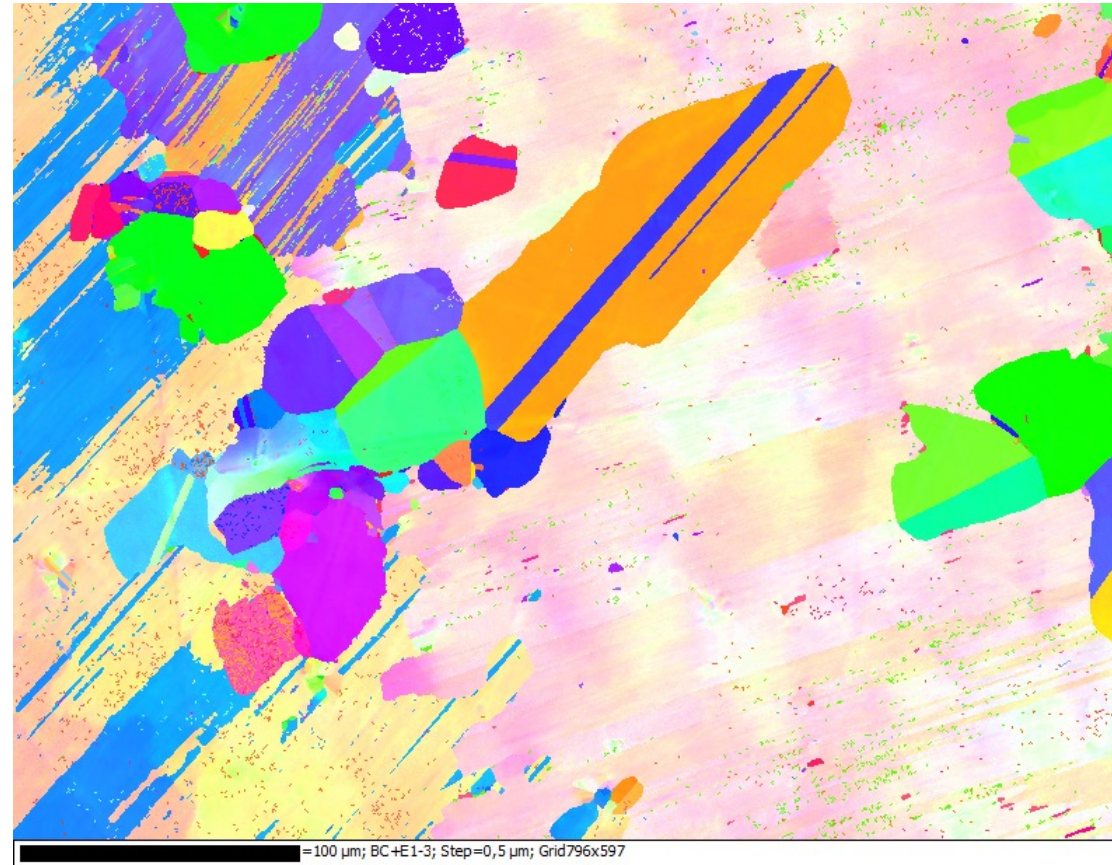
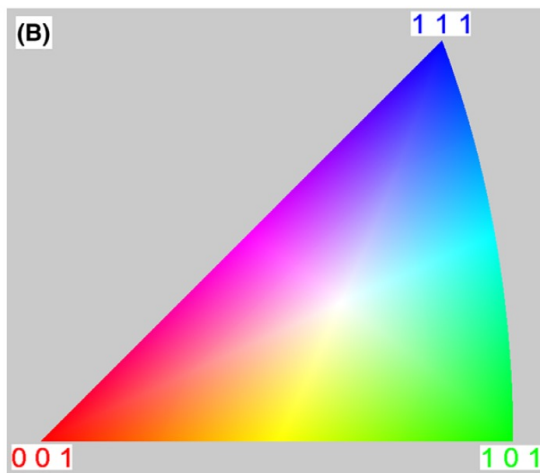
- EBSD involves directing a focused electron beam onto a tilted crystalline specimen
- The BSE form Kikuchi patterns, which are captured by a phosphor screen and recorded using a camera.
- These patterns are analyzed to extract information about the material's grain structure, orientation, and phase.



Example of extinction condition

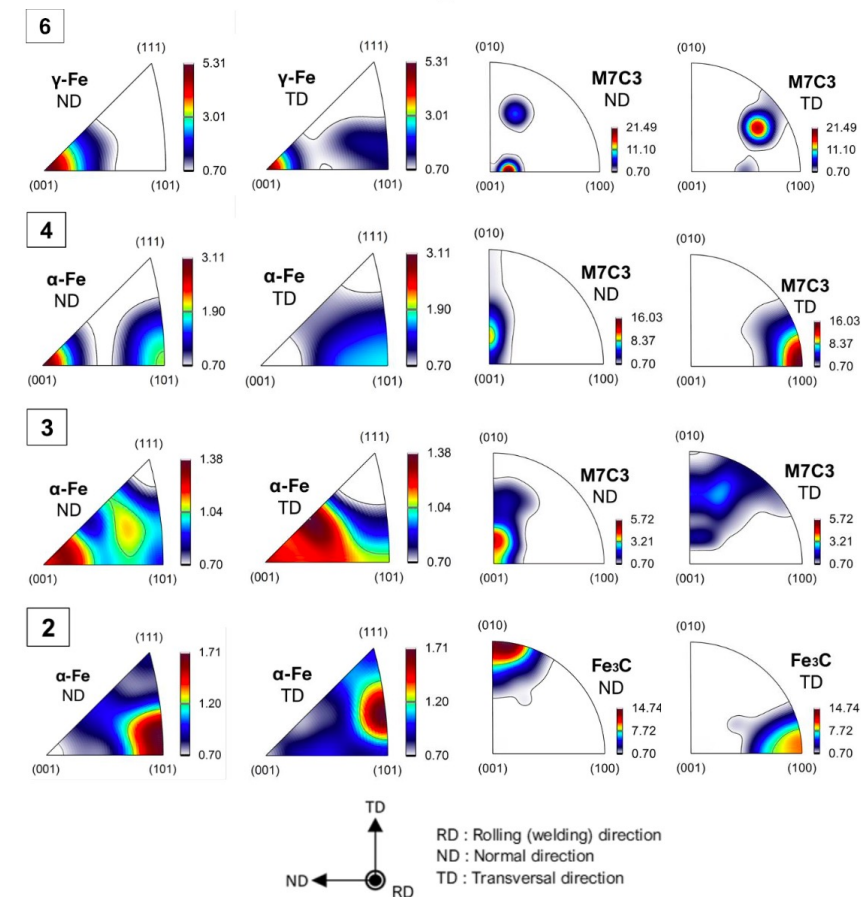
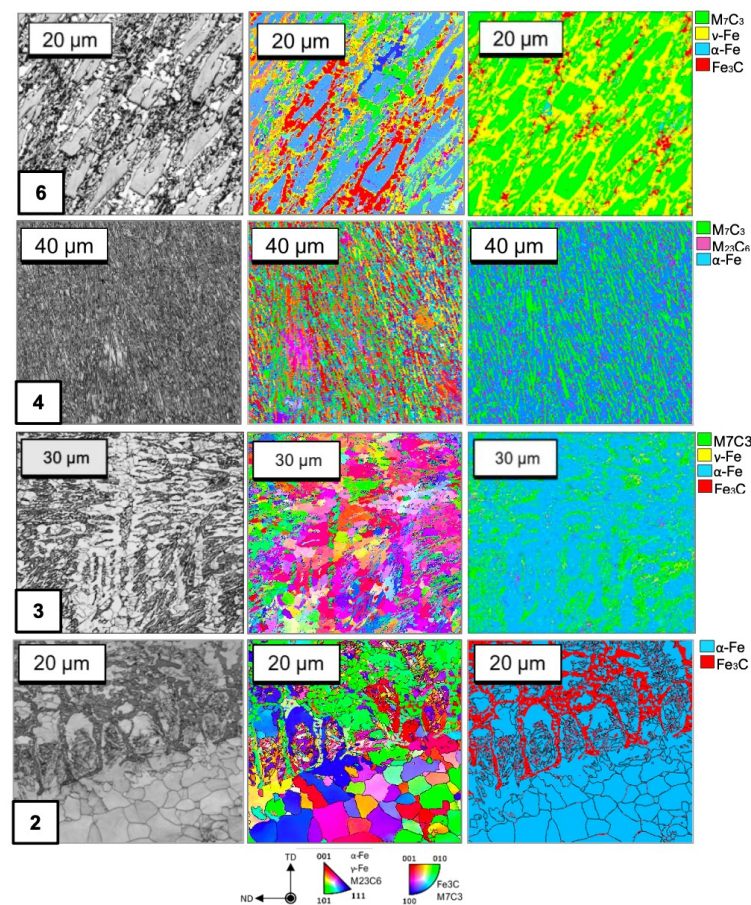
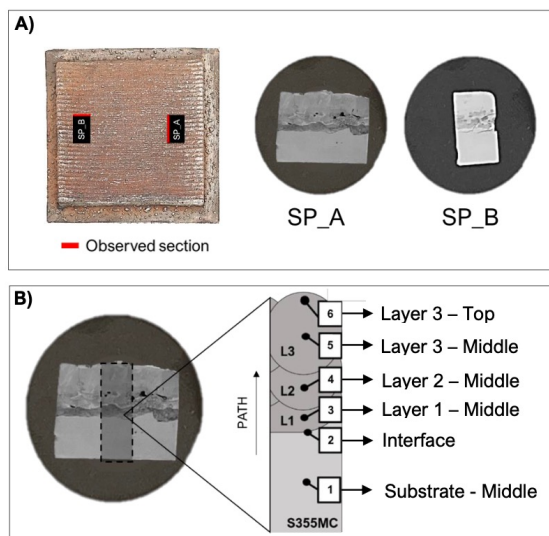
❖ Capabilities of EBSD:

- Orientation measurements
- Local misorientation measurements
- Grain identification
- Phase identification
- Statistics on the microstructure



Texture measurements by EBSD

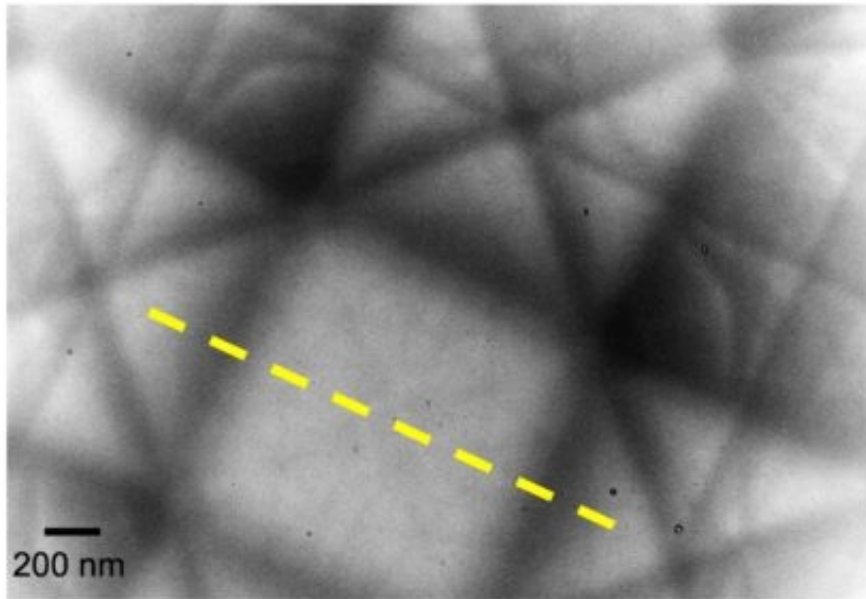
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Origin of the Electron Channeling Pattern (ECP)

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



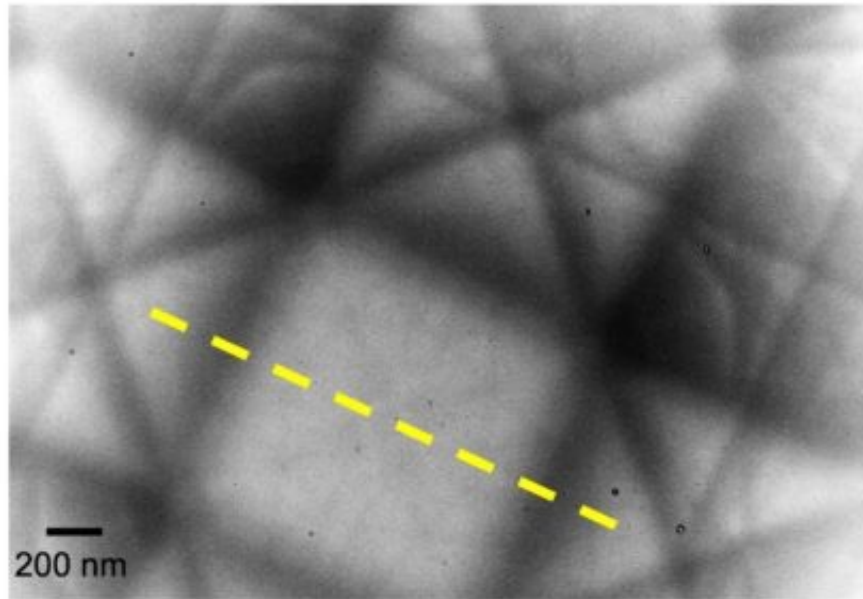
Electron Channeling Pattern "ECP"
obtained from Si single crystal.

Origin of the Electron Channeling Pattern (ECP)

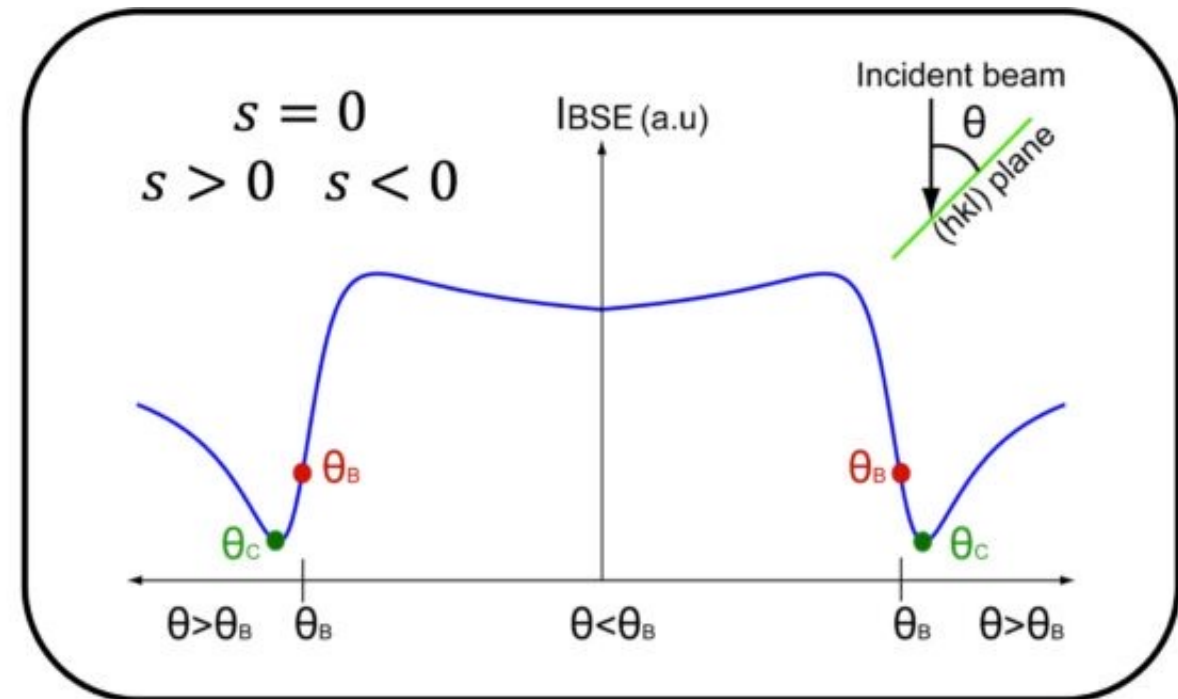
135

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

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

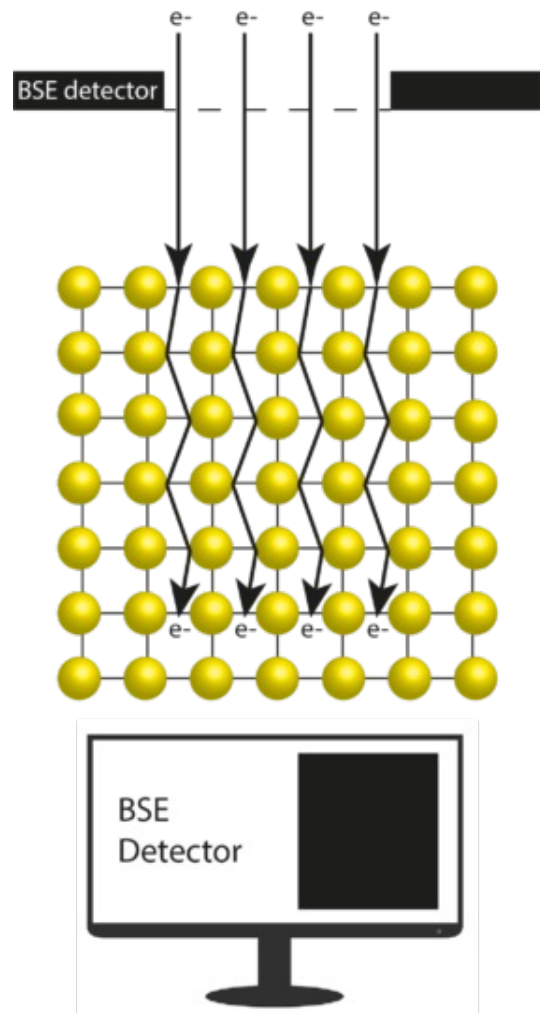


Electron Channeling Pattern "ECP" obtained from Si single crystal.



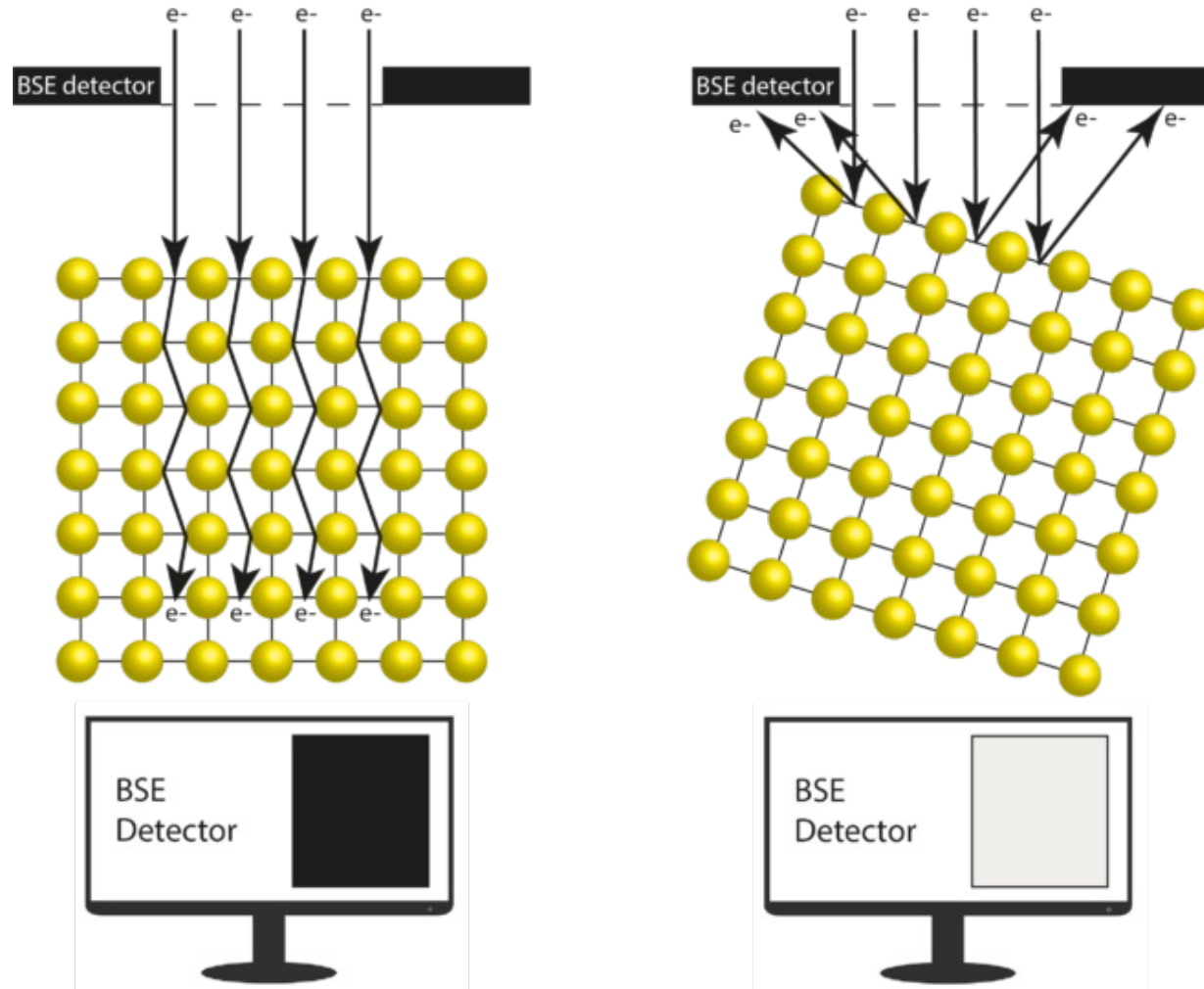
Channeling condition $\neq$ Bragg condition

What is the Electron Channeling Contrast Imaging? 136



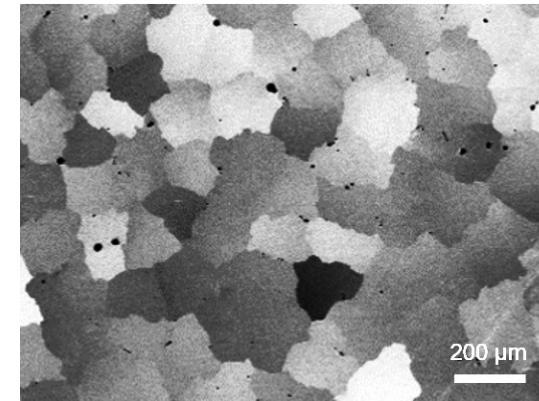
What is the Electron Channeling Contrast Imaging?

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This is not a polycrystal!

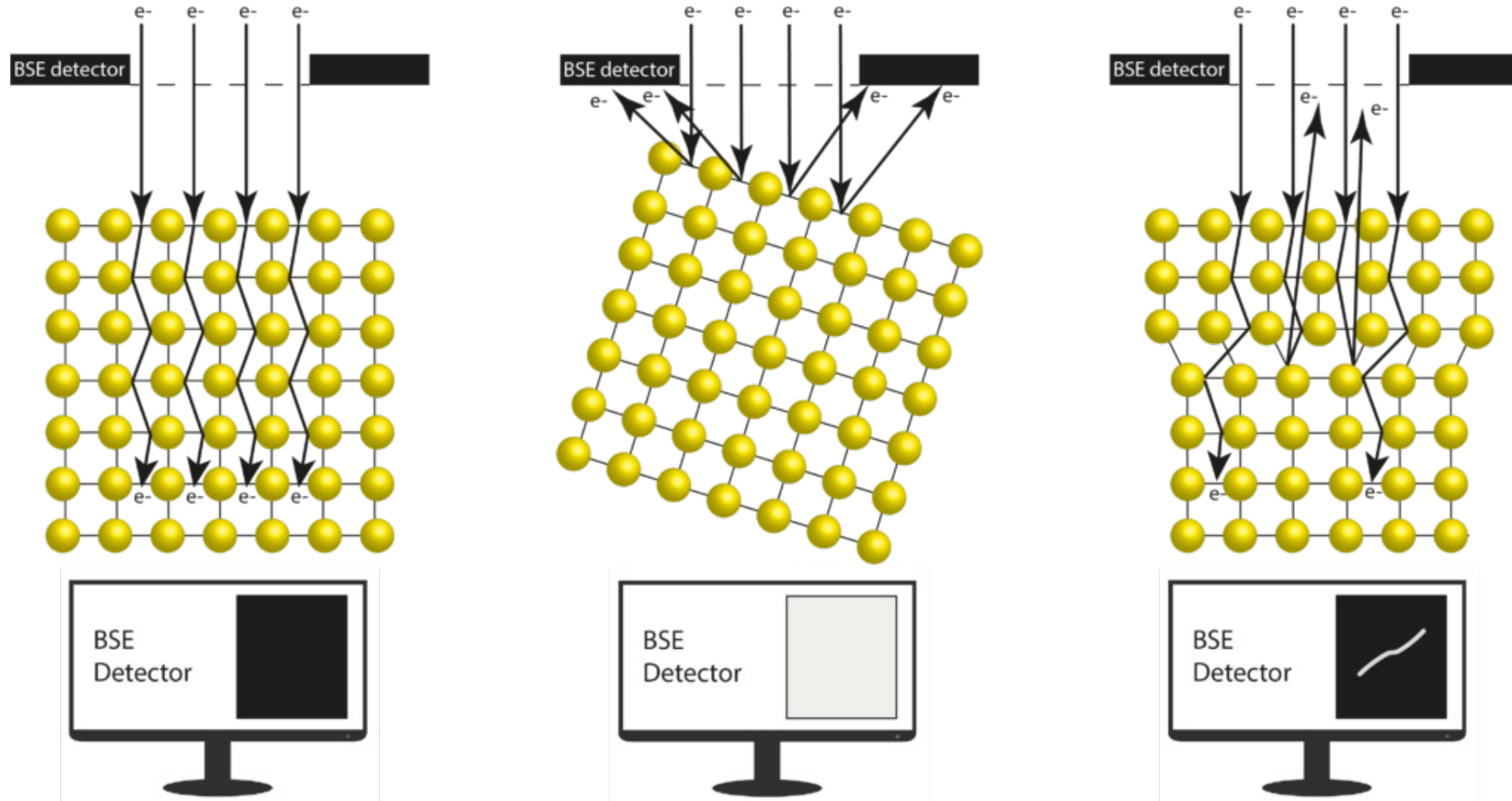
F. Habiyaemye et al., *MSEA* (2021)



Single crystal of CrCoNi with dendrites
(mean misorientation: $< 1^\circ$)

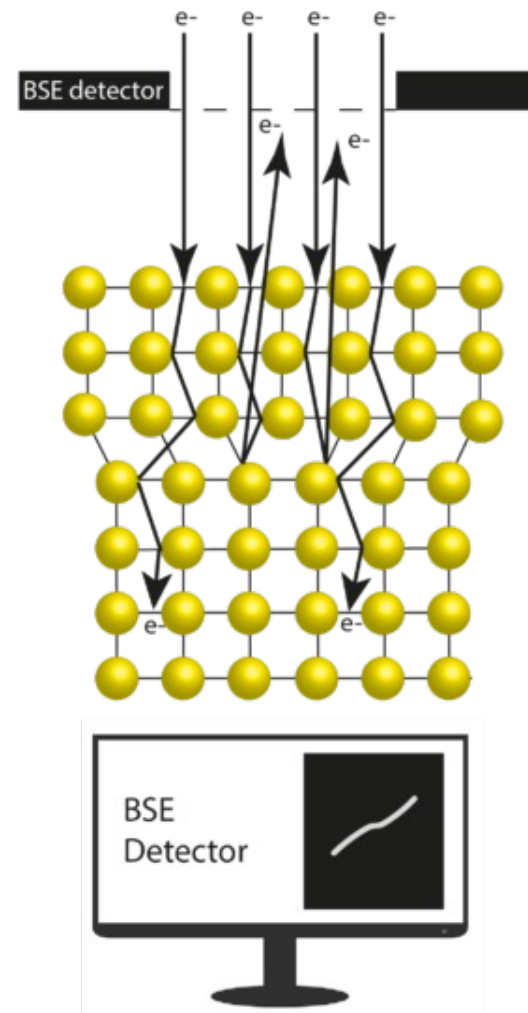
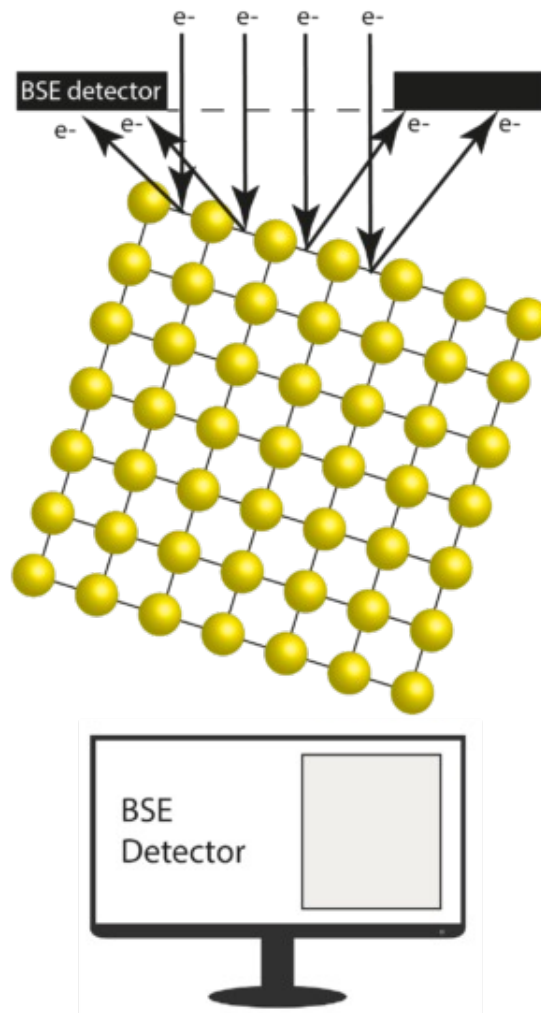
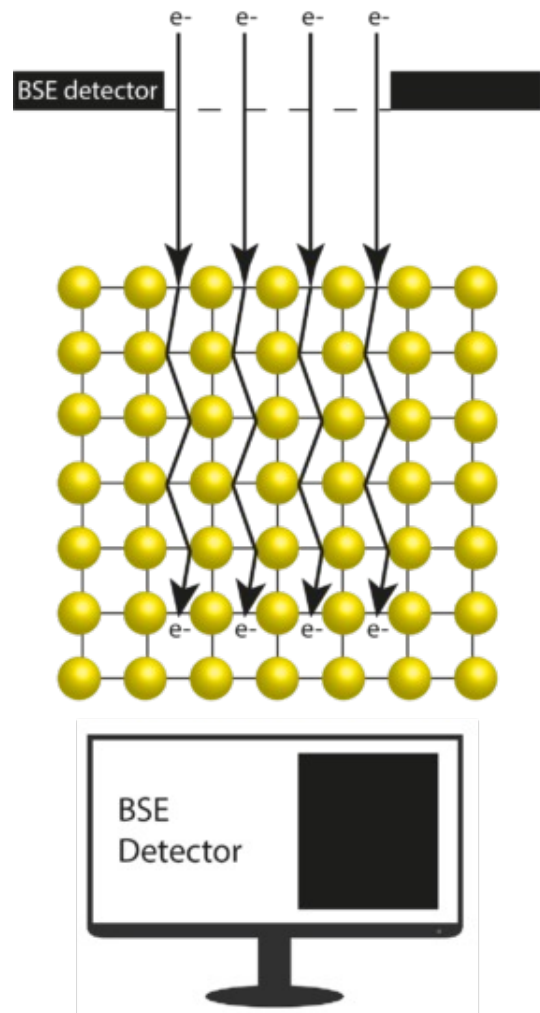
What is the Electron Channeling Contrast Imaging?

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What is the Electron Channeling Contrast Imaging?

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

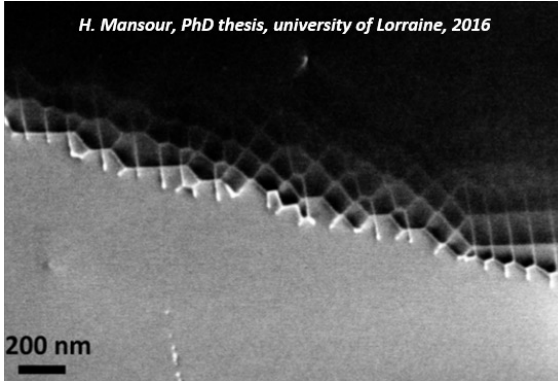
$$\text{Stacking fault} \Rightarrow \mathbf{g} \cdot \mathbf{R} = 0$$



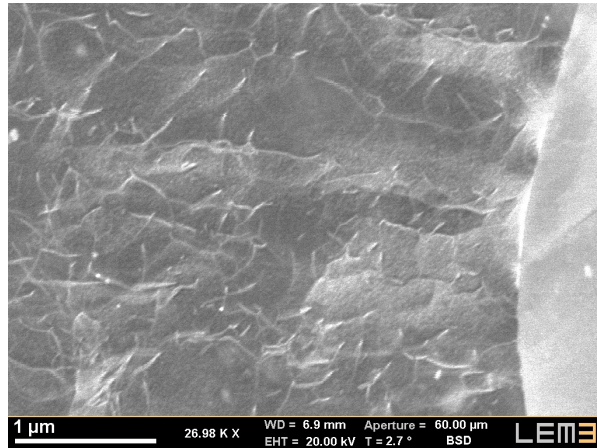
Nature of the crystalline defects

Detailed characterization of crystalline defects

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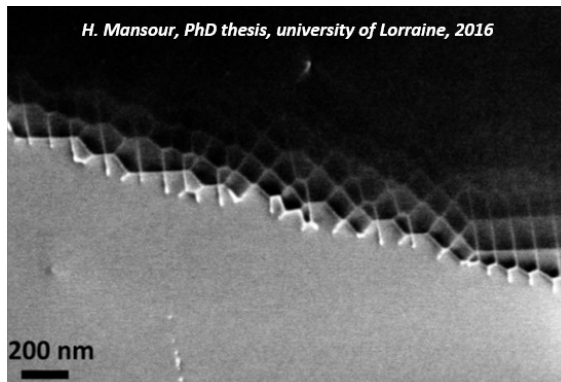


Ceramics (UO_2)

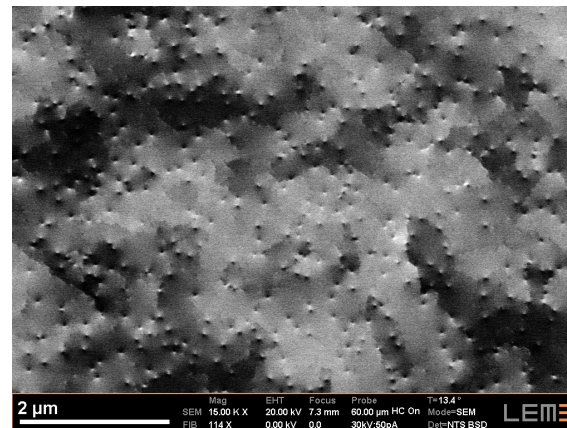


Ceramics (MAX phase)

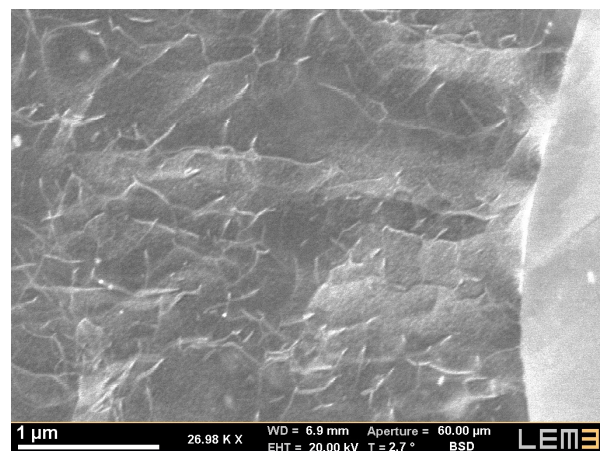
Detailed characterization of crystalline defects



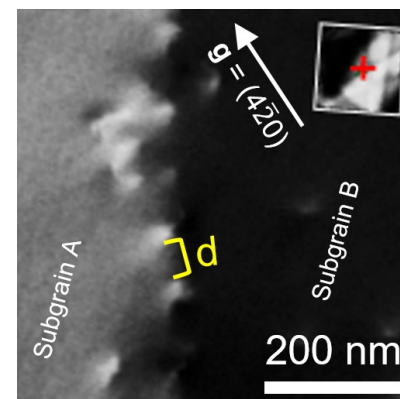
Ceramics (UO_2)



Semi-conductors (GaN)



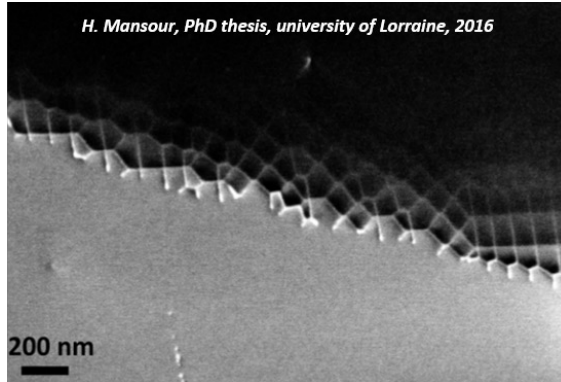
Ceramics (MAX phase)



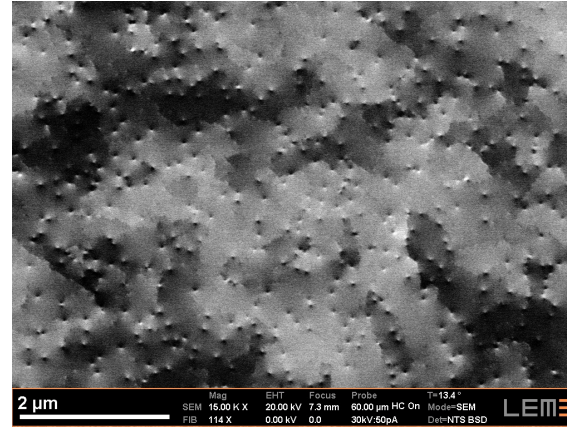
Metals (CrCoNi)

Detailed characterization of crystalline defects

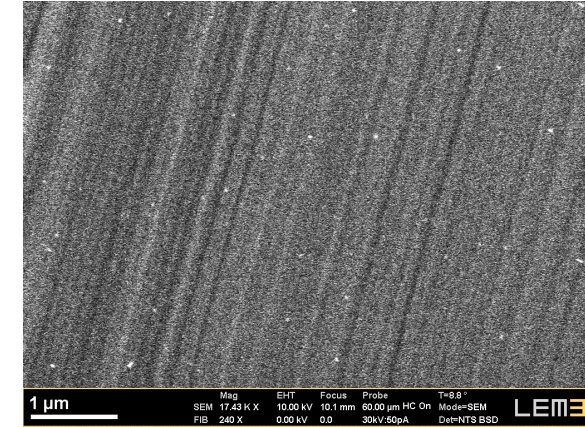
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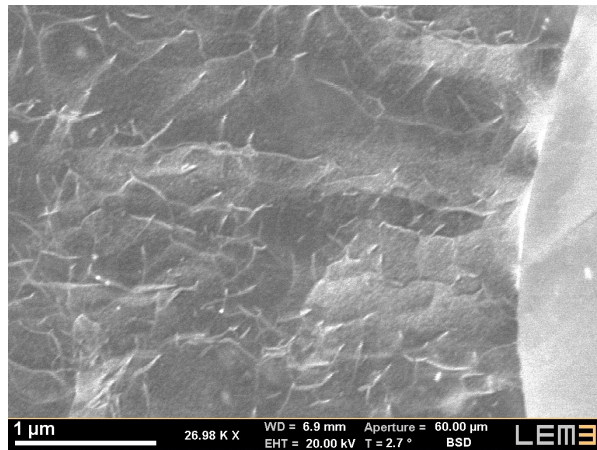
Ceramics (UO_2)



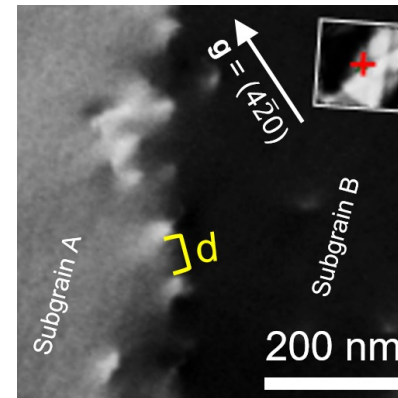
Semi-conductors (GaN)



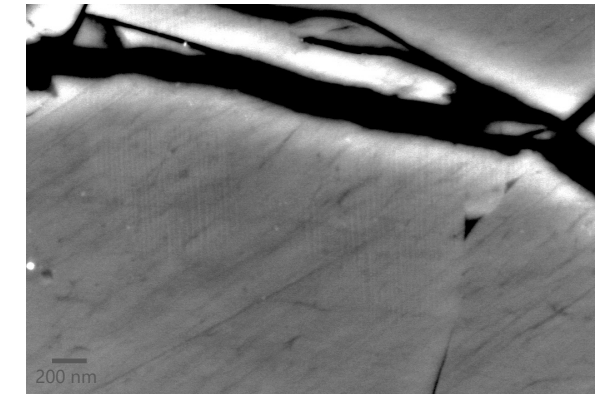
Diamond



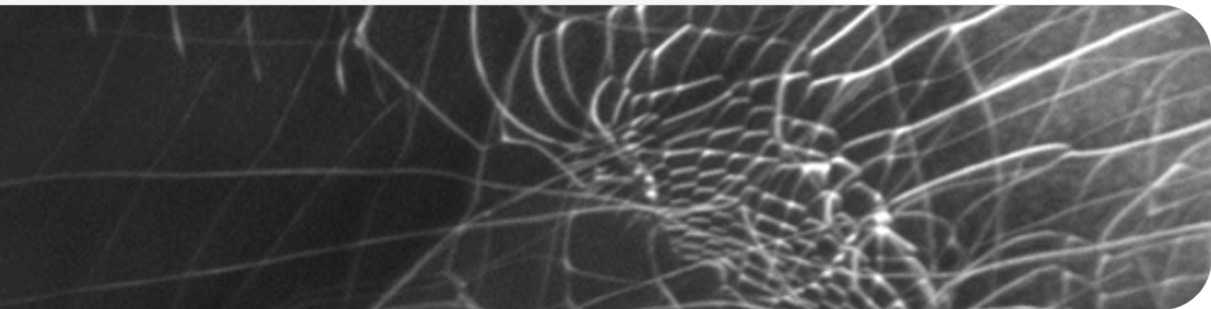
Ceramics (MAX phase)



Metals (CrCoNi)



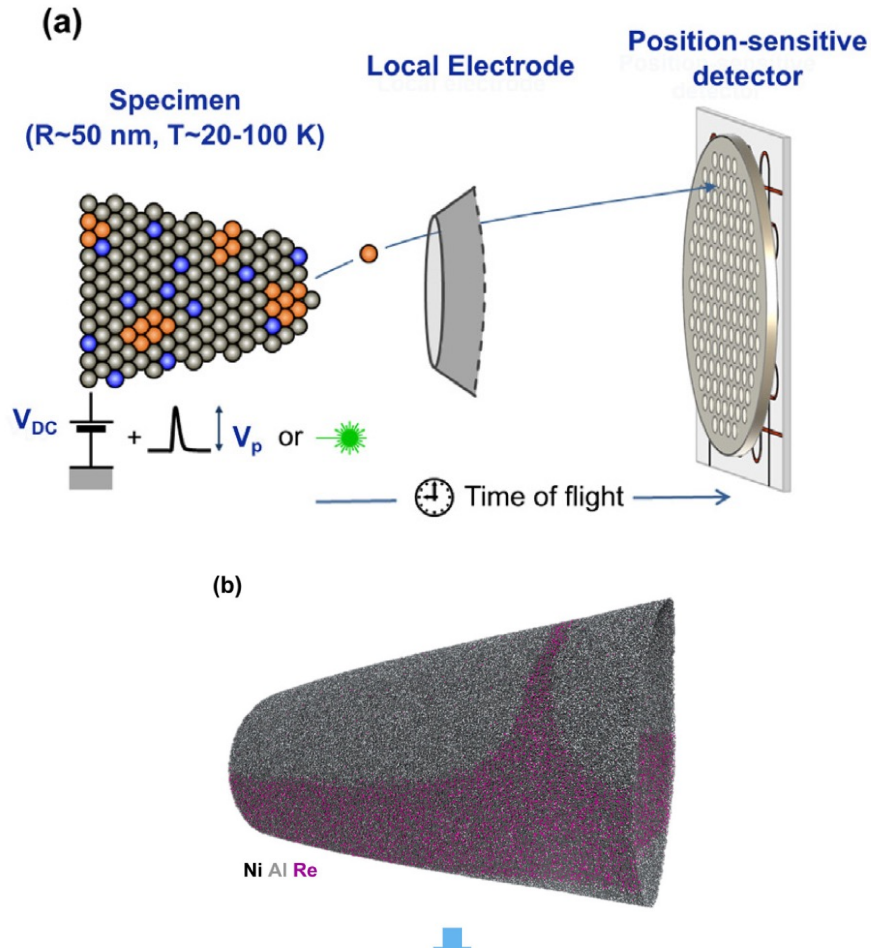
Rock (meteorite)



Atom Probe Tomography (APT)

Atomic scale

Basics of APT



❖ Definition:

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

❖ Principle:

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

❖ Spatial resolution:

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



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Thanks for your listening!

If you need further information:

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