Process-controlled microstructures for (mechanical) performance

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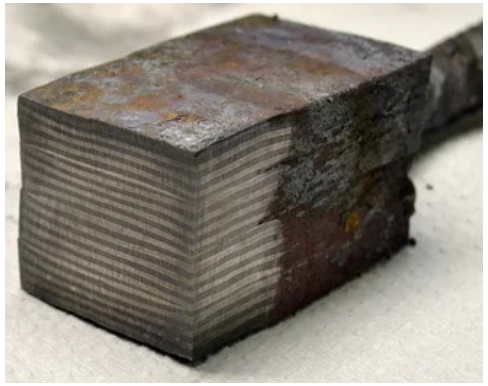
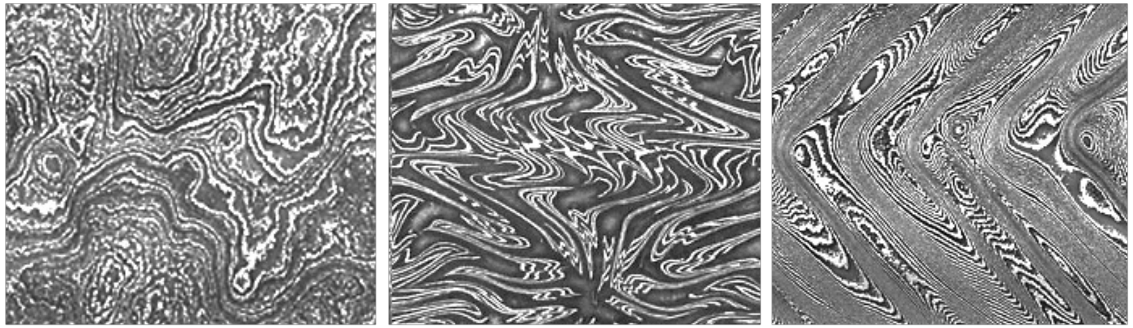
Damascus steel

- **Early evidence of process ⇒ microstructure ⇒ properties**
  - Medieval smiths empirically controlled folding, welding and forging routes to tailor mechanical behavior.
- **Layered microstructure as a natural composite**
  - Alternating high-carbon and low-carbon steels produced a heterogeneous microstructure with combined hardness and toughness.
- **Visible patterns reveal the internal structure**
  - The characteristic swirling motifs directly reflect microstructural gradients
- **Improved mechanical performance**
  - Hard, wear-resistant layers from high-carbon steel combined with tougher, more ductile low-carbon layers

    ⇒ An early form of microstructure tailoring to balance strength and toughness.
- **A precursor of modern engineered materials**
  - Conceptually linked to today's laminated steels, functionally graded materials, and architectured metallic microstructures.





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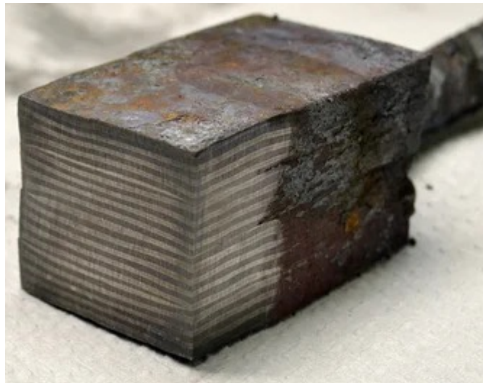

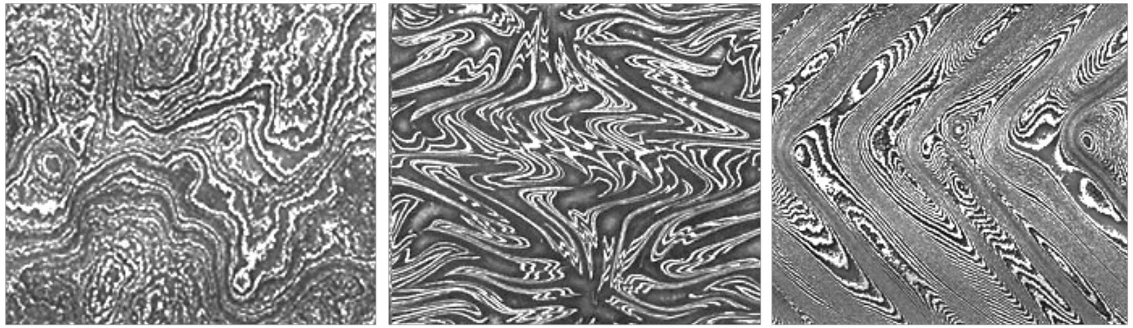

↳ Mastering the process means mastering the microstructure and ultimately the material's properties.

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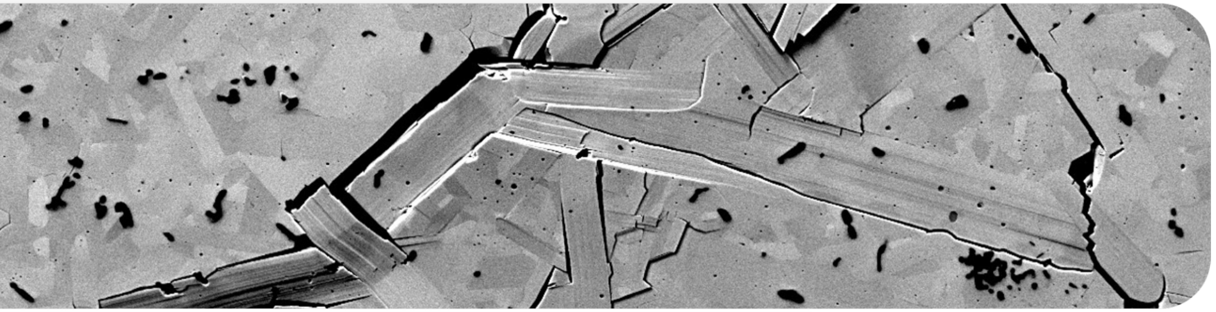
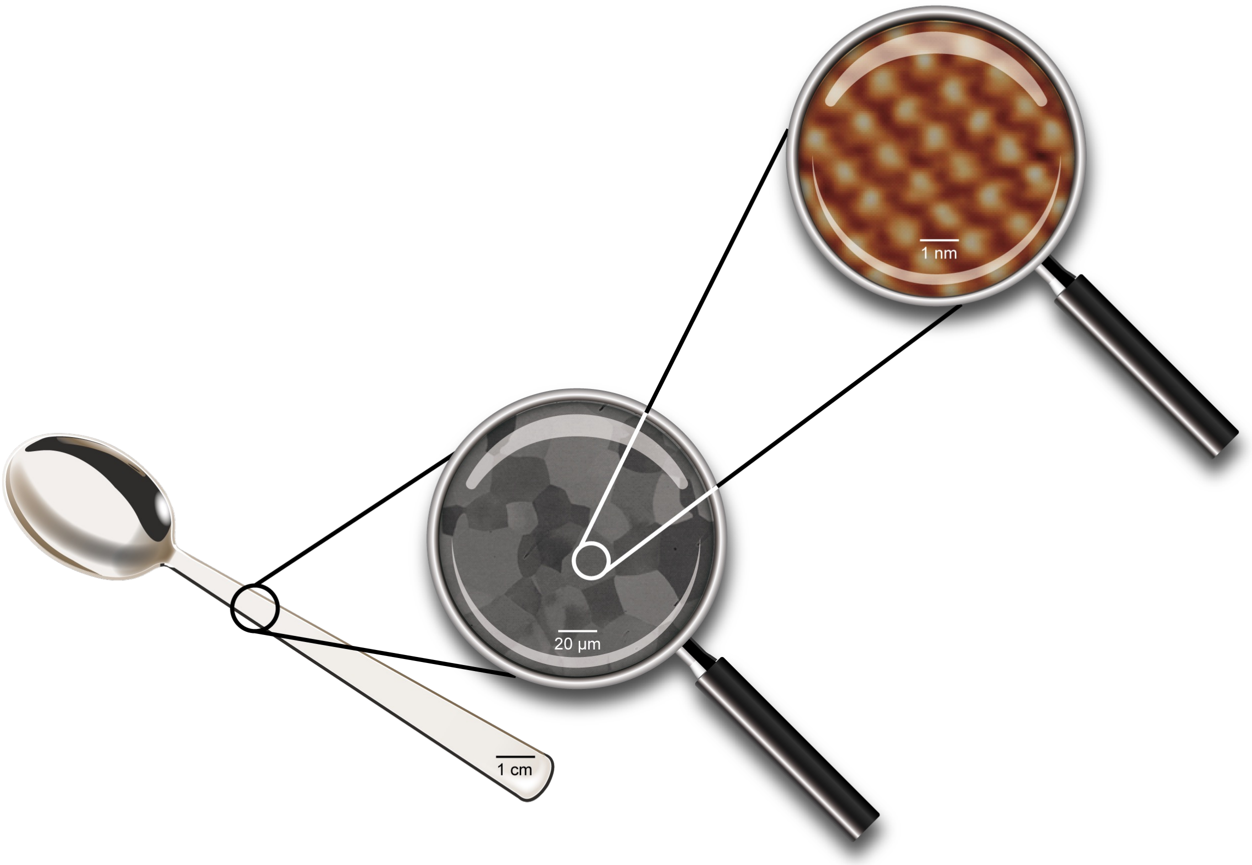


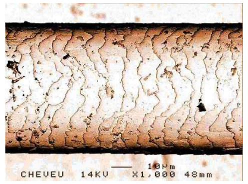
What is microstructure?

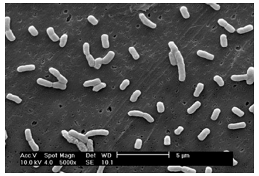
Micro-/nano-structures



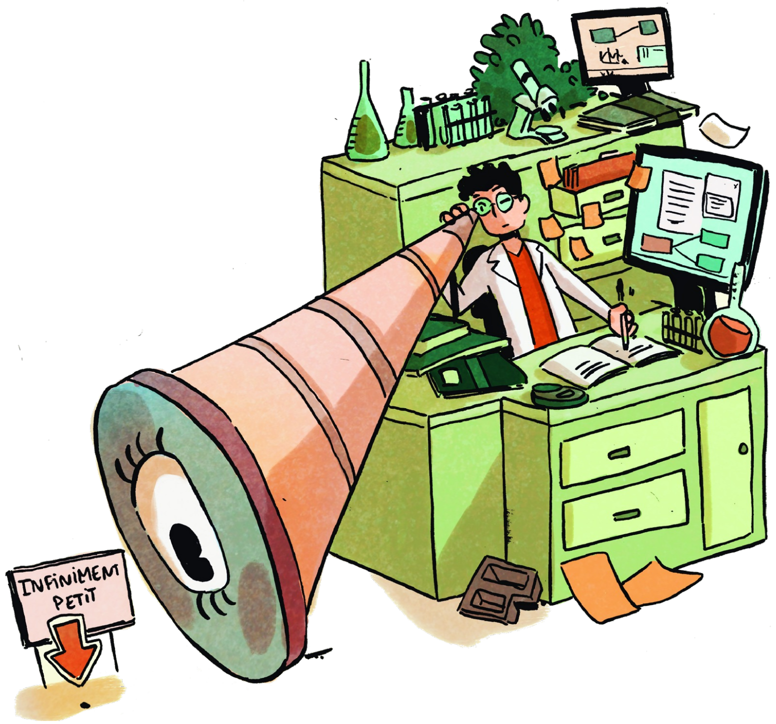
Some orders of magnitude

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





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



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

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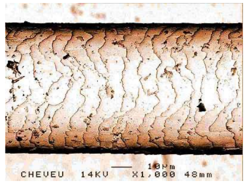

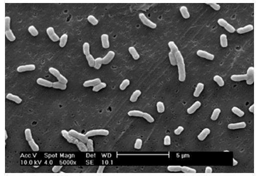

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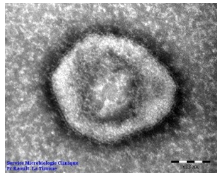
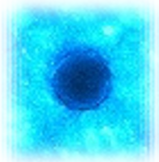
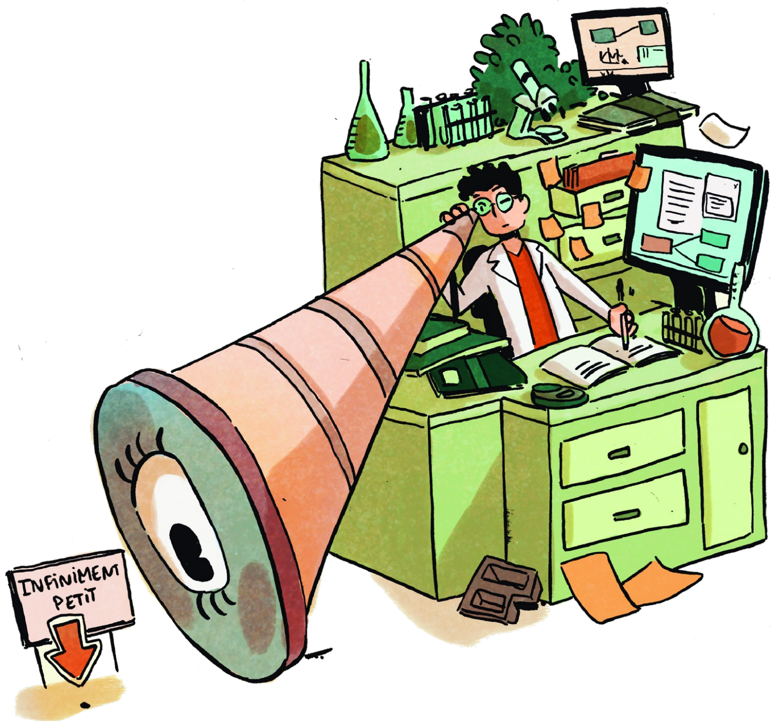




- Size of a virus = 20-450 nm



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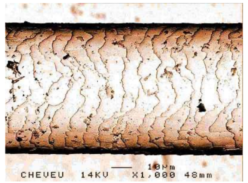

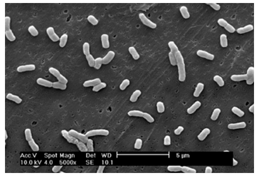

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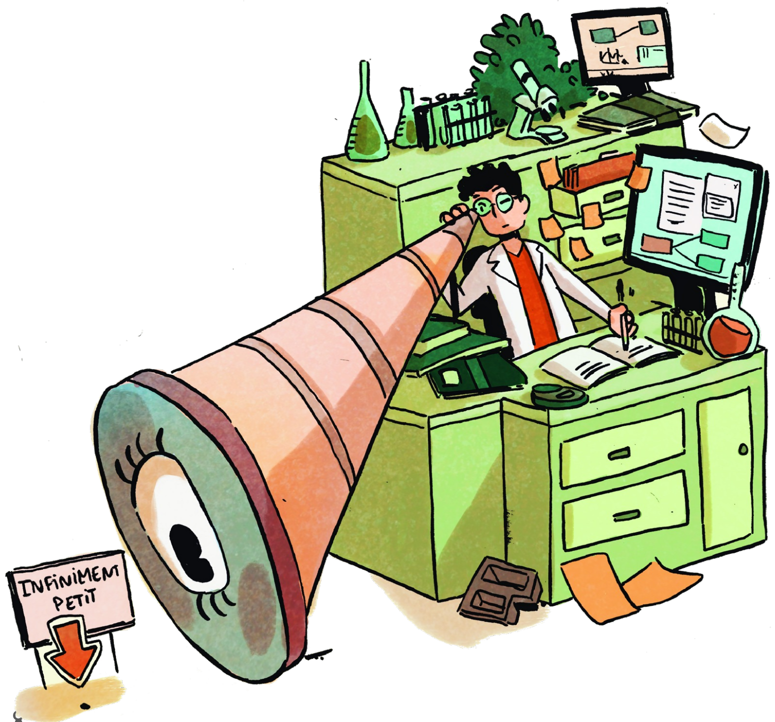

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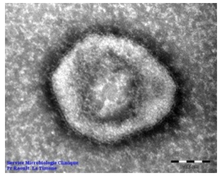
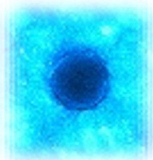

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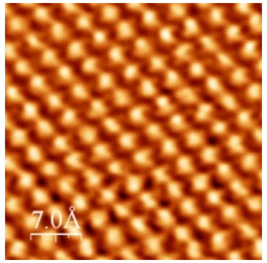
❖Atomic scale (1 Å = 0.0000000001 m = 10^{-10} m)



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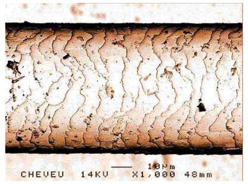
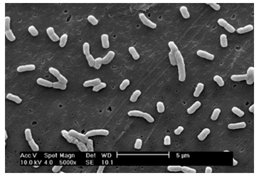

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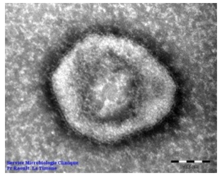
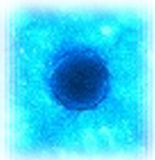

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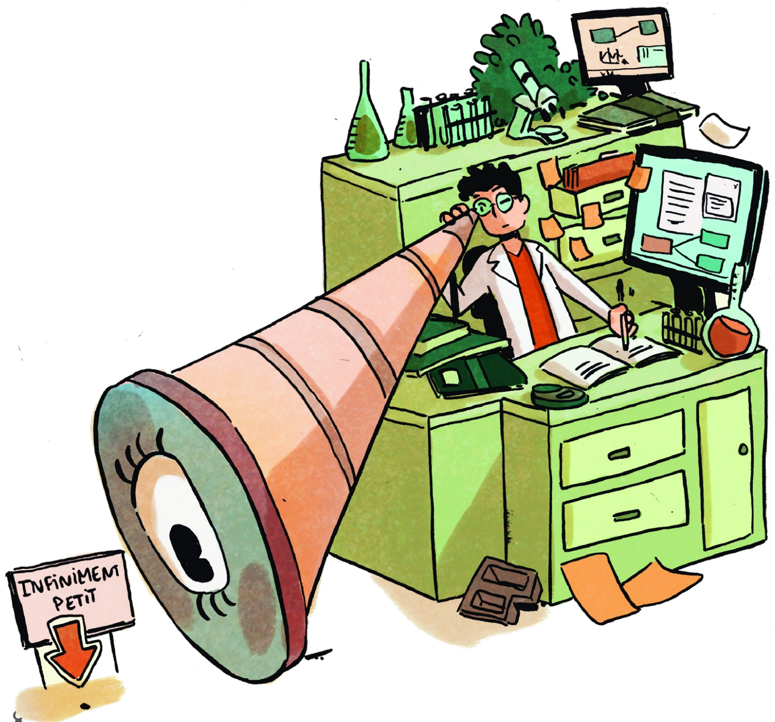

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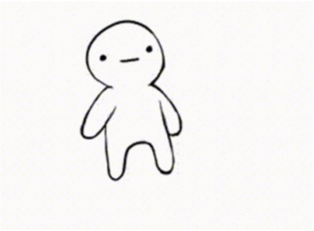
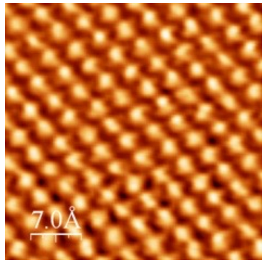

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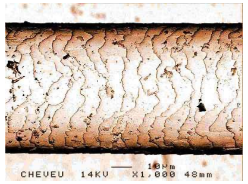
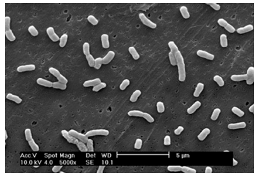

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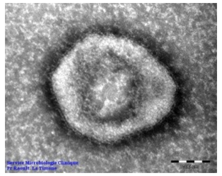
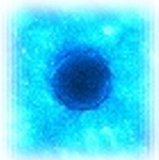

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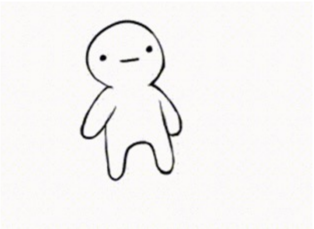
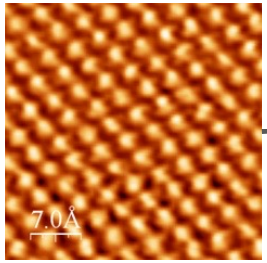

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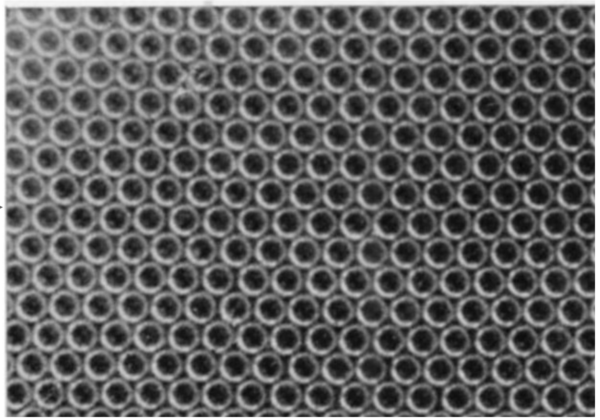
Stacking of spheres

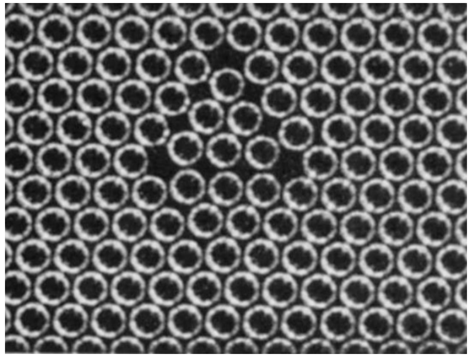
L. Bragg, J.F. Nye, *Proc. R. Soc. Lond.*, 1947



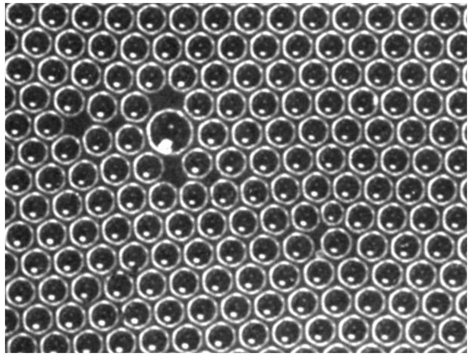
Defects in crystalline materials

0 dimension

Vacancy



Interstitial



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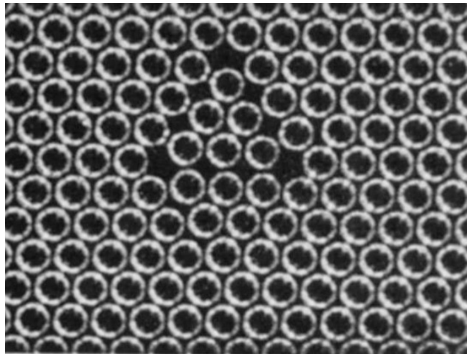

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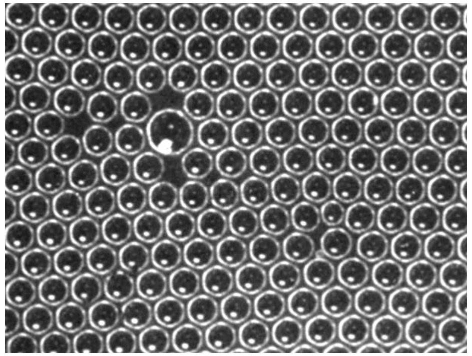
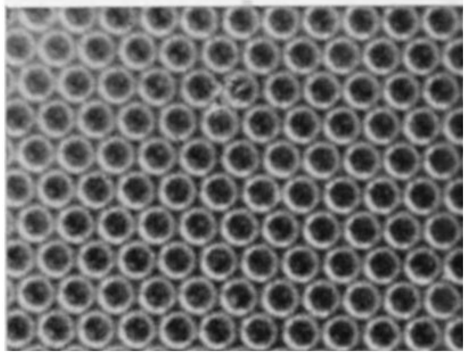


1 dimension

Dislocation



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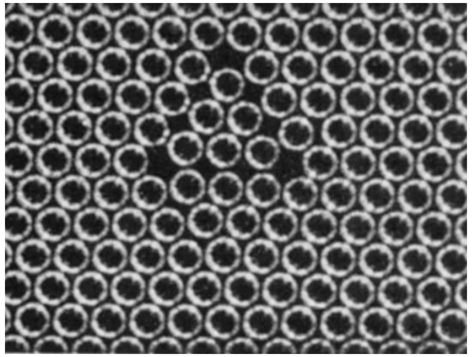

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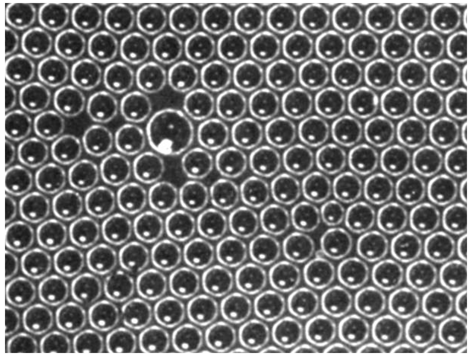

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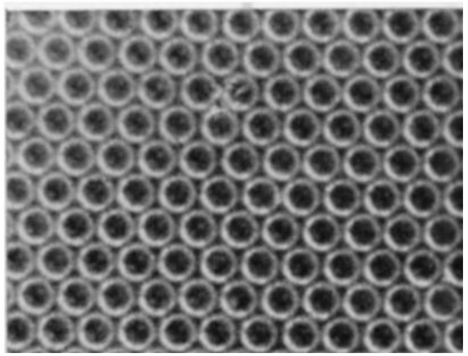
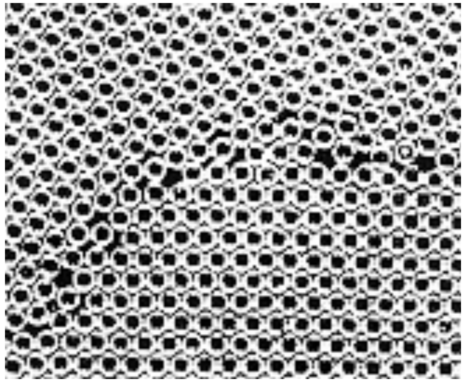


2 dimensions

Stacking fault

Grain boundaries



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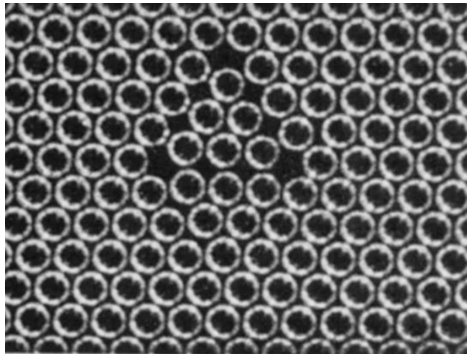

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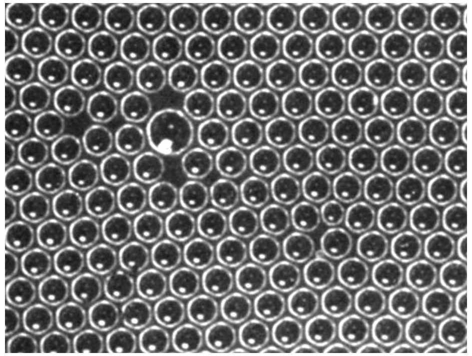

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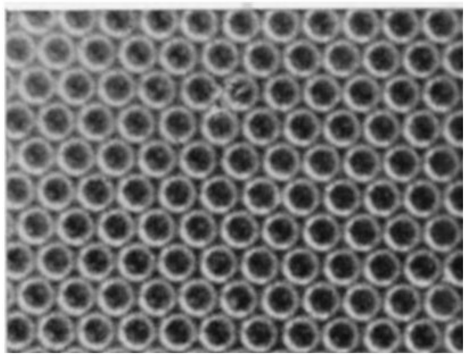

3 dimensions

Inclusions, precipitates...

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

2 dimensions

Stacking fault

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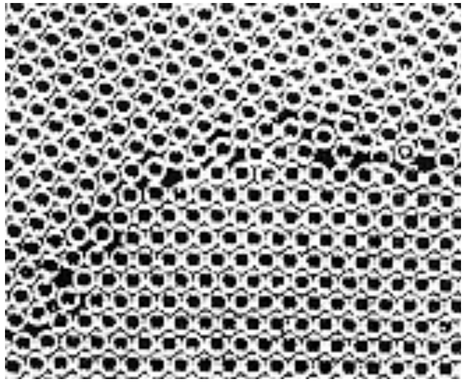

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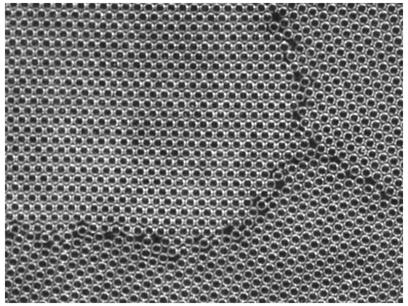
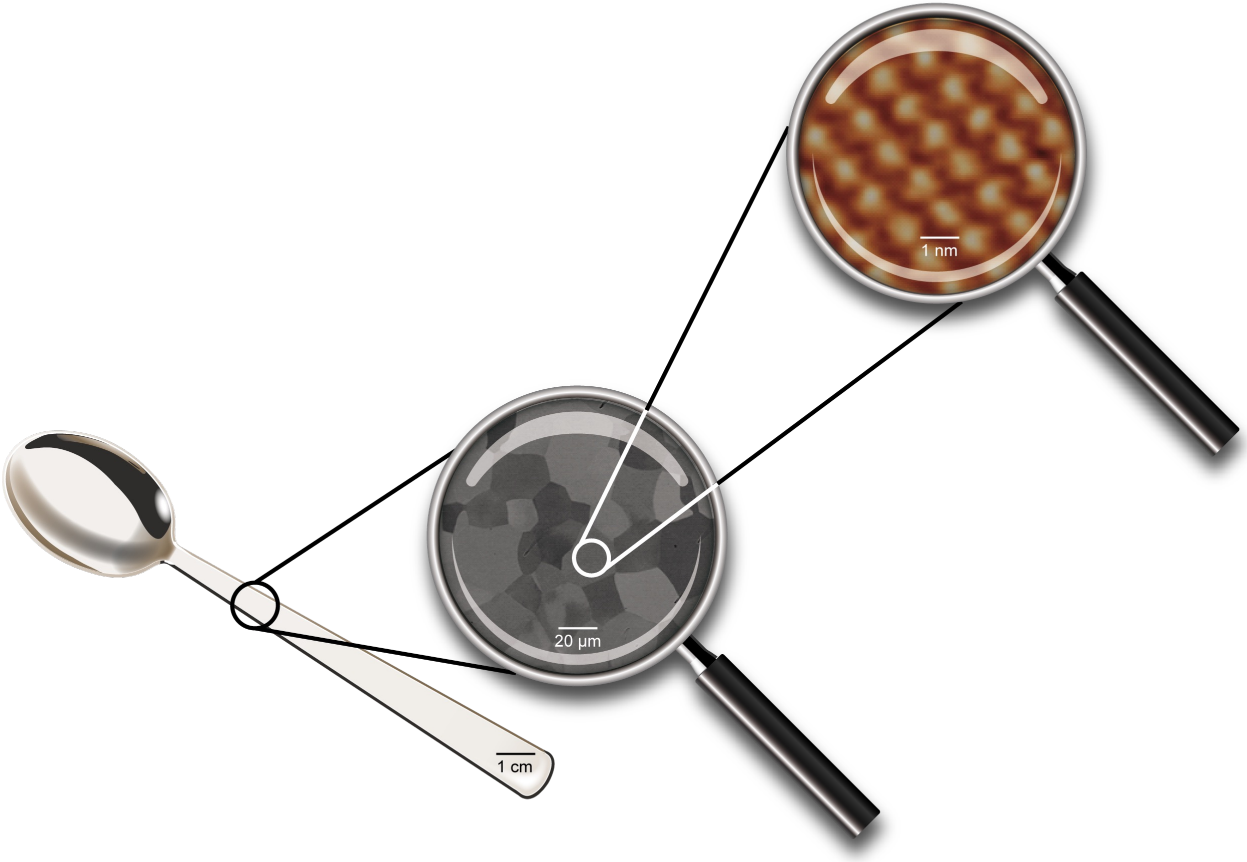
Micro-/nano-structures



Grains:

- Size
- Shape
- Chemistry
- Distribution



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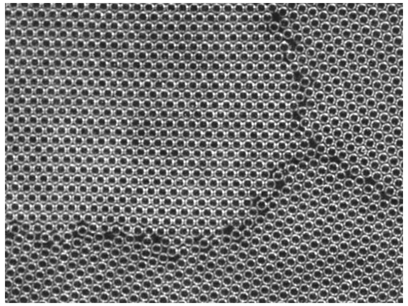

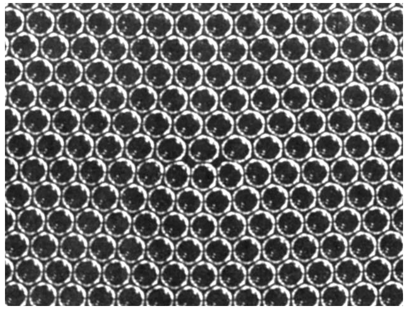

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- Types
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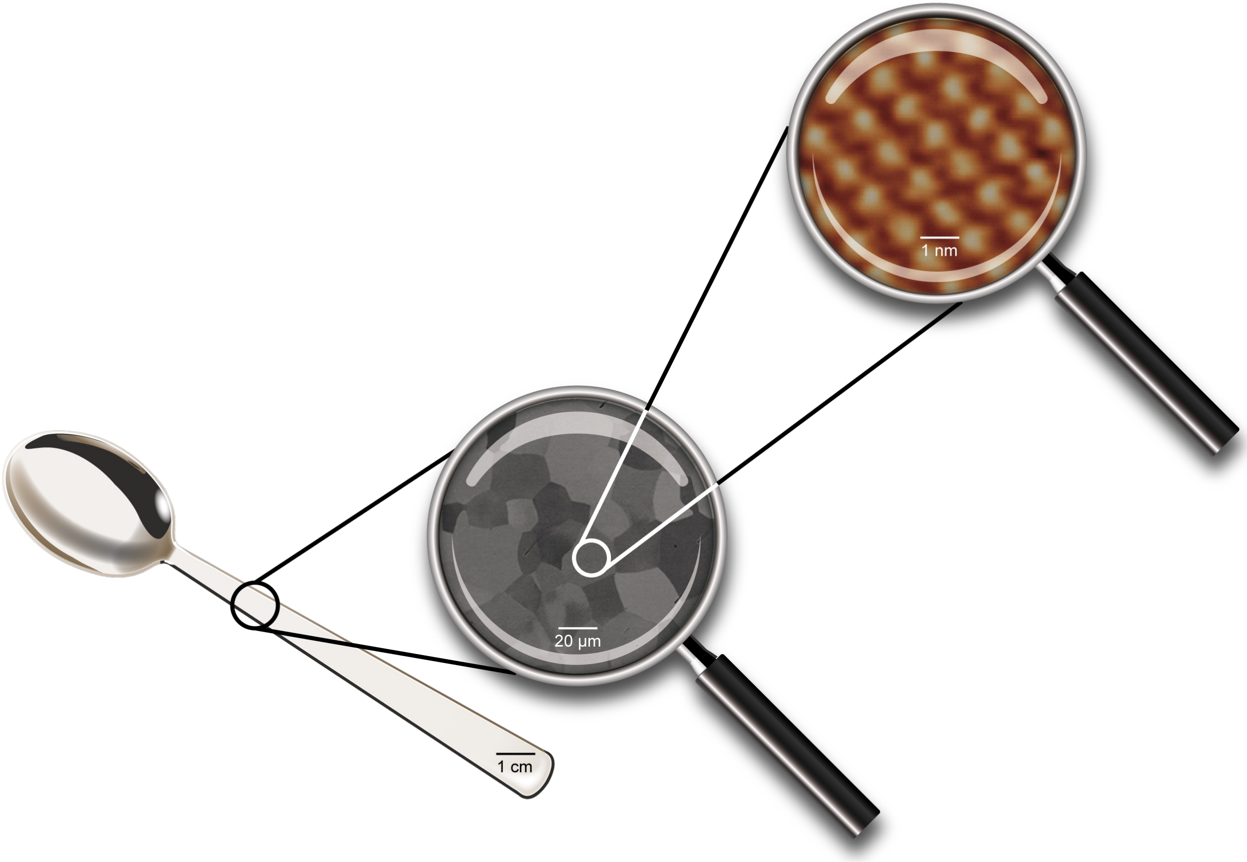

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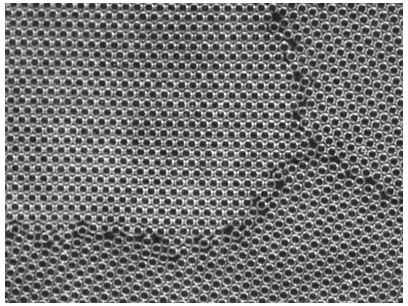

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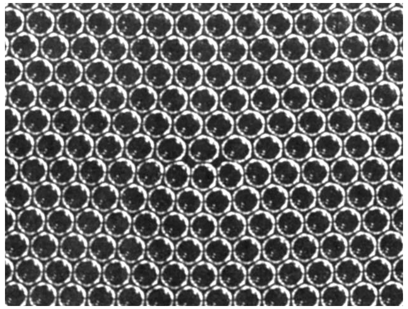

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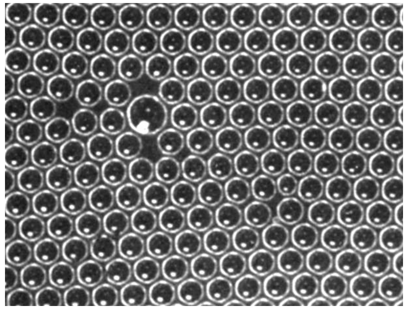

Solutes / Vacancies:

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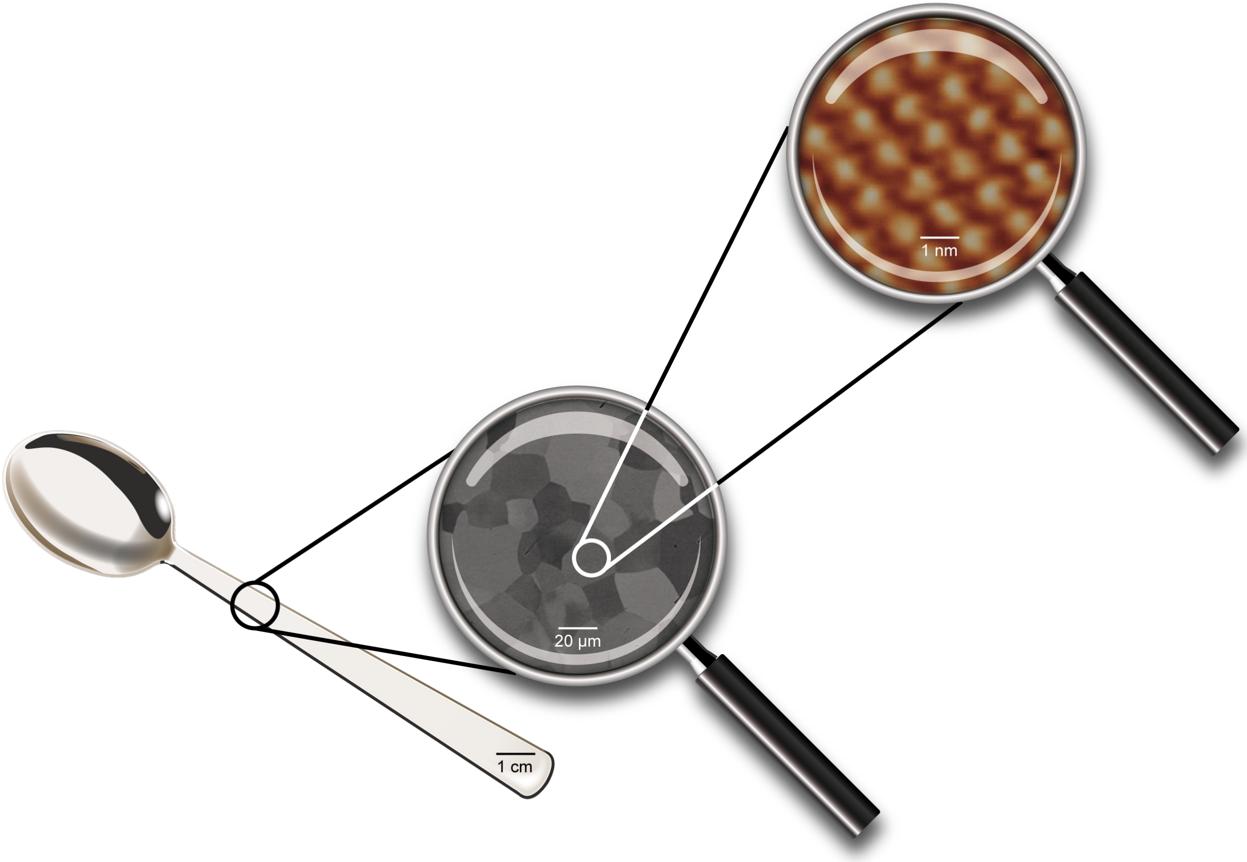
- Number
- Distribution



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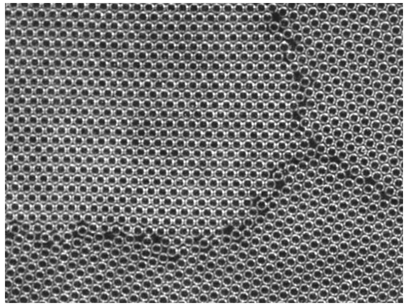

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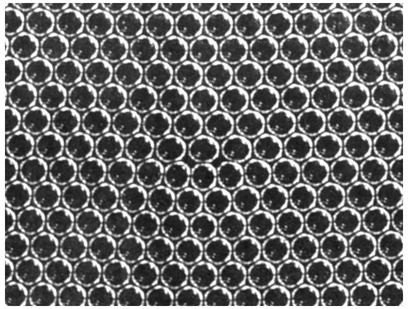

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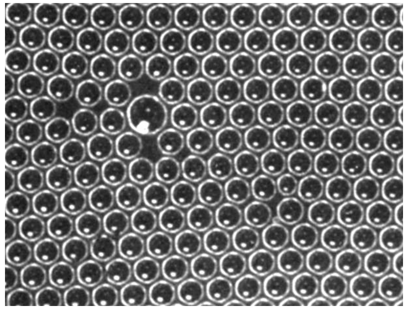

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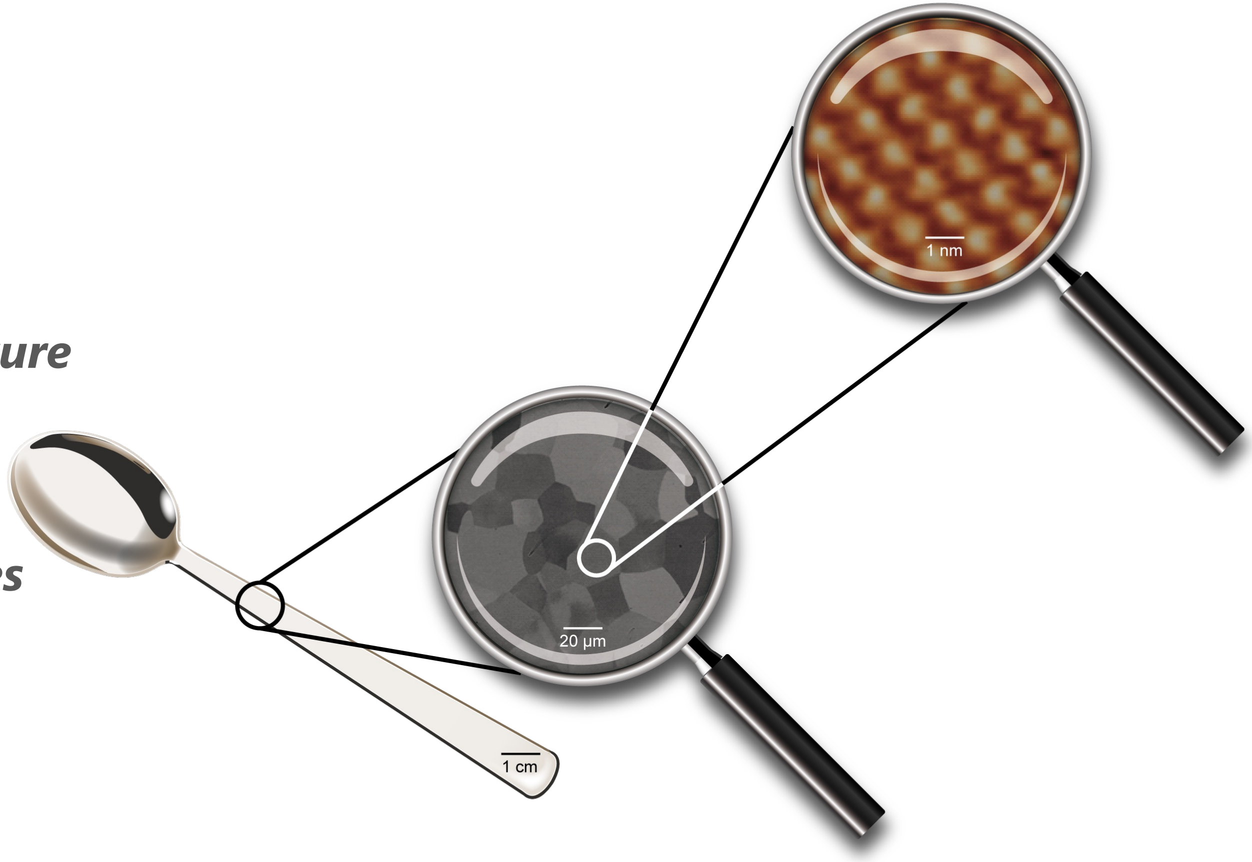
Microstructure

Properties



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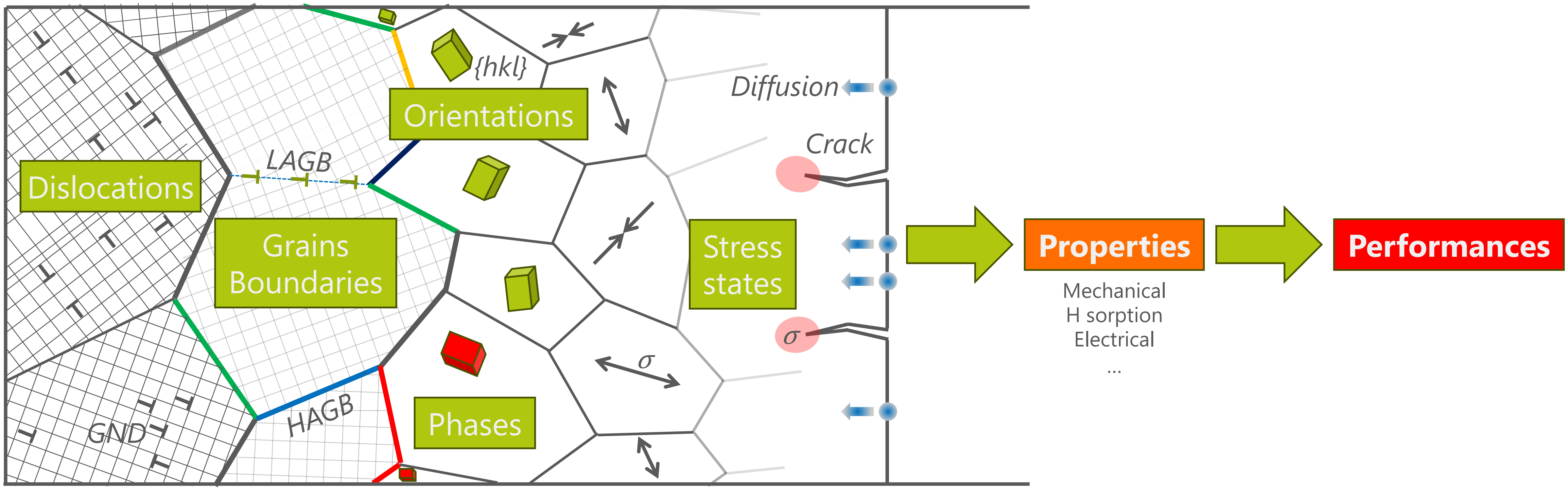
Micro-/nano-structures

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



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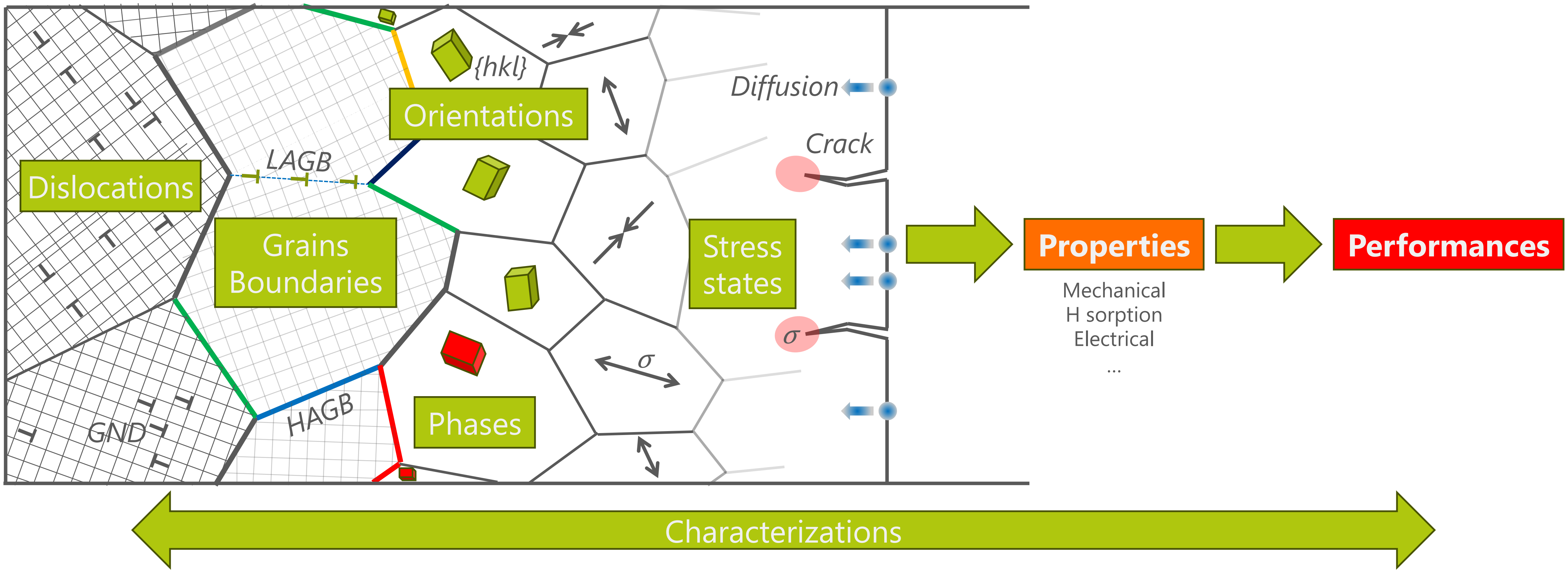
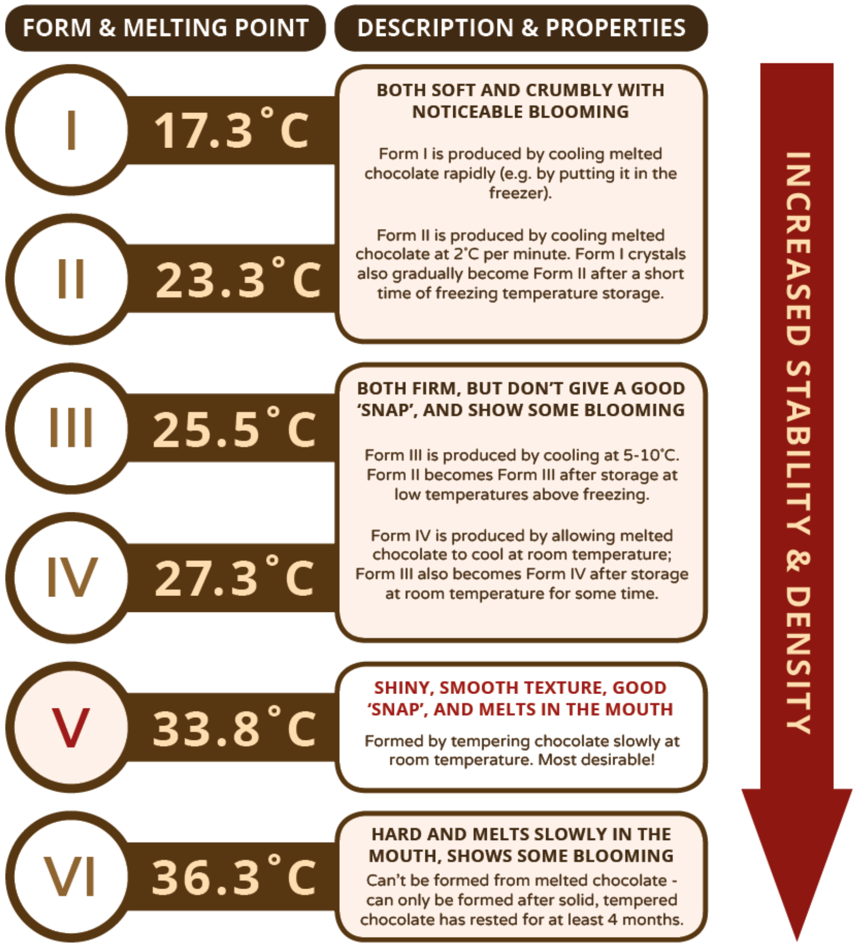


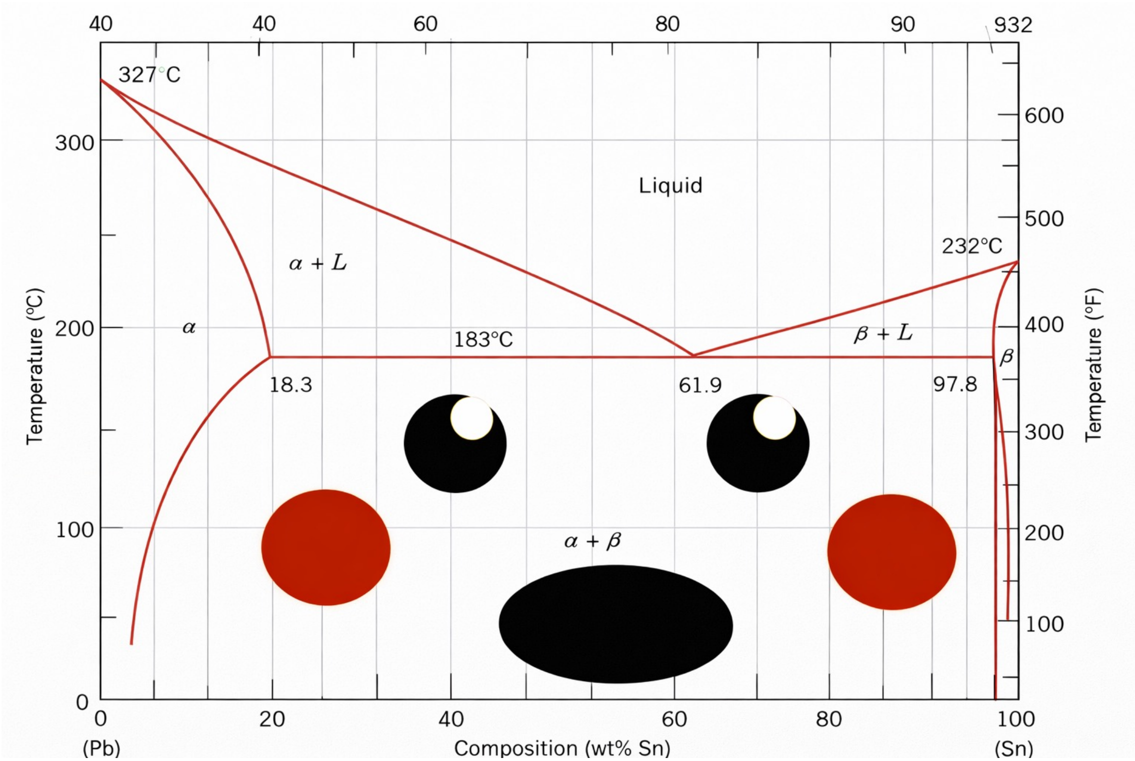
Phases



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

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

Phases



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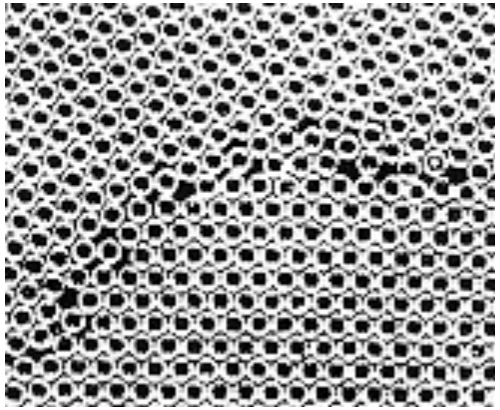
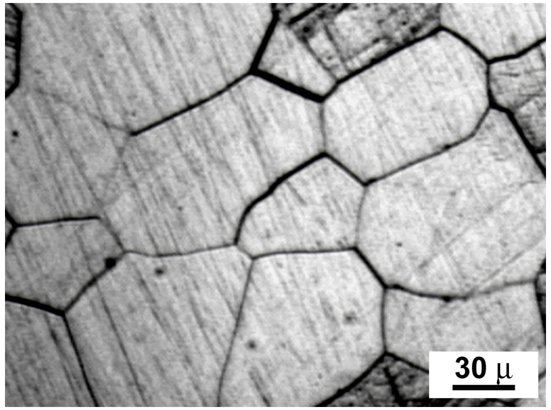
  - Heating and cooling control polymorphic transformation
- **Phase diagram:**
  - Graphically represents phase stability as a function of temperature and composition.
- **Tie line (isothermal line):**
  - A horizontal line drawn in a two-phase region to determine the composition of coexisting phases.
- **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

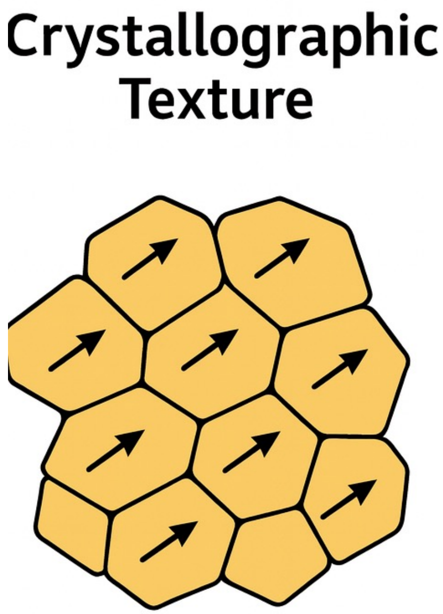
Texture:

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

Textures

❖ Crystallographic texture

- A non-random distribution of grain orientations in a polycrystalline material where many grains share the same preferred crystallographic direction.
- Grains oriented similarly rather than randomly, often seen as clusters or peaks in pole figures or inverse pole figures, or as elongated grains aligned after rolling.



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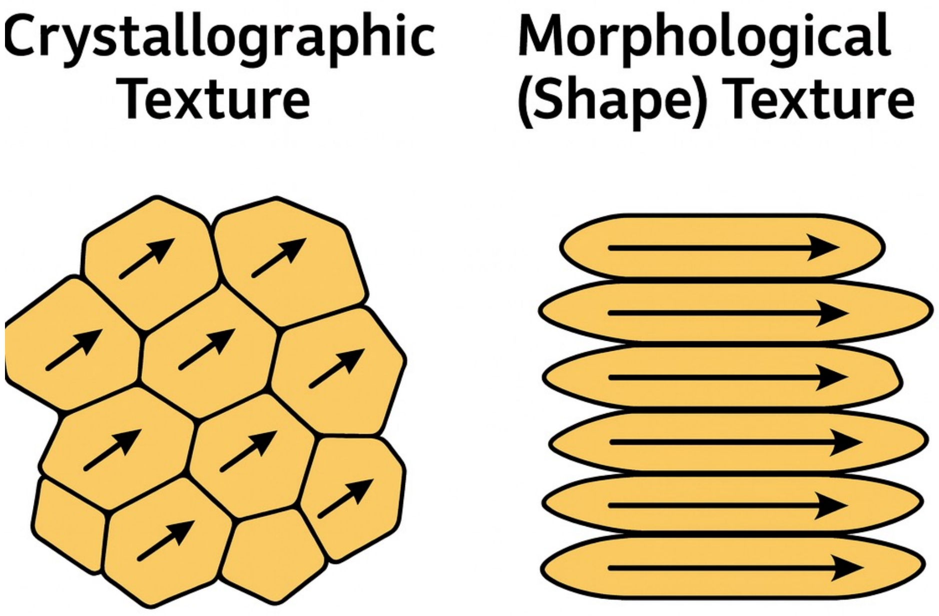
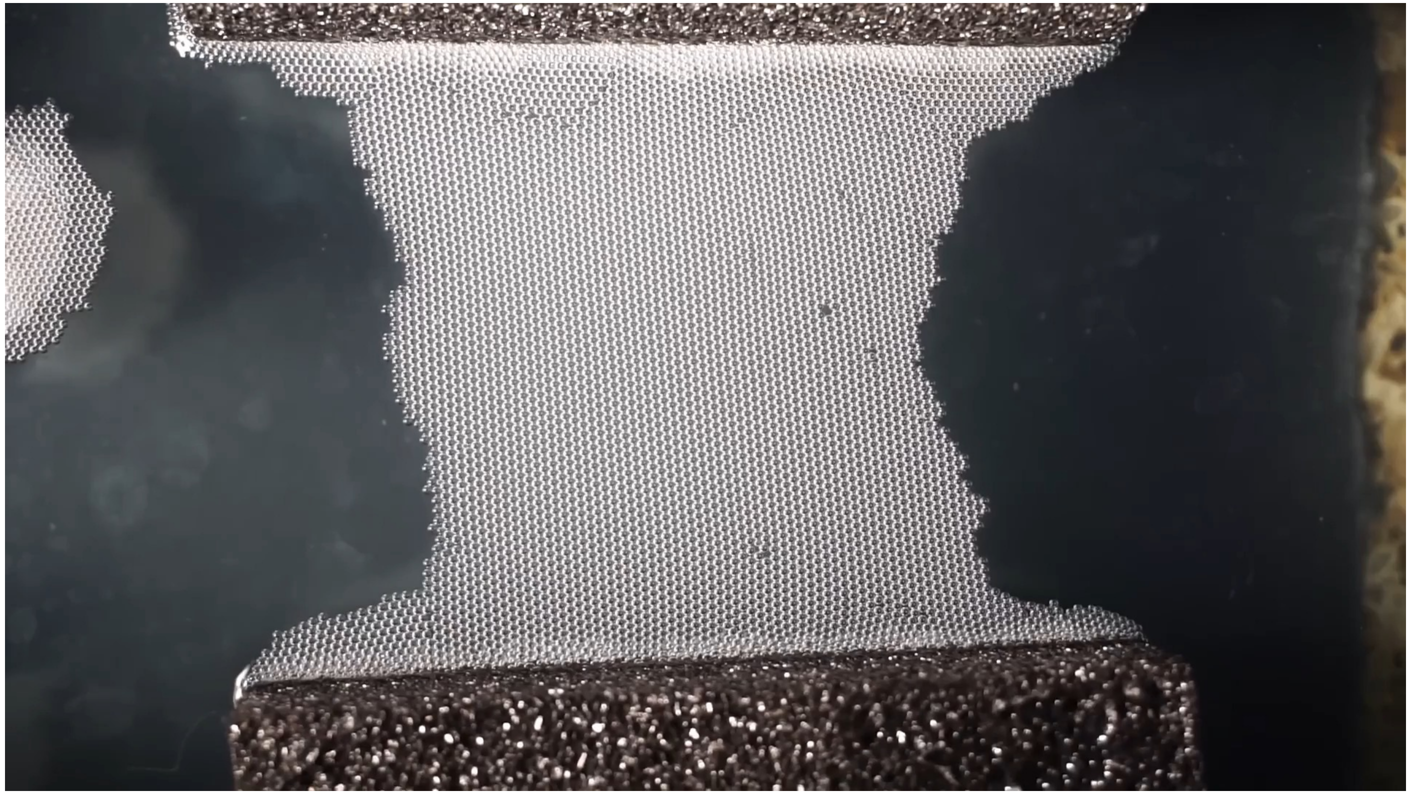
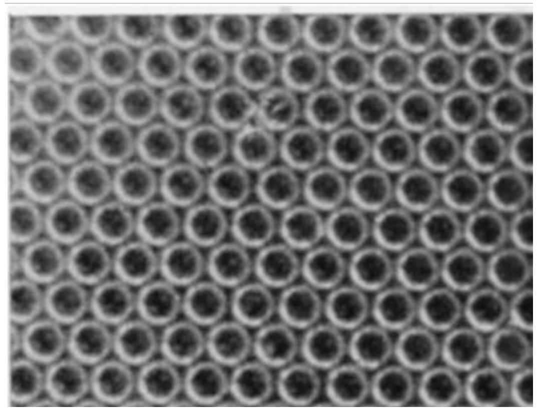
❖ Morphological (shape) texture

- A preferred orientation of the grain shapes themselves. Grains become elongated or flattened in the same direction.
- It appears as aligned, elongated grains in micrographs.



Dislocations





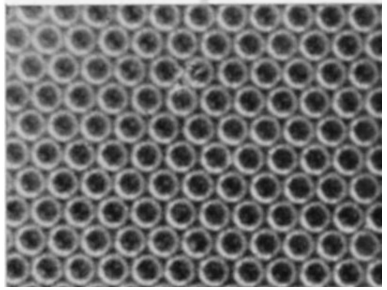
L. Bragg & J.F. Nye, *Proc. R. Soc. Lond.* (1947)

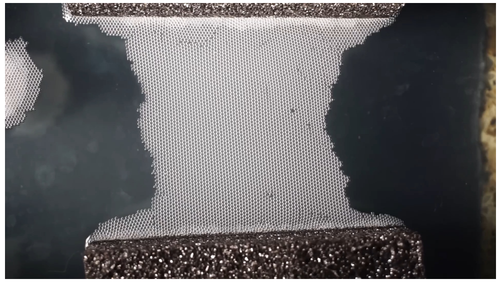
Dislocations

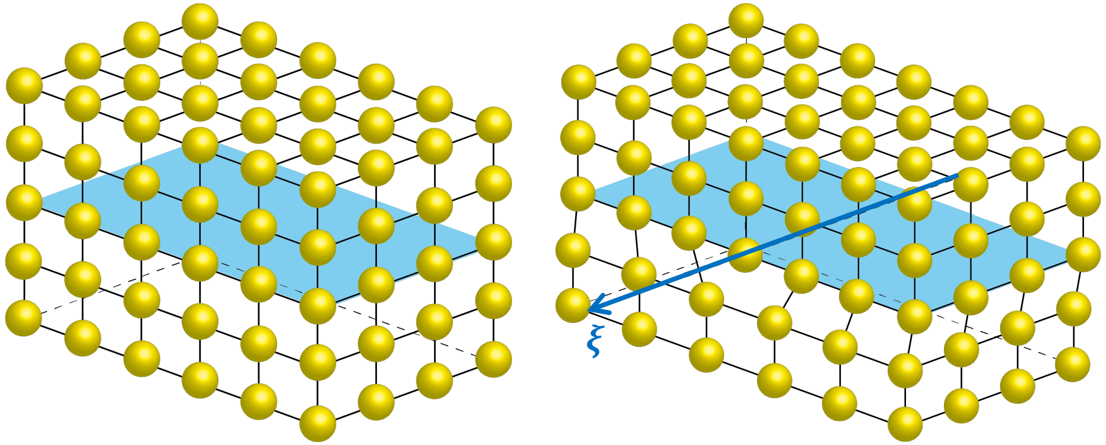
❖ **Definition:**

- 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 ξ.

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







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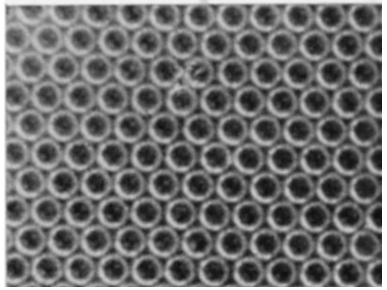

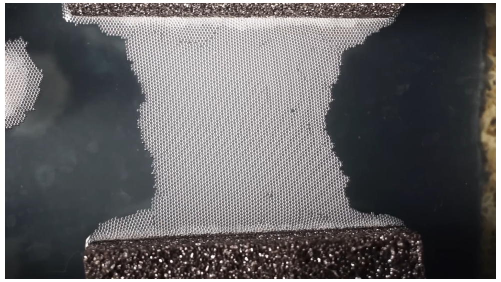

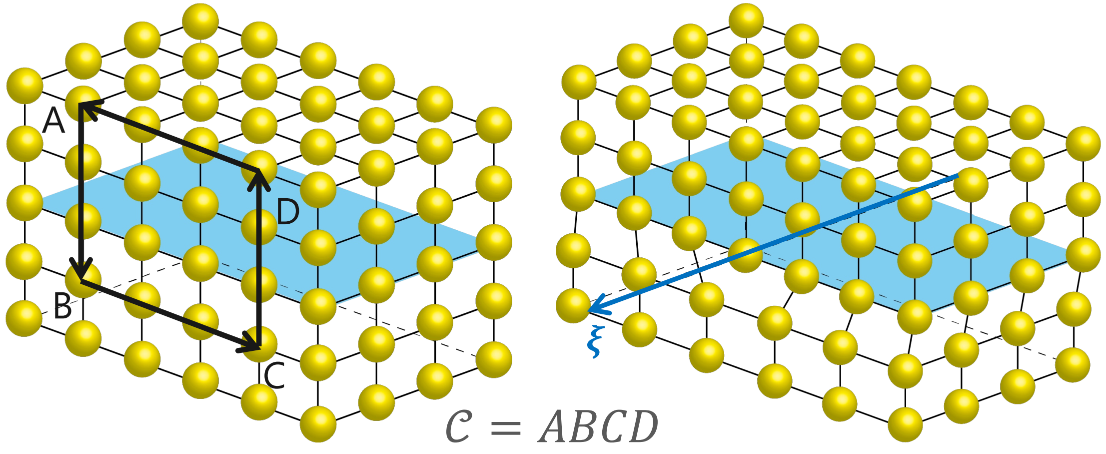

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- They represent a discontinuity in the crystal lattice that allows deformation to occur at lower stresses.
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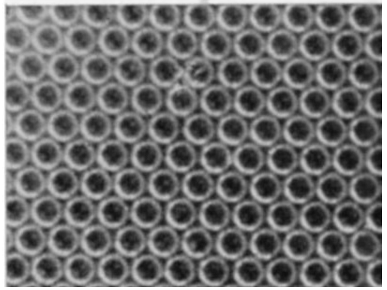
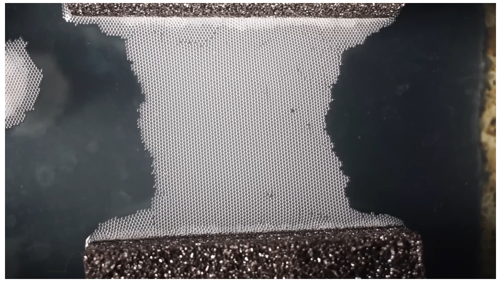
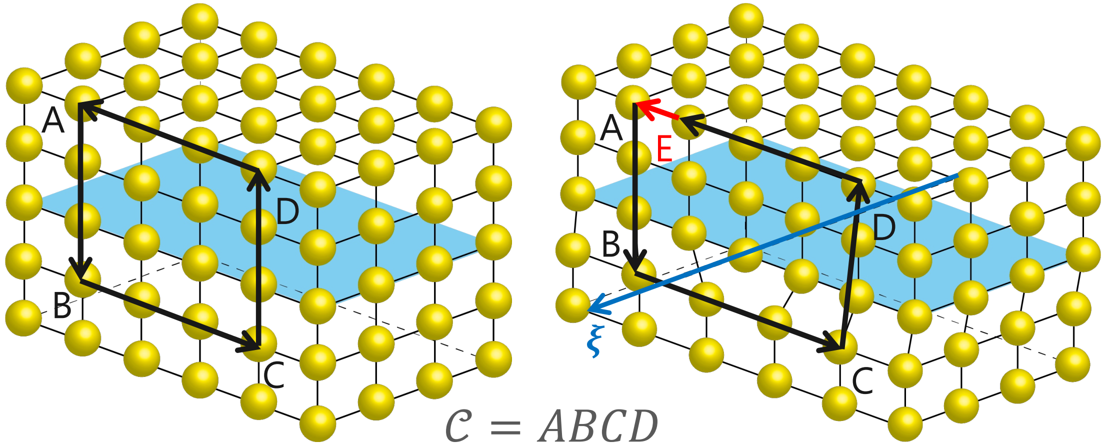
❖ Types:

- *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)





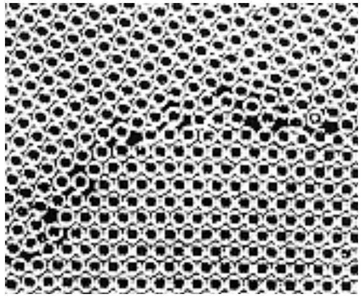


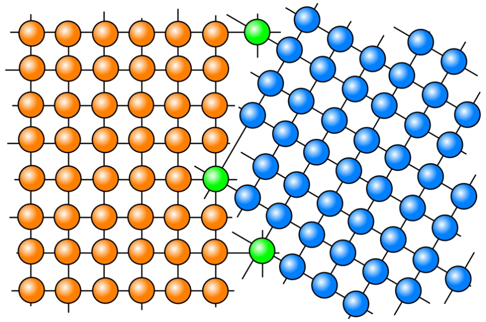
$$\mathcal{C} = ABCD$$

$$\boldsymbol{EA} = \boldsymbol{b}$$

Grain boundaries

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





❖ Definition:

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

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

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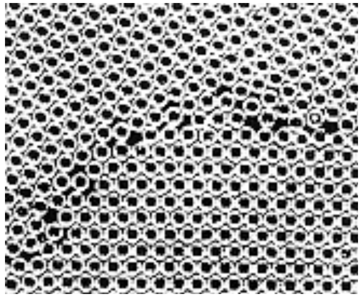

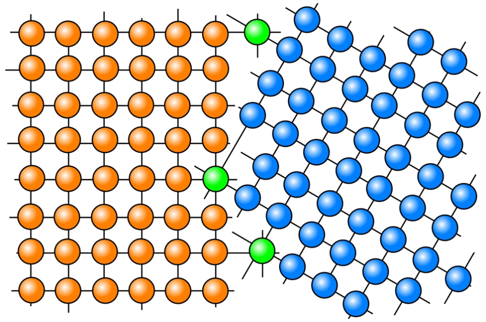

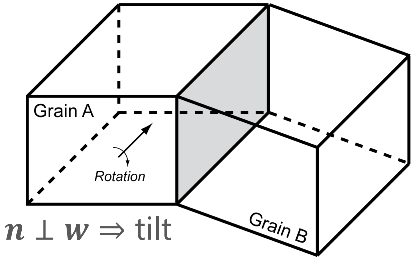

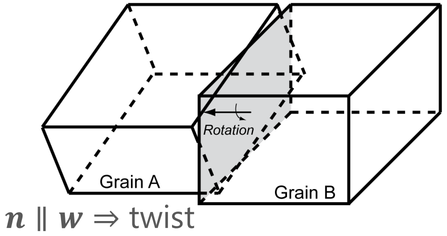

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  - *Tilt GB:* $\boldsymbol{n} \perp \boldsymbol{w}$
  - *Twist GB:* $\boldsymbol{n} \parallel \boldsymbol{w}$
  - *Mixed GB:* combination of tilt and twist components.

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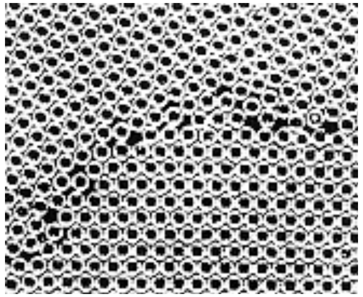
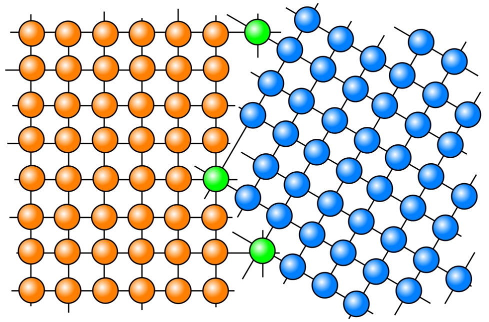
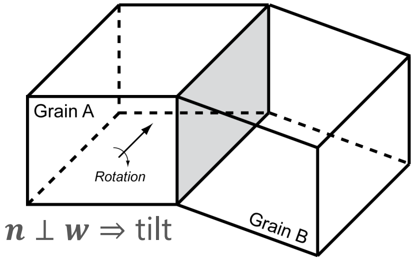
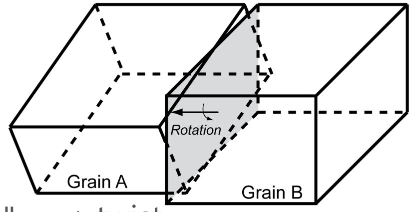






$\boldsymbol{n} \perp \boldsymbol{w} \Rightarrow$ tilt



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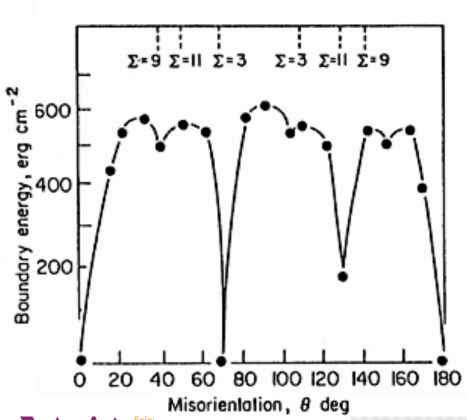

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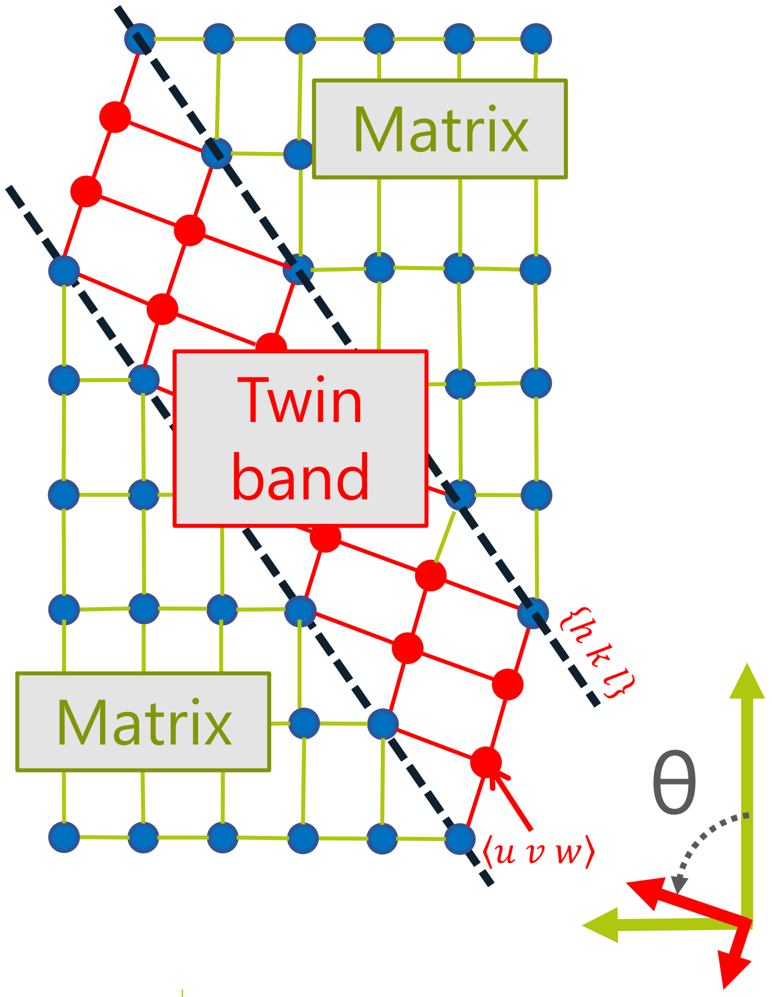
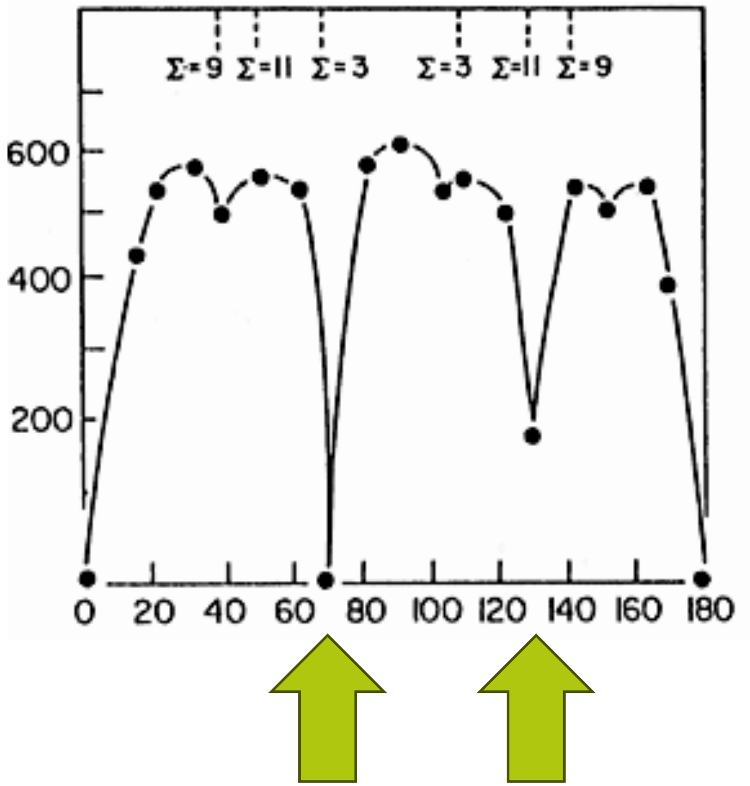
- *Mixed GB:* combination of tilt and twist components.
- Low-Angle GB (LAGB): $\theta \lessapprox 15°$
- High-Angle GB (HAGB): $\theta \gtrapprox 15°$

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

Focus on the twinning



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



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

Punctual defects

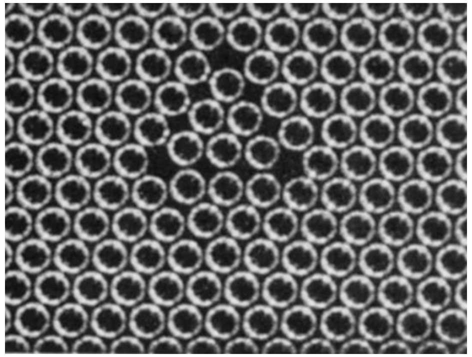
❖ Definition:

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

❖ Types:

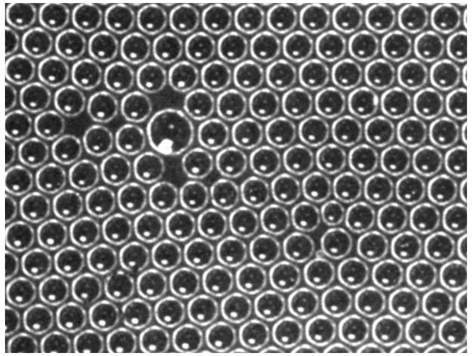
- *Vacancies:* Missing atoms in the lattice.
- *Interstitials:* Extra atoms positioned in the interstitial sites between the regular lattice points.
- *Substitutional defects:* Foreign atoms replacing host atoms in the lattice.

Vacancy



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

Interstitial



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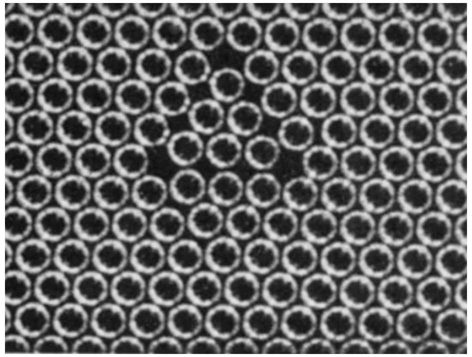
❖ Diffusion:

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

Vacancy



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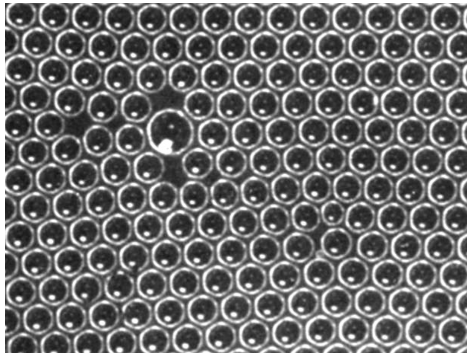
Interstitial



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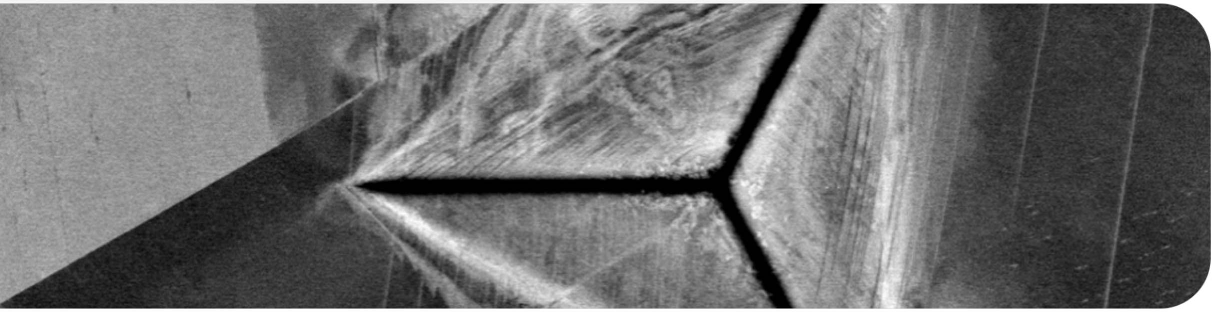

How processes Build microstructure

Metal casting

- **How it works?**
  - Molten metal is poured into a mold that defines the final shape of the part.
  - Heat is extracted through the mold walls, initiating solidification at the boundaries.

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  - Molten metal is poured into a mold that defines the final shape of the part.
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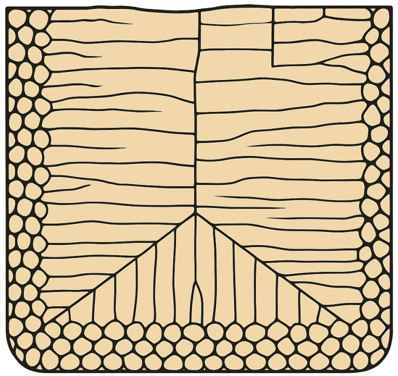
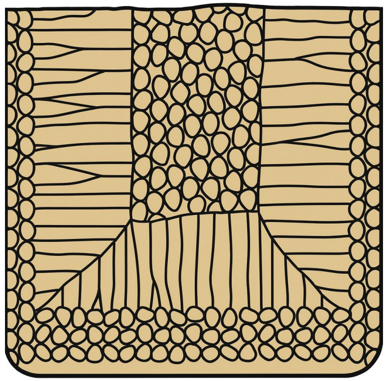
- ❖ **Key microstructure features for pure metals**
  - Formation of fine equiaxed grains near the mold walls due to rapid cooling.
  - Development of columnar grains growing opposite to the heat-flow direction.
  - Possible formation of a central equiaxed zone if sufficient undercooling develops.
  - No complex dendritic structures.
  - Risk of shrinkage porosity in the central region.

Pure metals





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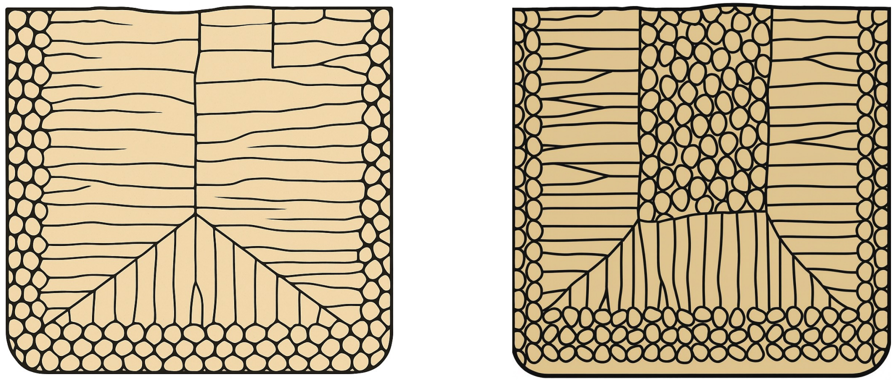
❖ **Key microstructure features for alloys**

- Solidification occurs over a temperature range.
- Formation of dendritic structures (primary and secondary arms).
- Solute segregation between dendrite arms.
- Possibility of eutectic or multiphase regions in the final microstructure.

Pure metals



Alloys

Deformation testing

- **How it works?**
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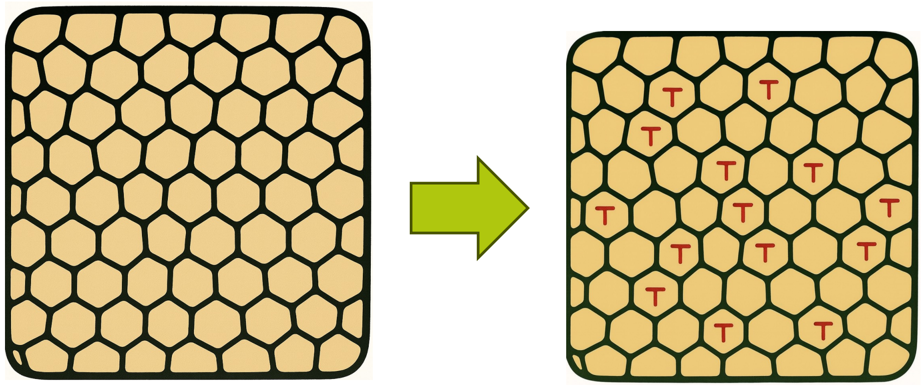
❖ **Key microstructure features at low deformation**
- Increase in dislocation density.
- Formation of dislocation tangles and cell structures.
- Grains mainly retain their original shape and orientation.

❖ **Key microstructure features at high deformation**
- Development of subgrains and dislocation walls.
- Grain elongation in the loading direction.
- Possible formation of shear bands.

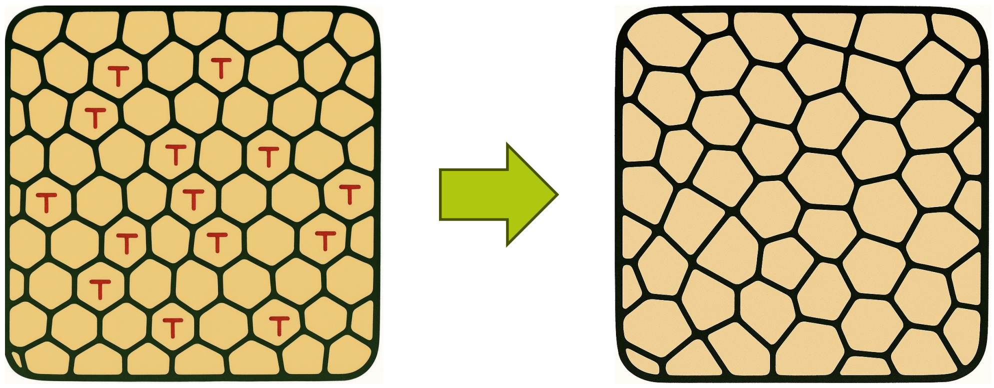
❖ **Key microstructure features at high temperature**
- Dynamic recovery: annihilation and rearrangement of dislocations.
- Dynamic recrystallization: formation of new equiaxed grains.
- Grain growth if temperature is sufficiently high.



Heat treatments

- **Annealing**
  - Recrystallization
    ⇒ formation of equiaxed, strain-free grains
  - Reduction in dislocation density
  - Grain growth at higher temperatures
- **Quenching**
  - Formation of lath or plate martensite
  - Retention of metastable phases (e.g., retained austenite)
- **Tempering / Aging**
  - Controlled precipitation of fine particles

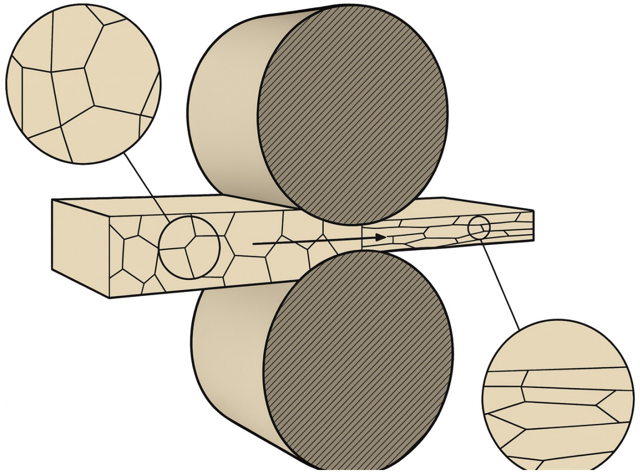
Annealing



Rolling

❖ **How it works?**

- A metal workpiece is passed between two rotating rolls.
- The rolls apply compressive forces, reducing the thickness.
- The material experiences plastic deformation and elongates in the rolling direction.
- Friction between rolls and workpiece ensures material entry.



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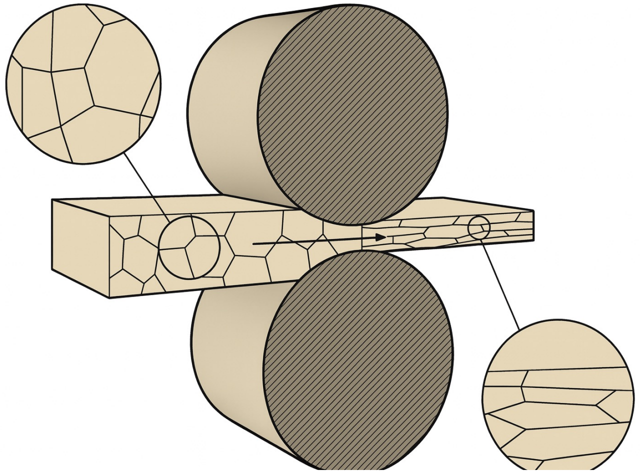
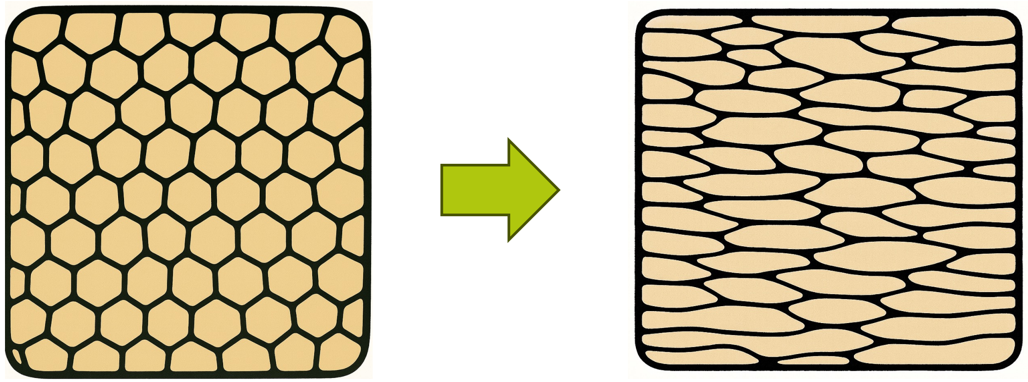
❖ Key microstructural features

- Elongation of grains in the rolling direction
- Increase in dislocation density due to plastic deformation (work hardening).
- Possible formation of subgrains and cell structures.
- Development of crystallographic texture (preferred orientation).
- If temperature is high enough

  ⇒ dynamic recrystallization (formation of new equiaxed grains).





Severe plastic deformation (SPD)

❖ **How it works?**

- Very large plastic strains are applied
- Deformation refines the microstructure through dislocation accumulation and grain subdivision.
- SPD can be applied either to the whole volume or only to the surface of the material.

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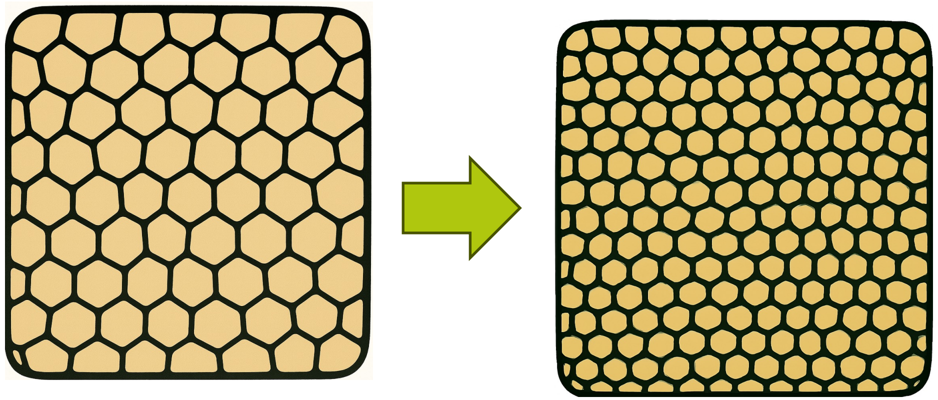
- Very large plastic strains are applied
- Deformation refines the microstructure through dislocation accumulation and grain subdivision.
- SPD can be applied either to the whole volume or only to the surface of the material.

❖ **Key microstructure features for volume SPD**
- Formation of ultrafine-grained (UFG) or nanocrystalline structures throughout the bulk.
- Extremely high dislocation density leading to grain subdivision.
- Development of high-angle grain boundaries after severe deformation.

Volume SPD



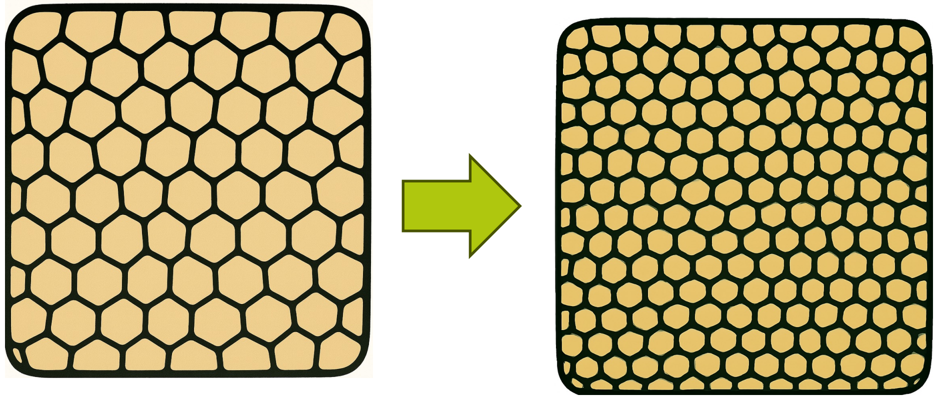
Severe plastic deformation (SPD)

❖ **How it works?**
- Very large plastic strains are applied
- Deformation refines the microstructure through dislocation accumulation and grain subdivision.
- SPD can be applied either to the whole volume or only to the surface of the material.

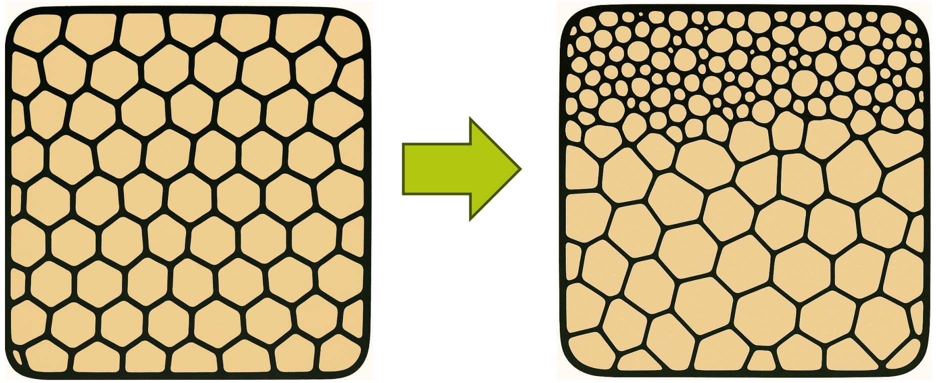
❖ **Key microstructure features for volume SPD**
- Formation of ultrafine-grained (UFG) or nanocrystalline structures throughout the bulk.
- Extremely high dislocation density leading to grain subdivision.
- Development of high-angle grain boundaries after severe deformation.

❖ **Key microstructure features for surface SPD**
- Formation of a nanocrystalline surface layer.
- Gradient microstructure: nanograins at the top
- Introduction of compressive residual stresses.

Volume SPD



Surface SPD



Surface thermochemical treatments

❖ How it works

- Atoms (C, N, B…) diffuse from the surface into the material at high temperature.
- The surface composition is modified

Surface thermochemical treatments

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- Atoms (C, N, B…) diffuse from the surface into the material at high temperature.
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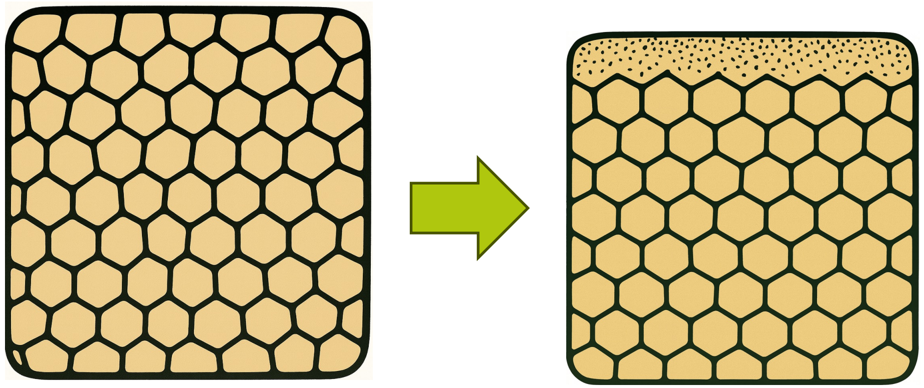
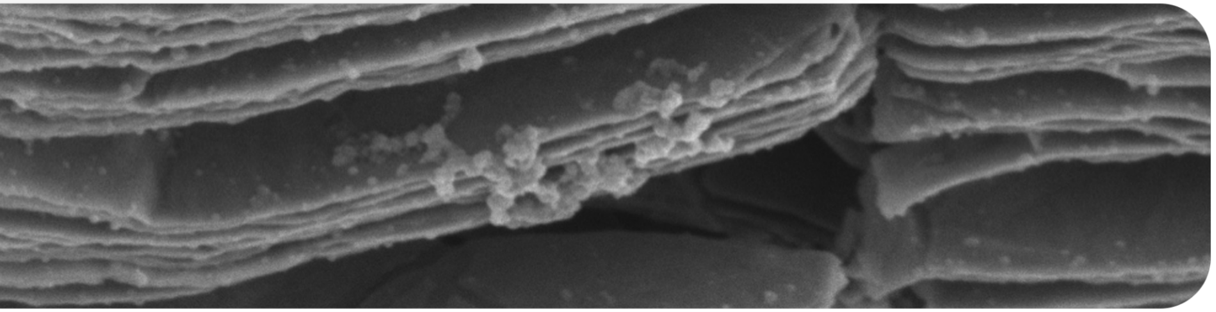
❖ Key microstructural features

- Composition gradient from surface to core.
- Presence of fine precipitates (carbides, nitrides, borides…) within the diffusion zone.
- Possible formation of a compound layer at the extreme surface.
- Grain refinement or transformation products near the surface (e.g., martensite).
- Introduction of compressive residual stresses in the modified layer.





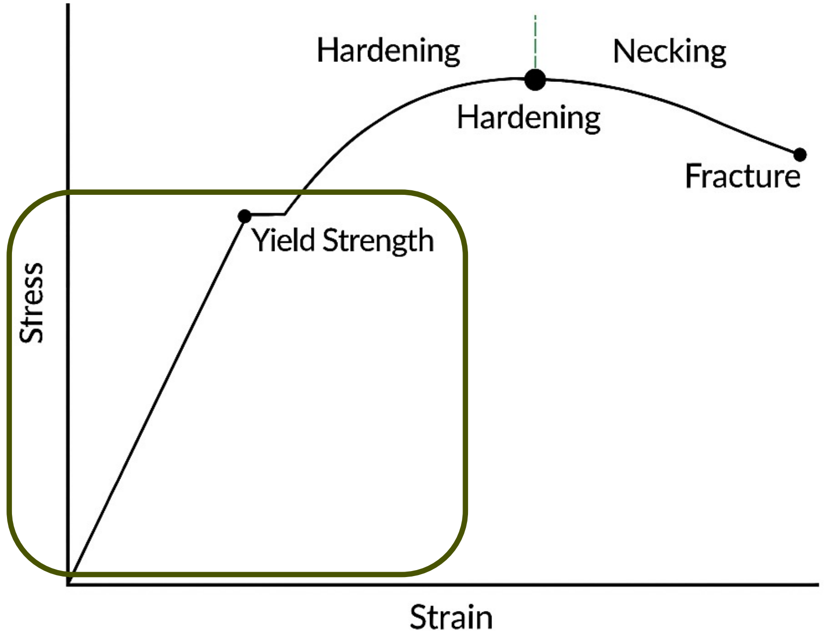
How microstructure controls mechanical behavior?

Essentials of elasticity

❖ **1D Hooke's law:**

$$\sigma = E\varepsilon$$

σ: stress; ε: strain; E: Young's modulus



Essentials of elasticity

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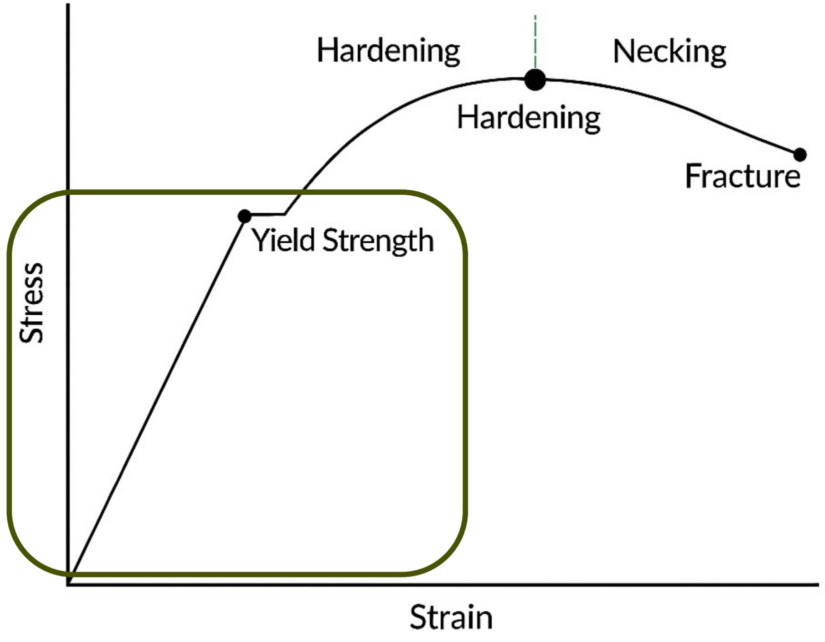
- **1D Poisson's law:**

$$\varepsilon_{\perp} = -\nu\varepsilon_{\parallel}$$

$\varepsilon_{\perp}$: transversal strain; $\varepsilon_{\parallel}$: longitudinal strain; ν: Poisson coefficient



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- **3D generalization for isotropic materials:**

$$\sigma_{ij} = \lambda\delta_{ij}\,\mathrm{tr}(\boldsymbol{\varepsilon}) + 2\mu\varepsilon_{ij}$$

$\lambda = \frac{E\nu}{(1+\nu)(1-2\nu)}$; $\mu = \frac{E}{2(1+\nu)}$; σ_{ij}: stress tensor; ε_{ij}: strain tensor

- **Strain tensor:**

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

(u_i: displacement field)

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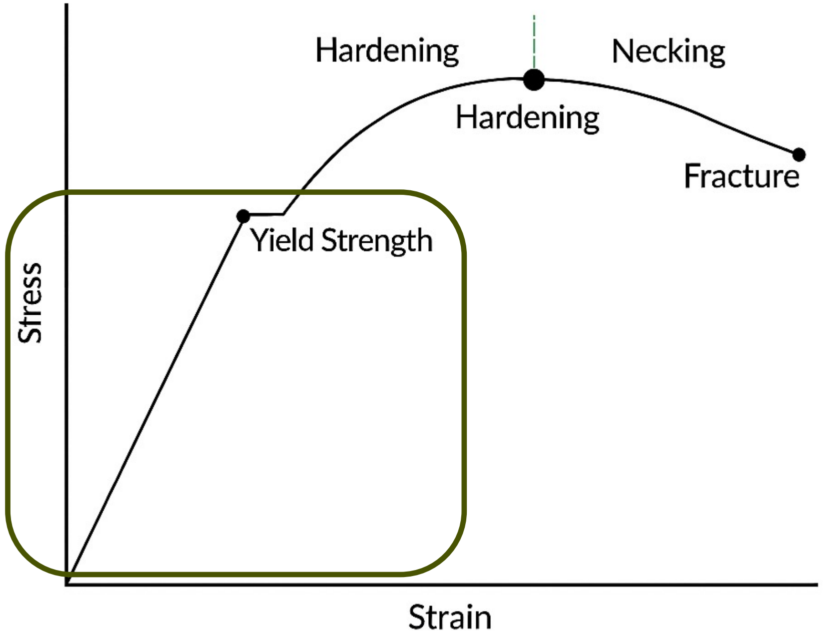
❖ **3D generalization for anisotropic materials:**

$$\boldsymbol{\sigma} = \mathbb{C} : \boldsymbol{\varepsilon} \Leftrightarrow \sigma_{ij} = C_{ijkl}\varepsilon_{kl}$$

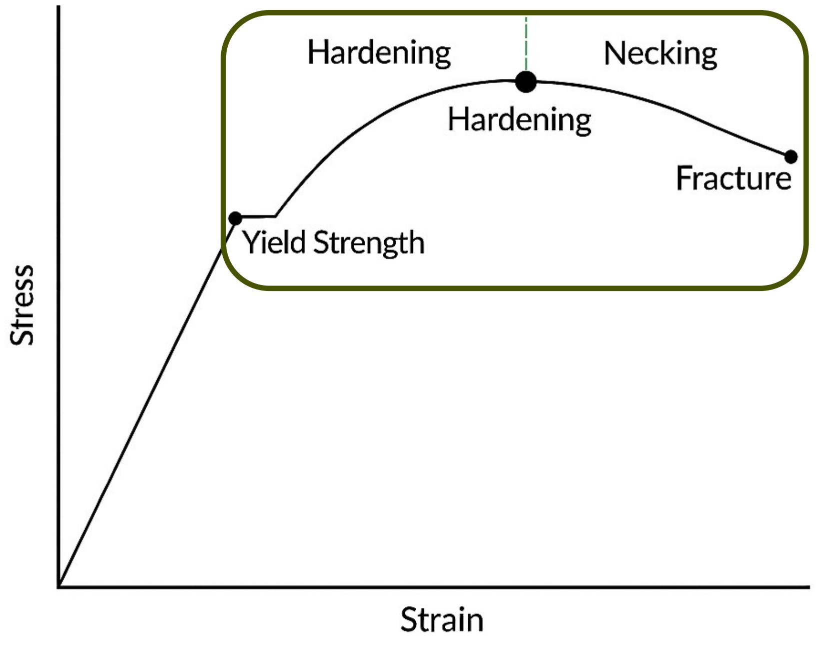
$\mathbb{C}, C_{ijkl}$: stiffness tensor



First law of plasticity

❖ **Historical context:**

- 1909: Paul Ludwik introduced the first empirical law describing the plastic behavior of ductile metals.
- At the time, most materials science focused on elasticity; Ludwik was among the first to address permanent deformation quantitatively.



P. Ludwik, Elemente der technologischen Mechanik, *Springer, Berlin* (1909)

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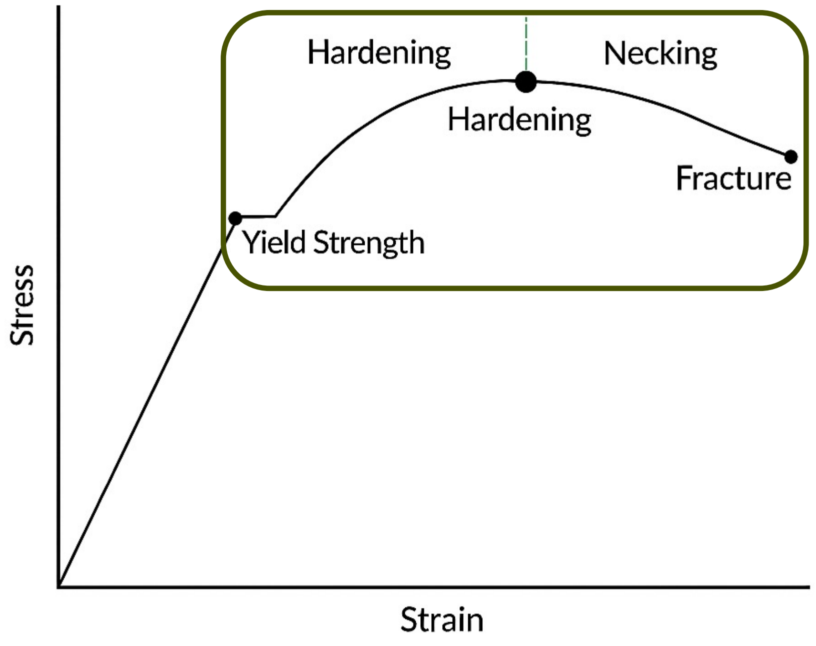
- At the time, most materials science focused on elasticity; Ludwik was among the first to address permanent deformation quantitatively.

❖ **Ludwik's law (1909):**

$$\sigma_T = \sigma_0 + K\varepsilon_T^n$$

$\sigma_T = \frac{F}{A_{inst}}$: true stress; $\varepsilon_T = \int_{L_0}^{L} \frac{\mathrm{d}L}{L} - \varepsilon_e$: true plastic strain;
K: strength coefficient; n: strain hardening exponent;
σ_0: yield stress (threshold for plastic flow)

- If $\sigma_0 = 0 \Rightarrow \sigma_T = K\varepsilon_T^n$ (Hollomon's law)



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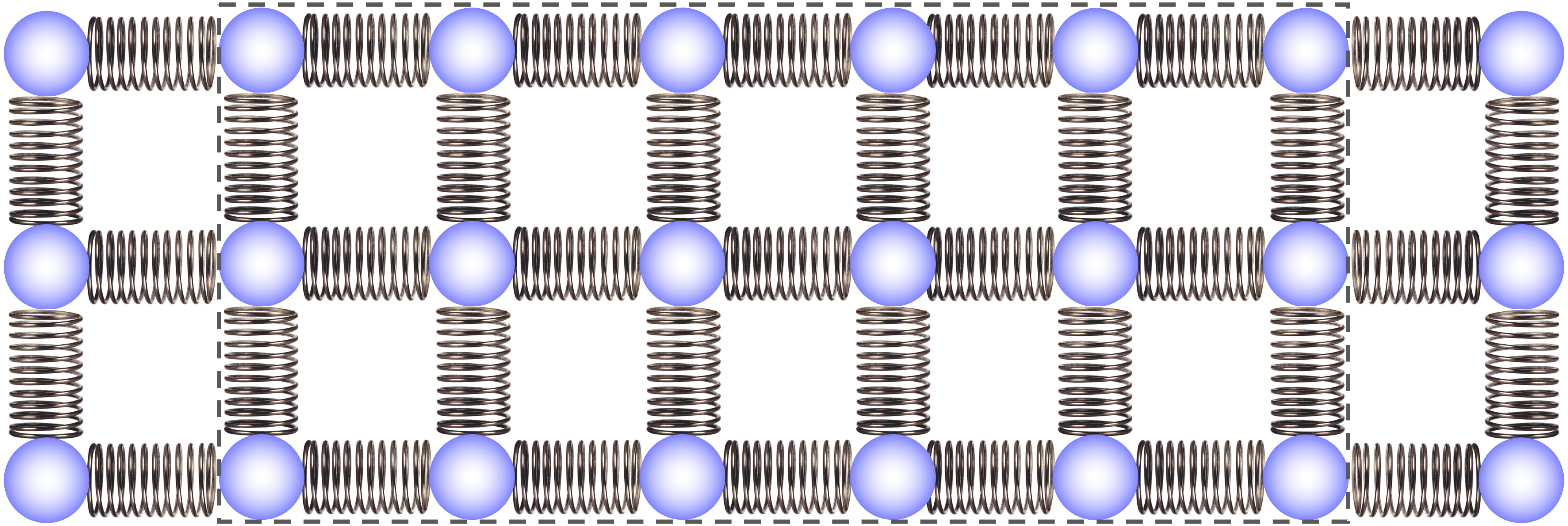
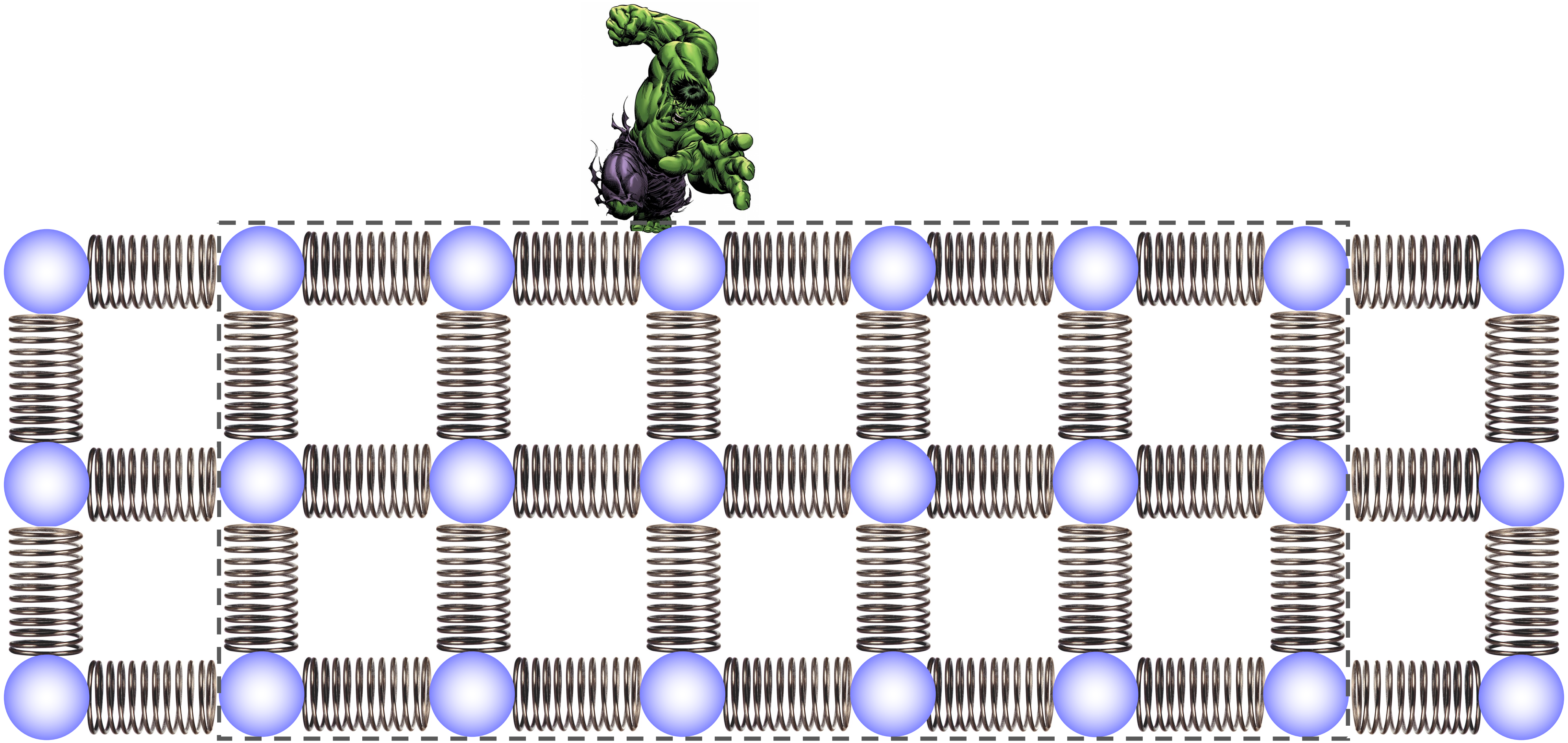
Dislocations are the carriers of plastic deformation

Initial size of the structure $L \times l \times h$



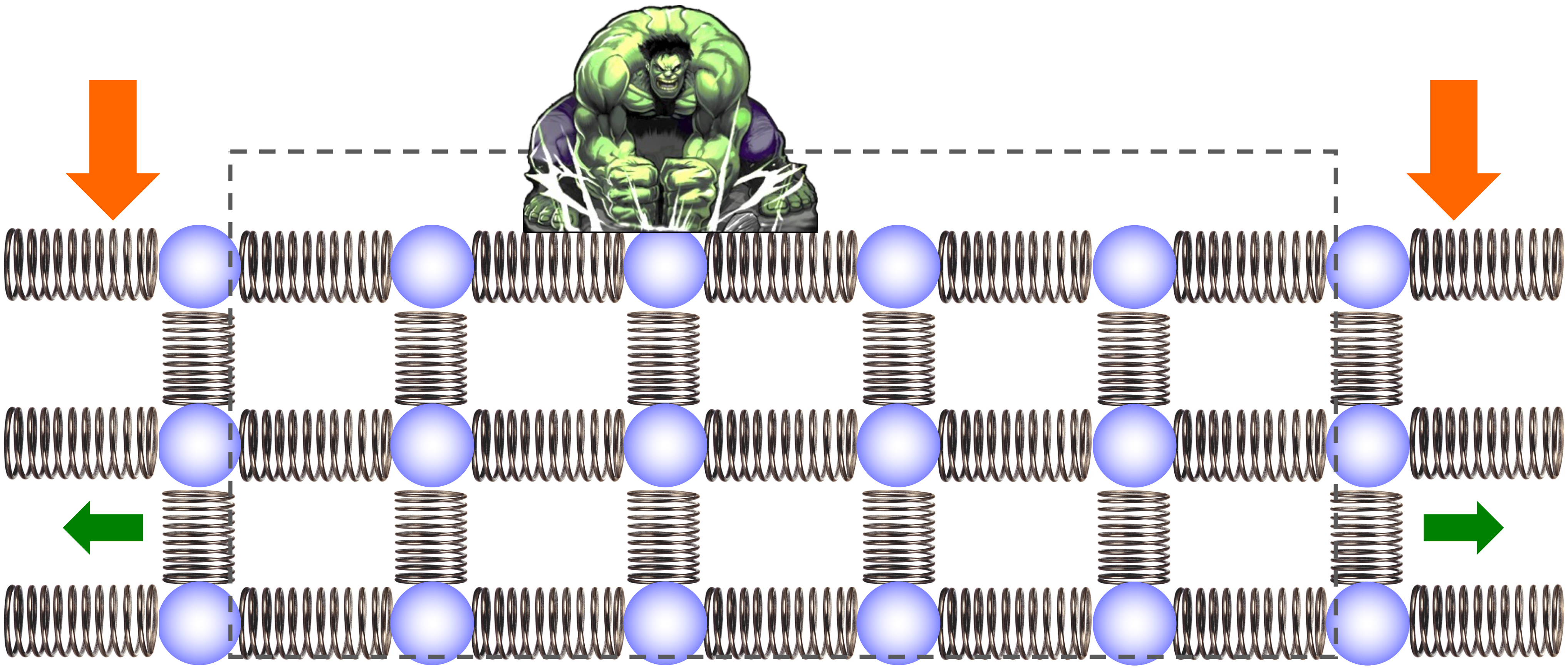
Dislocations are the carriers of plastic deformation

Application of a vertical load



Dislocations are the carriers of plastic deformation

Under the load, the vertical springs are compressed and the horizontal ones are stretched.



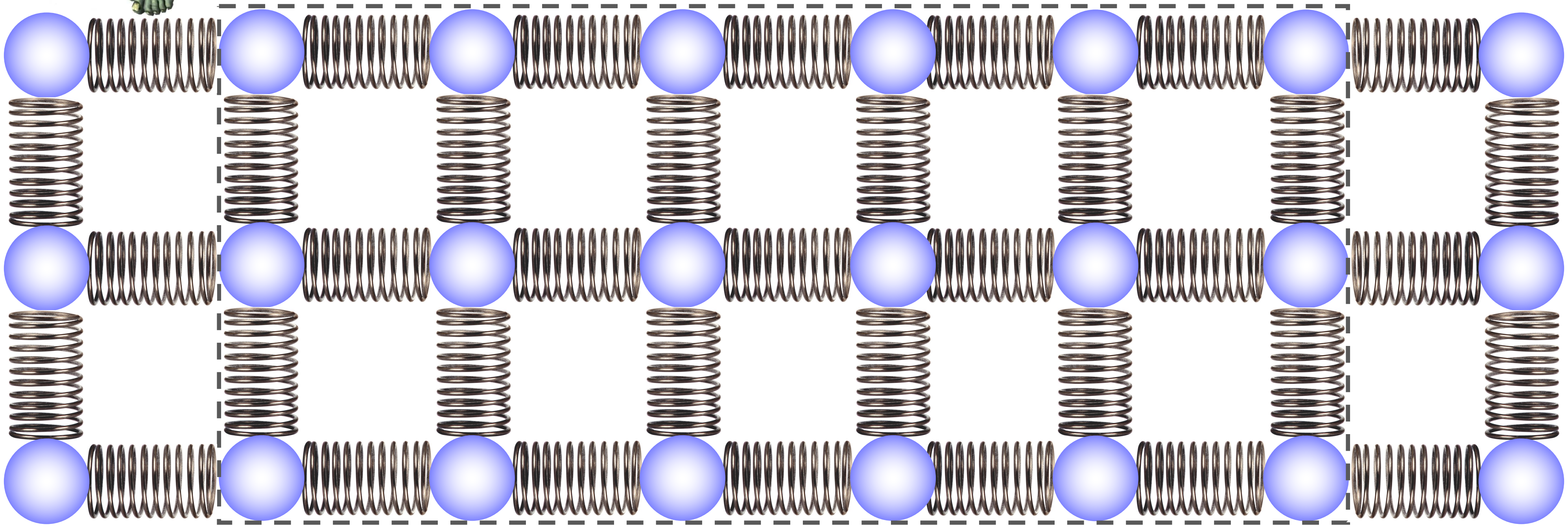
Dislocations are the carriers of plastic deformation

When the load is removed, the structure returns to its original dimensions.

Reversible deformation

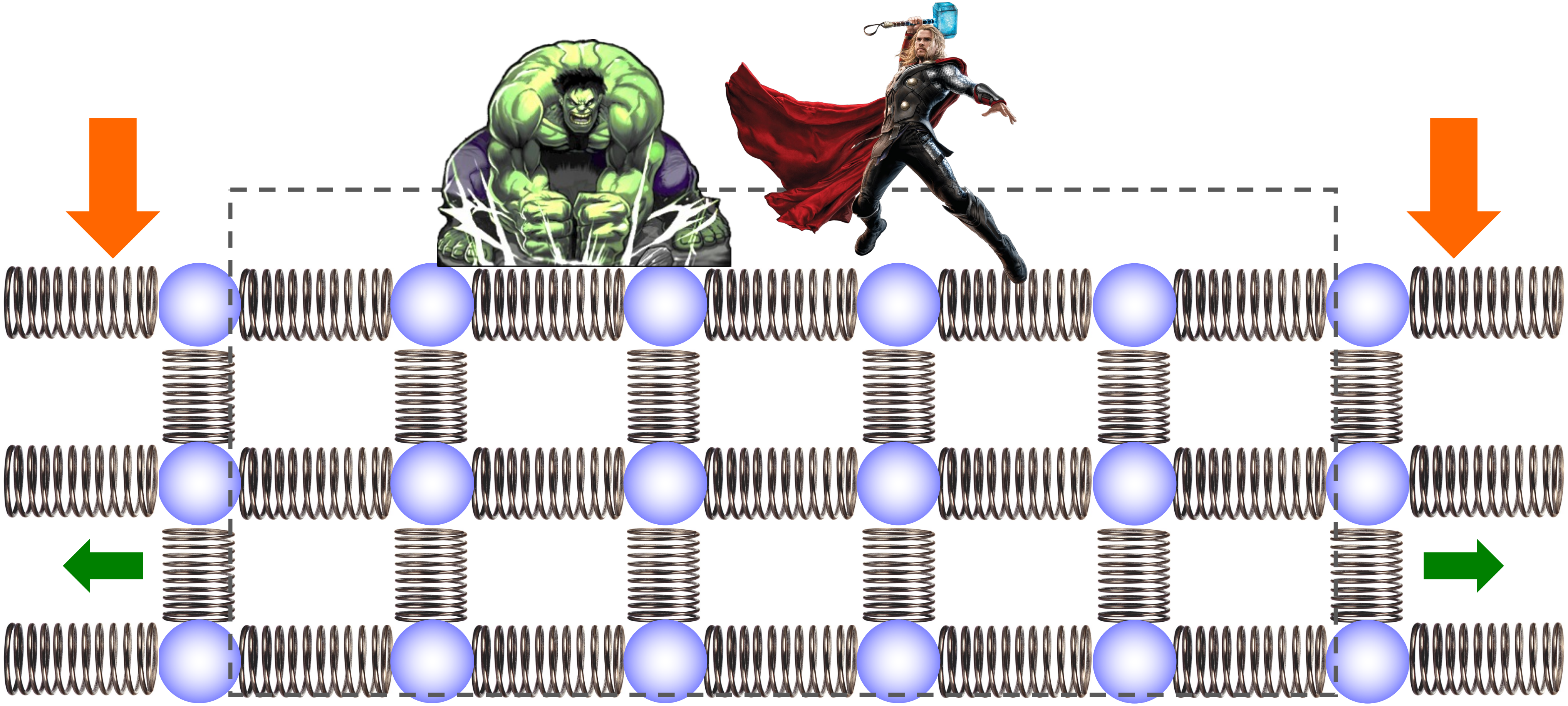
=

Elastic deformation



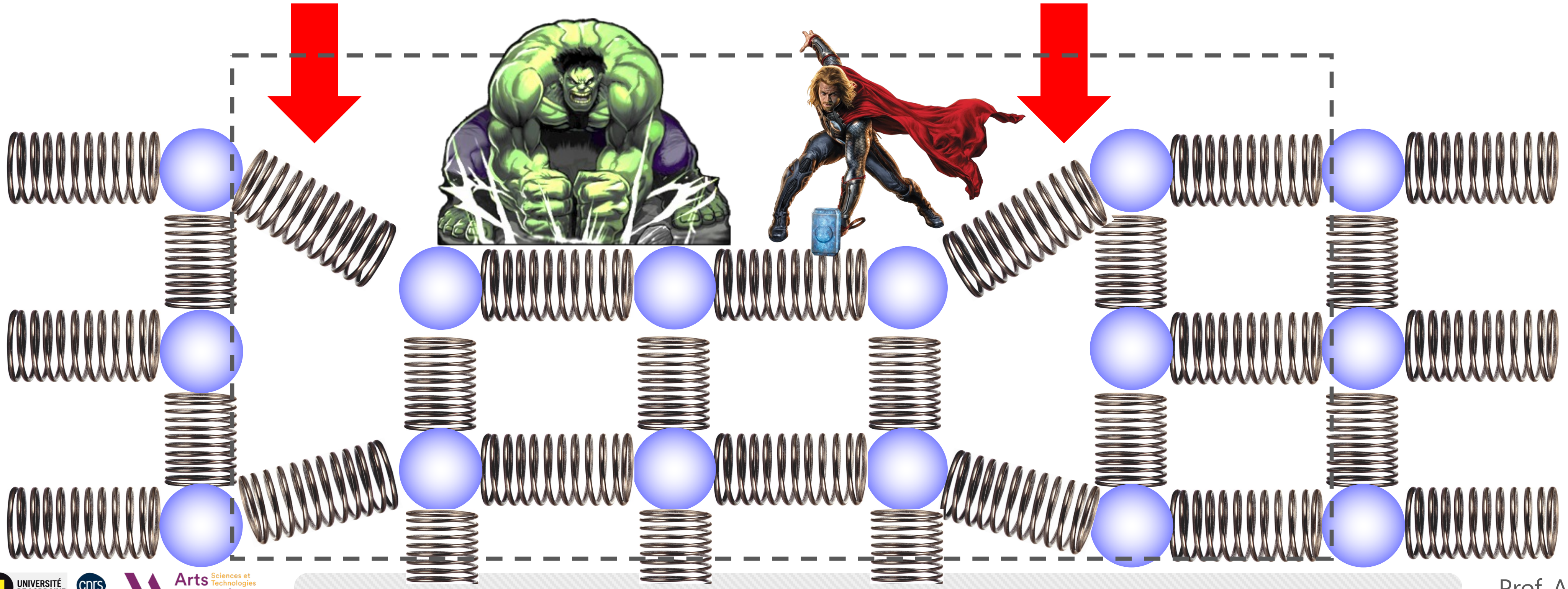
Dislocations are the carriers of plastic deformation

Under the load, the vertical springs are compressed and the horizontal ones are stretched.
A higher load is applied.



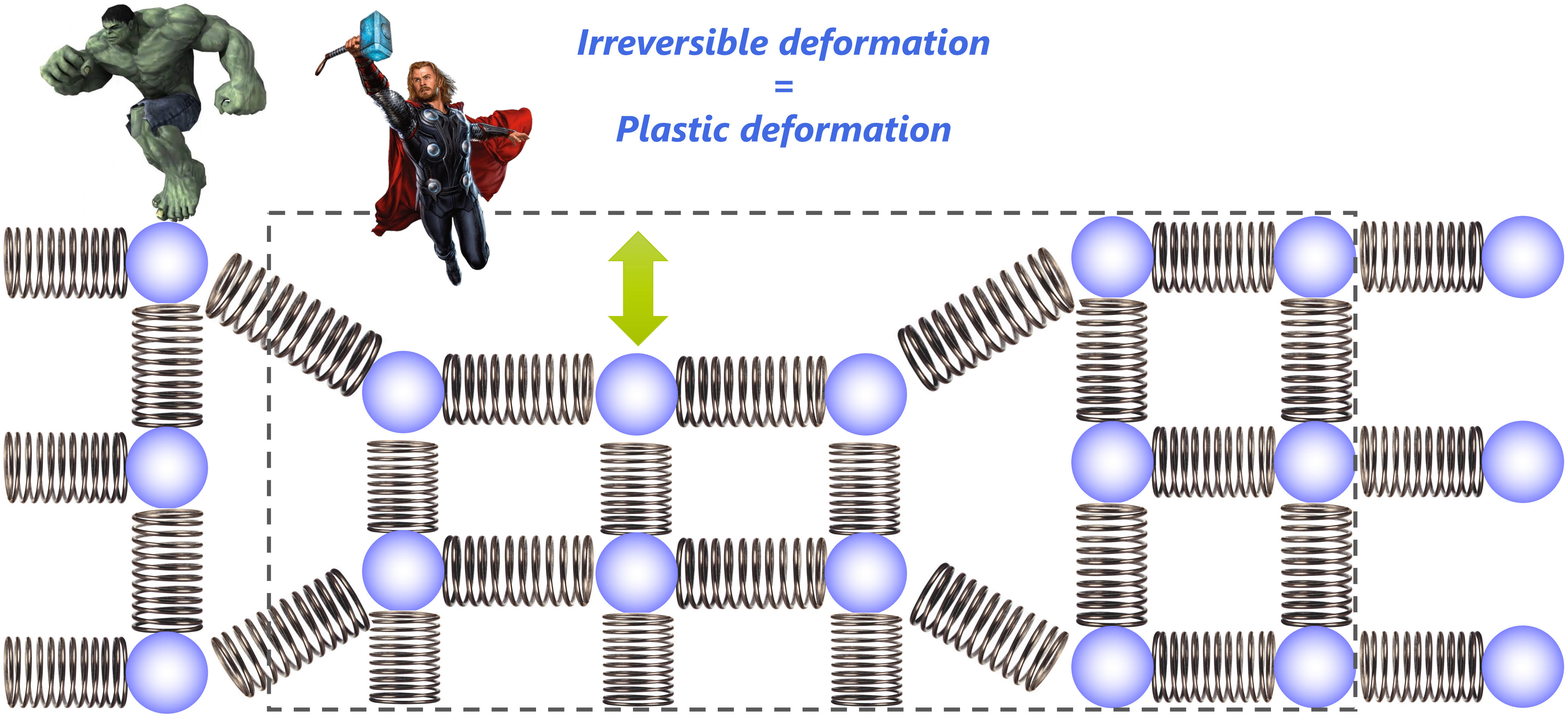
Dislocations are the carriers of plastic deformation

Under the load, the vertical springs are compressed and the horizontal ones are stretched.
Some springs are broken.



Dislocations are the carriers of plastic deformation

Since some springs are broken, the structure does not return to its original shape.



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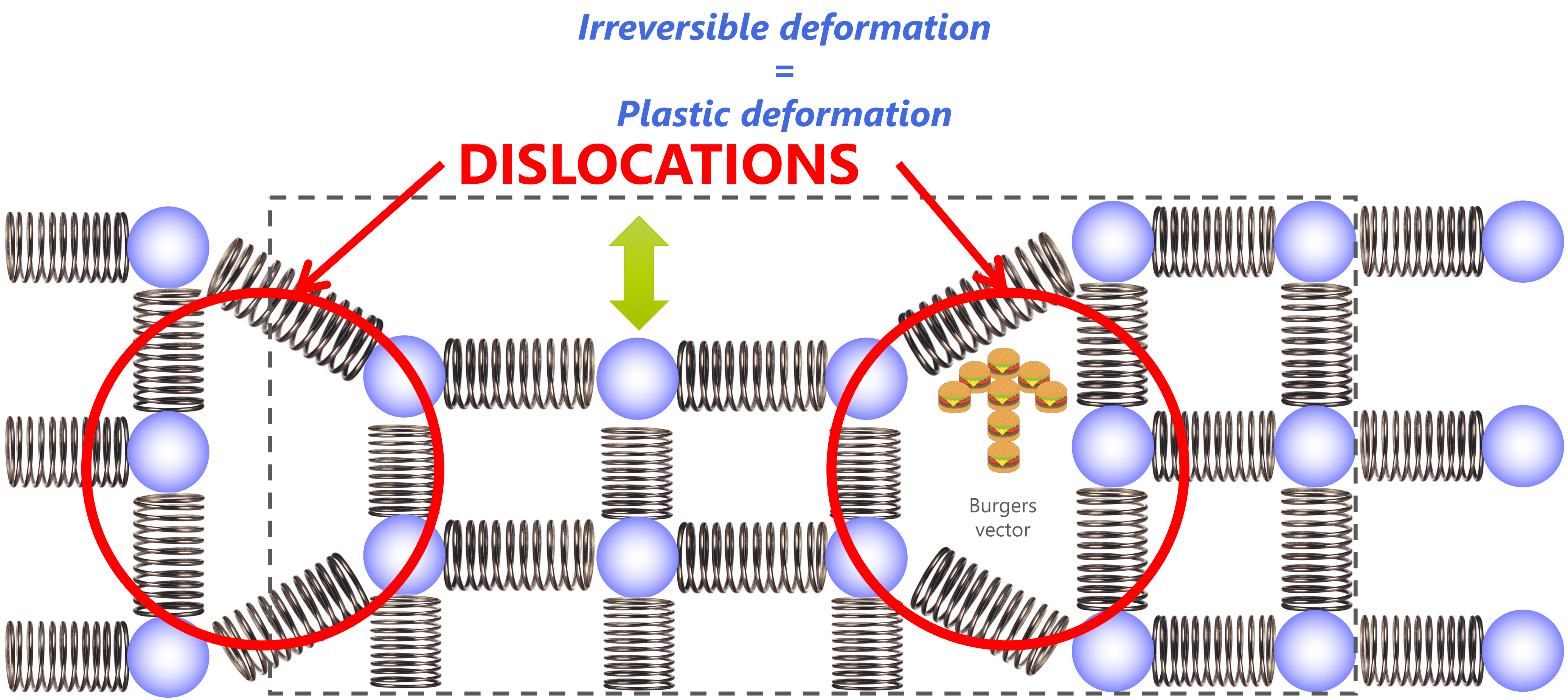
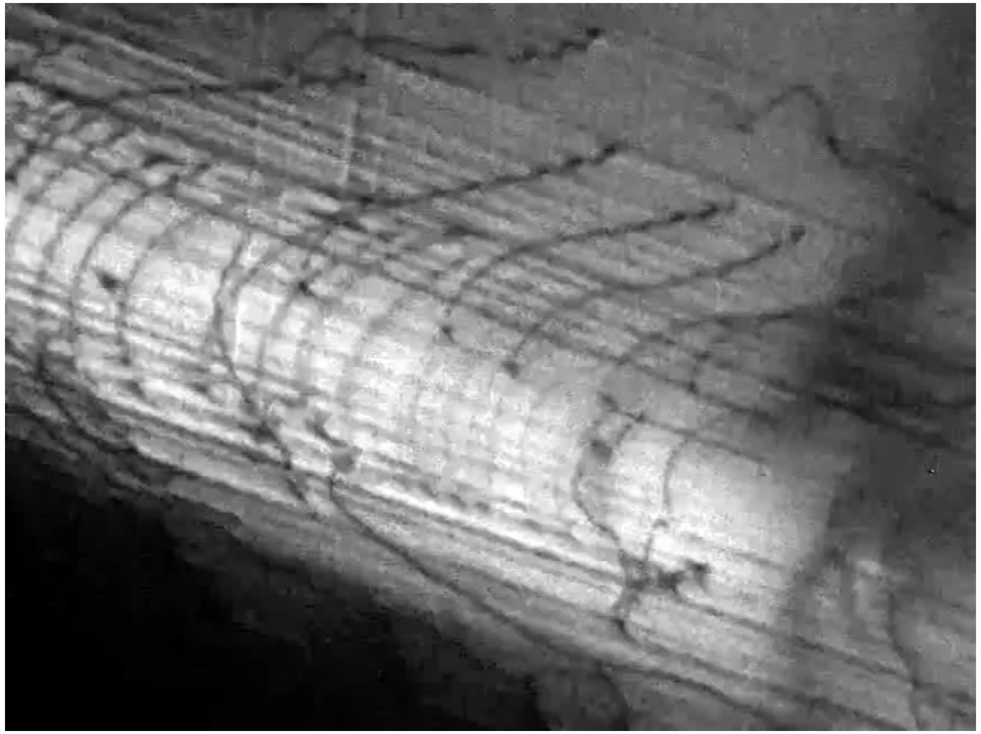


Dislocation glide

Dislocation glide



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

Dislocation glide

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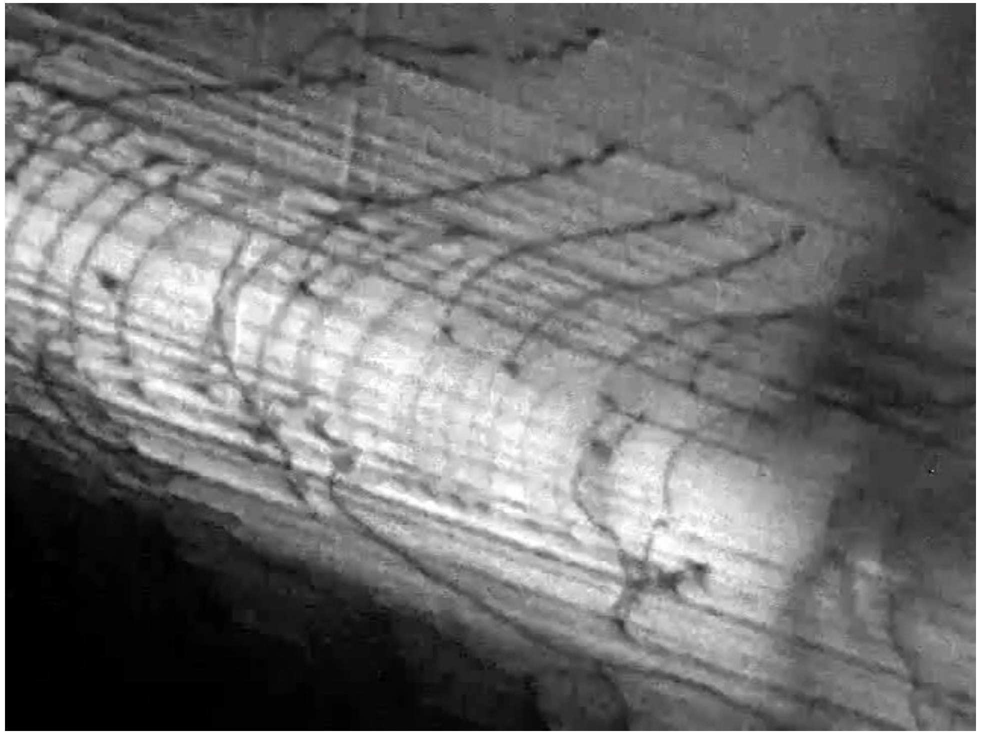
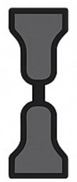


Forest interaction

↳Blocking dislocation glide ⇒ Strength increases

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

The yield strength ($\sigma_{0.2}$/ $R_{p\,0.2}$)



Material class	Approx. yield strength (MPa)
Foams	0.01–5
Polymers	10–100
Aluminum alloys	150–400
Steels	250–1500
Titanium alloys	800–1200
Composites (carbon)	500–1500
Ceramics	n.a.

- **Definition:**
  - The stress at which a material begins to deform plastically, as determined by the 0.2% offset method.
  - Below this point, deformation is considered elastic and reversible.
- **Unit:** Pa
- **Determines the maximum stress a material can withstand without permanent deformation**

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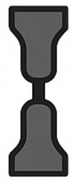

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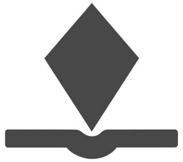
- **Relationship with microstructural features:**
  - Grain size: Smaller grains ⇒ $\sigma_{0.2}$ ↗ (Hall-Petch).

    $$\sigma_{0.2} = \sigma_0 + Kd^{-\frac{1}{2}}$$
  - Dislocation density: Higher density ⇒ $\sigma_{0.2}$ ↗ (work hardening).

    $$\sigma_{0.2} = \alpha G b \sqrt{\rho}$$
  - Precipitates/particles: Block dislocations ⇒ $\sigma_{0.2}$ ↗
  - Solid solution atoms: Lattice distortion impedes dislocations ⇒ $\sigma_{0.2}$ ↗

Hardness



Material class	Hardness (HV)
Foams	1-20
Polymers	5-30
Aluminum alloys	30-150
Steels	120-800
Titanium alloys	150-400
Composites (carbon)	50-200 (matrix); 800-1500 (fiber)
Ceramics	500-2000

- **Definition:**
  - Resistance of a material to local plastic deformation, typically measured by indentation (Vickers, Brinell, Rockwell).
- **Unit:** HV, HB, HRC, Pa
- **Importance in materials engineering:**
  - Provides a simple, reliable indicator of the microstructure (phases, strengthening, heat treatments).
  - Used for quality control, heat-treatment verification, and surface assessment.
  - Helpful when tensile testing is impractical or when only small volumes of material are available.
  - Critical for applications involving wear resistance, contact stresses, and surface durability.

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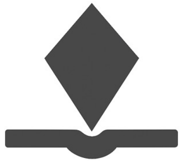

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- Precipitates/particles: Block dislocations $\Rightarrow H \nearrow$
- Solid solution atoms: Lattice distortion impedes dislocations $\Rightarrow H \nearrow$

The ultimate tensile strength (UTS)

Material class	Approx. UTS (MPa)
Foams	0.01–2
Polymers	20–150
Aluminum alloys	200–550
Steels	400–2000
Titanium alloys	900–1400
Composites (carbon)	600–2000 (direction-dependent)
Ceramics	100–1000

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  - The maximum stress a material can withstand in tension before necking or failure occurs.
  - It is the peak point on the engineering stress–strain curve.
- **Unit:** Pa

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- **Unit:** Pa
- **Relationship with microstructural features:**
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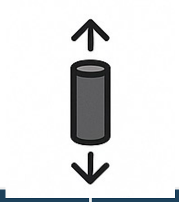
  - Dislocation density: Higher density ⇒ $UTS \nearrow$ (work hardening).
  - Precipitates/particles: Block dislocations ⇒ $UTS \nearrow$
  - Solid solution atoms: Lattice distortion impedes dislocations ⇒ $UTS \nearrow$

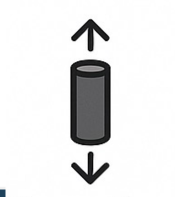
Ductility: elongation ($\%EL$)



Material class	Approx. %EL (%)
Foams	< 5
Polymers	10–800
Aluminum alloys	5–20
Steels	10–40
Titanium alloys	10–30
Composites (carbon)	1–2 (direction-dependent)
Ceramics	< 1

- **Definition:**
  - The strain at fracture *i.e.*, how much a material stretches before breaking under tension
  - It includes both elastic and plastic deformations.
- **Unit:** %

Ductility: elongation ($\%EL$)



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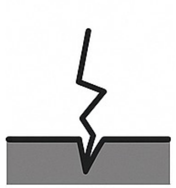
  - It includes both elastic and plastic deformations.
- **Unit:** %
- **Relationship with microstructural features:**
  - Grain size: Larger grains ⇒ $\%EL$ ↗
  - Dislocation density: Higher density ⇒ $\%EL$ ↘
  - (less ability to accommodate plastic strain).
  - Precipitates/particles: Crack initiation sites⇒ $\%EL$ ↘
  - Solid solution atoms: hindering dislocation motion ⇒ $\%EL$ ↘

The fracture toughness (K_{1C})



Material class	Approx. K_{1C} ($MPa{\cdot}m^{1/2}$)
Foams	< 0.05
Polymers	1–5
Aluminum alloys	20–40
Steels	50–150
Titanium alloys	40–100
Composites (glass fibers)	10–30 (in plan)
Ceramics	0.5–5

- **Definition:**
  - The ability of a material containing a crack to resist fracture
  - Mode I critical stress intensity factor

$$K_{1C} = Y\sigma_r\sqrt{\pi a_c}$$

- **Unit:** $MPa{\cdot}m^{1/2}$

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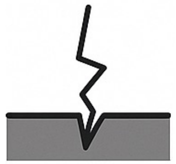

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$$K_{1C} = Y\sigma_r\sqrt{\pi a_c}$$

- **Unit:** $MPa \cdot m^{1/2}$
- **Relationship with microstructural features:**
  - Grain size: Larger grains ⇒ K_{1C} ↗ (more crack deflection and energy absorptions).
  - Dislocation density: Very higher density ⇒ K_{1C} ↘ (reduced ability to blunt cracks).
  - Precipitates/particles: H ard particules ⇒ K_{1C} ↘ (crack initiation sites)
  - Solid solution atoms: K_{1C} ↗ or K_{1C} ↘

Fatigue resistance

Material class	Fatigue strength at 10^7 cycles (MPa)
Foams	< 1
Polymers	2-10
Aluminum alloys	20–80 (no true fatigue limit)
Steels	130-500 (no true fatigue limit)
Titanium alloys	150-400
Composites (glass fibers)	300-800 (in fiber direction)
Ceramics	<10

- **Definition:**
  - The ability of a material to withstand repeated cyclic loading without failing.
  - Characterized by the fatigue limit (for steels and some Ti alloys) or by S–N curves showing stress amplitude vs. number of cycles to failure.
  - Failure occurs by initiation and propagation of microcracks driven by cyclic plasticity.
- **Unit:** stress amplitude (MPa) specified at a given number of cycles (N).

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  - Failure occurs by initiation and propagation of microcracks driven by cyclic plasticity.
- **Unit:** stress amplitude (MPa) specified at a given number of cycles (N).
- **Relationship with microstructural features:**
  - Grain size: Finer grains ⇒ better resistance to crack initiation.
  - Dislocation density: Very high density reduces fatigue resistance (more initiation sites).
  - Precipitates/particles: Can improve or degrade resistance depending on size and coherency.
  - Solid solution: May enhance resistance by stabilizing the microstructure.

Creep resistance (high-temperature deformation)

Material class	Creep strength (MPa) at high T (typical range)
Foams	Negligible
Polymers	< 1 at moderate T
Aluminum alloys	5-20 (≈ 150-250°C)
Steels	50-150 (≈ 500-600°C)
Titanium alloys	80-200 (≈ 400-500°C)
Composites (glass fibers)	150-300+ (≈ 700-900°C)
Ceramics	Limited usable creep due to brittleness

- **Definition:**
  - The ability of a material to resist time-dependent plastic deformation under constant load at elevated temperature.
  - Creep behavior is described by a stress–strain–time relationship and depends strongly on temperature, microstructure, and diffusion-controlled mechanisms.
- **Unit: S**tress (MPa) required to cause a specified amount of creep (e.g. 1% creep strain or rupture at 10^4-10^5 h).

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Ceramics	Limited usable creep due to brittleness

- **Definition:**
  - The ability of a material to resist time-dependent plastic deformation under constant load at elevated temperature.
  - Creep behavior is described by a stress–strain–time relationship and depends strongly on temperature, microstructure, and diffusion-controlled mechanisms.
- **Unit: S**tress (MPa) required to cause a specified amount of creep (e.g. 1% creep strain or rupture at 10^4-10^5 h).
- **Relationship with microstructural features:**
  - Grain size: Larger grains ⇒ higher creep resistance (fewer grain-boundary sliding paths).
  - Precipitates/particles: Stable high-temperature precipitates pin dislocations ⇒ improved creep strength.
  - Dislocation density: Low initial dislocation density is beneficial (reduces recovery and creep rate).
  - Solid solution: Solute atoms slow diffusion and dislocation motion ⇒ enhanced creep resistance.

Microstructure controls mechanical performance

- **Grain refinement**
  - Small grains ⇒ more GBs
    ⇒ dislocation blocking

  ⇒ **Yield strength increases (Hall–Petch effect)**

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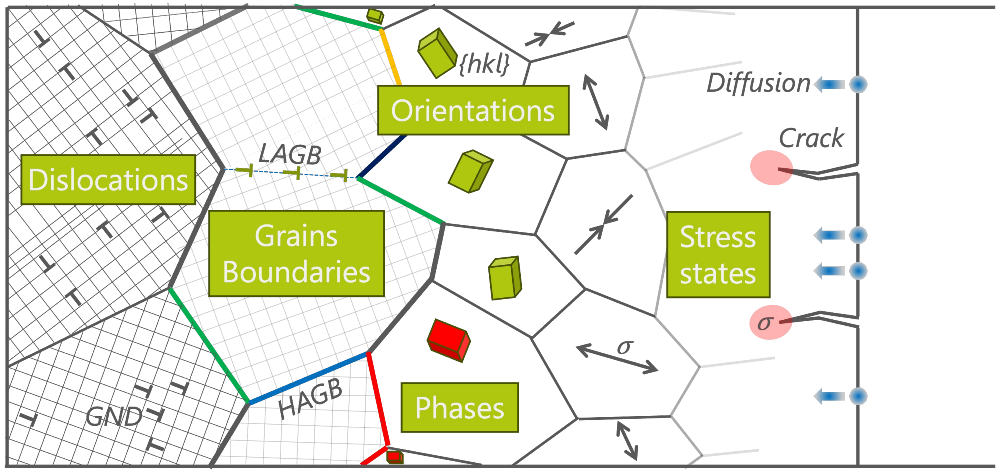
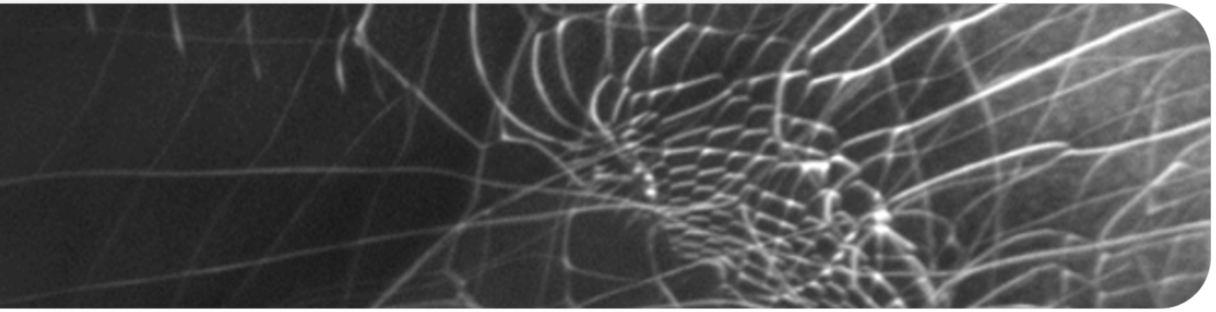
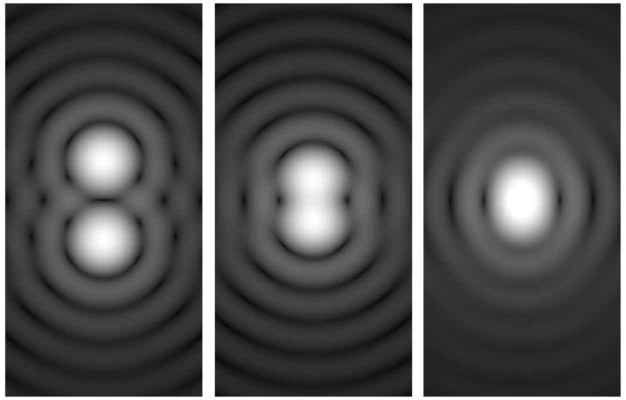




How we measure microstructure?

Limit of resolution



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

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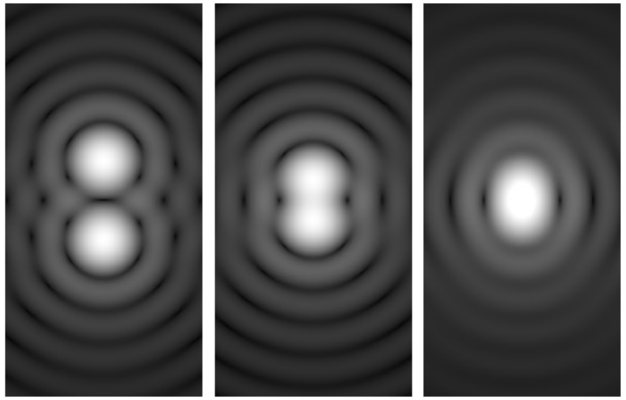

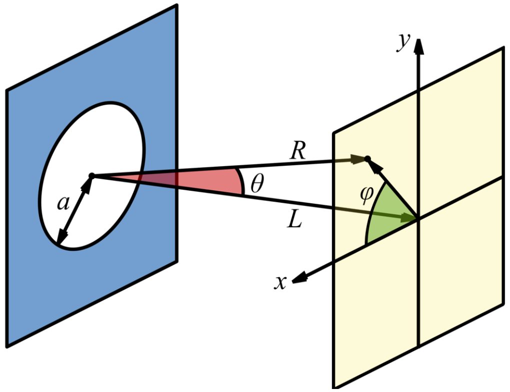

❖ Airy disk and Rayleigh criterion

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

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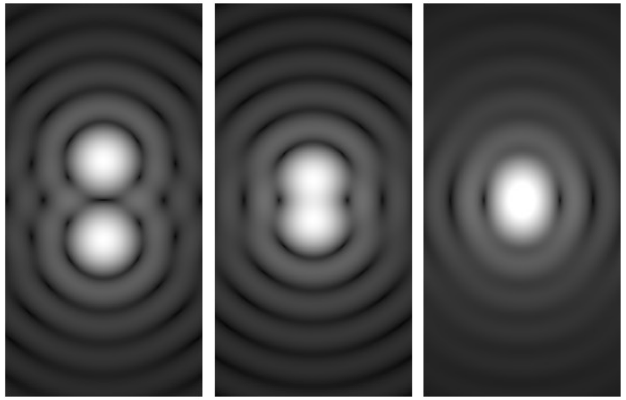

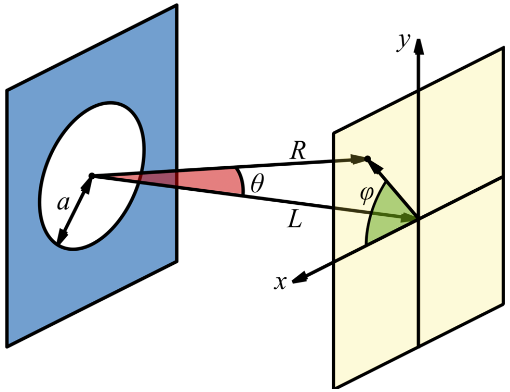

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$$\sin\theta = 1.22\frac{\lambda}{a} \sim \theta$$

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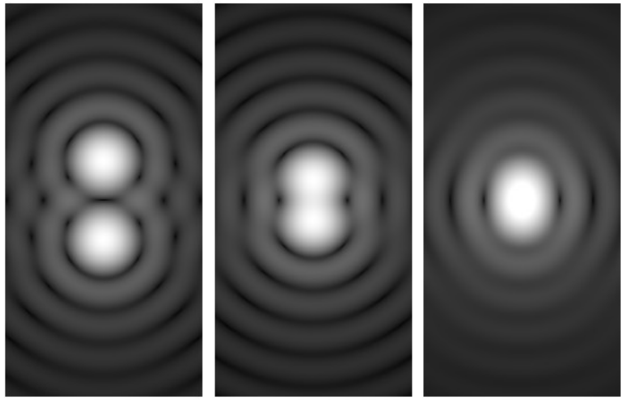

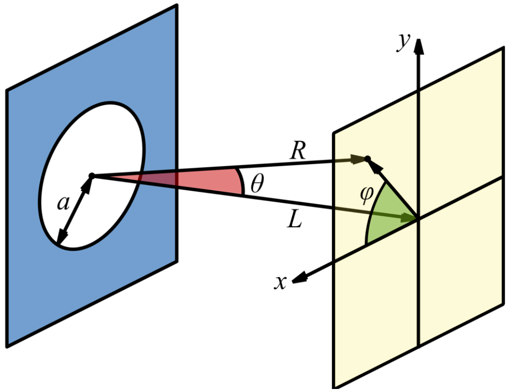

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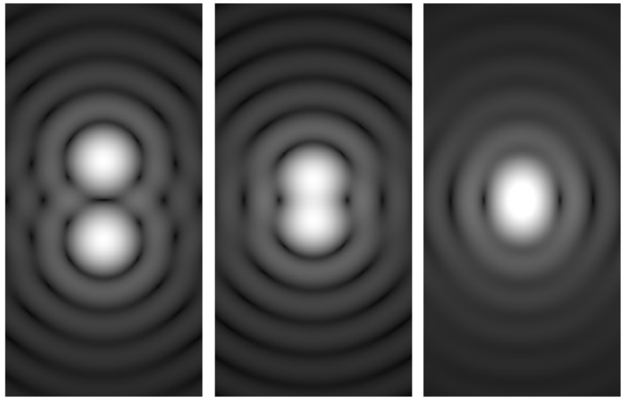

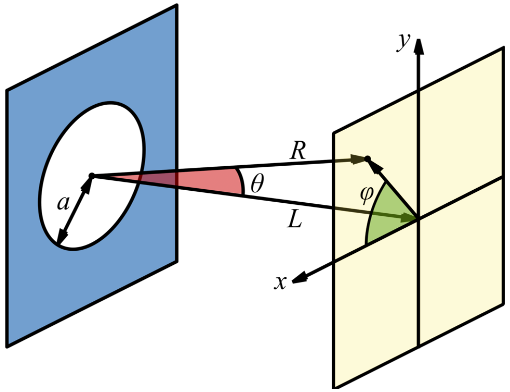

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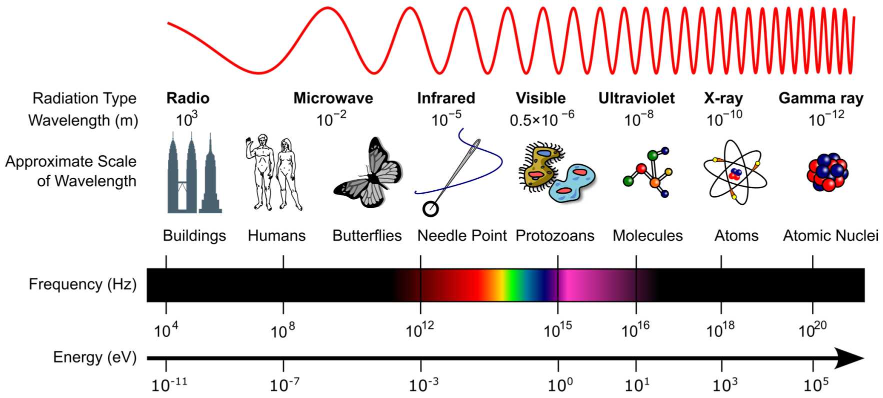
↳ Two objects cannot be resolved if they are closer than one wavelength λ.

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 removed from atoms or molecules, thereby ionizing them ($E_{ion}\sim$ 10 eV).

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



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

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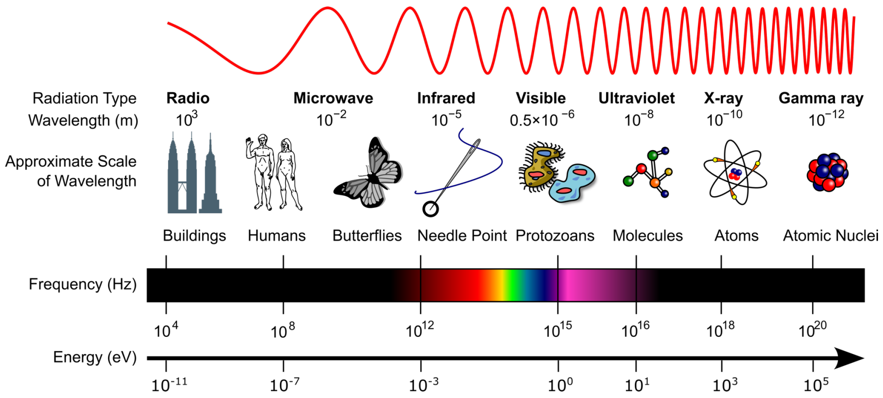

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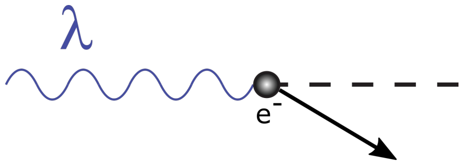
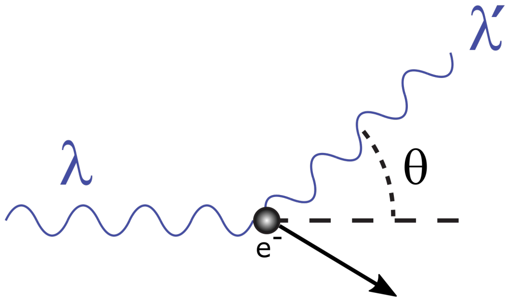
Photon-matter interaction

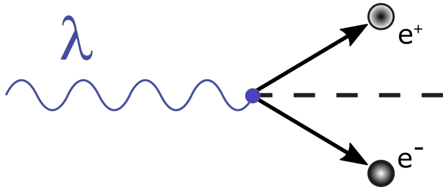
- **Photoabsorption effect:** A photon transfers all its energy to an electron, ejecting it if the energy exceeds the electron's binding energy.



- **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.

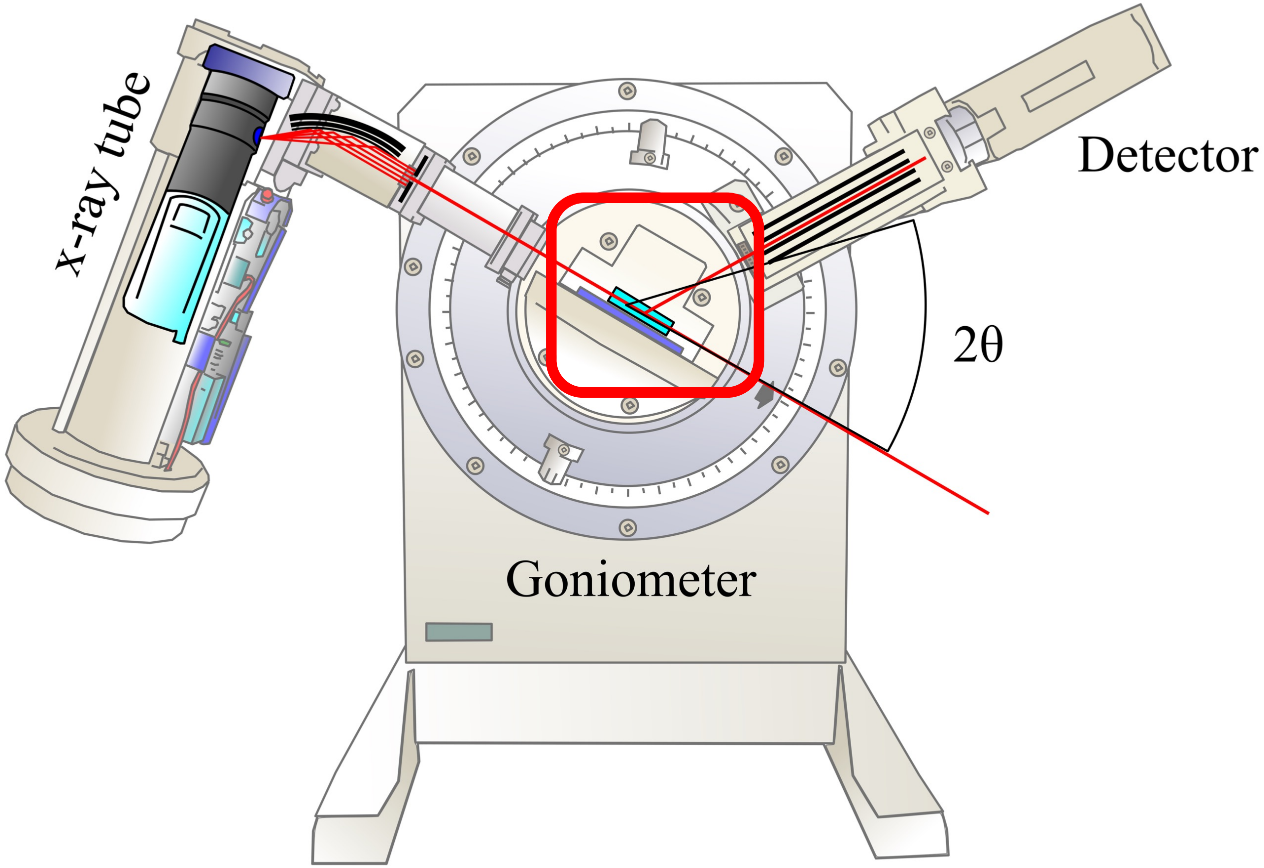


- **Pair production:** A high-energy photon interacts with an atom's nucleus and converts into an electron-positron pair.

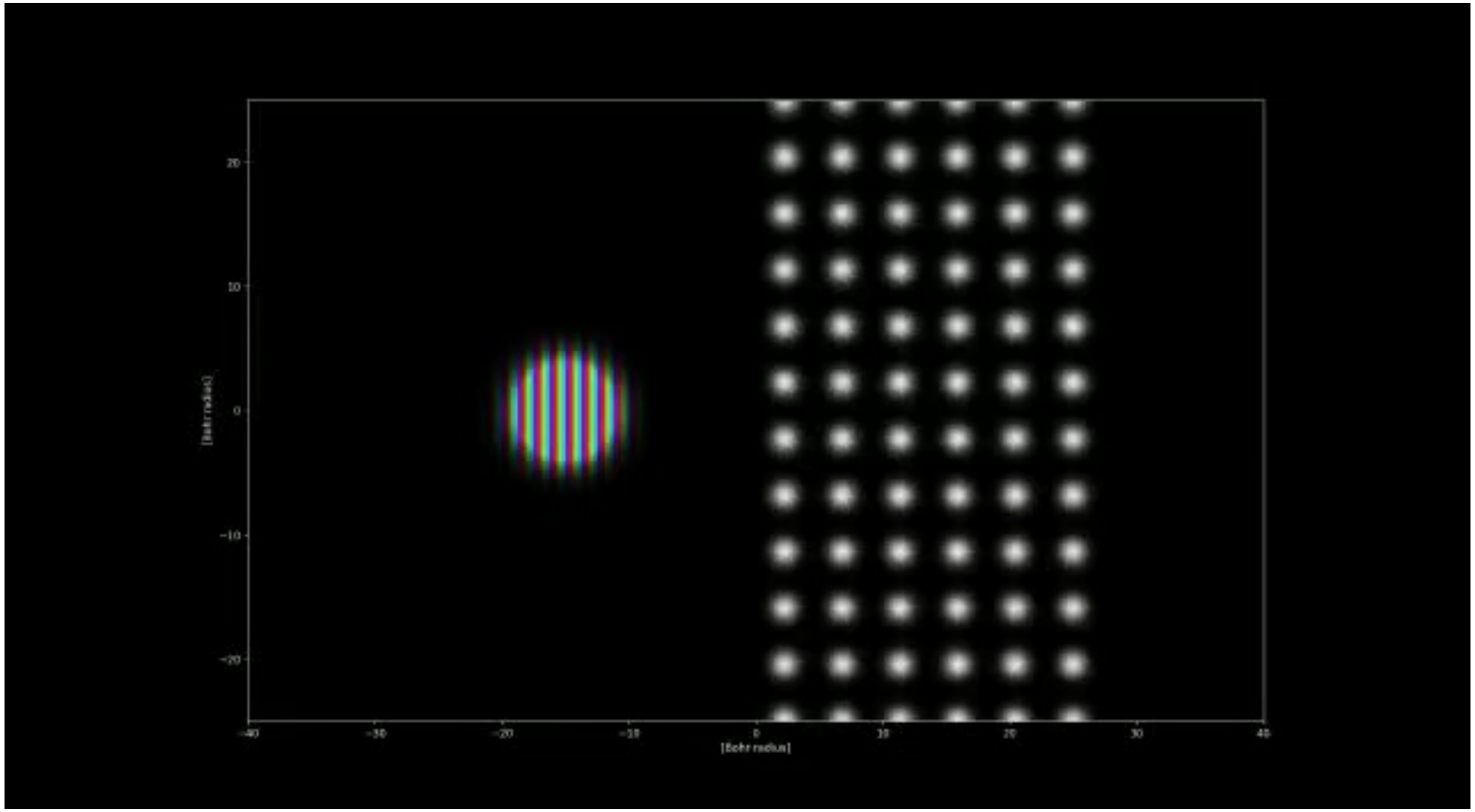


The experimental setup: the diffractometer

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.

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

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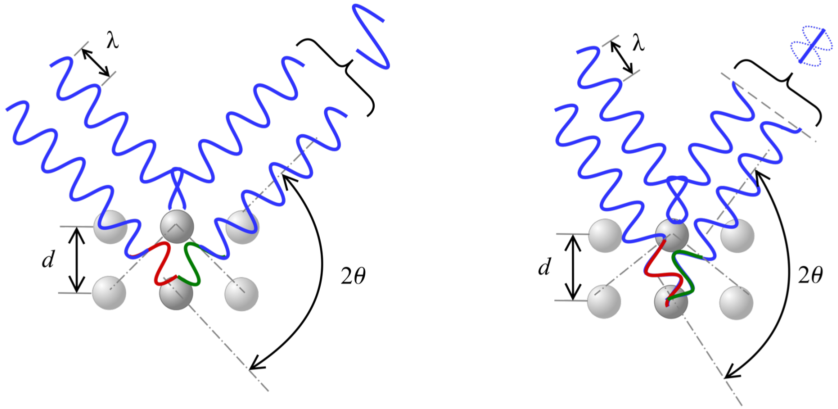

- **Liquids or amorphous materials:**
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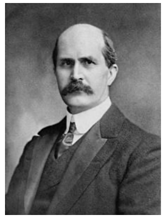
- **In crystalline materials:**
  - X-rays produce sharp and intense peaks of radiation at certain angles

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



William Henry BRAGG
(1862 – 1940)

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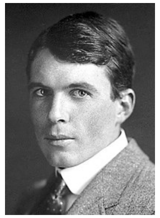
(1890 – 1971)

2/2 Nobel Prize (1915)



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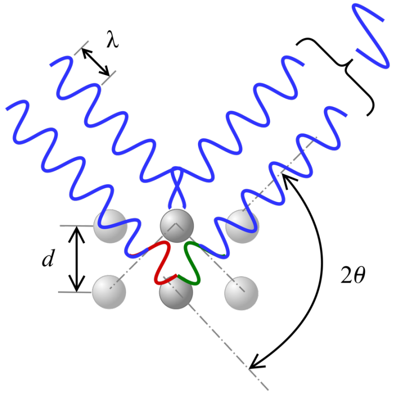

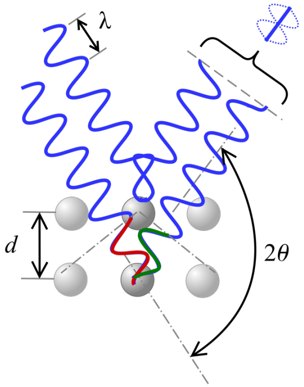

↳ $n\lambda = 2d\sin\theta$

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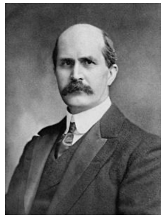

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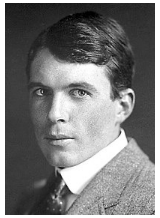

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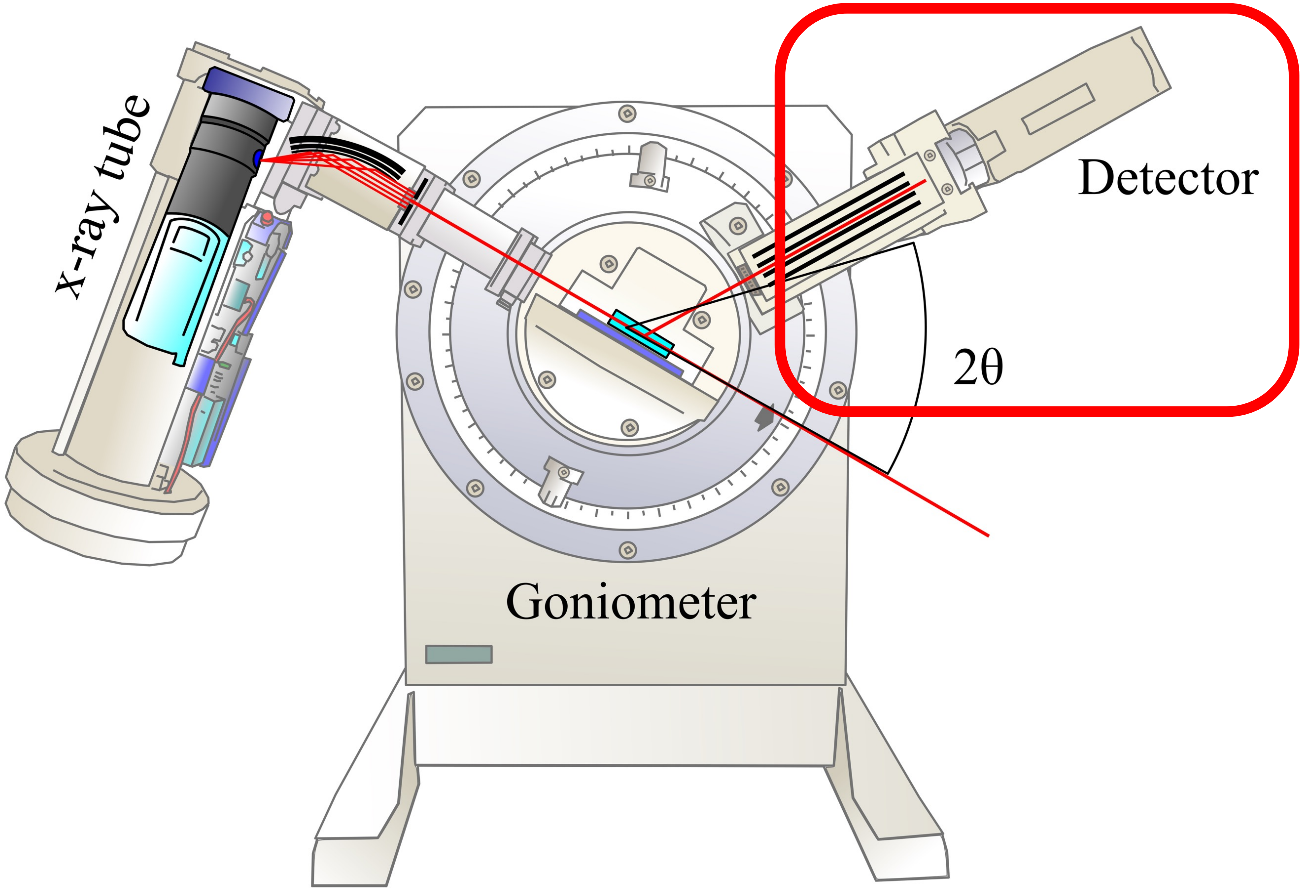


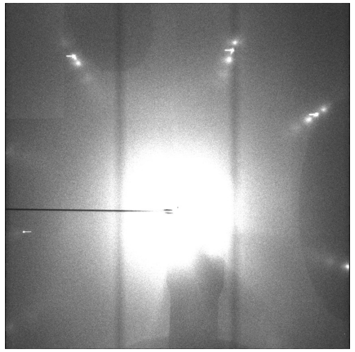
X-ray diffraction techniques

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**



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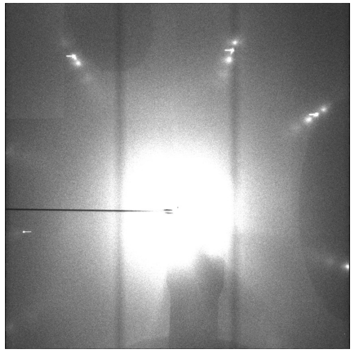

Monochromatic beam

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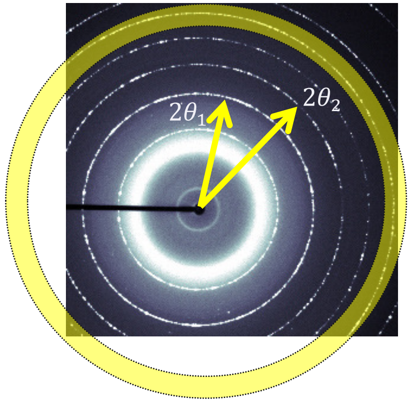
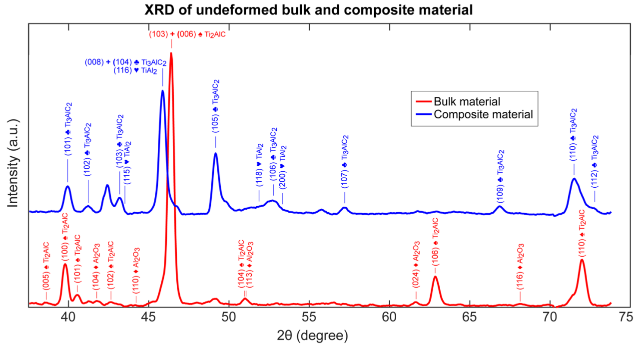
⇒ **Concentric rings or peak diffractograms**



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



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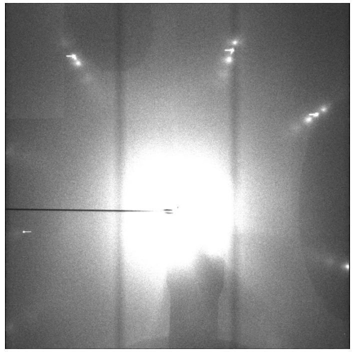

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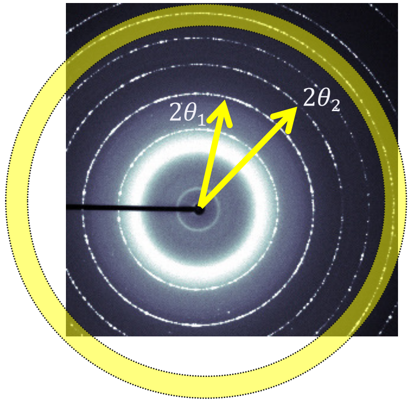

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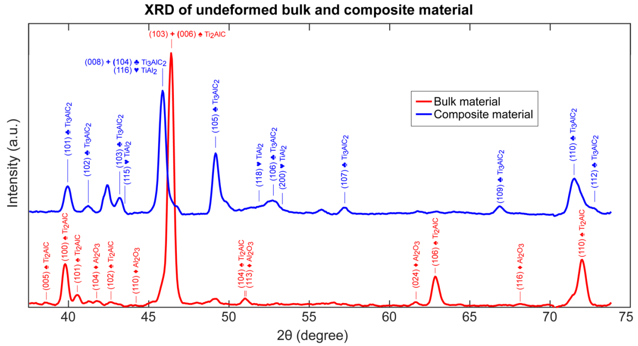
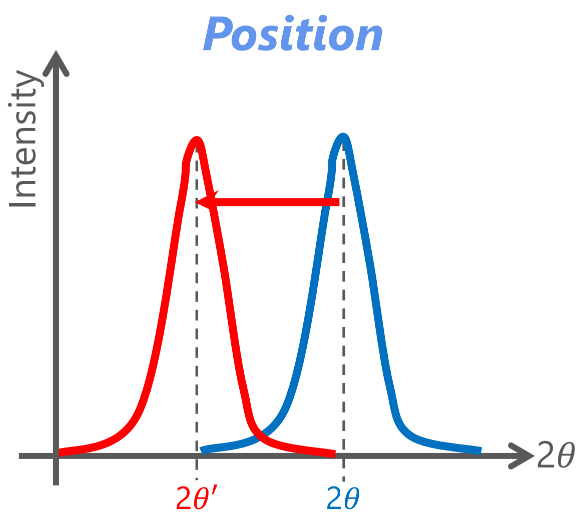


One crystal, many wavelengths – that's Laue.
Many crystals, one wavelength – that's powder.

Shape of the diffracted peaks

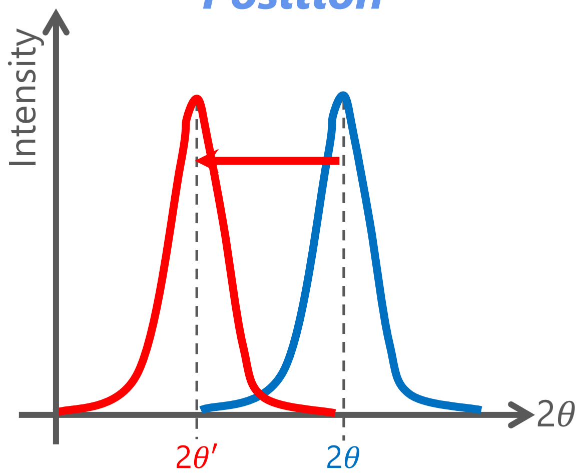


- **Peak shift:**
  - $n\lambda = 2d_{hkl}\sin\theta$
    - ✓ $2\theta \searrow \Rightarrow \sin\theta \searrow \Rightarrow d_{hkl} \nearrow \Rightarrow$ macrocompression
    - ✓ $2\theta \nearrow \Rightarrow \sin\theta \nearrow \Rightarrow d_{hkl} \searrow \Rightarrow$ macrotraction
- **Average elastic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

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

Shape of the diffracted peaks

Position



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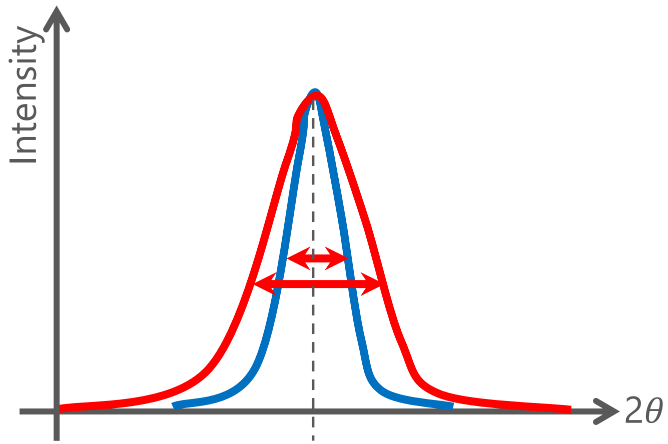


- **Peak width:**
  - Crystallite size (L)

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

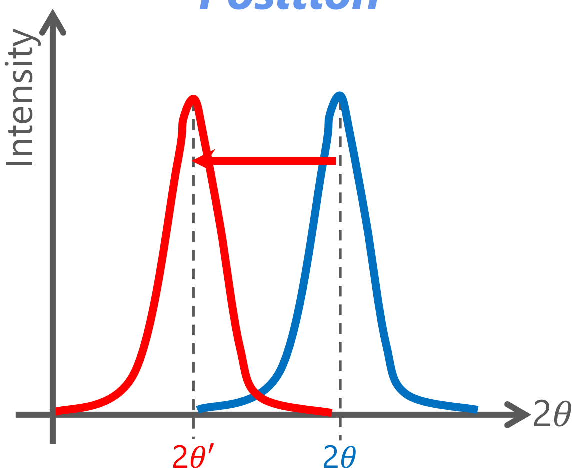
  - Lattice strain heterogeneities
    - ✓ Dislocation density (ρ)

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

    - ✓ Microstrain

Shape of the diffracted peaks

Position



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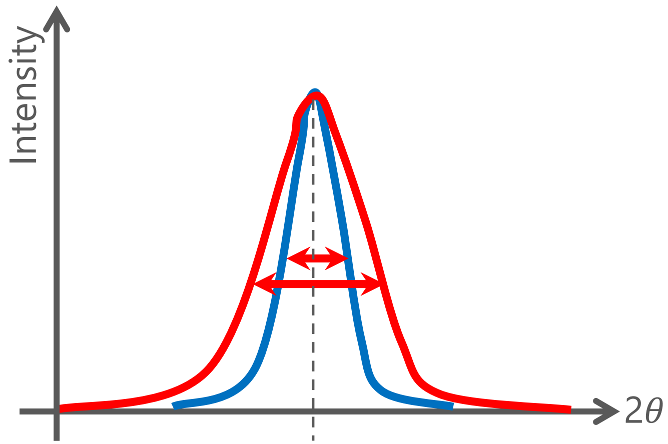

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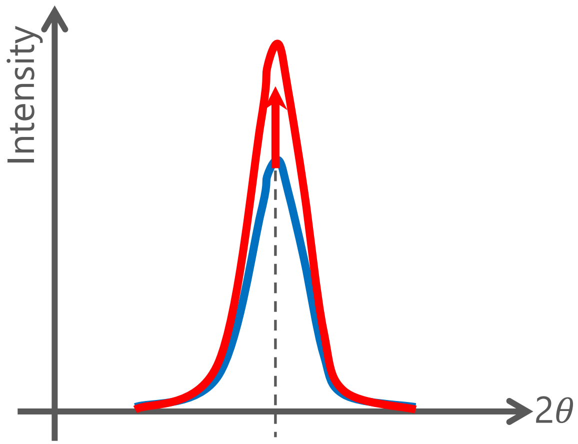
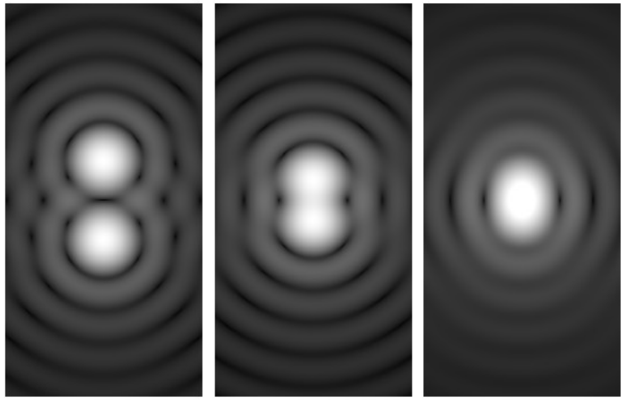
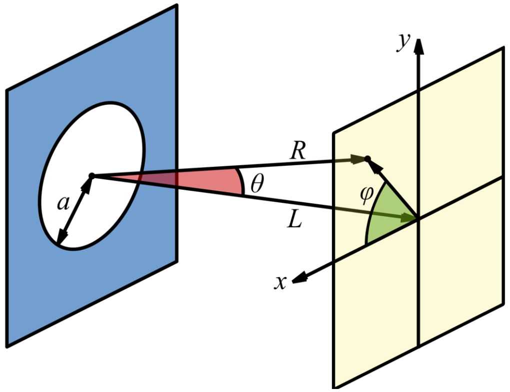


- **Peak height:**
  - Structure factor (F)
  - Phases
  - Texture

Limit of resolution





❖ Airy disk and Rayleigh criterion

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

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

- The first dark ring is at:

$$\sin\theta = 1.22\frac{\lambda}{a} \sim \theta$$

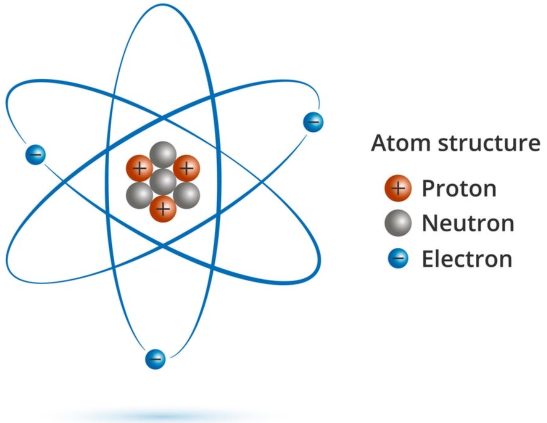
- The 1st minimum coincides with the maximum of the other.

$$\Rightarrow \Delta r = L\tan\left(1.22\frac{\lambda}{a}\right) \sim 1.22L\frac{\lambda}{a}$$

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

Focus on the electron

Particle



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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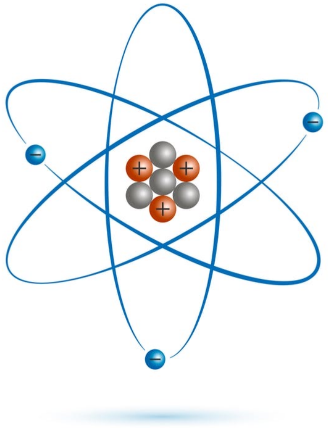

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Wave

Louis DE BROGLIE
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1 Nobel Prize (1929)

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

Electrons act not only as particles but as waves too.

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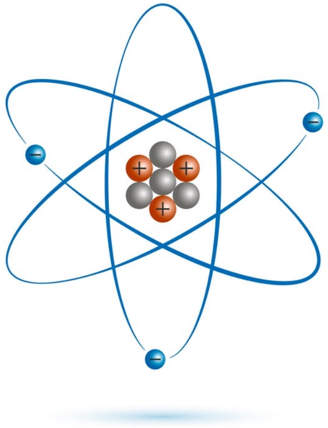

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$$E = eV = \frac{mv^2}{2} \Leftrightarrow v = \sqrt{\frac{2eV}{m}} \Rightarrow \lambda = \frac{h}{\sqrt{2meV}}$$

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

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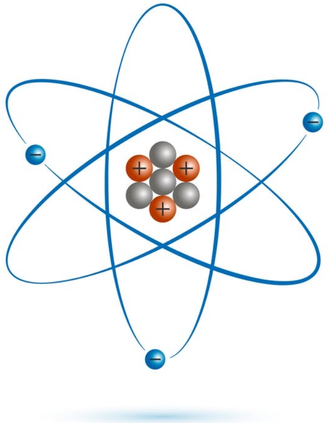

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$\lambda(30 \text{ kV}) = 7 \text{ pm}$
$\lambda(200 \text{ kV}) = 2.5 \text{ pm}$

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

The 2 types of electron microscopes

Transmission electron microscope (TEM)

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

↳Nanometric/atomic scale

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↳ Meso-/micro-/nano-metric scale

↳ **The two microscopes are complementary.**

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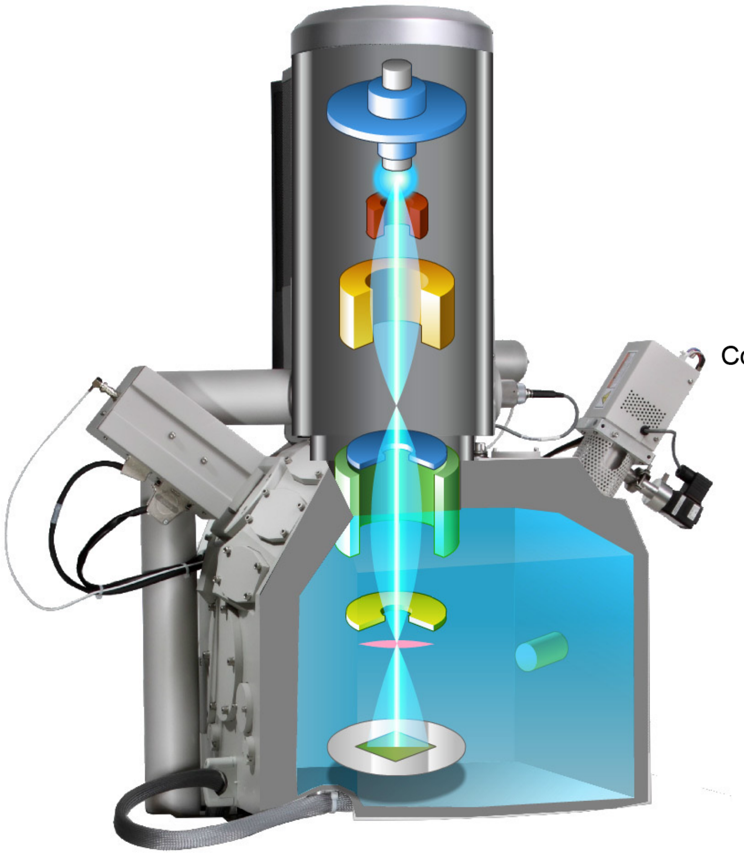
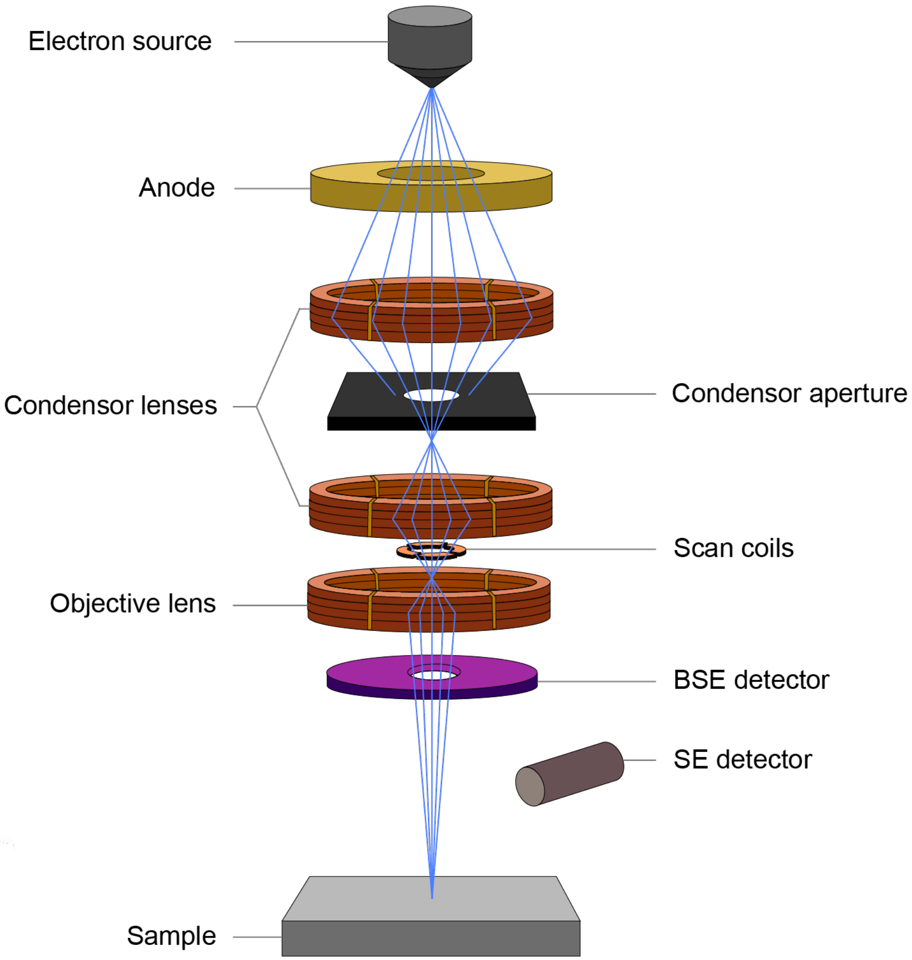
Basics of SEM

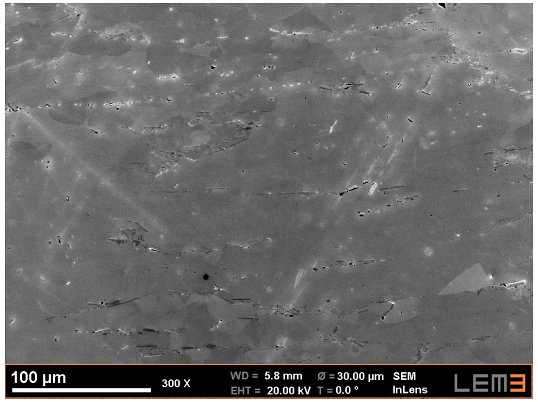




J. Aubourg, *PhD Thesis (2024)*

SEM imaging

Secondary electrons (SE)

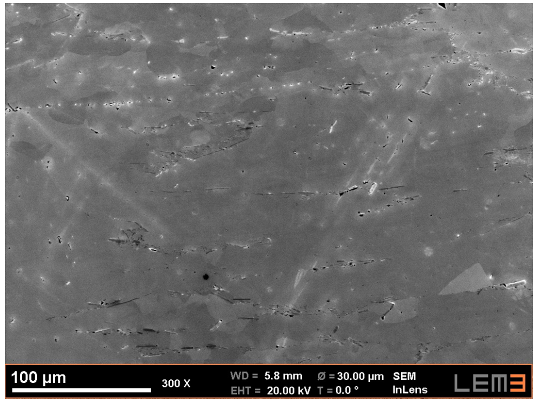


- Inelastic scattering between incident electrons and outer-shell electrons of sample atoms ⇒ emission of SE
- Inelastic scattering involving inner-shell electrons, followed by electronic transitions → emission of characteristic X-rays
  ⇒ EDS

↳Topographic contrast

SEM imaging

Secondary electrons (SE)



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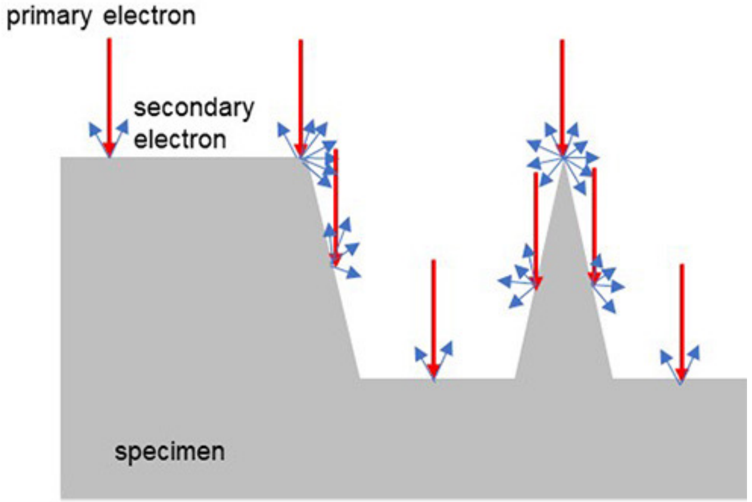
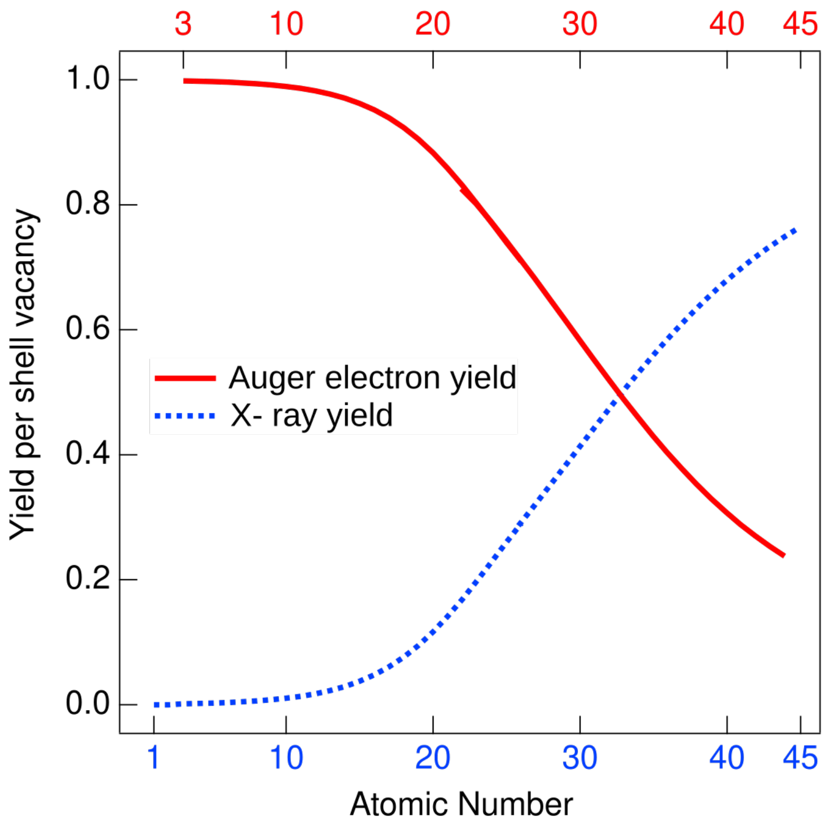
The edge effect

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



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

Energy Dispersive X-ray Spectroscopy (EDS)



❖ **Inner-shell ionization:**

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

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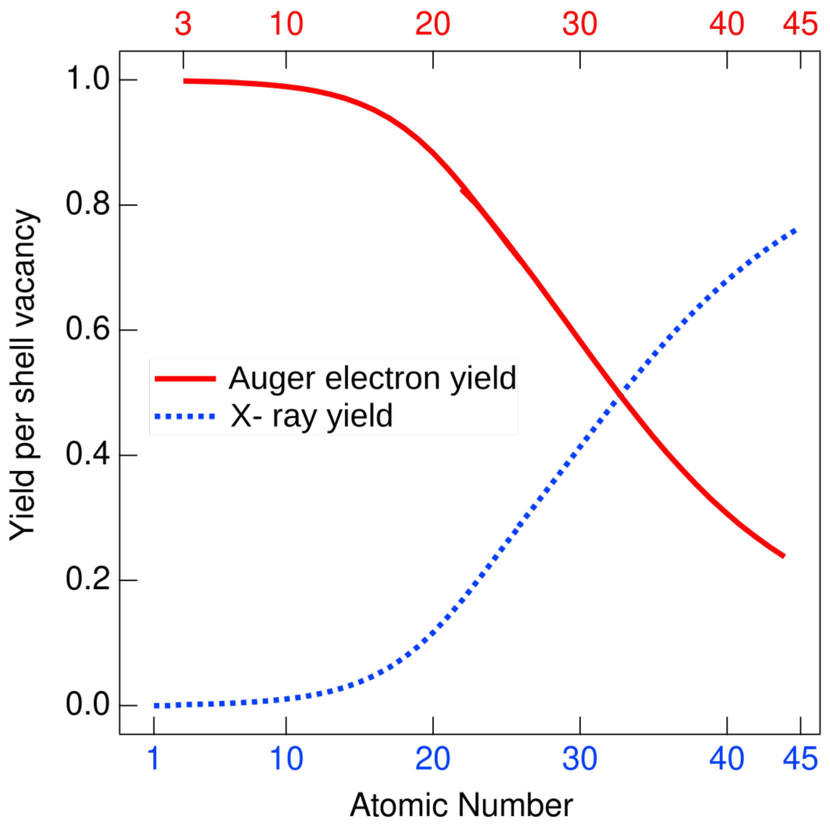

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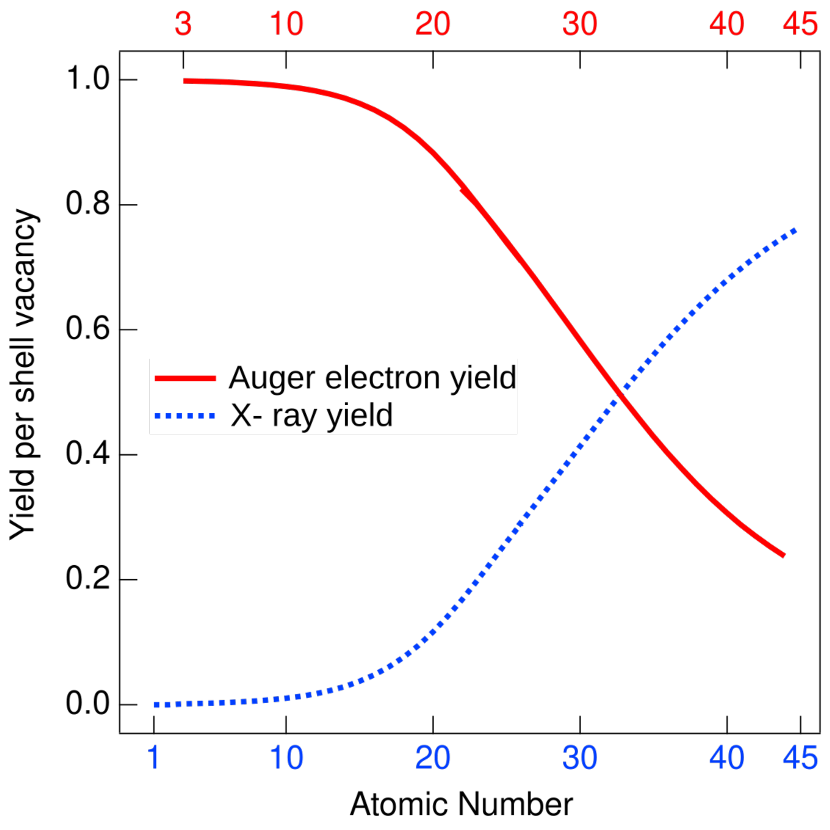

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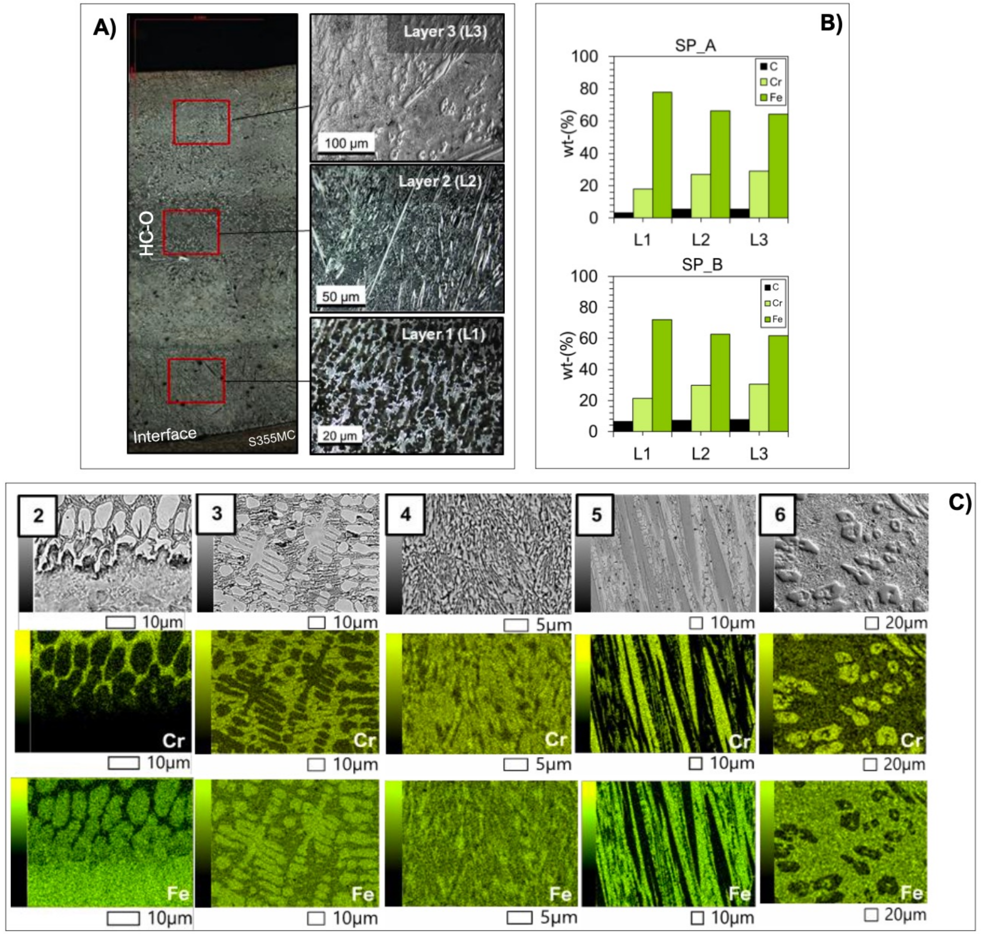

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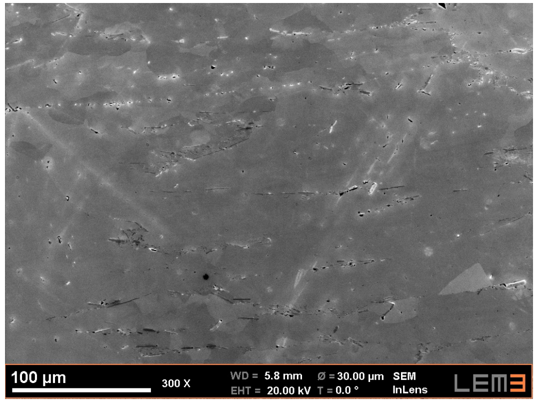
- **Detection:**
  - An EDS detector measures the energy of these X-rays, allowing us to:
    - ✓ Identify the elements present (qualitative analysis)
    - ✓ Estimate their relative amounts (semi-quantitative analysis but not accurate for low Z elements)

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

SEM imaging

Secondary electrons (SE)



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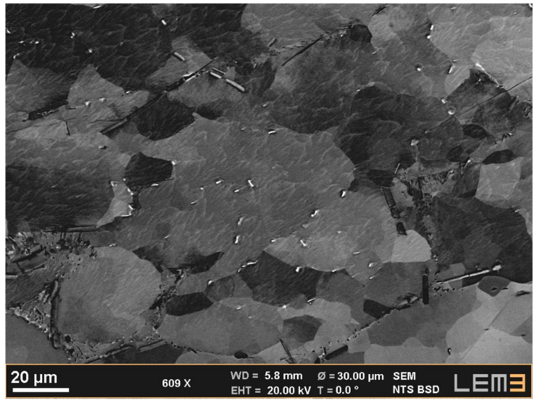
  ⇒ EDS

↳ Topographic contrast

BackScattered Electrons (BSE)



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

  ⇒ EBSD, ECCI

↳ Topographic contrast
↳ Chemistry contrast
↳ Orientation contrast

↳ The two imaging modes are complementary.

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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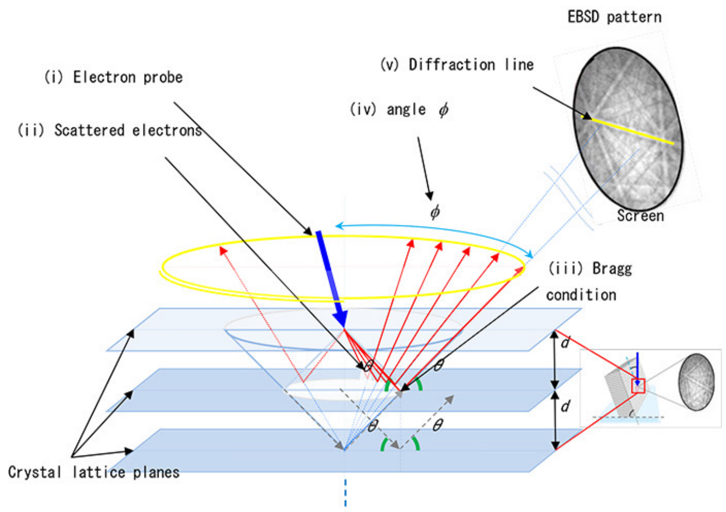
  - The BSE cross section increases with Z
- **Other energy losses (heat, phonons, electronic excitations)**

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

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

Basics of the Electron BackScattered Diffraction (EBSD)



❖ Generation of Kikuchi patterns:

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

www.jeol.co.jp

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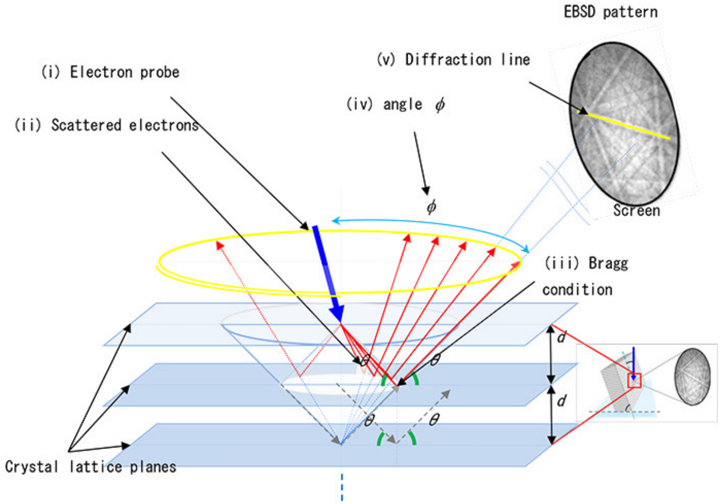

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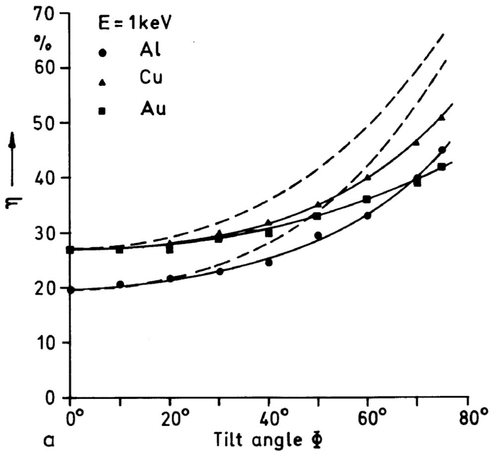
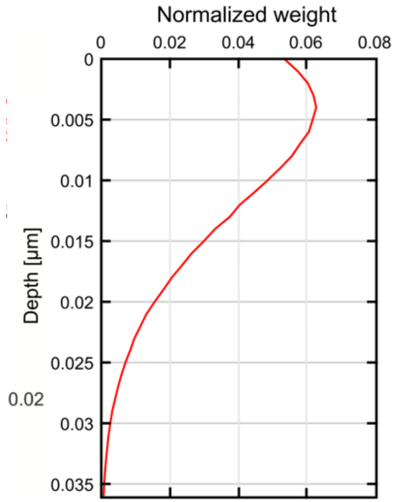
⮱The BSE yield increases with increasing tilt angle





⮱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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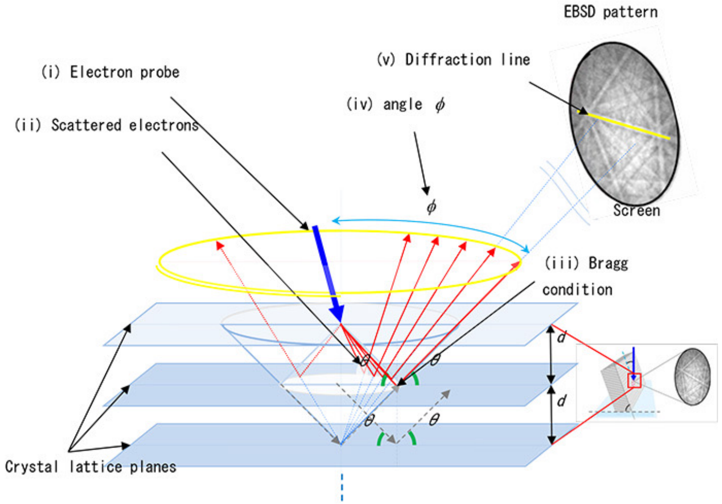

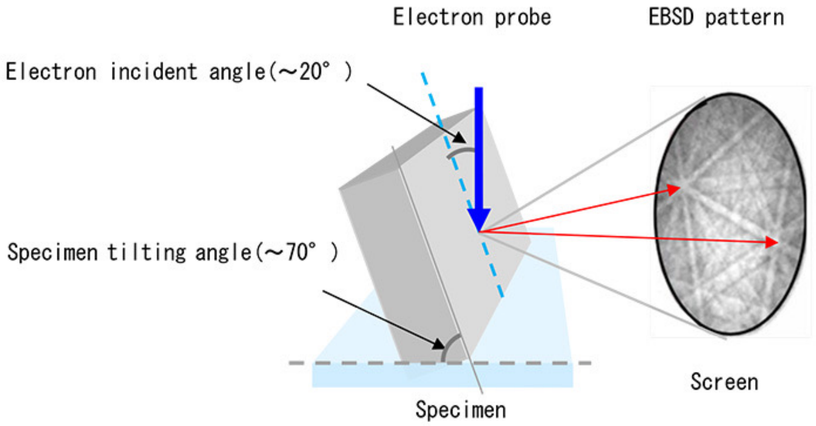

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❖ Basics of the EBSD:

- 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.

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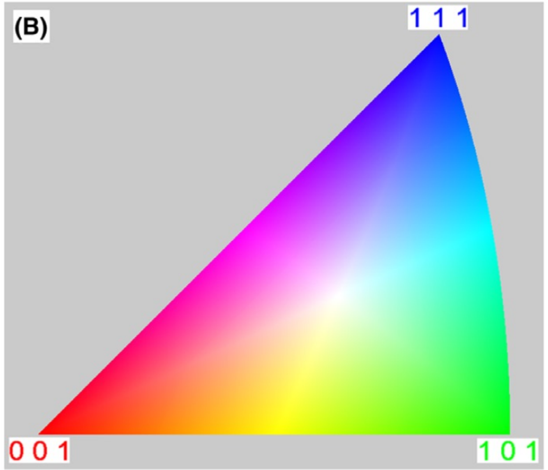
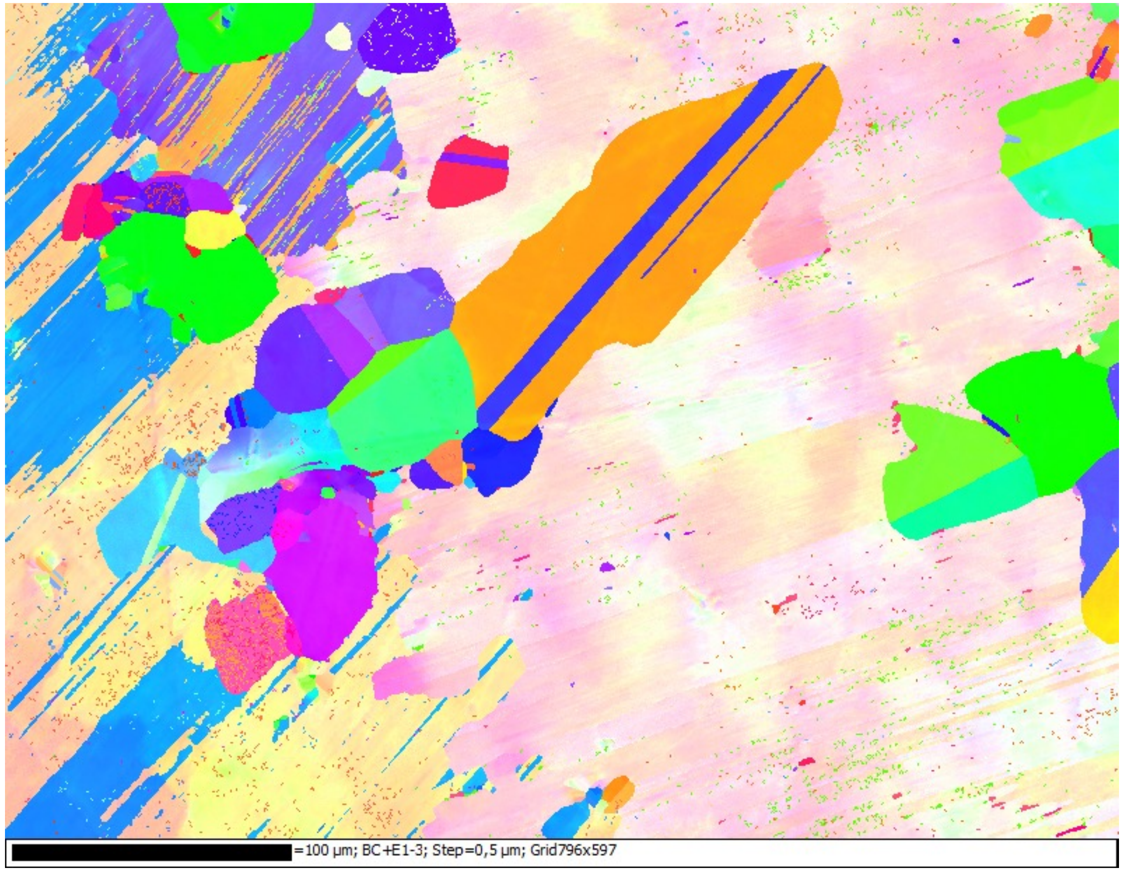
Example of extinction condition

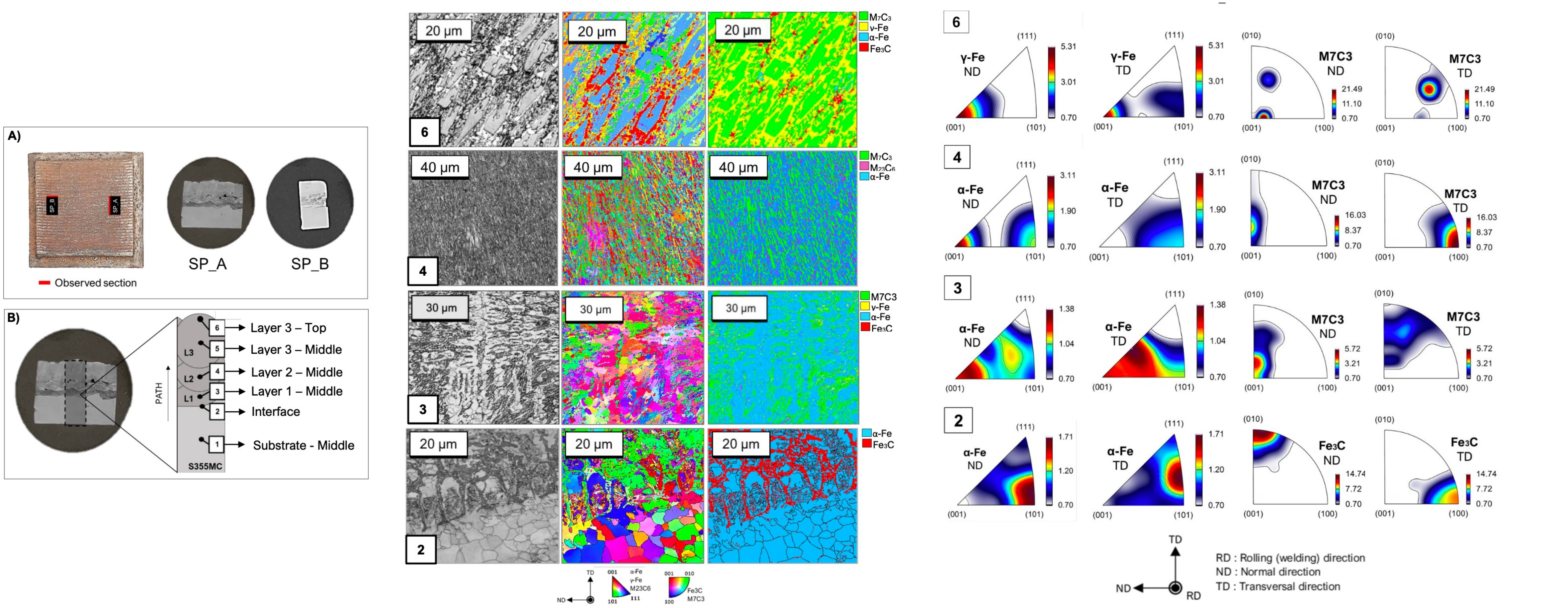
- **Capabilities of EBSD:**
  - Orientation measurements
  - Local misorientation measurements
  - Grain identification
  - Phase identification
  - Statistics on the microstructure





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

Texture measurements by EBSD



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

Thanks for your listening!

If you need further information:

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LABORATOIRE D'ÉTUDE DES MICROSTRUCTURES ET DE MÉCANIQUE DES MATÉRIAUX

UNIVERSITÉ DE LORRAINE

enim École Nationale d'Ingénieurs de Metz