Conventional Transmission Electron Microscopy (CTEM)

understanding contrast of crystal defects

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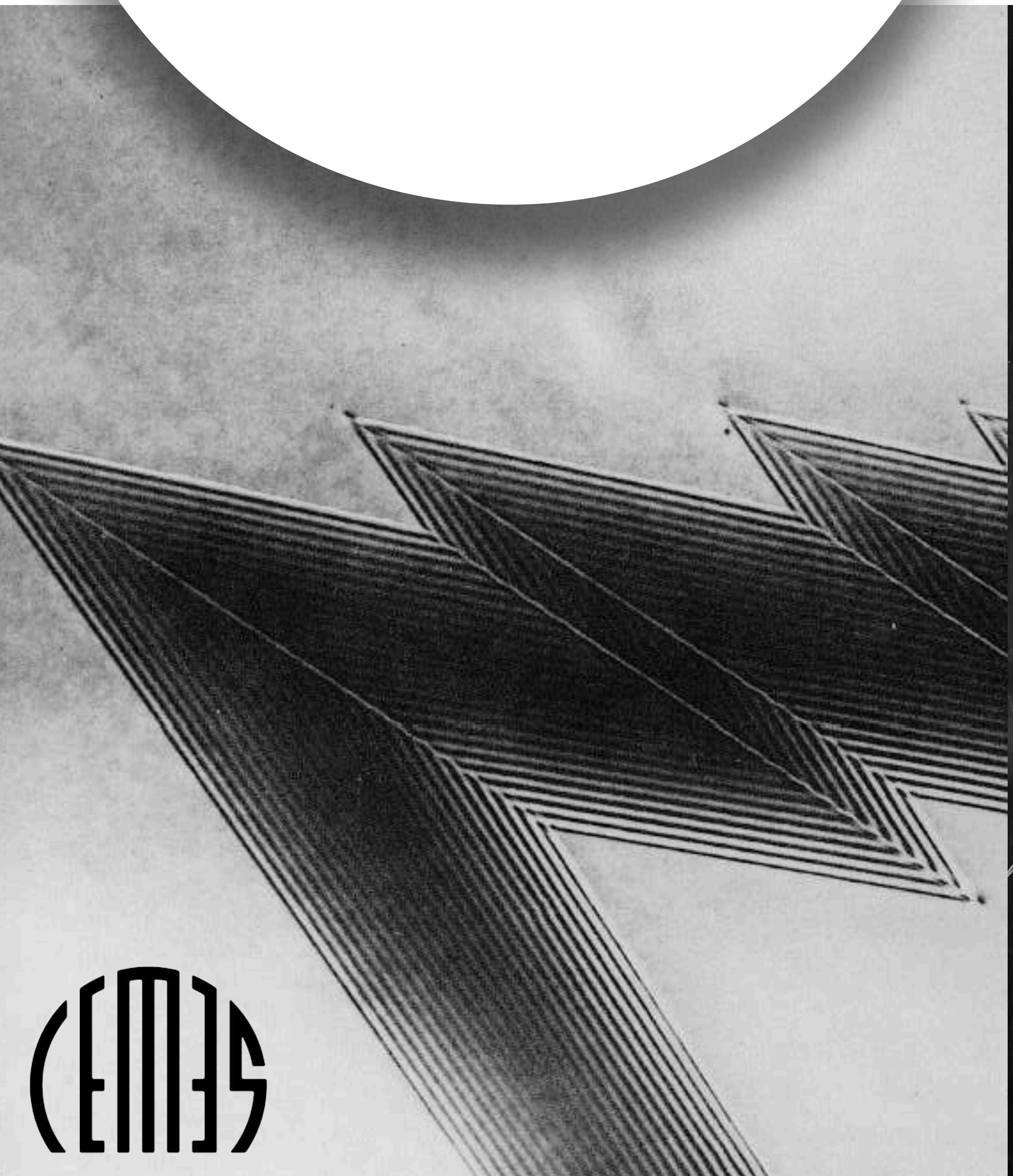
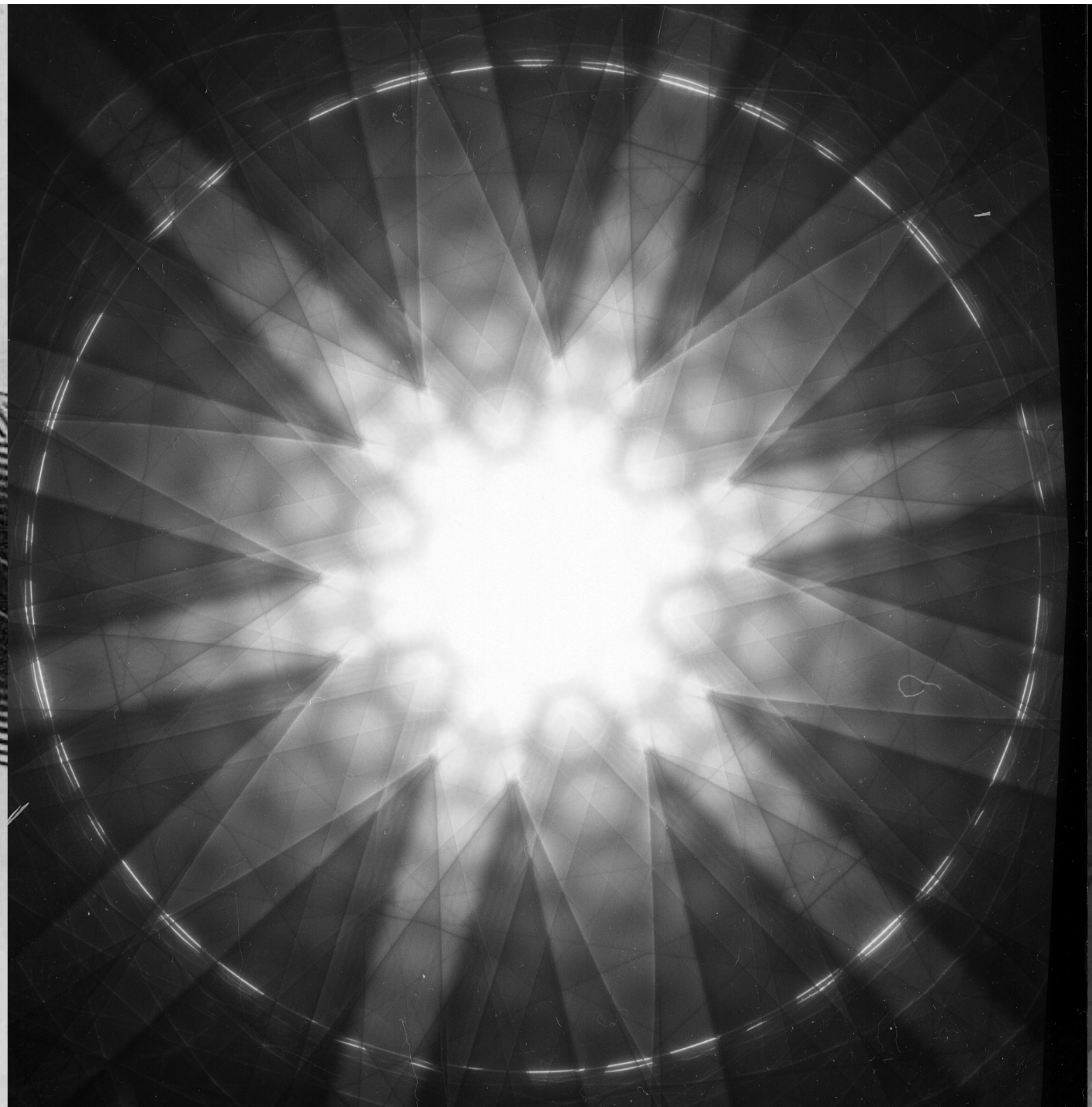
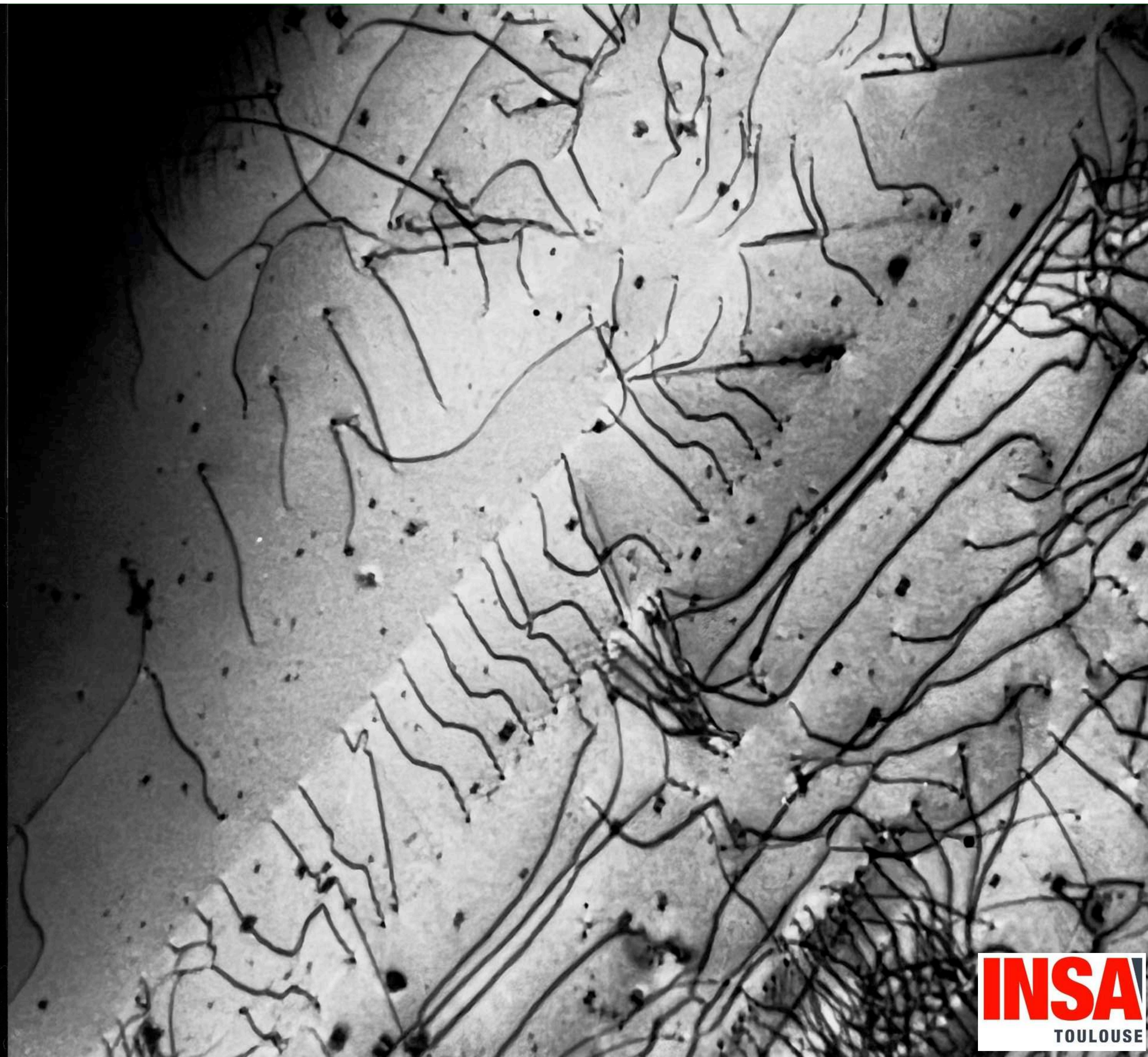




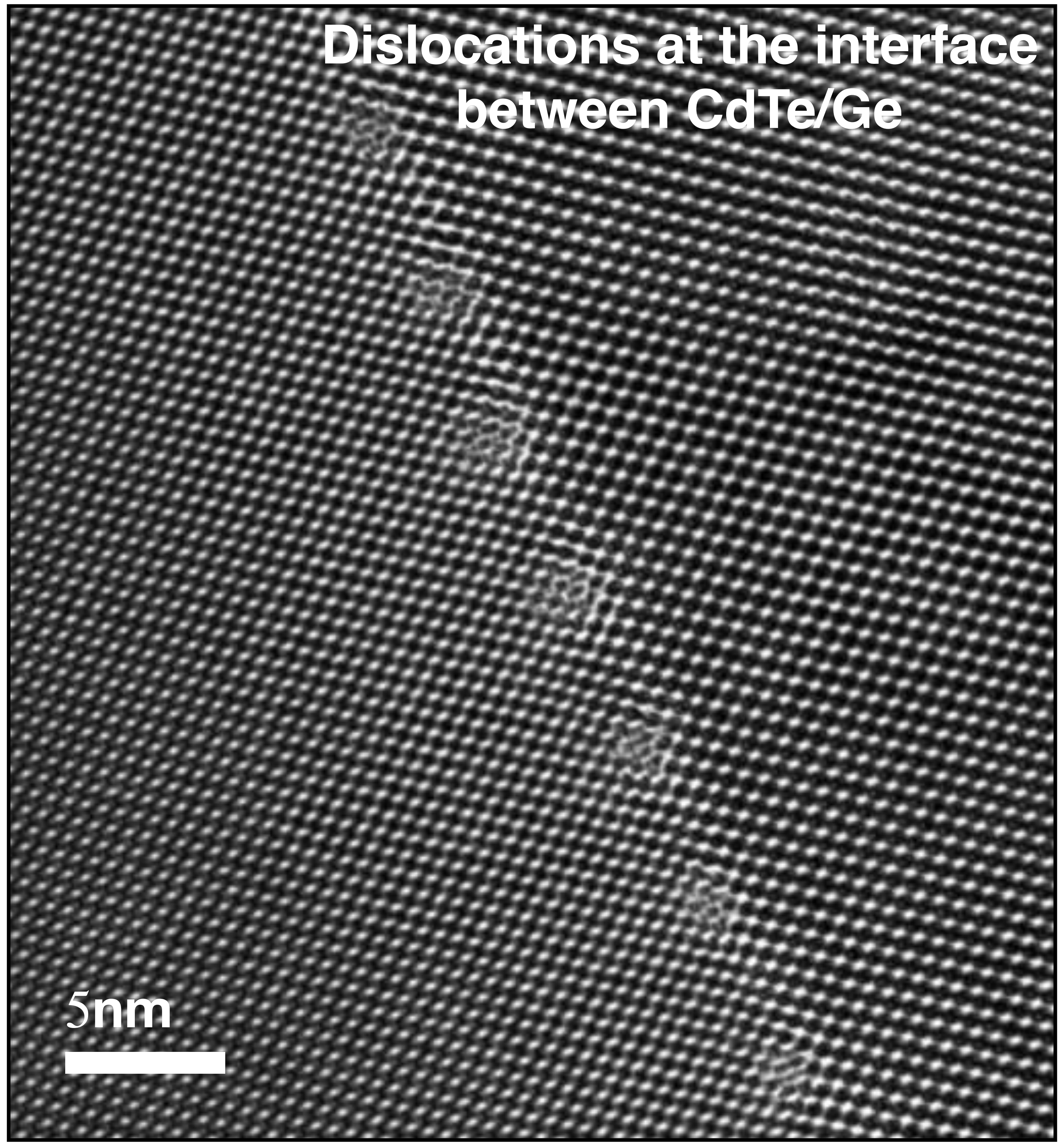


What is CTEM ?

What is conventional microscopy ?
Structural characterization :

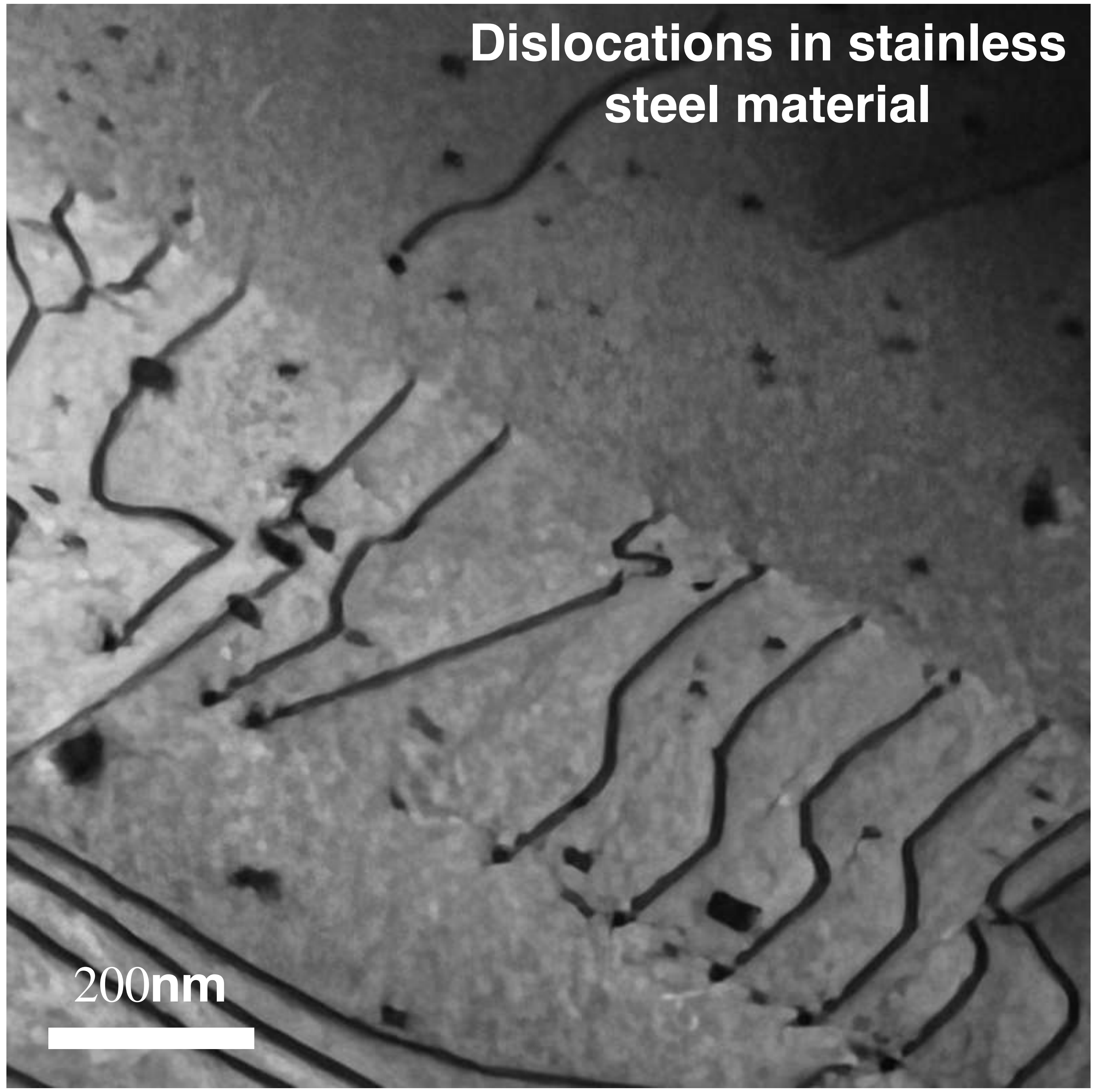
- Sample morphology (grain size, precipitate size, etc.)
- Crystal defects (dislocations, faults, grain boundaries, etc.)
- Crystalline symmetries (point and space groups)
- Crystalline and non-crystalline materials

Atomic scale ?



VS

Diffraction contrast !



Why CTEM ?

Example : what kind of informations are required to study dislocations in materials ?

local
(the core of the dislocations governs their mobility)

global
(geometry and organisation)

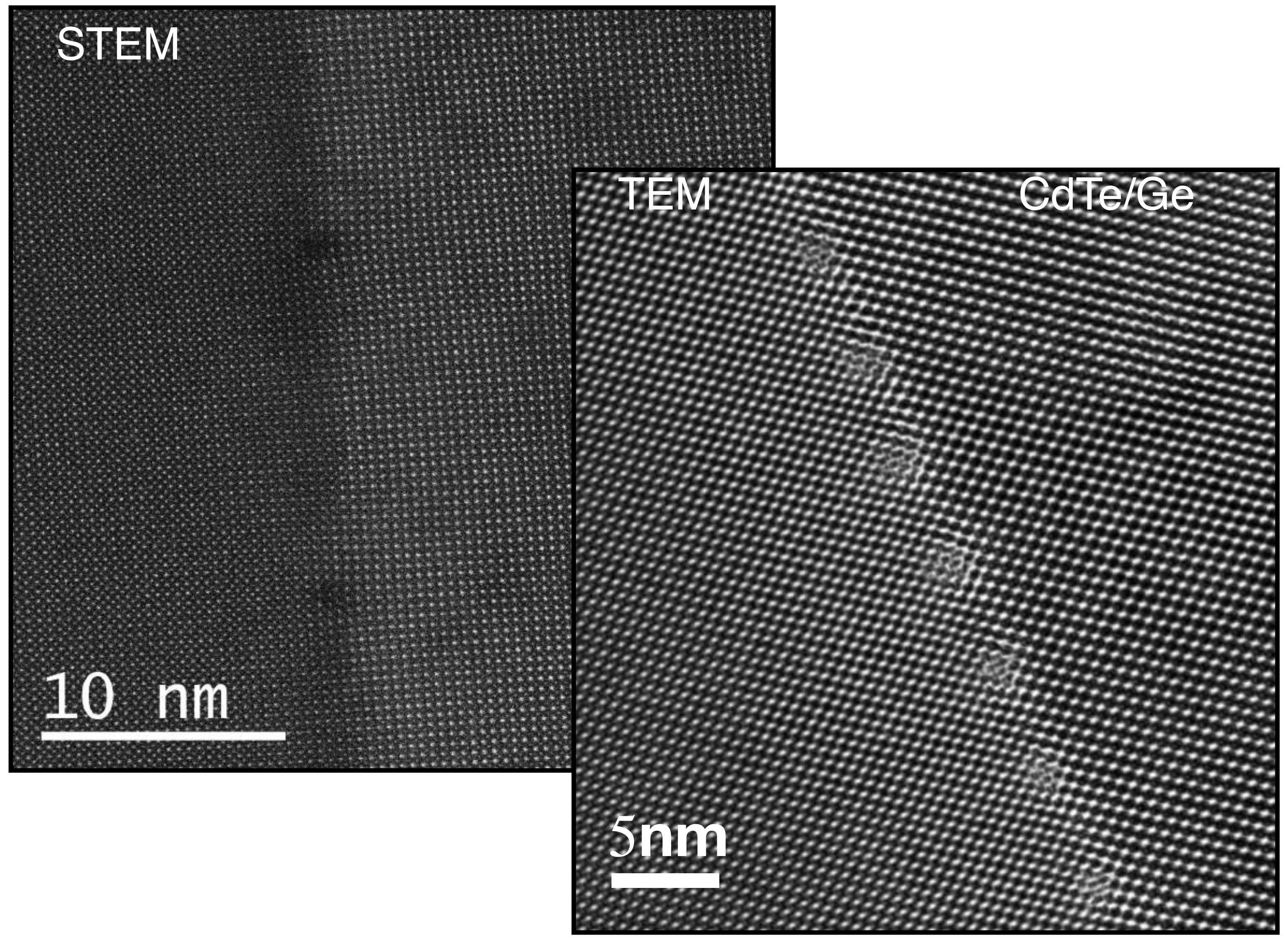
Nanometric scale :
High resolution

Mesoscopic domain :

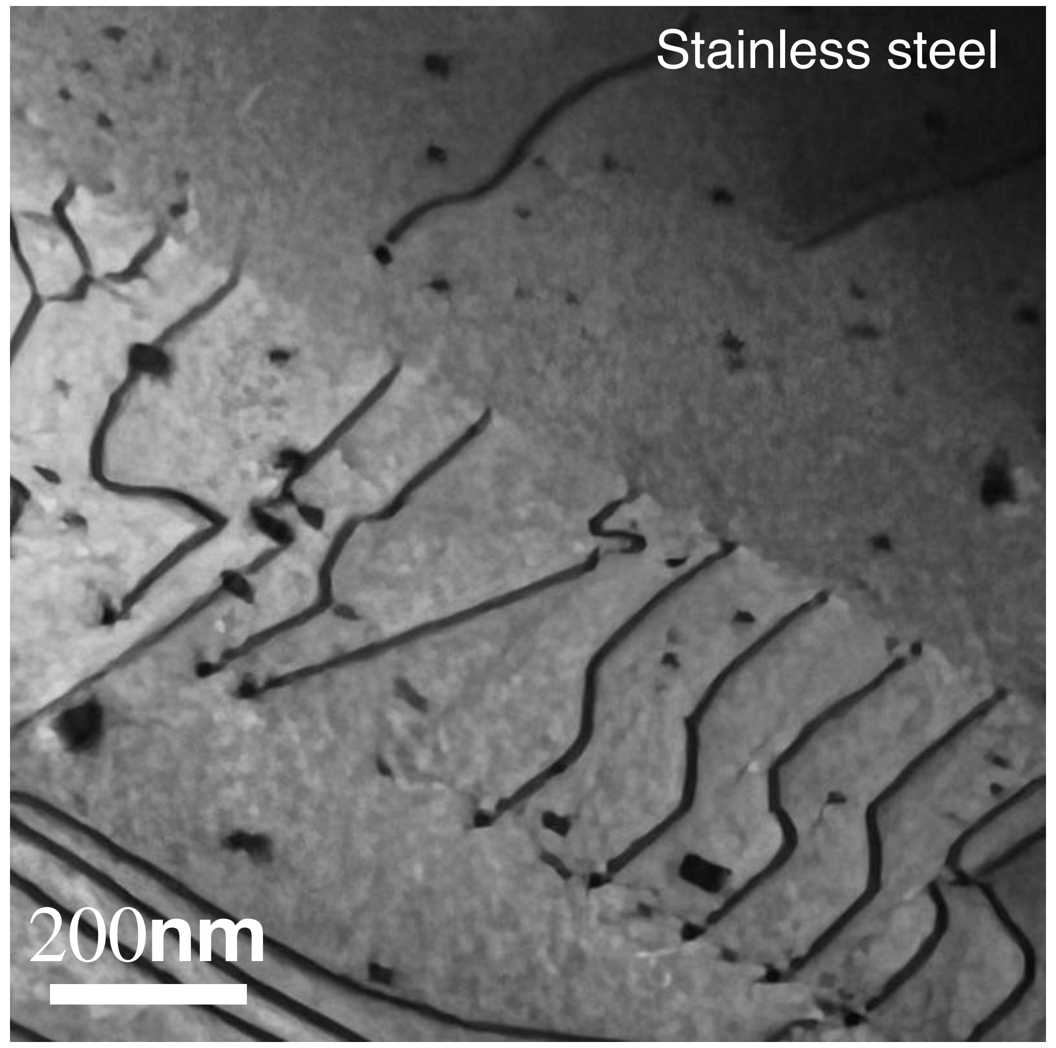
Macroscopic region (SEM or Optical methods)

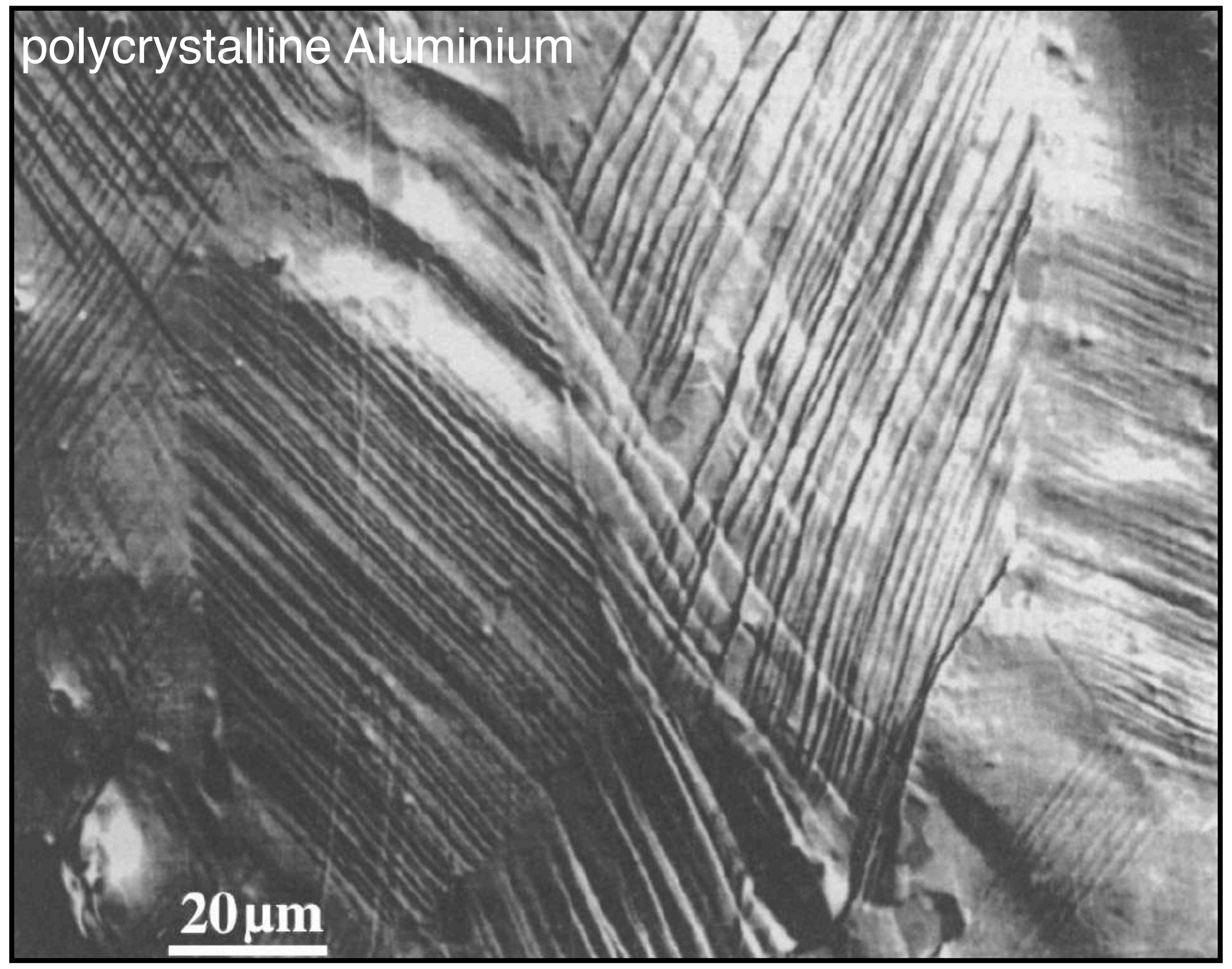
- Very fine structure (atomic scale)
- Not easy to use (surface-dependent)

- Slip traces
- Slip plans
- Burgers vectors activated
- No local information



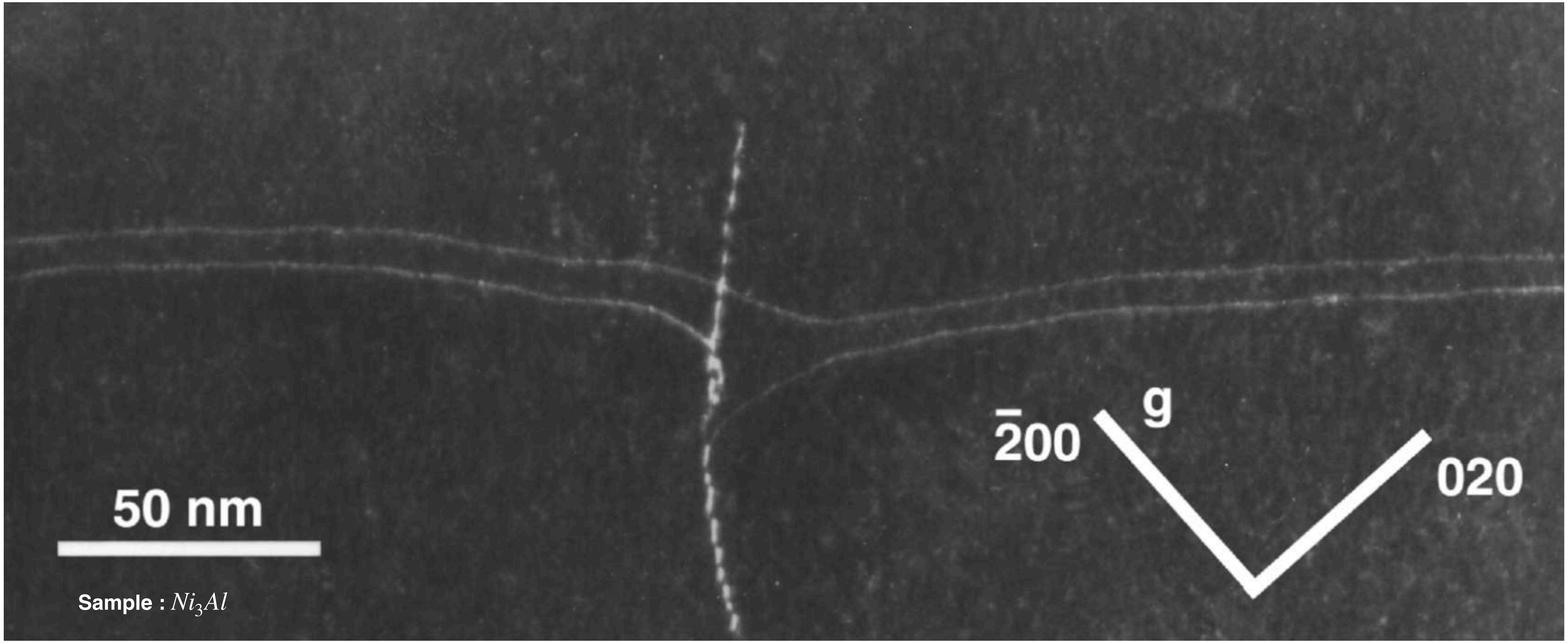
Conventional transmission microscopy





Why CTEM ?

- Magnification neither too small nor too large : x 10 000 to x 100 000
- Area observed : from $50\mu m^2$ to $1\mu m^2$
- Requires a standard microscope (not necessary coherent nor corrected)



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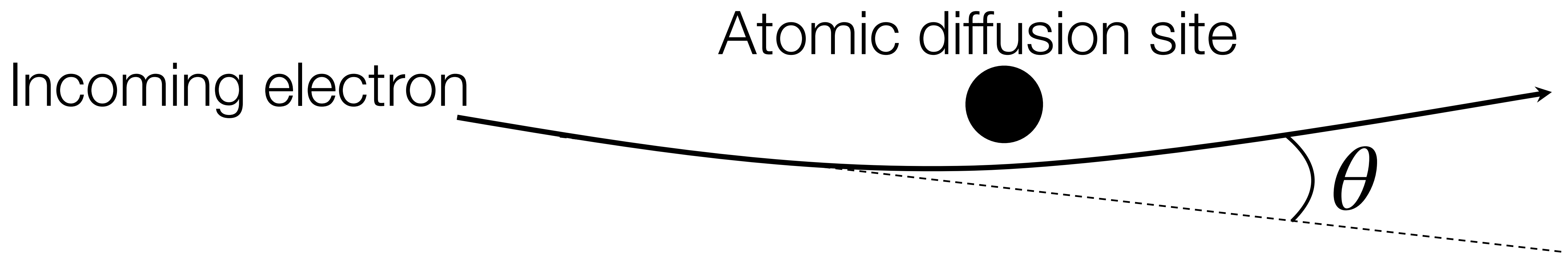
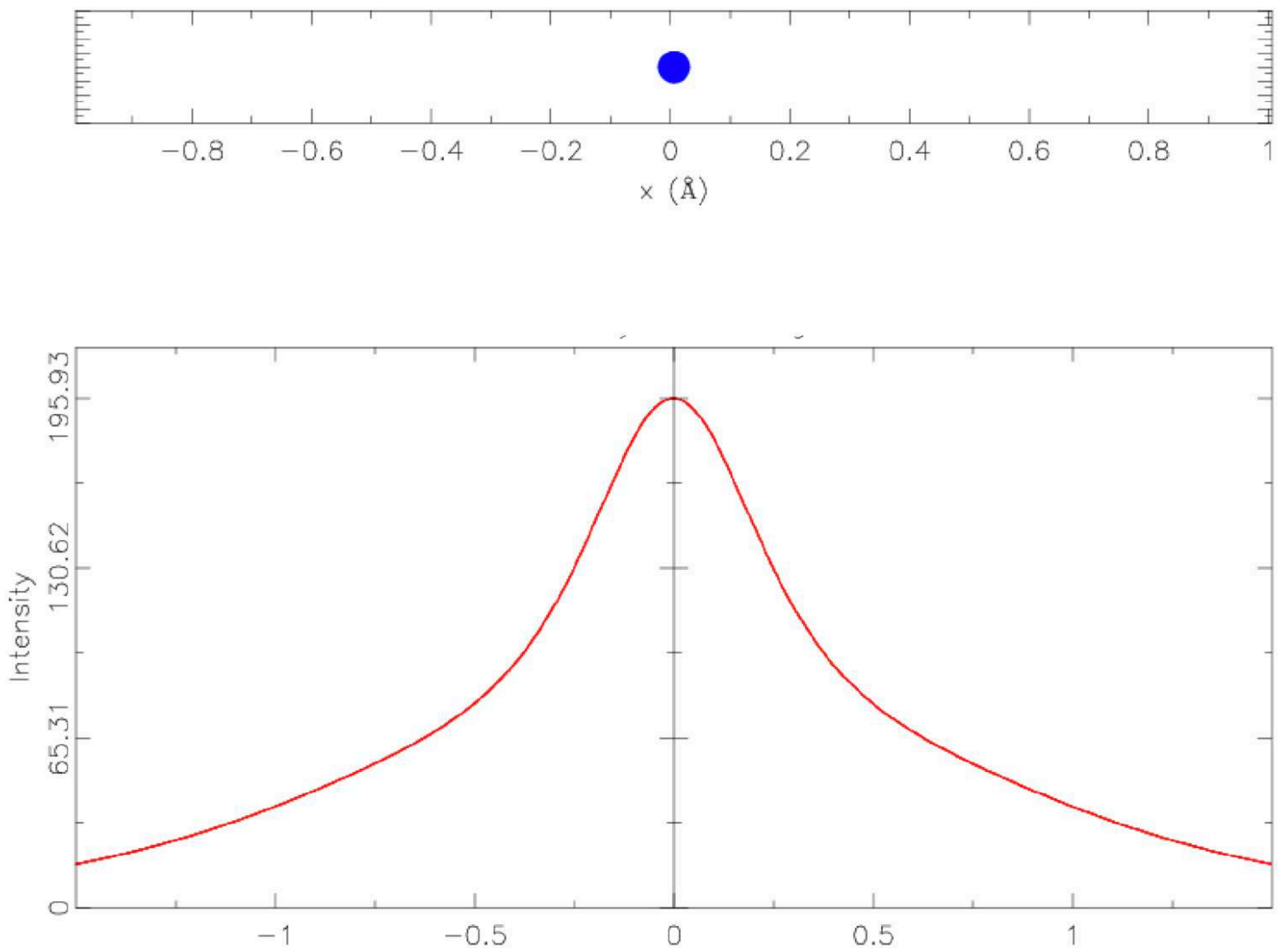
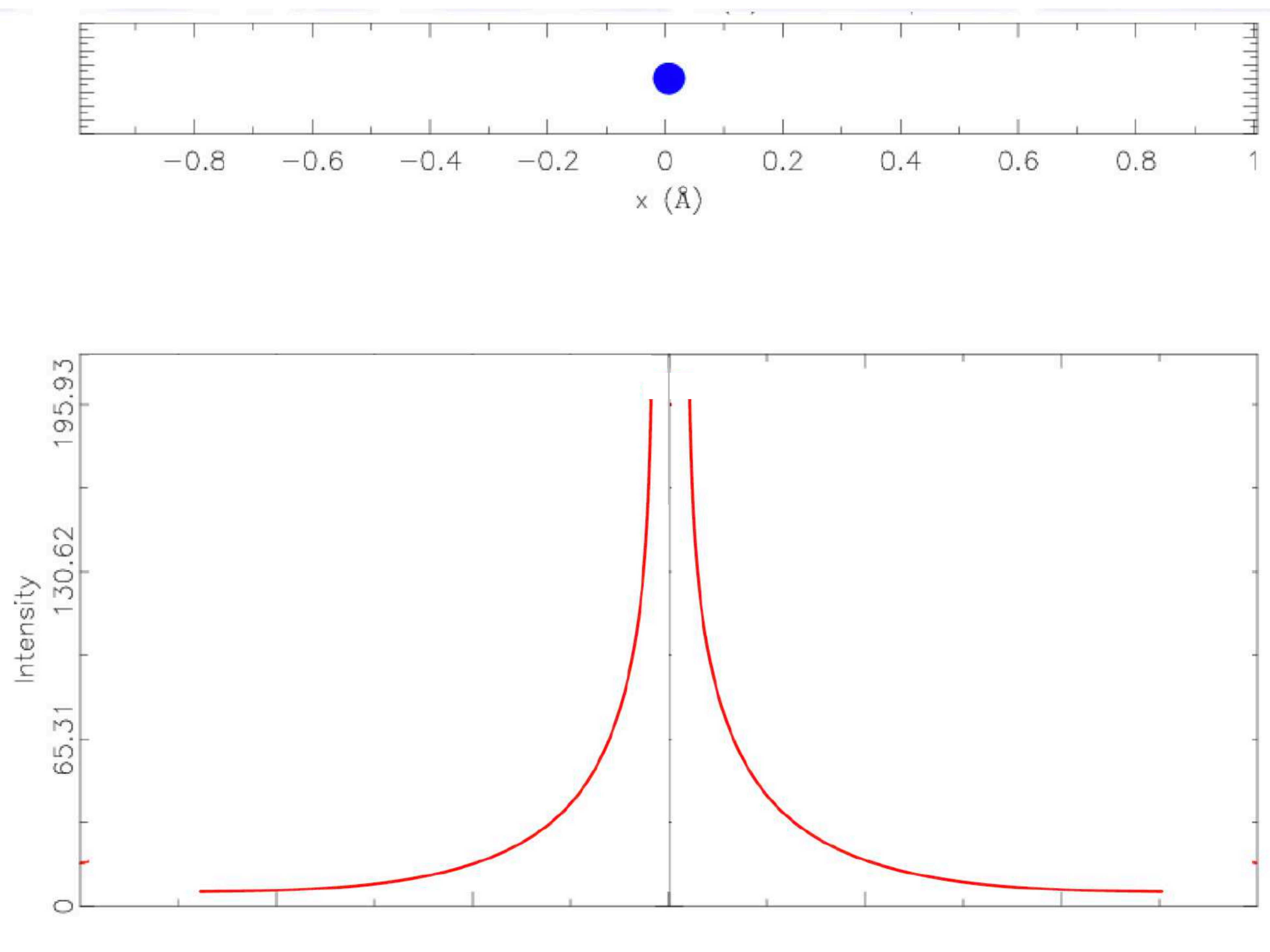
Diffraction theory of perfect crystal

Kinematical theory
Amplitude-Phase diagram
Howie-Whelan approach
to dynamical effect

Atomic form factor





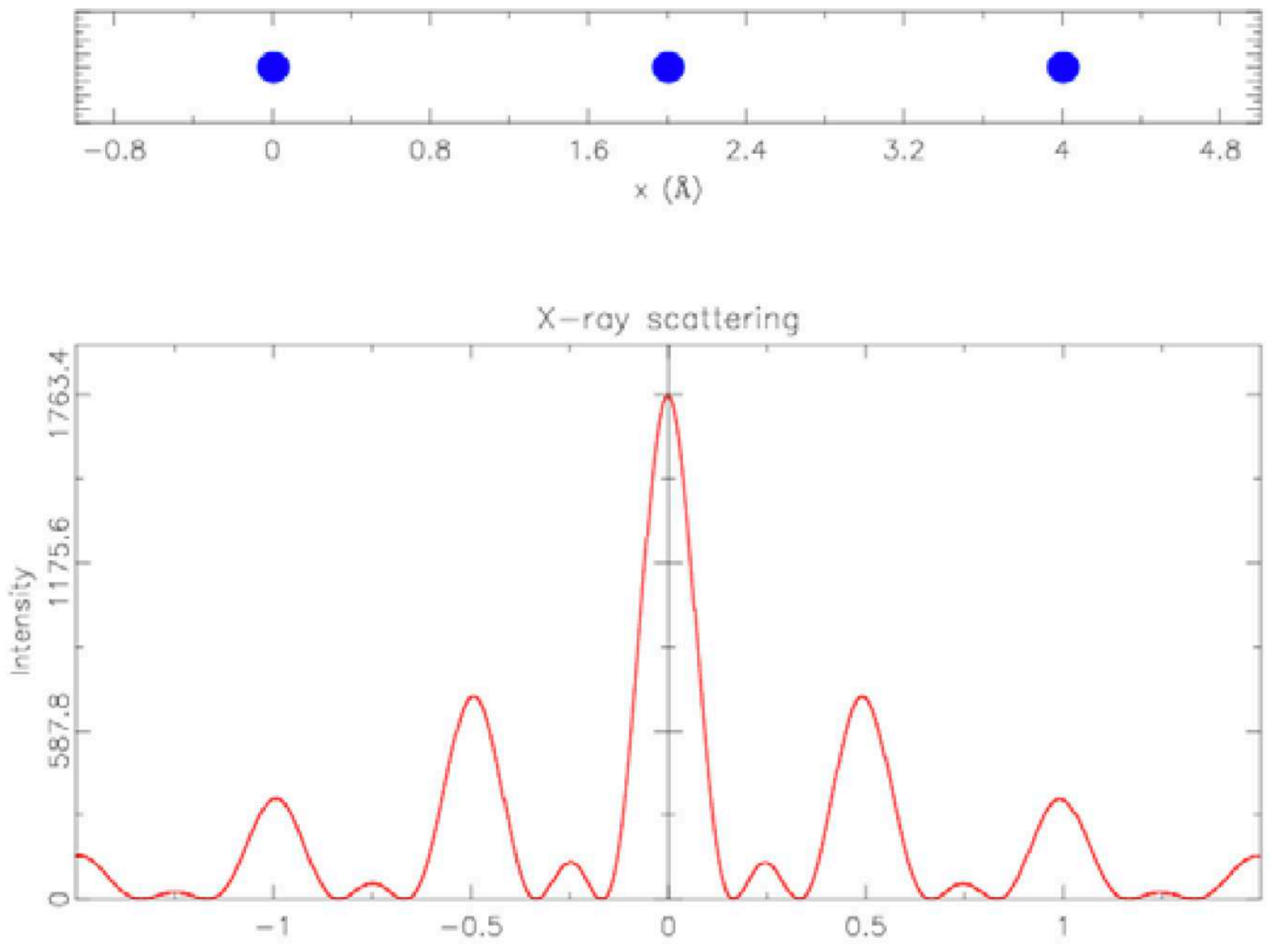


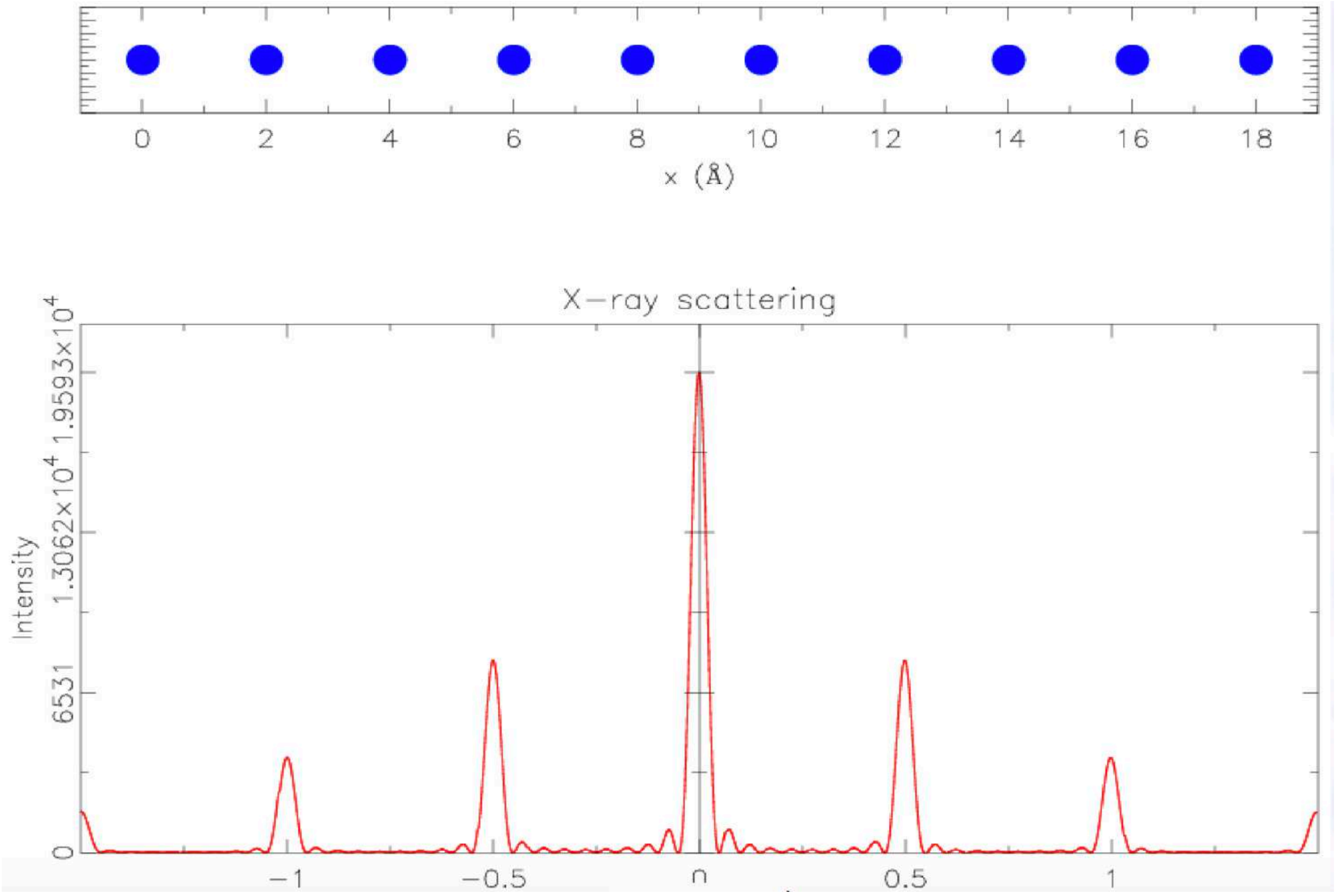
$$f_i^X(s) = \sum_{i=1}^{4} A_j e^{-B_j s^2} + C$$

$$f_i^e(s) = \frac{me^2}{2h^2}\frac{Z_i - f_i^X(s)}{s^2}$$

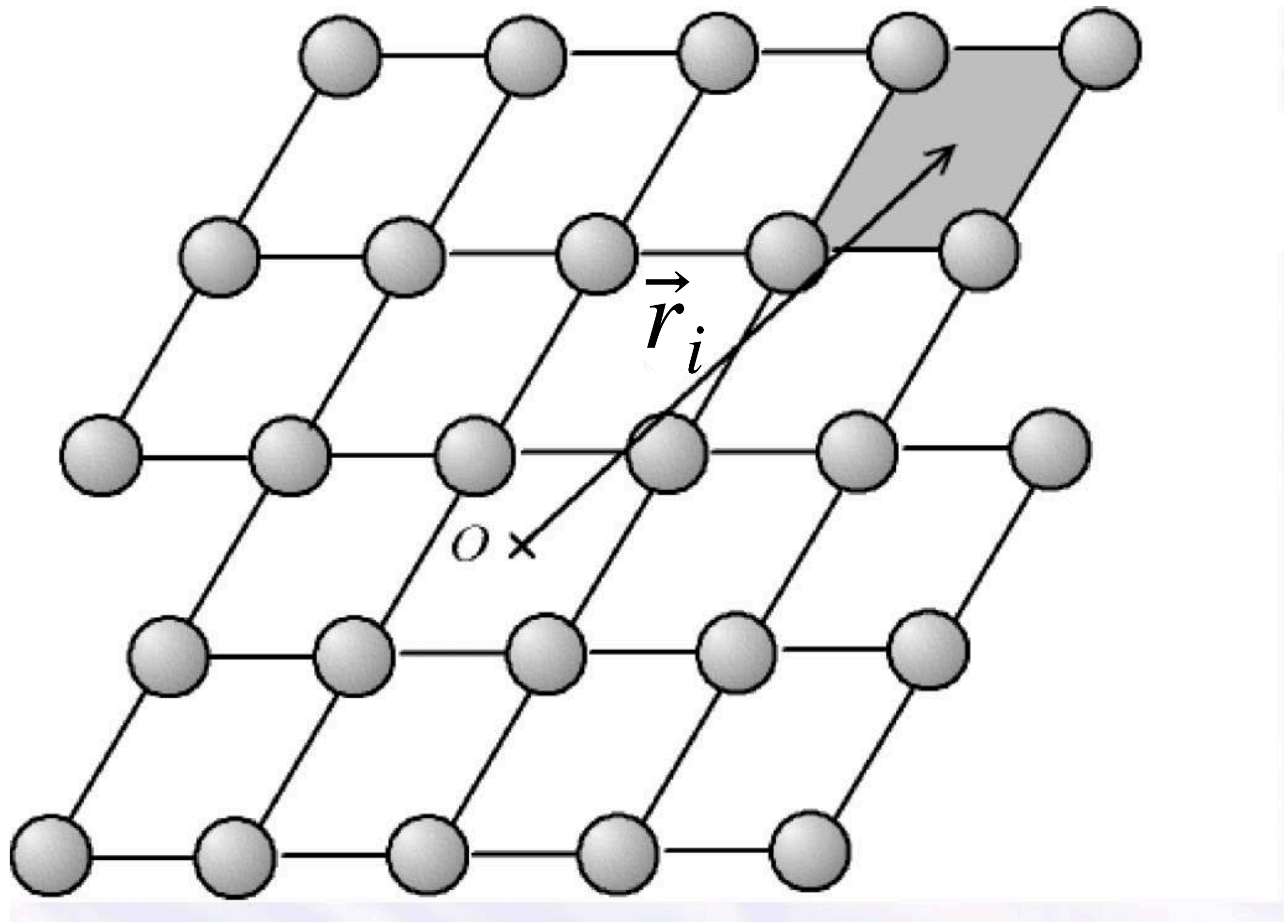
See *Doyle-Turner* or *Weickenmeir-Kohl* diffusion coefficients in International table of crystallographic

Diffraction by a periodic set of diffusion sites





Crystal lattice



Structure factor

$$F_{\vec{g}} = \sum_i f_i^e(s) e^{-B_i s^2} e^{-2\pi \vec{g}.\vec{r}_i}$$

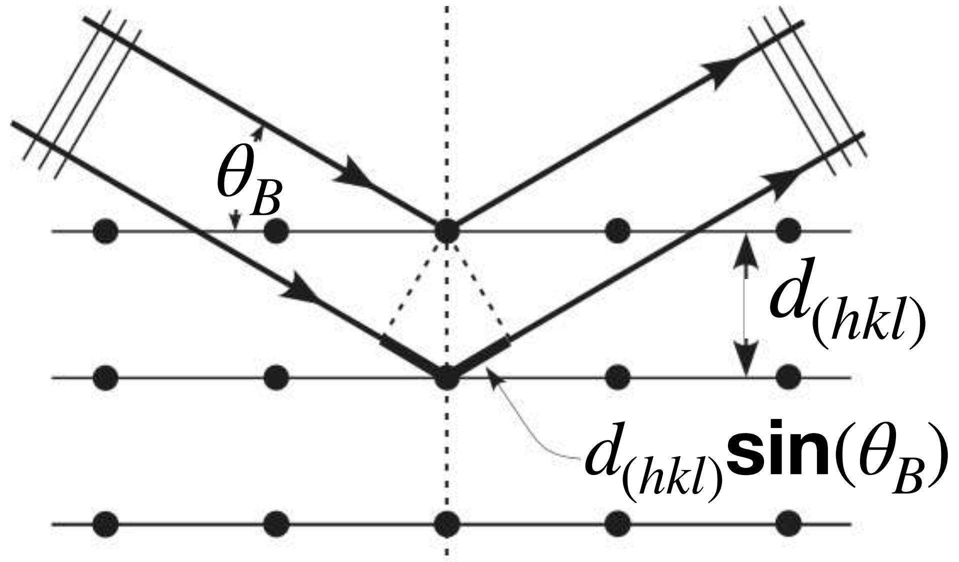
Debye-Waller factor
(damping due to thermal vibration)

$$B_i = 8\pi^2 < u^2 >$$

$$S_{\vec{K}}$$

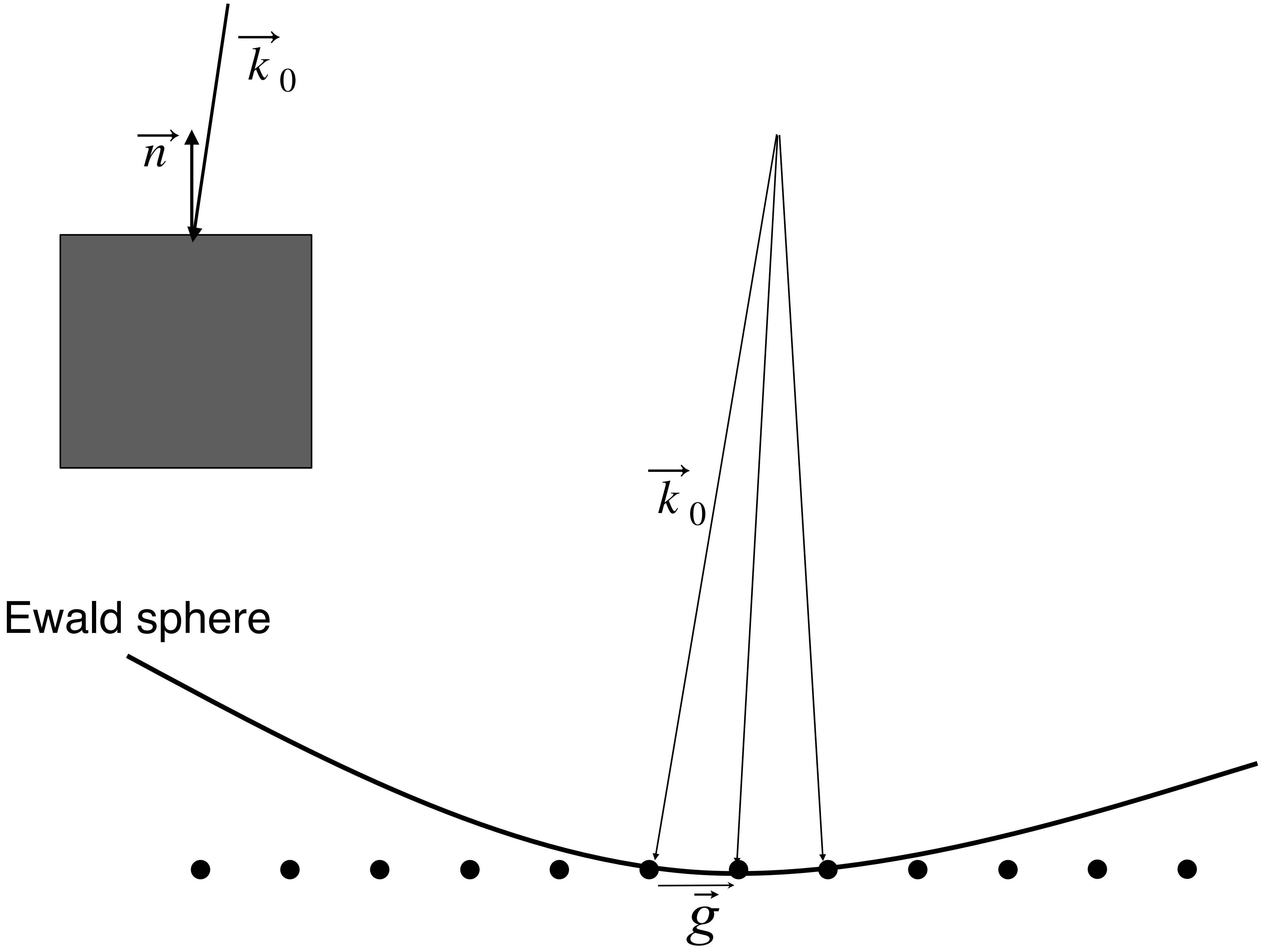
$$I_{\vec{g}} \approx |F_{\vec{g}}|^2 |S_{\vec{K}}|^2$$

Introduction to electron diffraction theory : the Bragg law

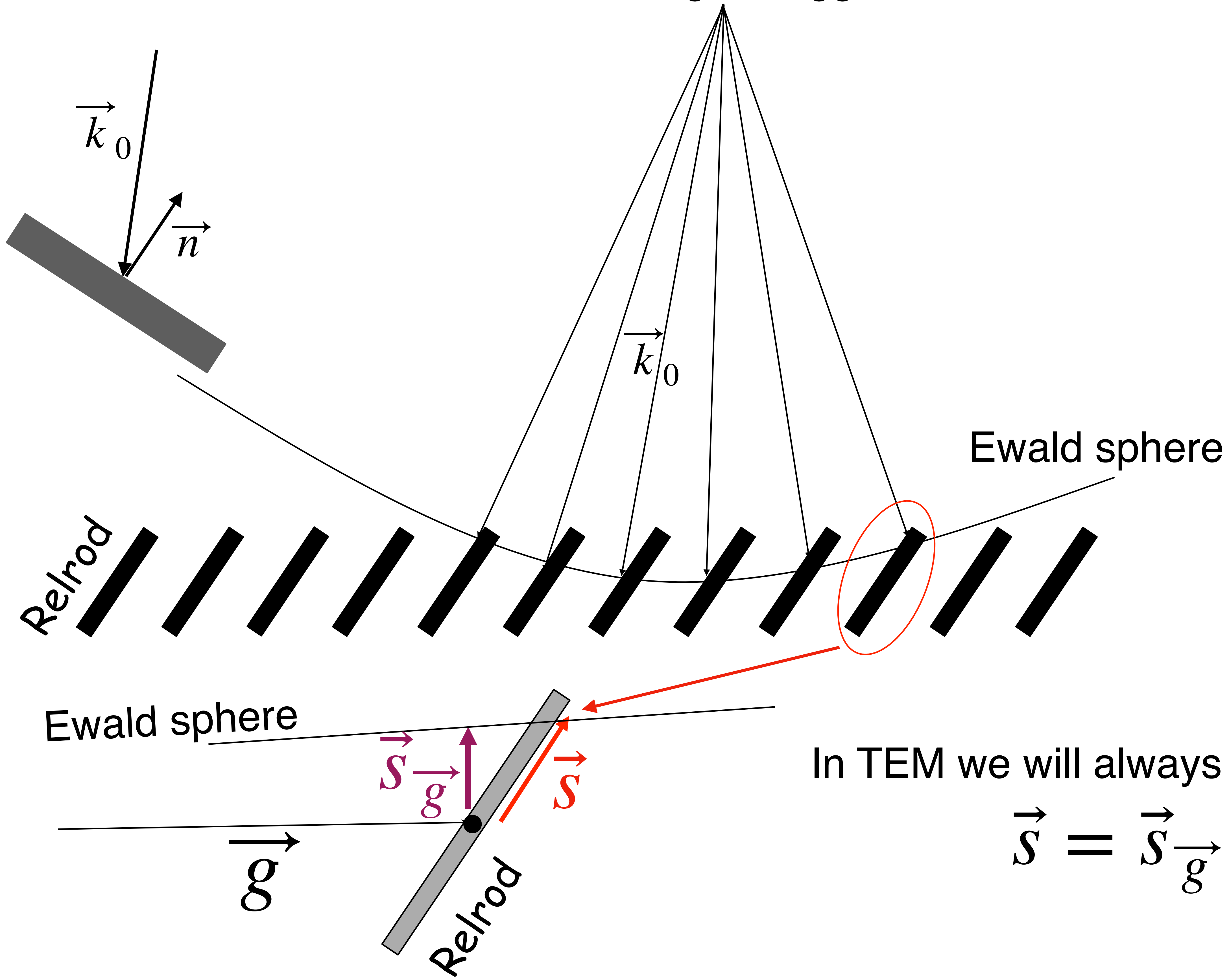


$$2d_{(hkl)}\sin(\theta_B) = \lambda$$

The diffraction pattern can be deduced from the intersection of a sphere called the Ewald sphere with the reciprocal crystal lattice :



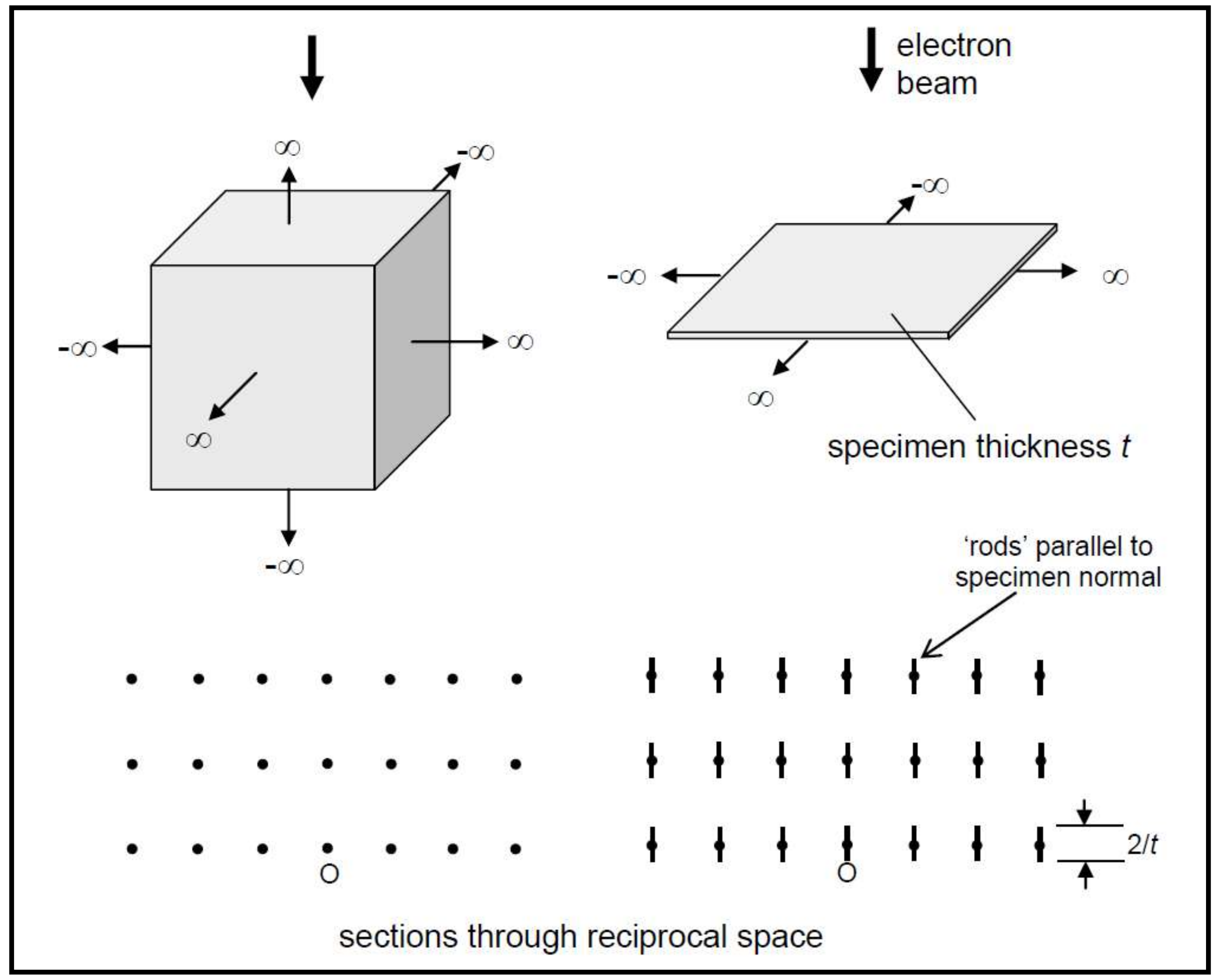
➡ Because of thinning : Bragg relaxation

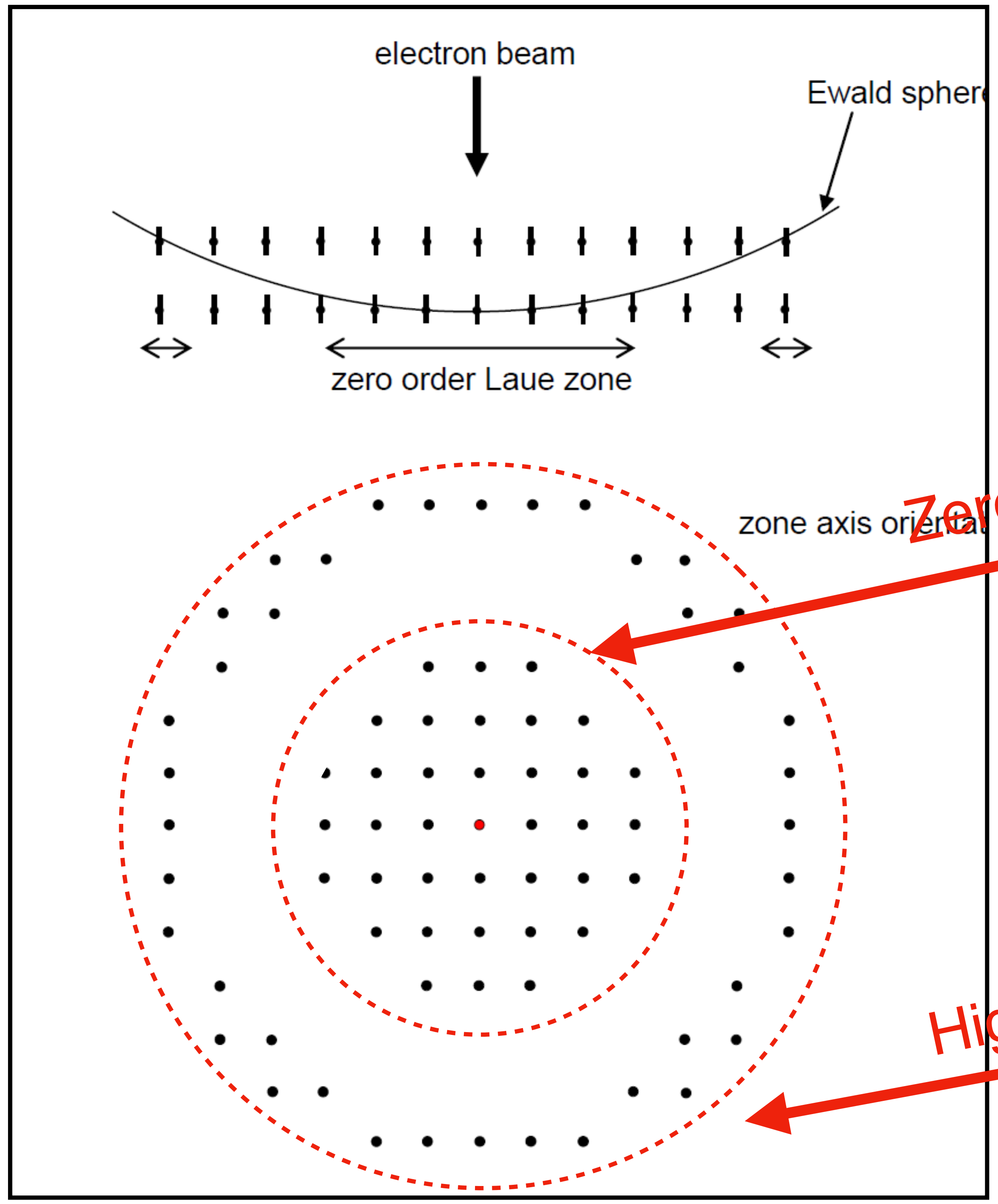


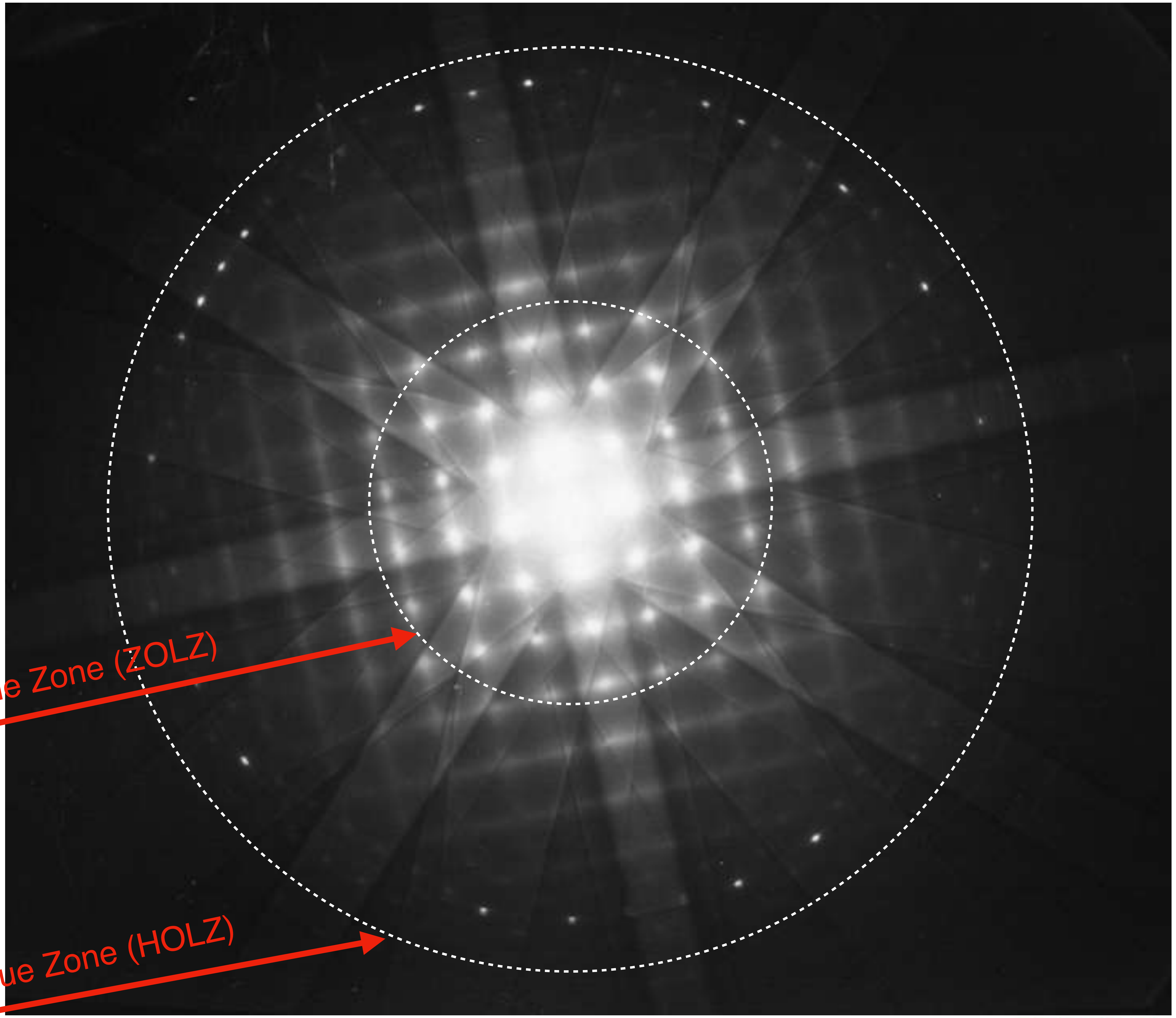
In TEM we will always assume

$$\vec{s} = \vec{s}_{\vec{g}}$$

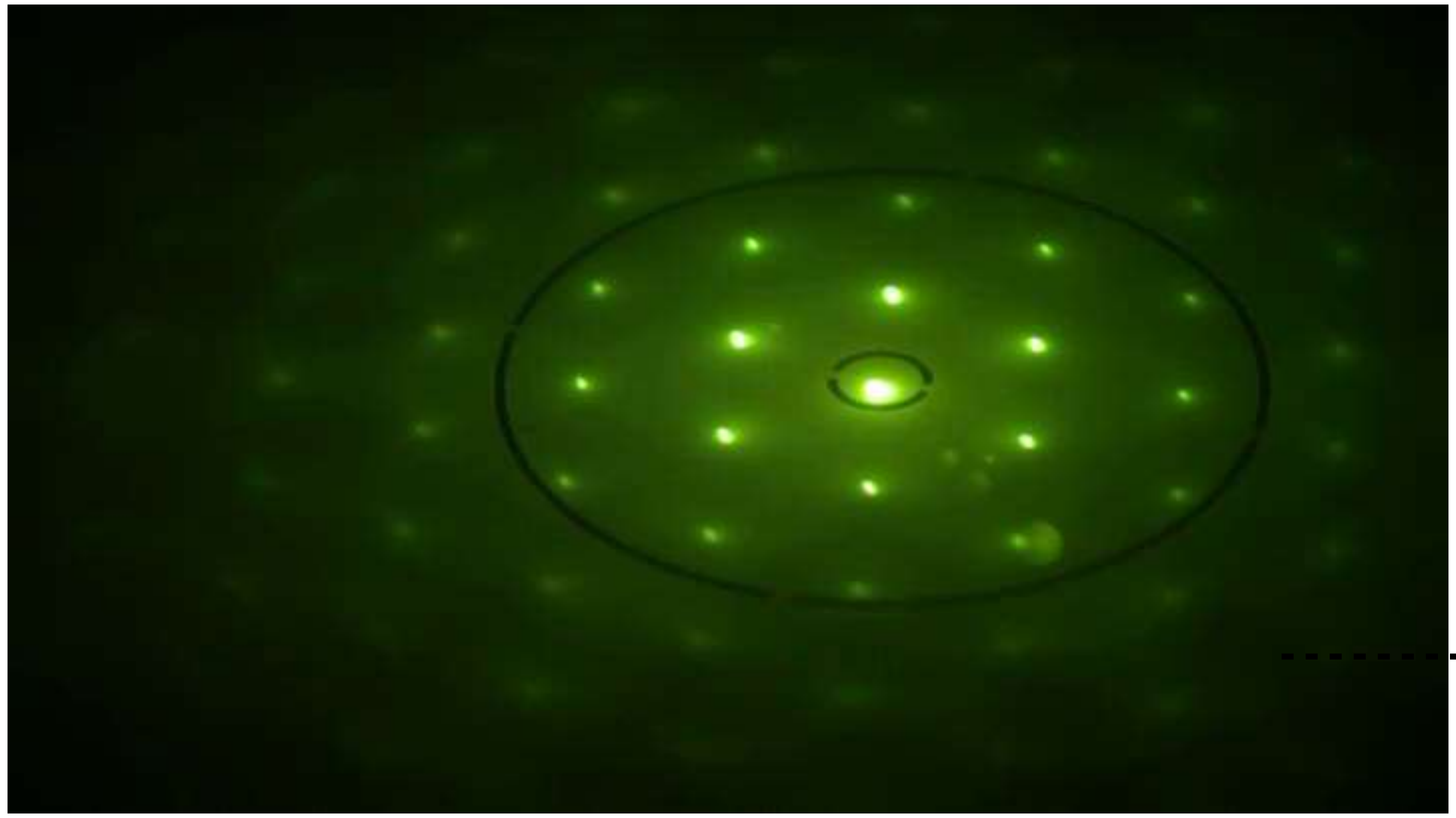
Introduction to electron diffraction theory : geometry of a diffraction pattern

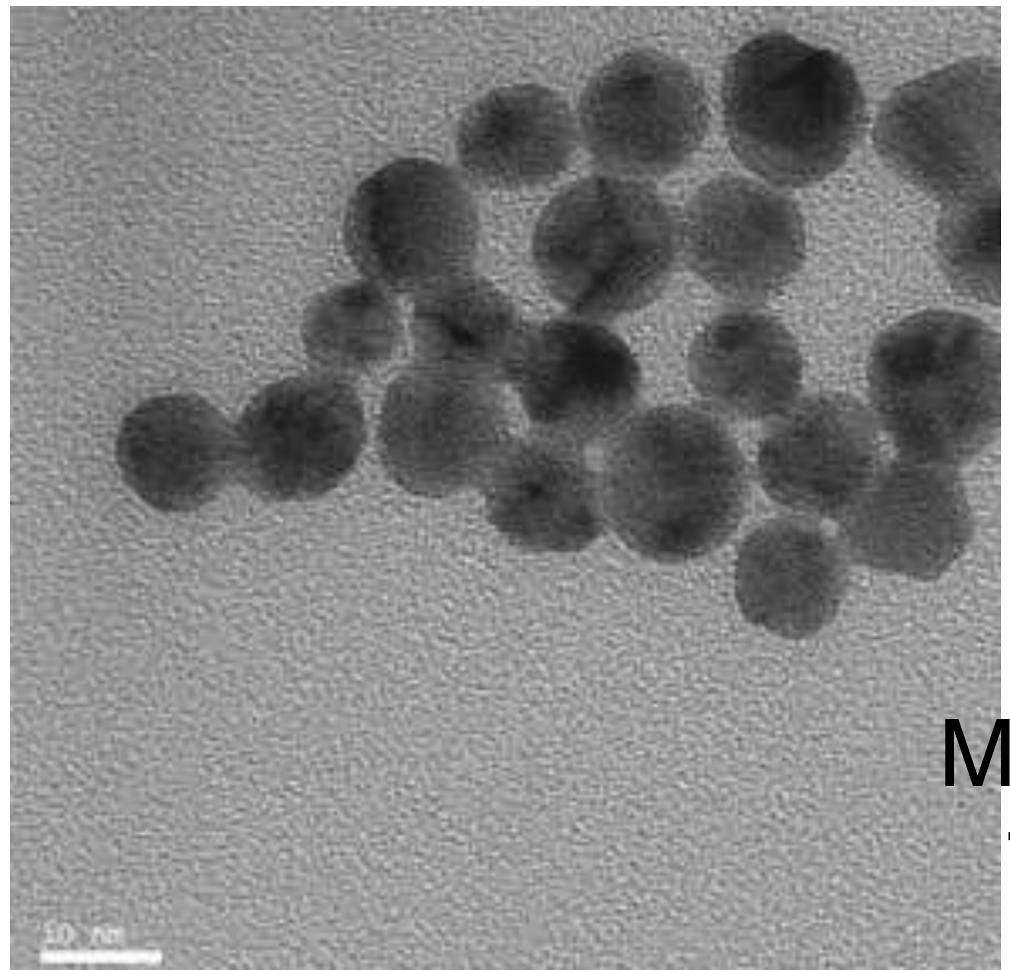


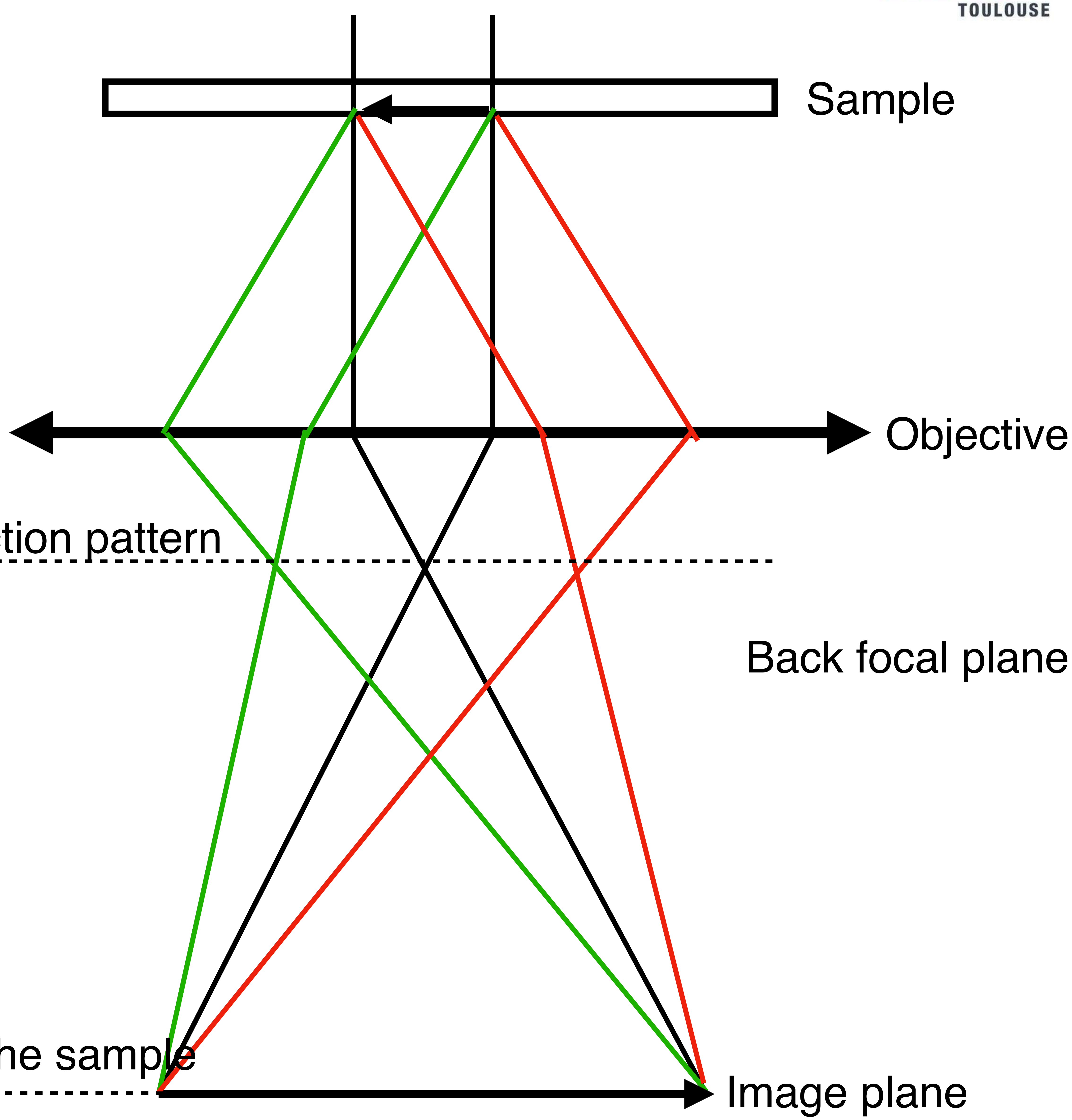




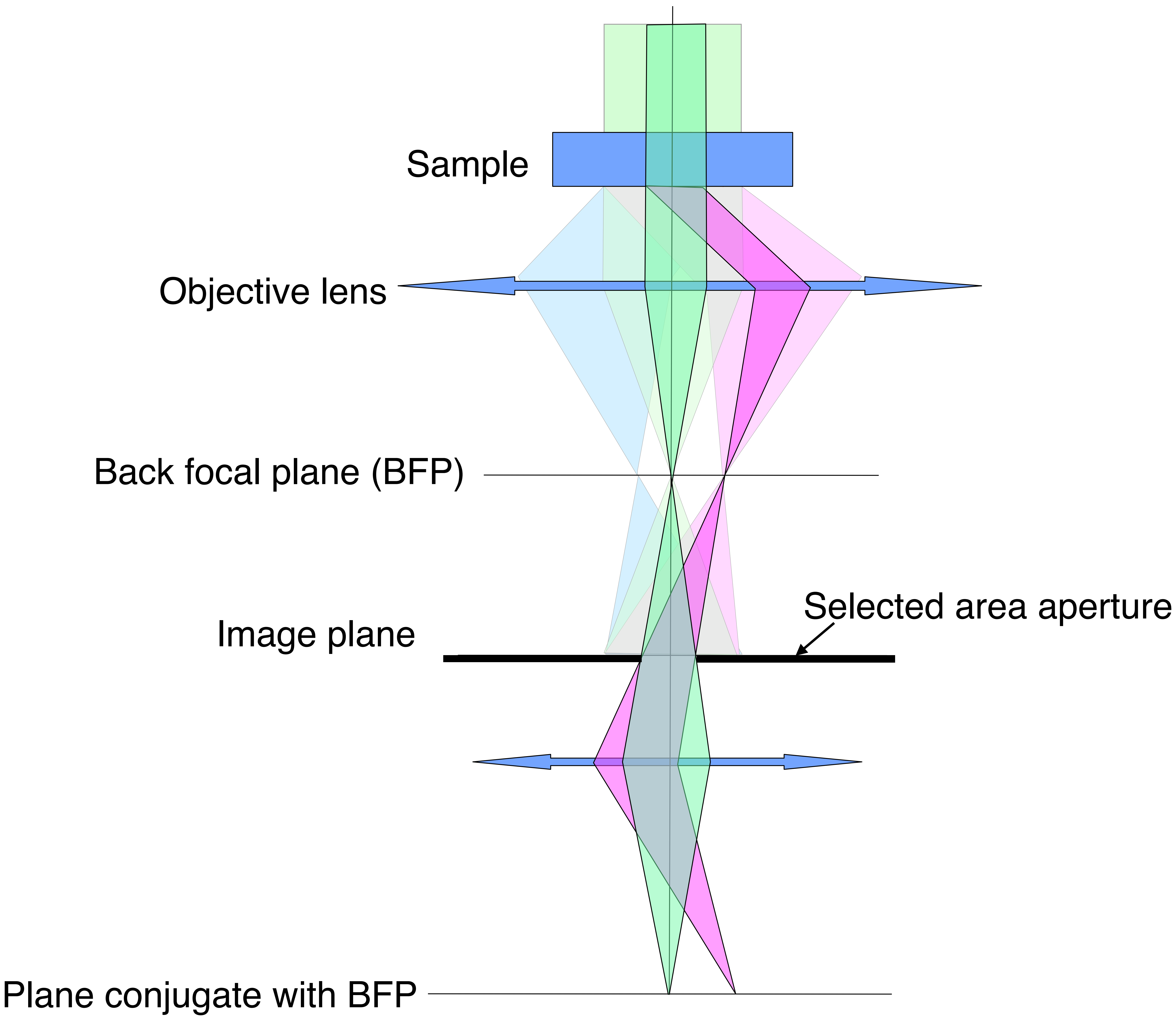
Diffraction in a TEM and image formation : the Abbe theory of imaging

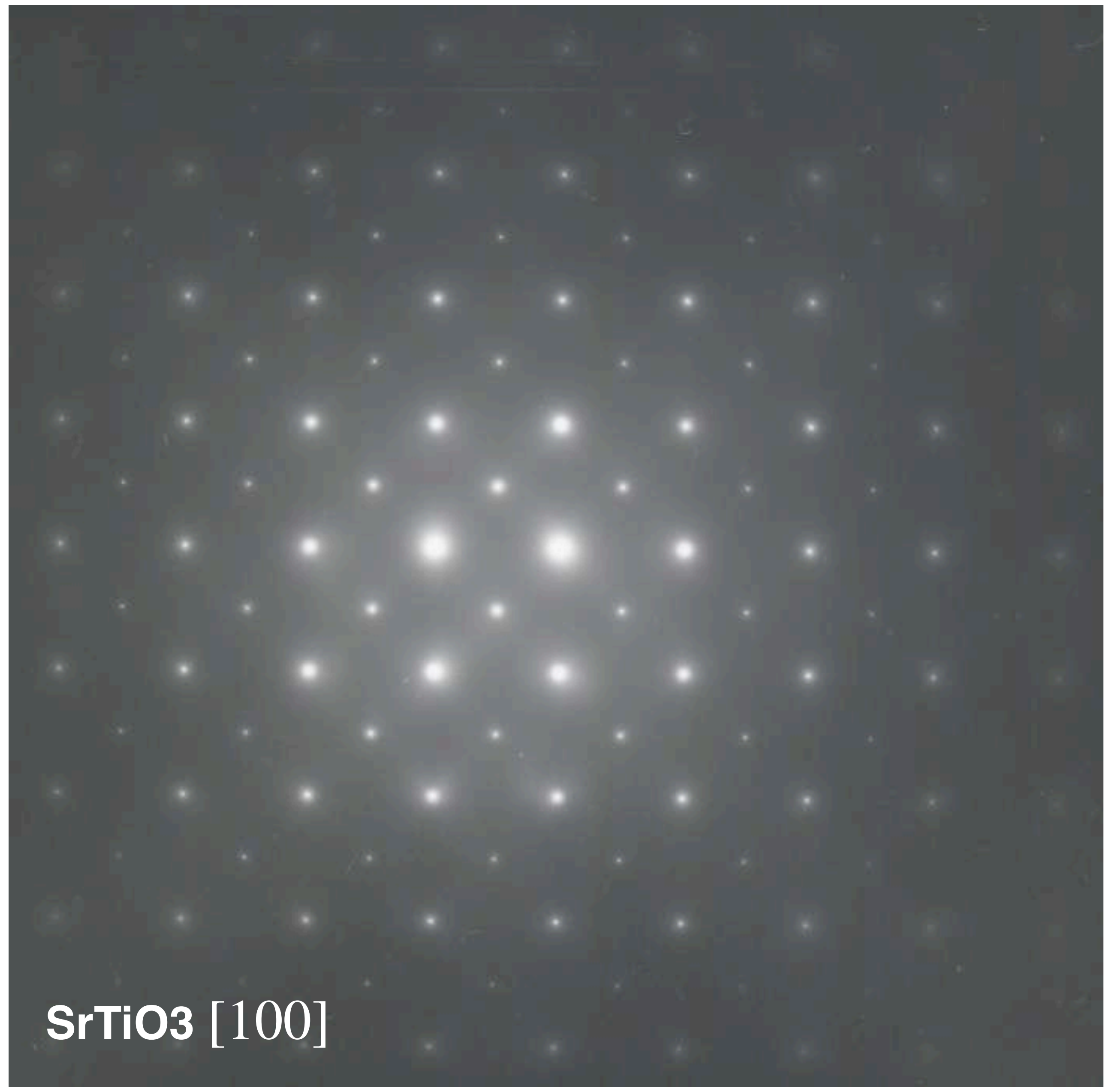


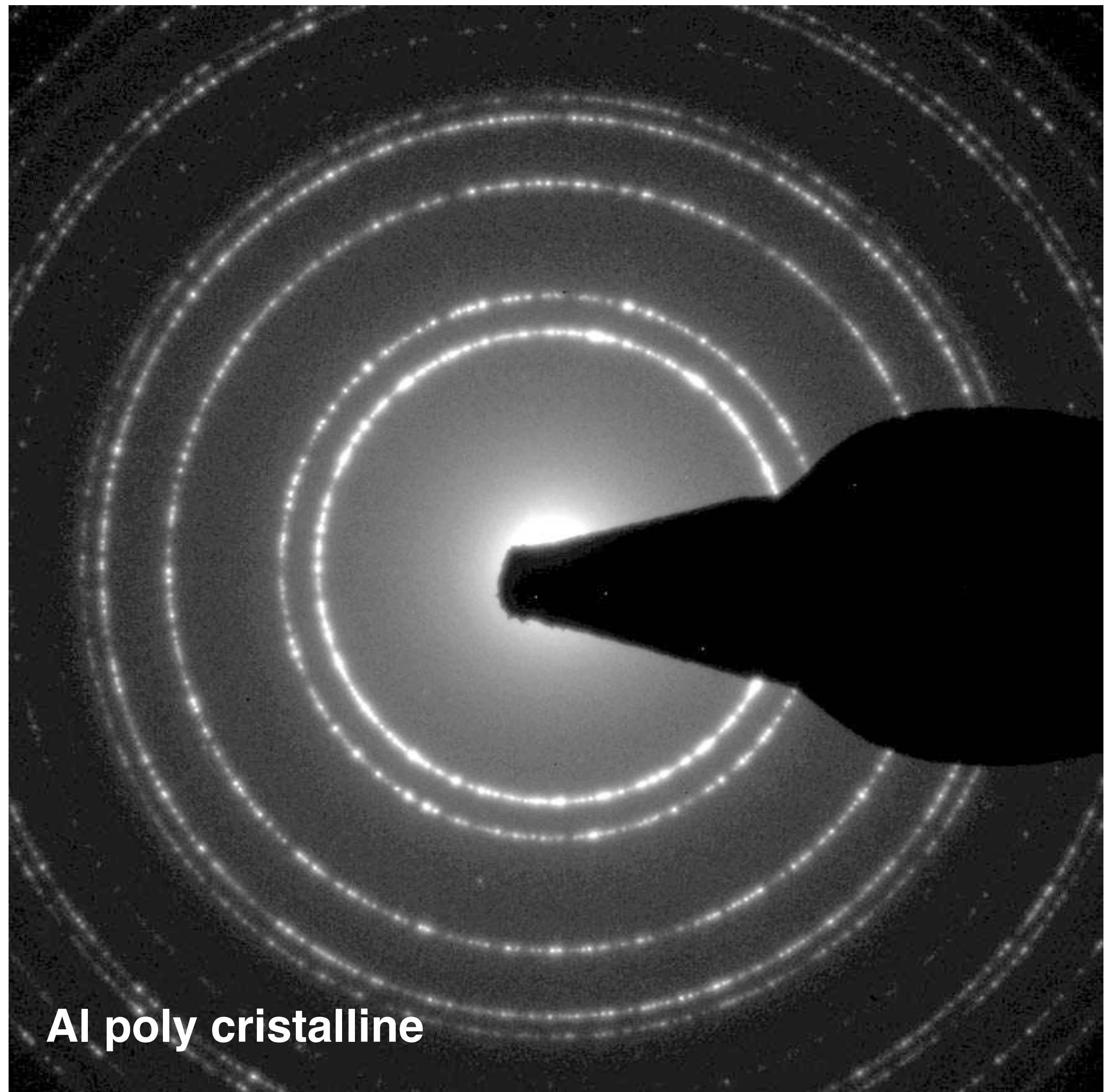




Selected area electron diffraction (SAED) : strength of the TEM

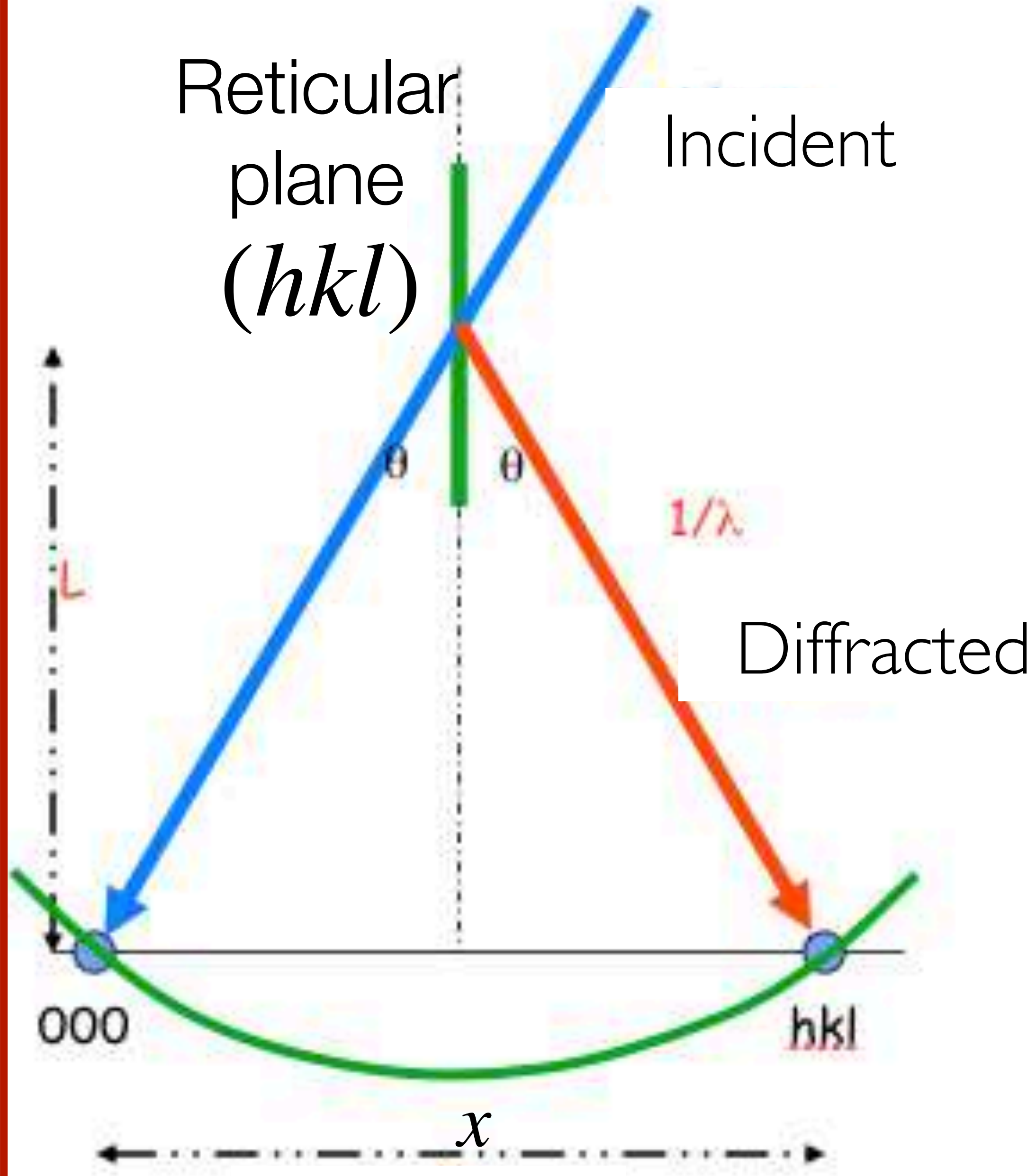




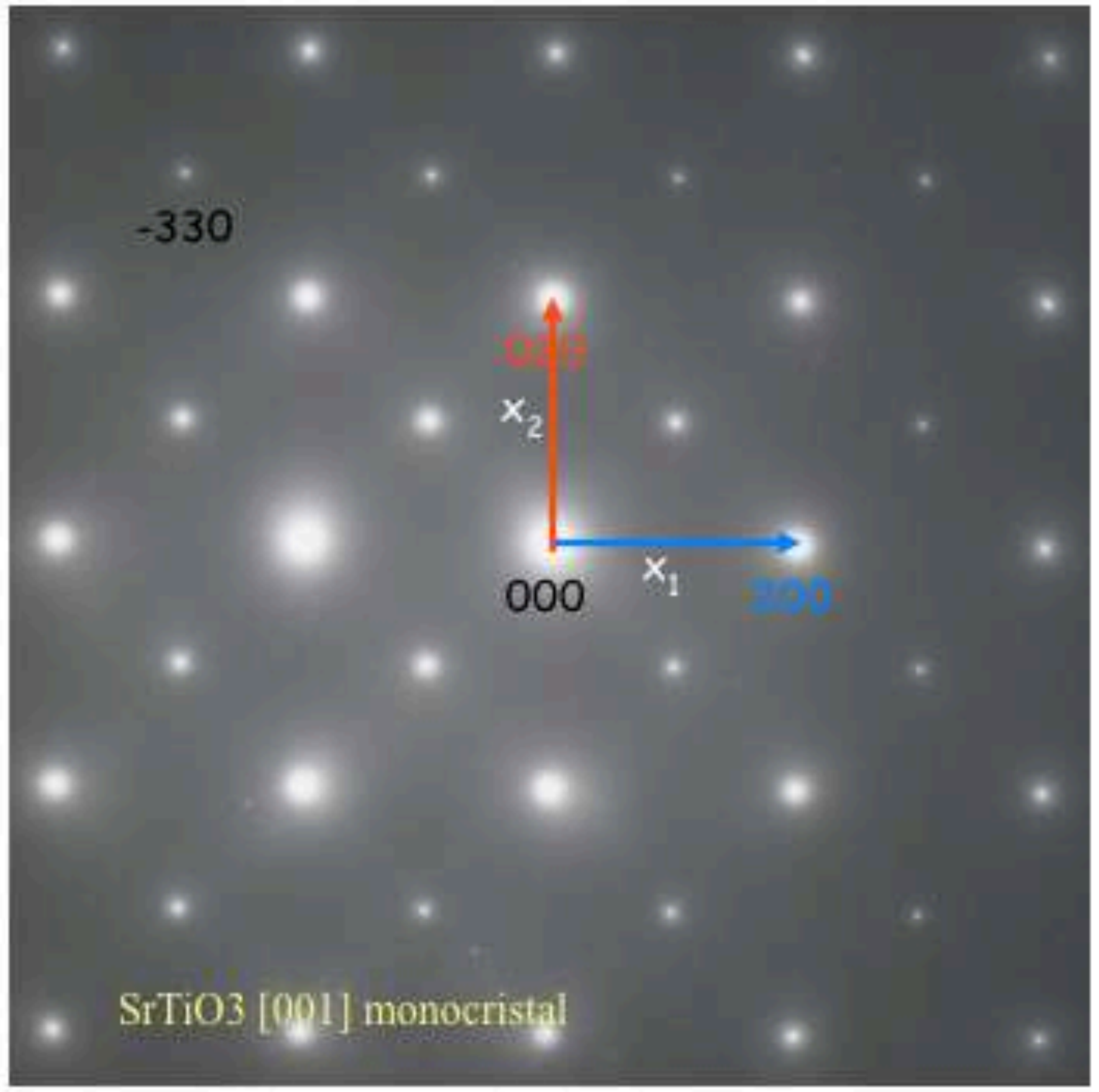


Example of conventional diffraction analysis : mono vs poly / crystal

Monocrystal



$$x d_{(hkl)} = \lambda L$$



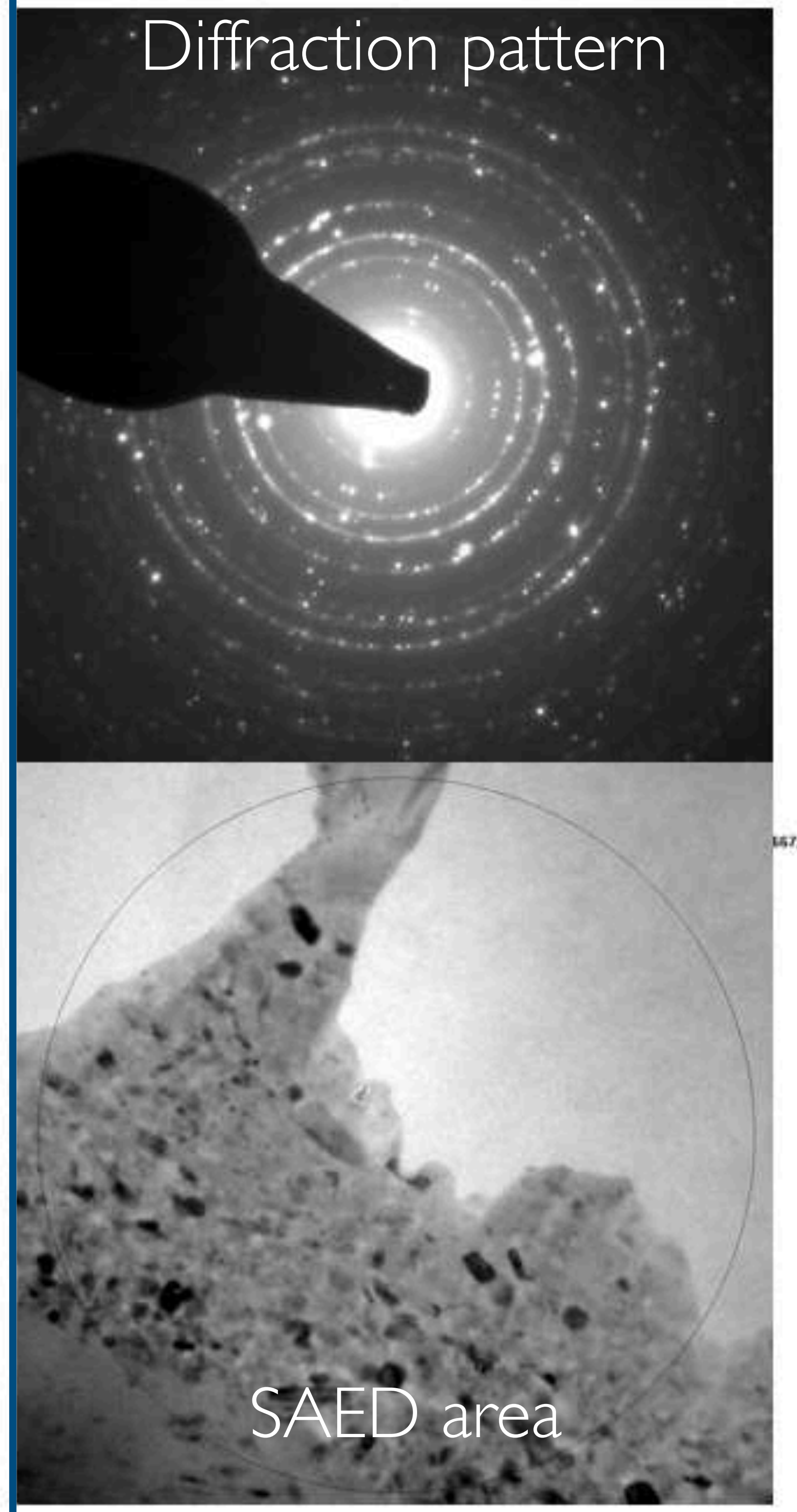
Zone axis

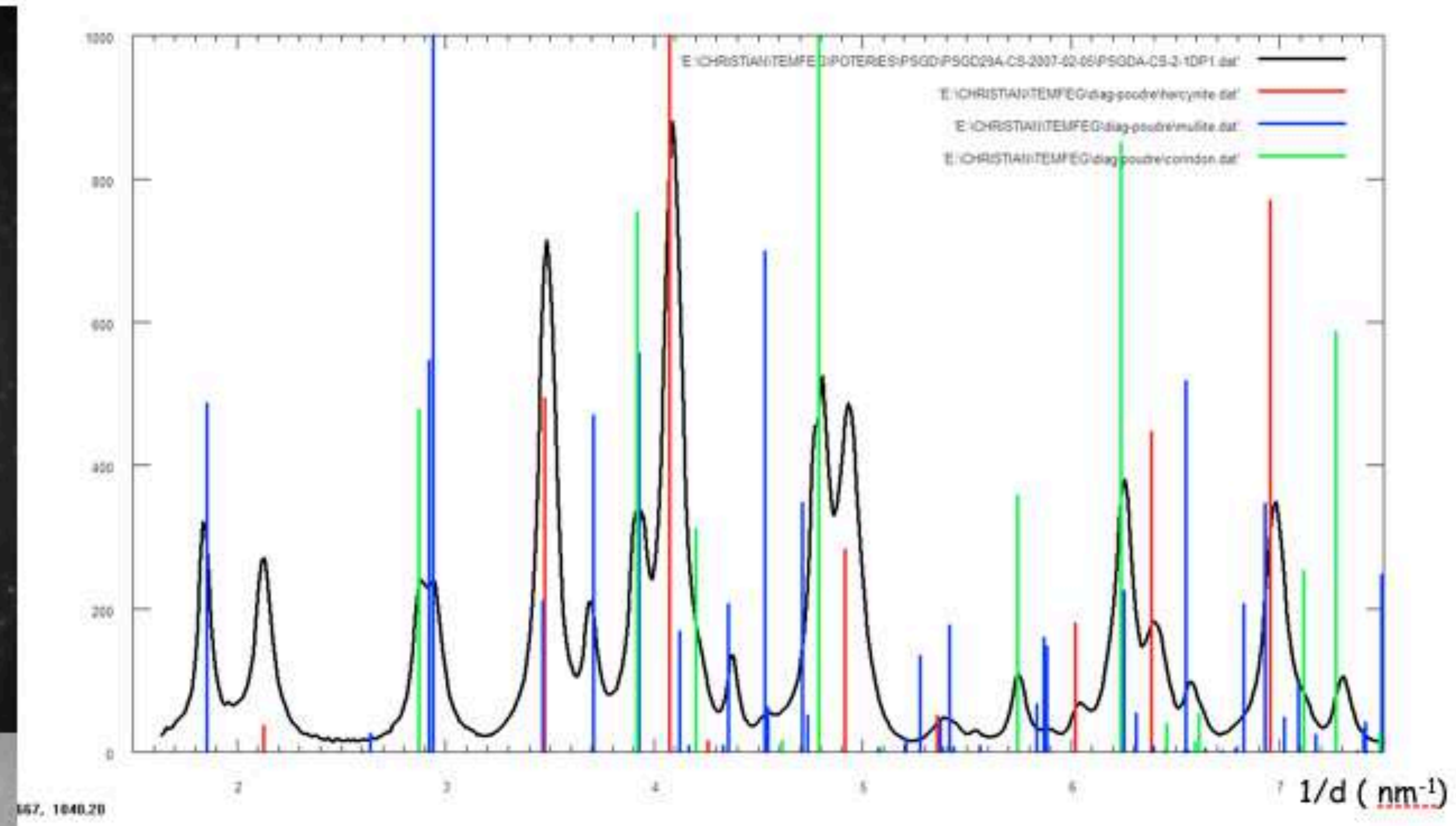
→ x_1 ⟶ d_{hkl1} ⟶ 200

x_2 ⟶ d_{hkl2} ⟶ 020

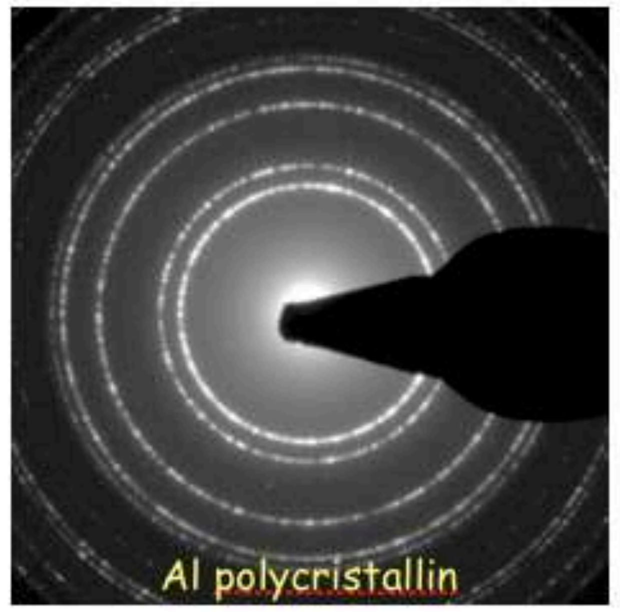
$$200 \wedge 020 \implies [001]$$

Polycrystal



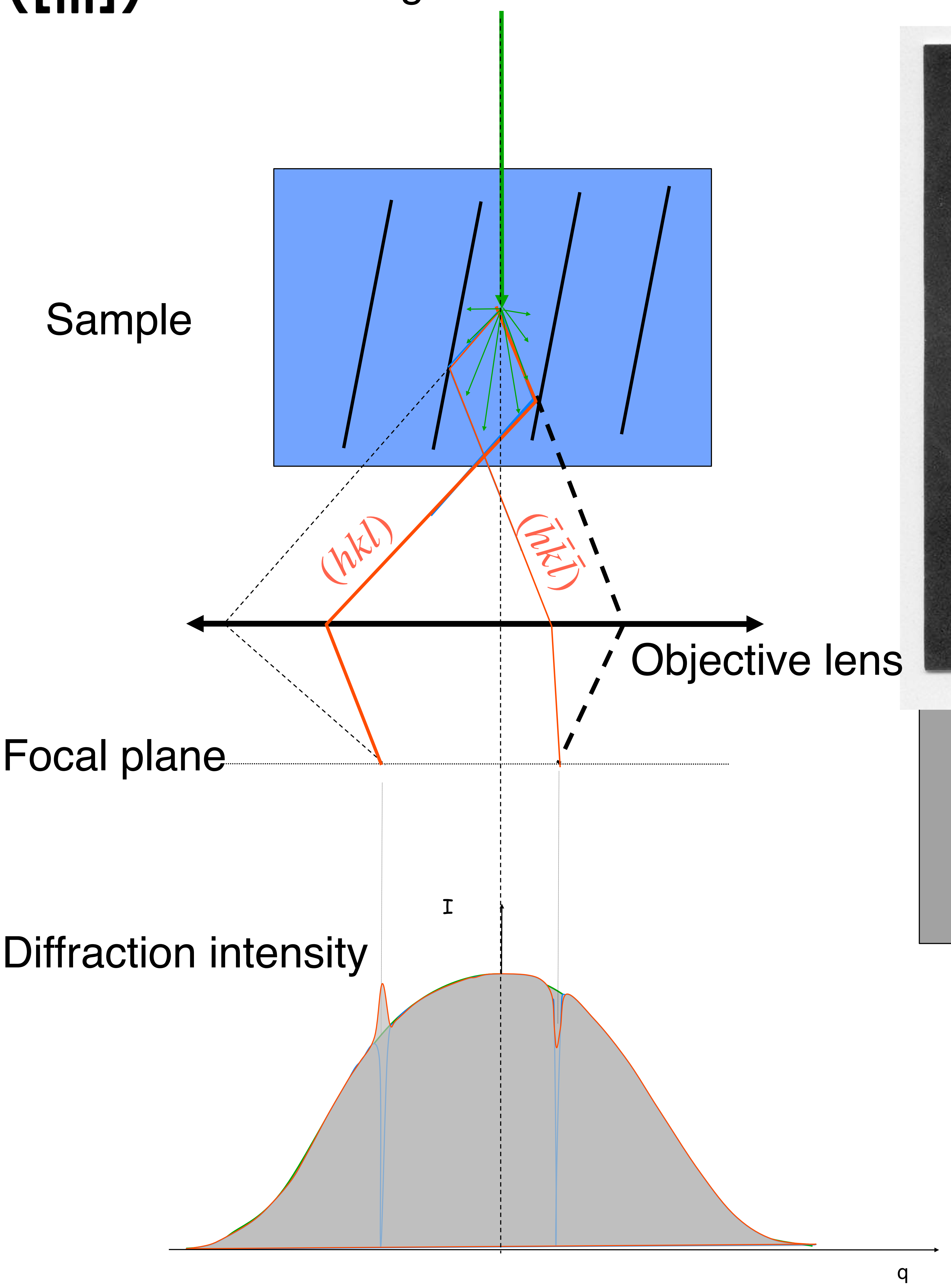


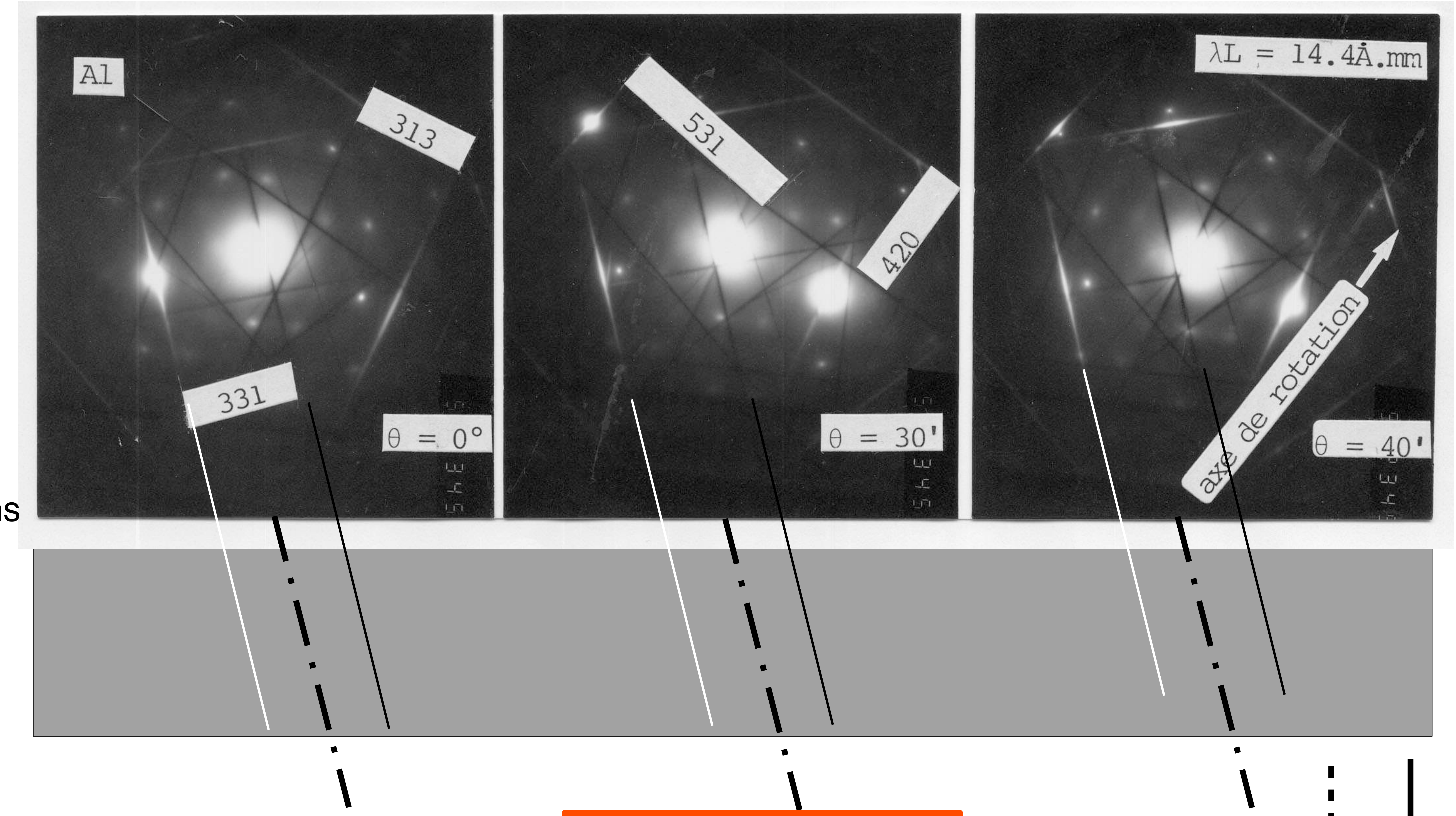
$$d_{(hkl)} = \frac{\lambda L}{R}$$



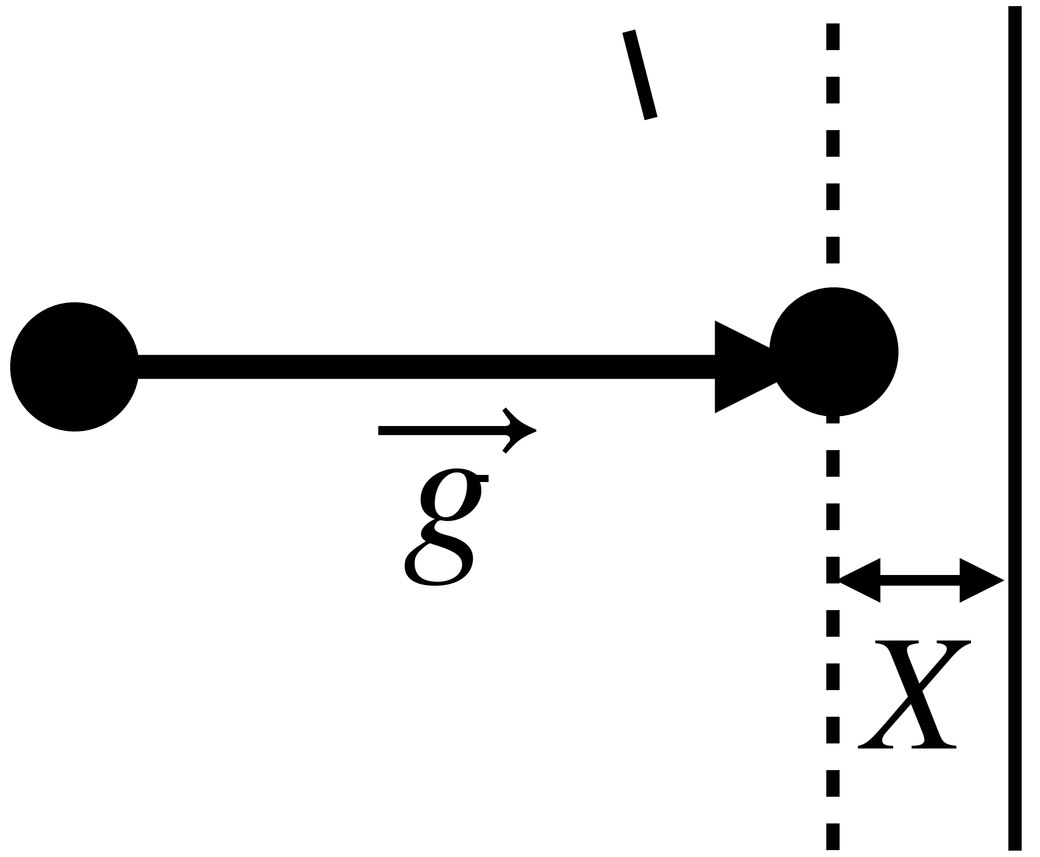
Calibration

Kikuchi lines



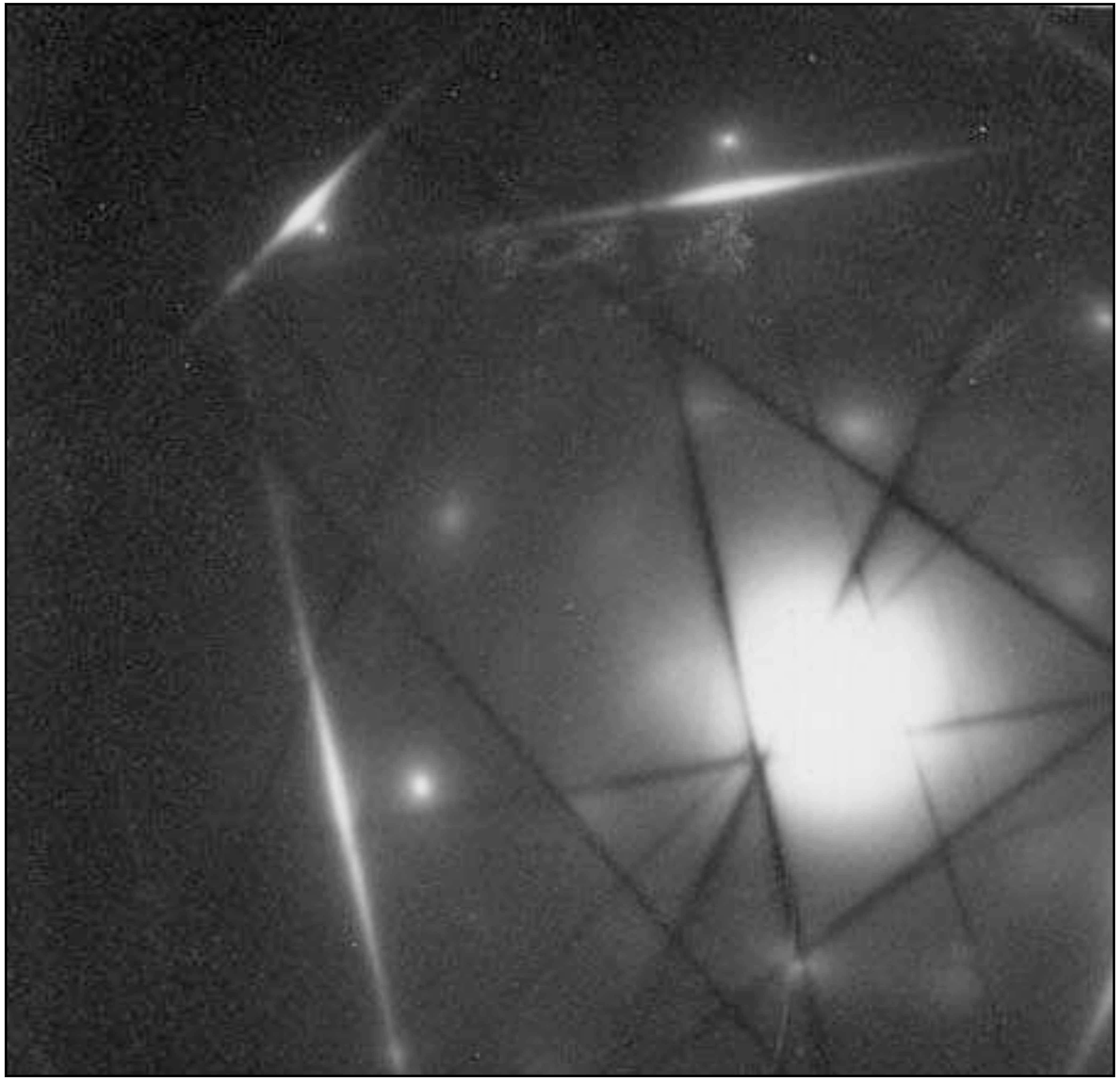


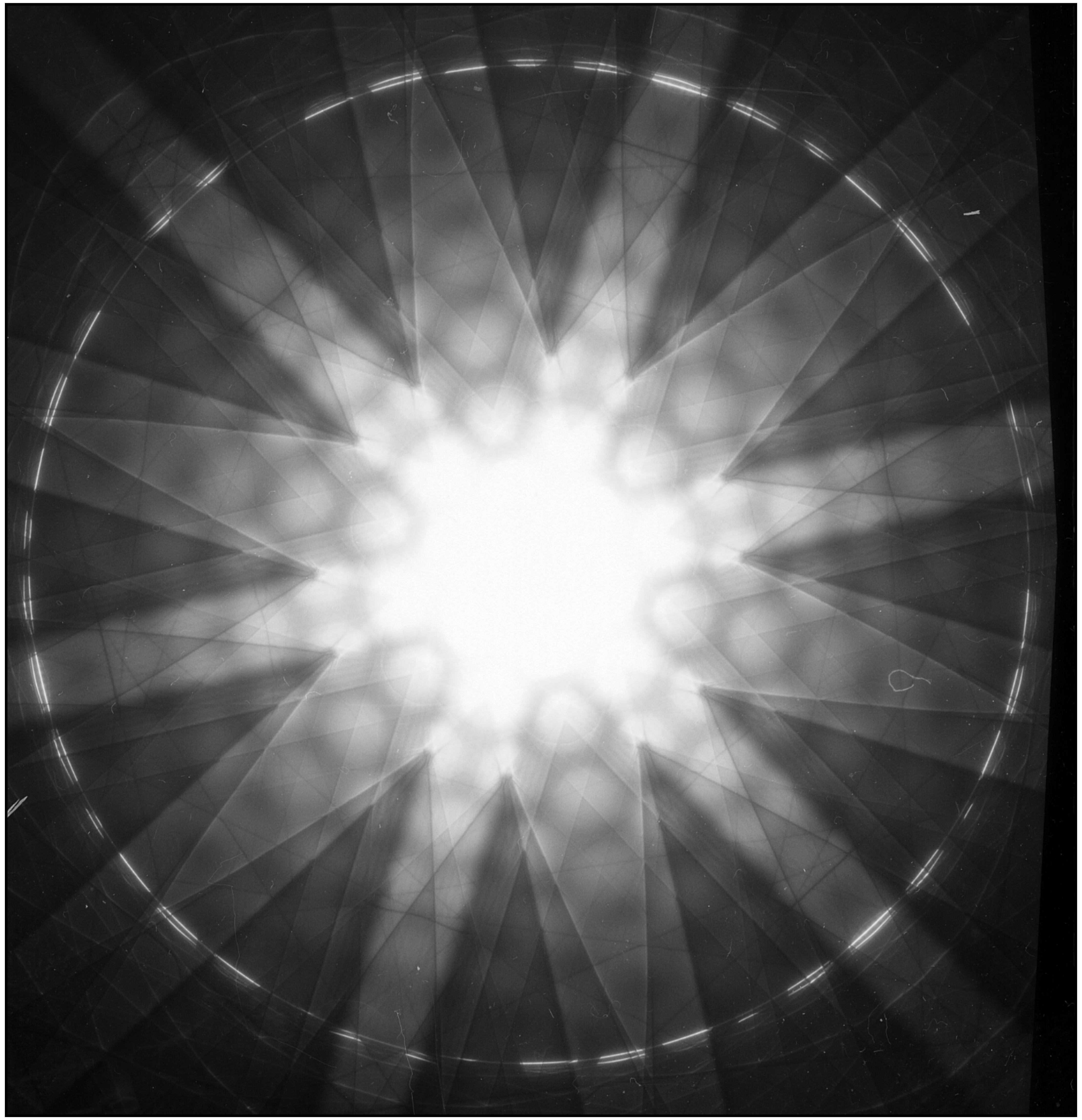
$$s = \frac{X}{g}\frac{\lambda}{d_{(hkl)}^2}$$



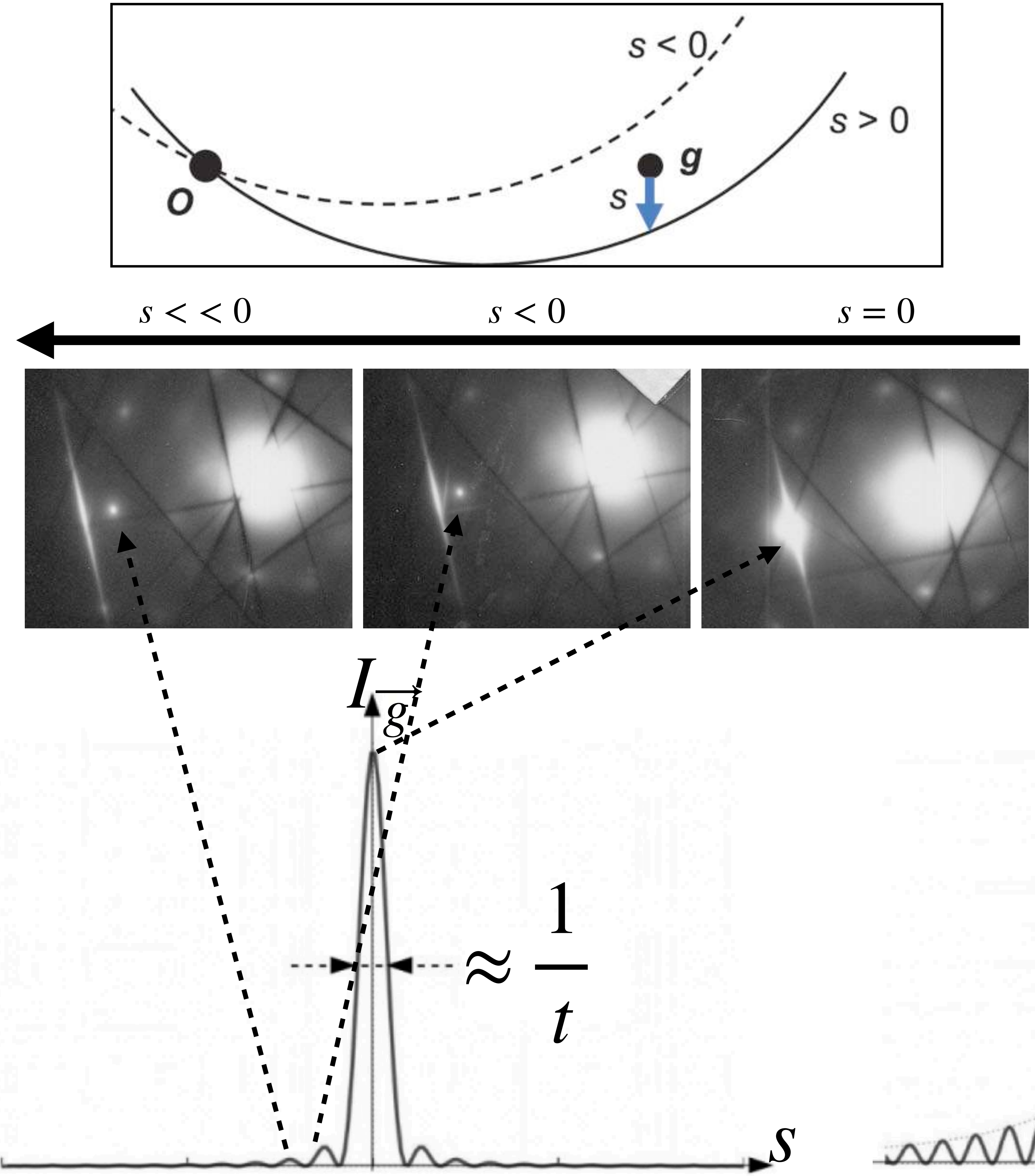
- The spots positions are « attached » to the illumination condition (Ewald sphere)
- The kikuchi lines (and the spot intensities) are « attached » to the sample tilt

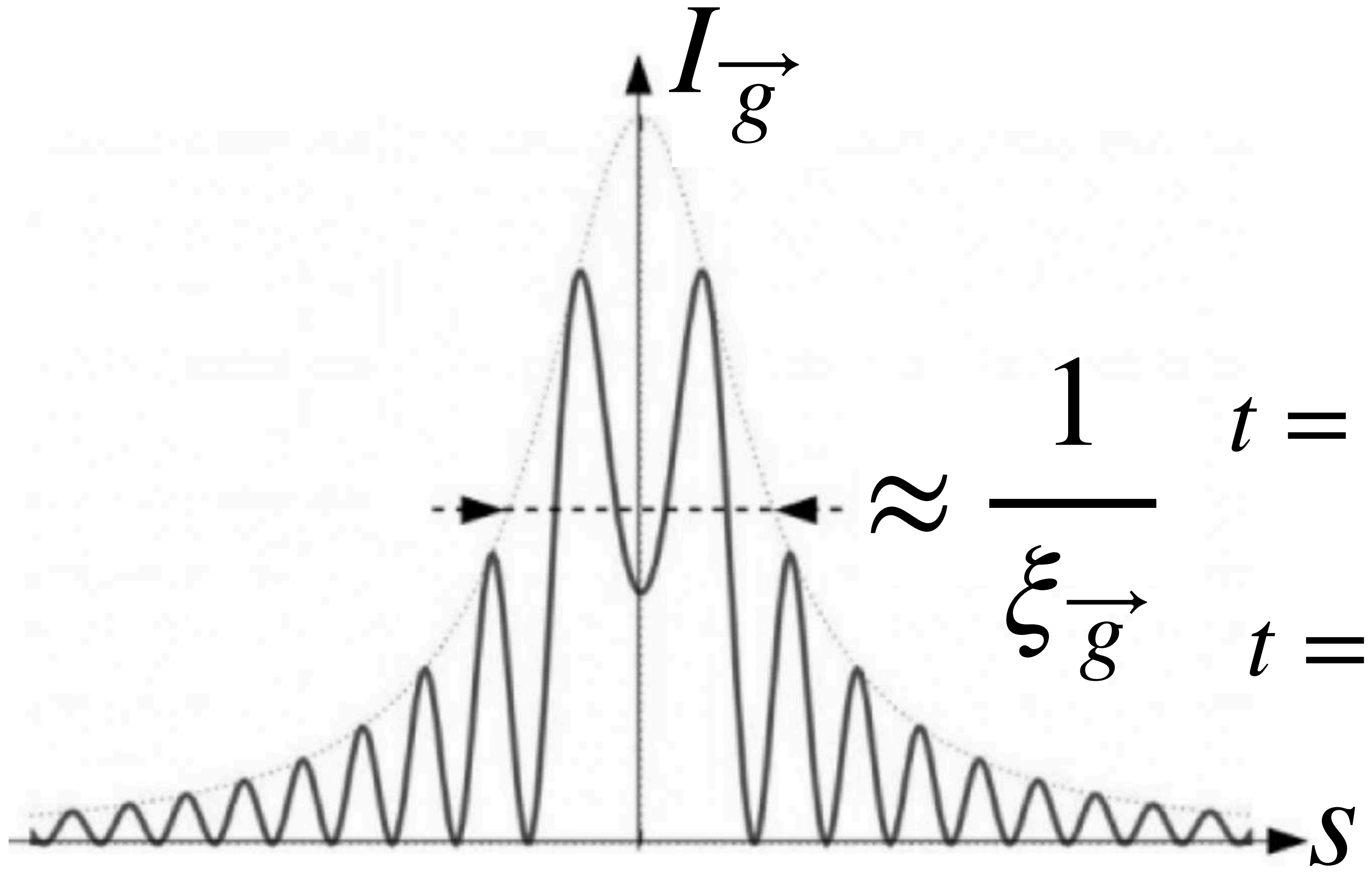
Kikuchi lines : zone axis pattern (ZAP)

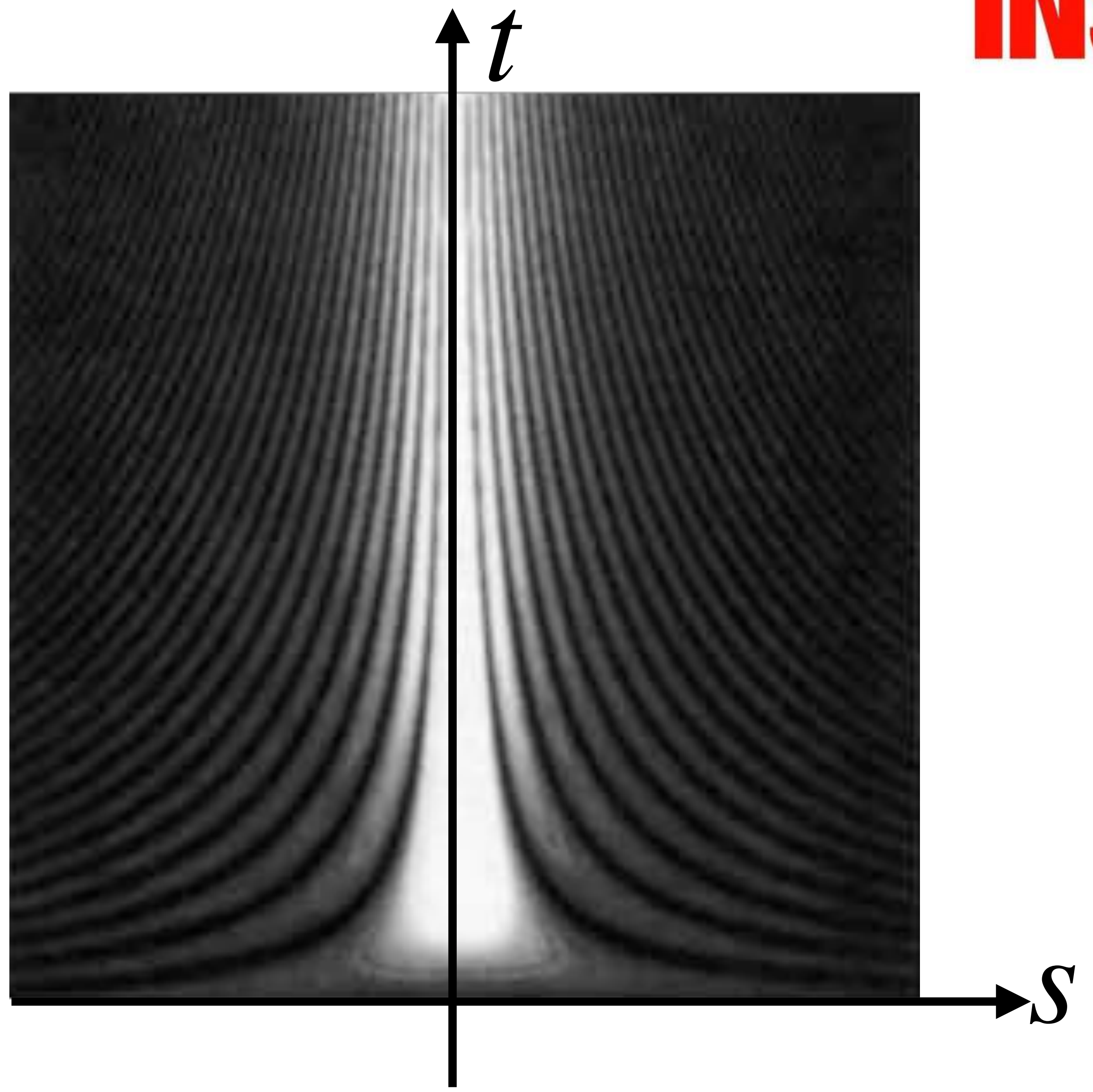


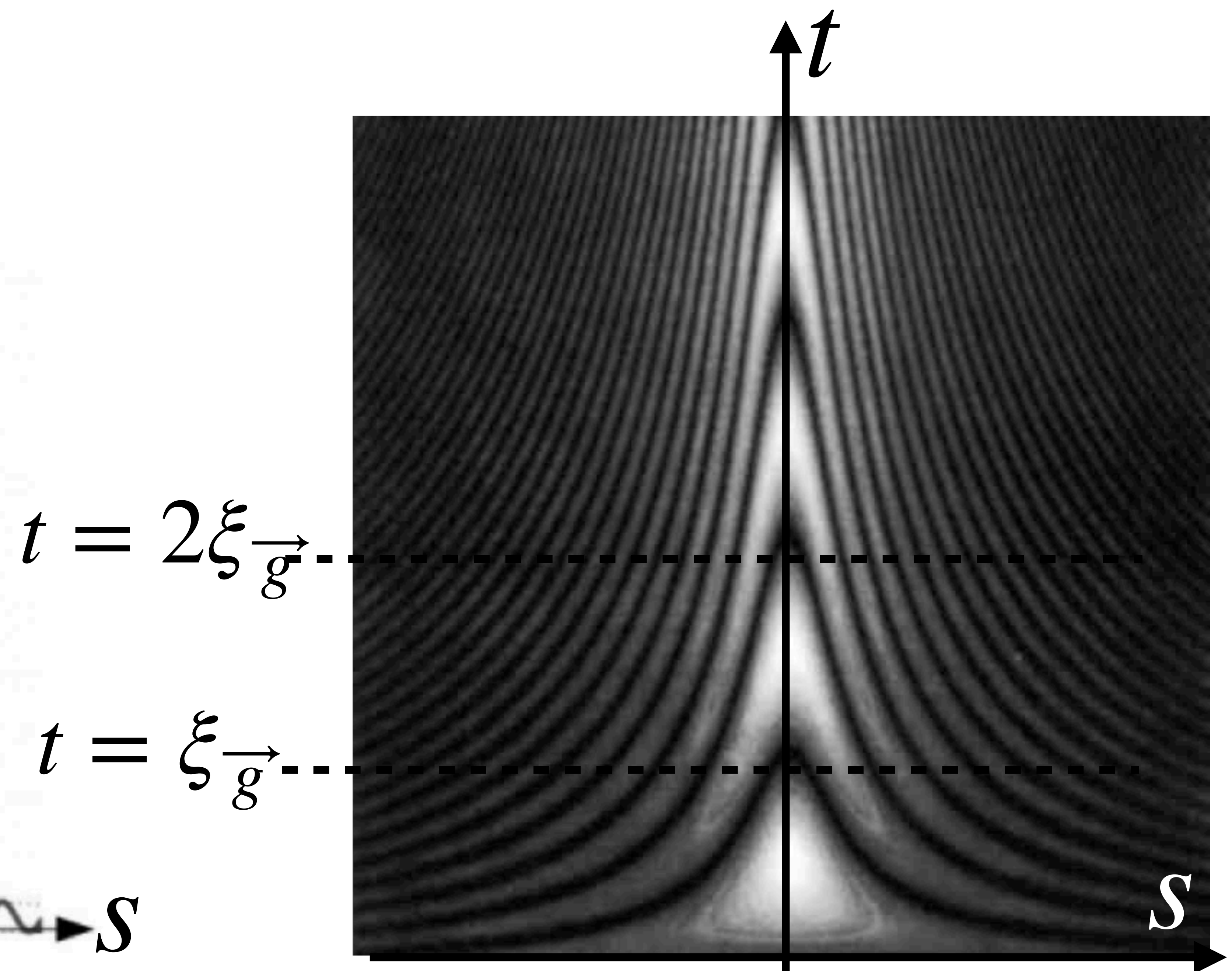


The rocking curve

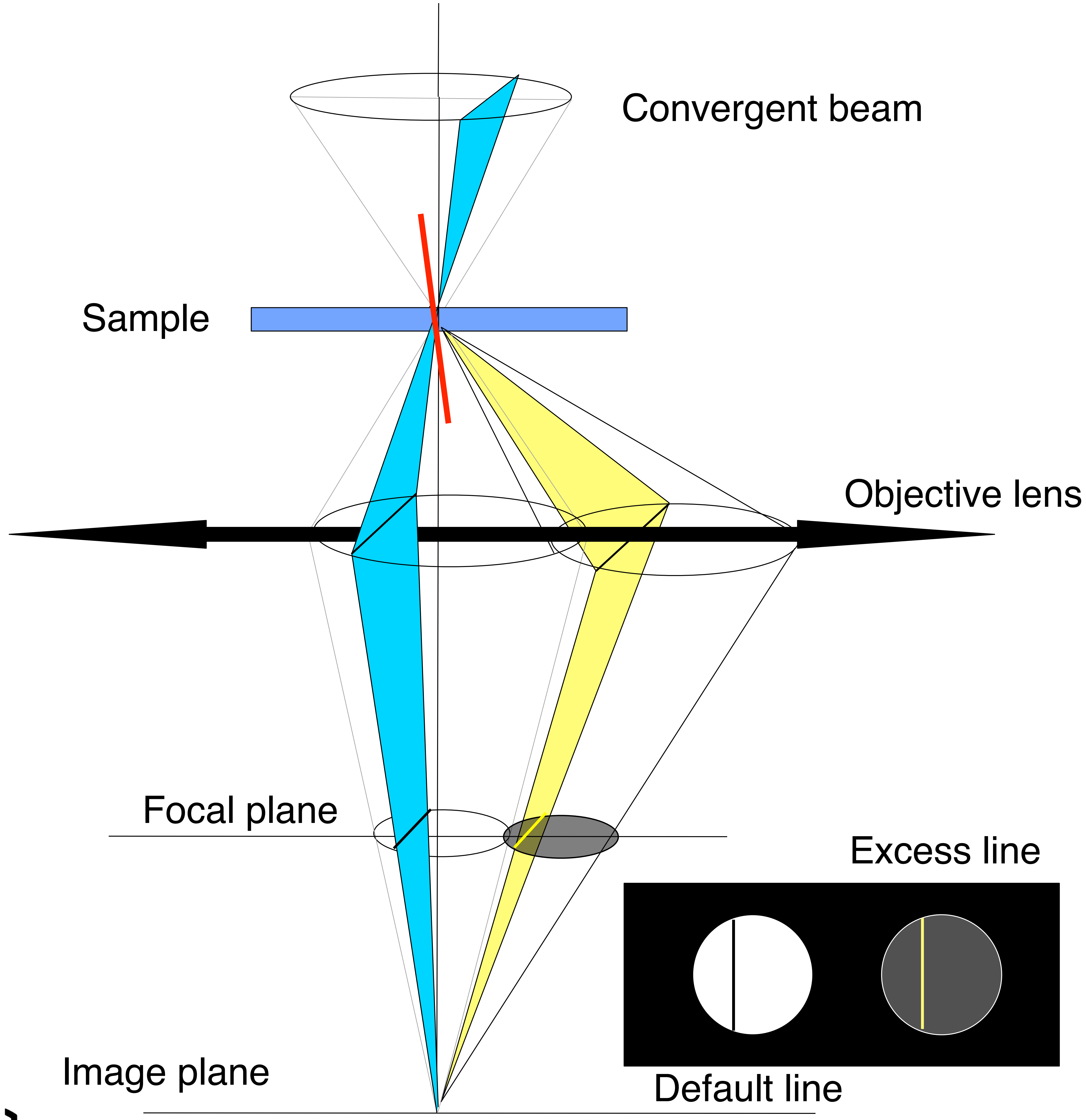


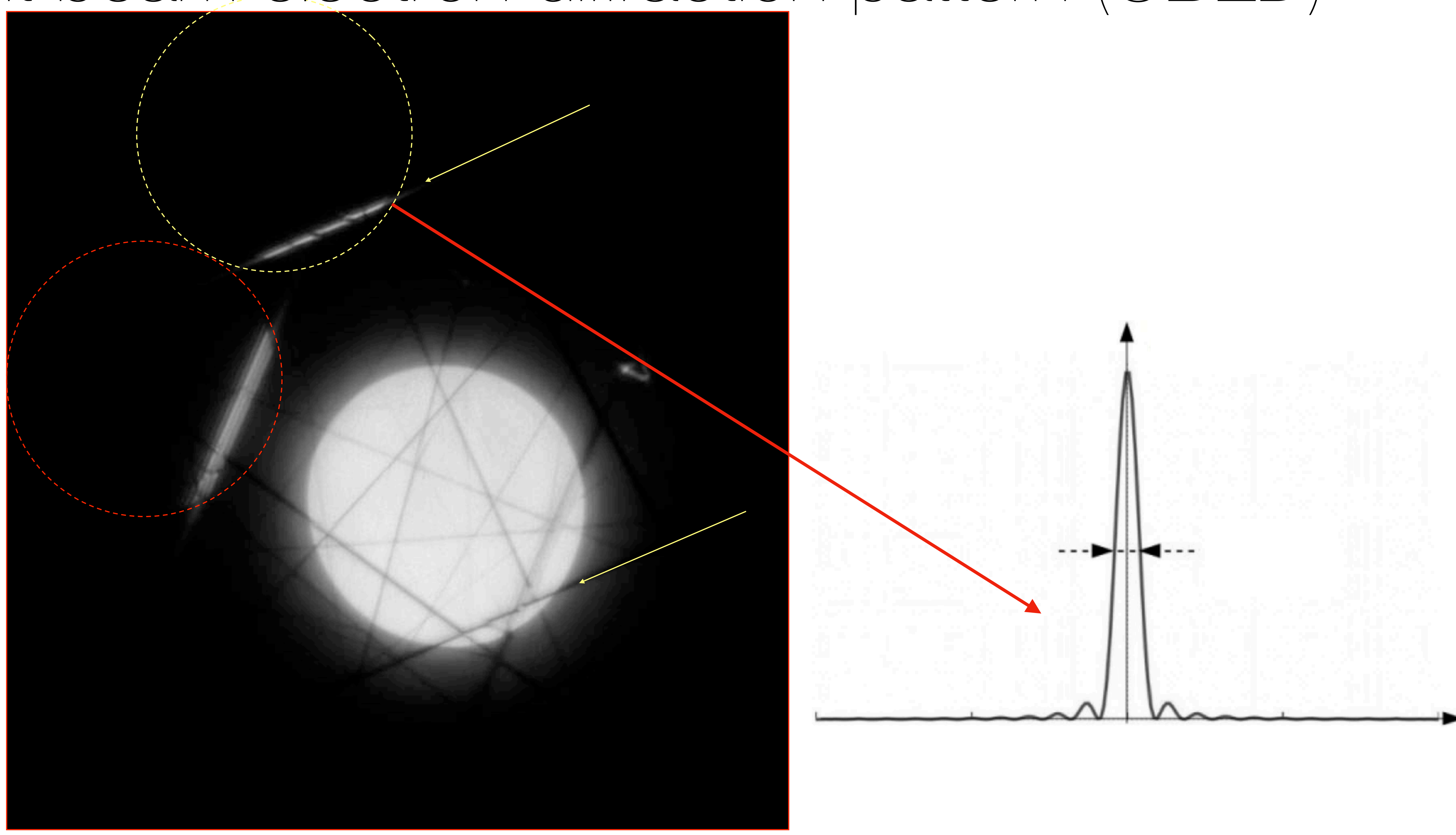


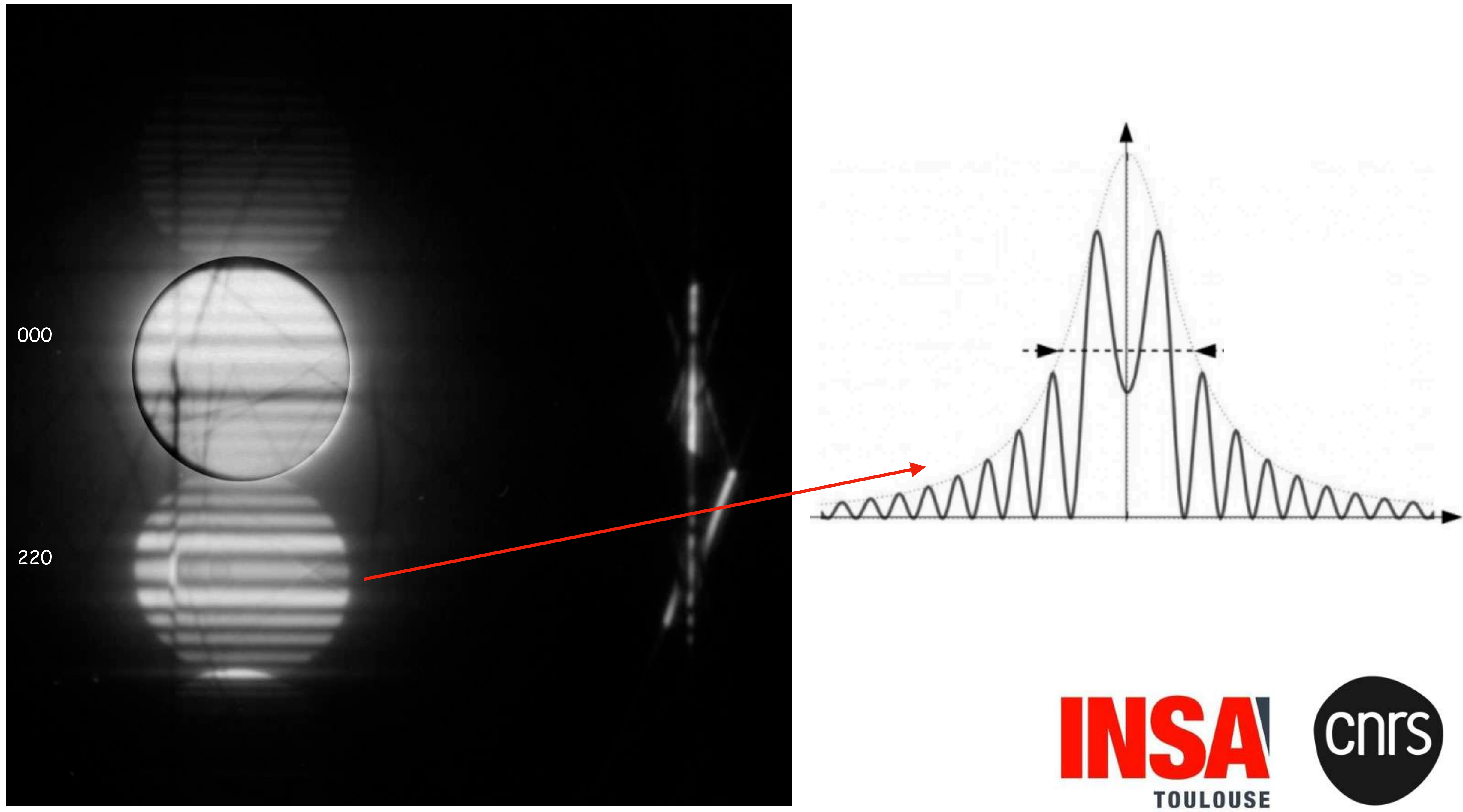




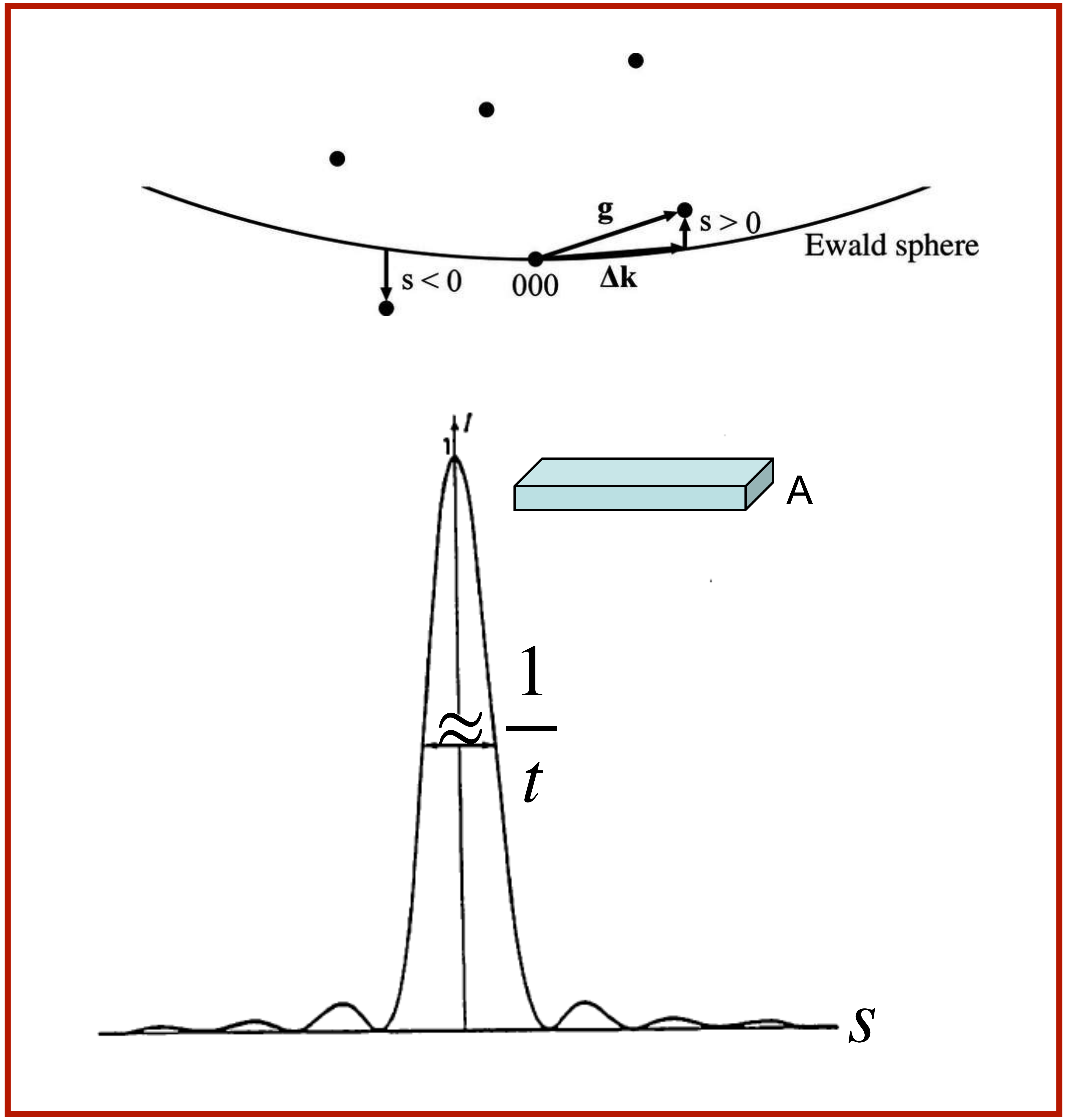
Mapping of the rocking curves : the convergent beam electron diffraction pattern (CBED)

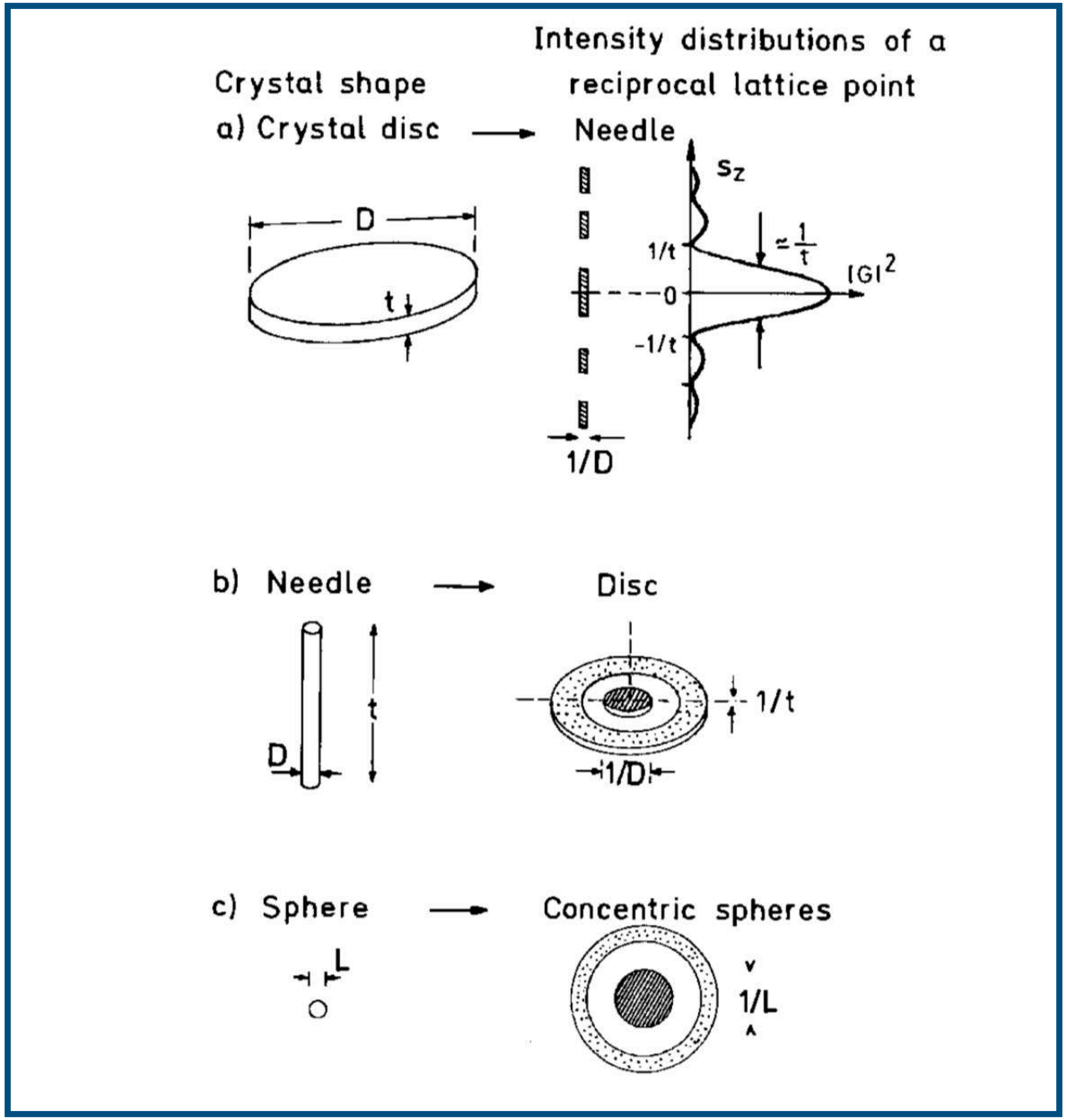




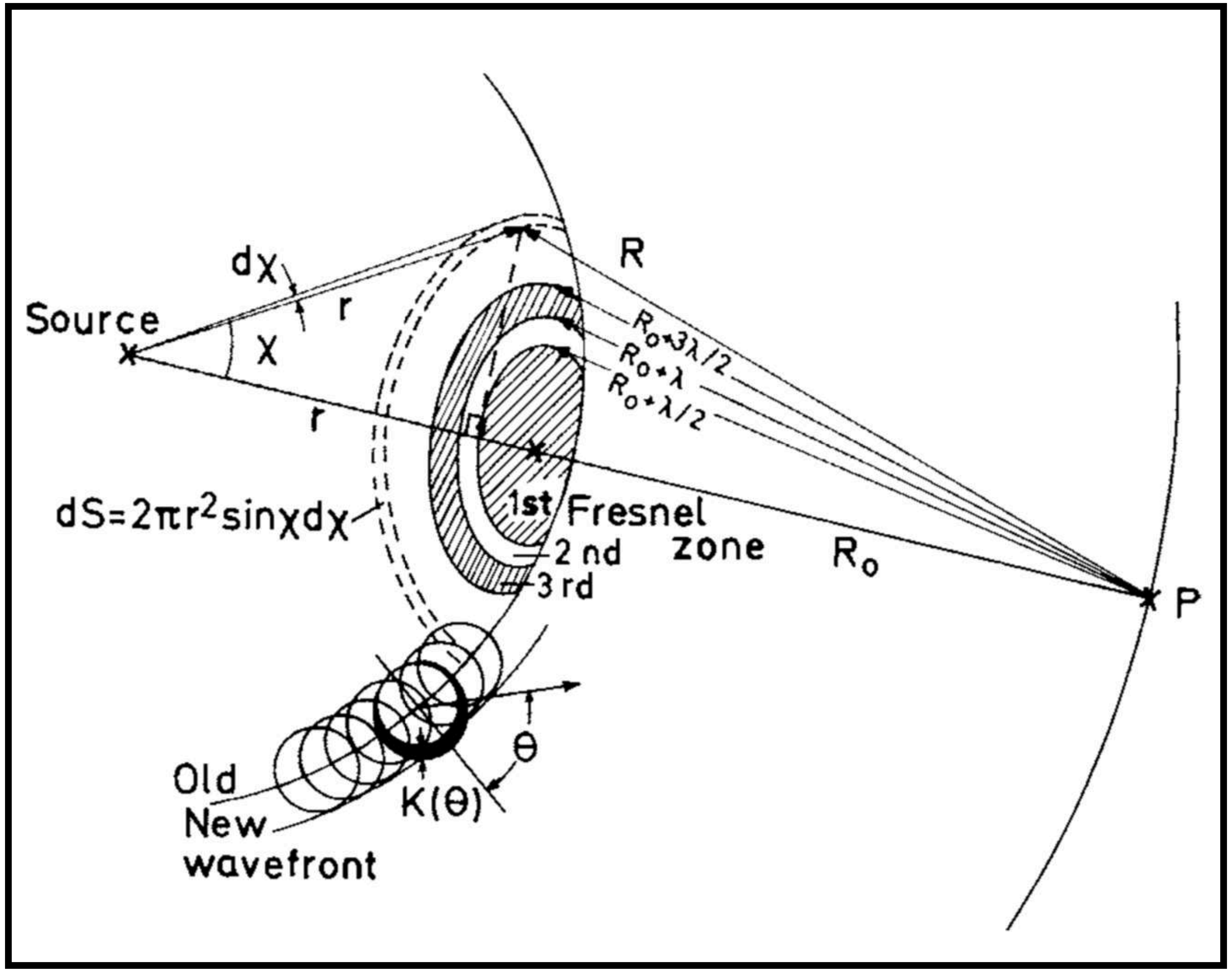


Shape transforms





Huygens-Fresnel principle : spherical wave propagation



Huygens-Fresnel principle :
The secondary wave amplitude at the point P
is obtained by summing the amplitudes
of the spherical wavelets from a spherical wavefront of radius r.

(I) Incoming spherical wave :

$$\psi = A_Q \frac{e^{2\pi i k r}}{r}$$

(II) **Huygens-Fresnel principle** : from each point, emission of wavelets :

$$d\psi = K(\theta)\psi \frac{e^{2\pi i k R}}{R} dS \qquad K(\theta) = \frac{1+\cos(\theta)}{2}\frac{1}{i\lambda} = A(\theta)\frac{1}{i\lambda}$$

$$\Rightarrow \psi(P) = \iint_S d\psi = \iint_S K(\theta)\psi \frac{e^{2\pi i k R}}{R} dS$$

(III) Integration variable change due to wavefront geometry

$$dS = r d\chi 2\pi r \sin(\chi) \qquad R =^2 = r^2 + (r+R_0)^2 - 2r(r+R_0)\cos(\chi)$$

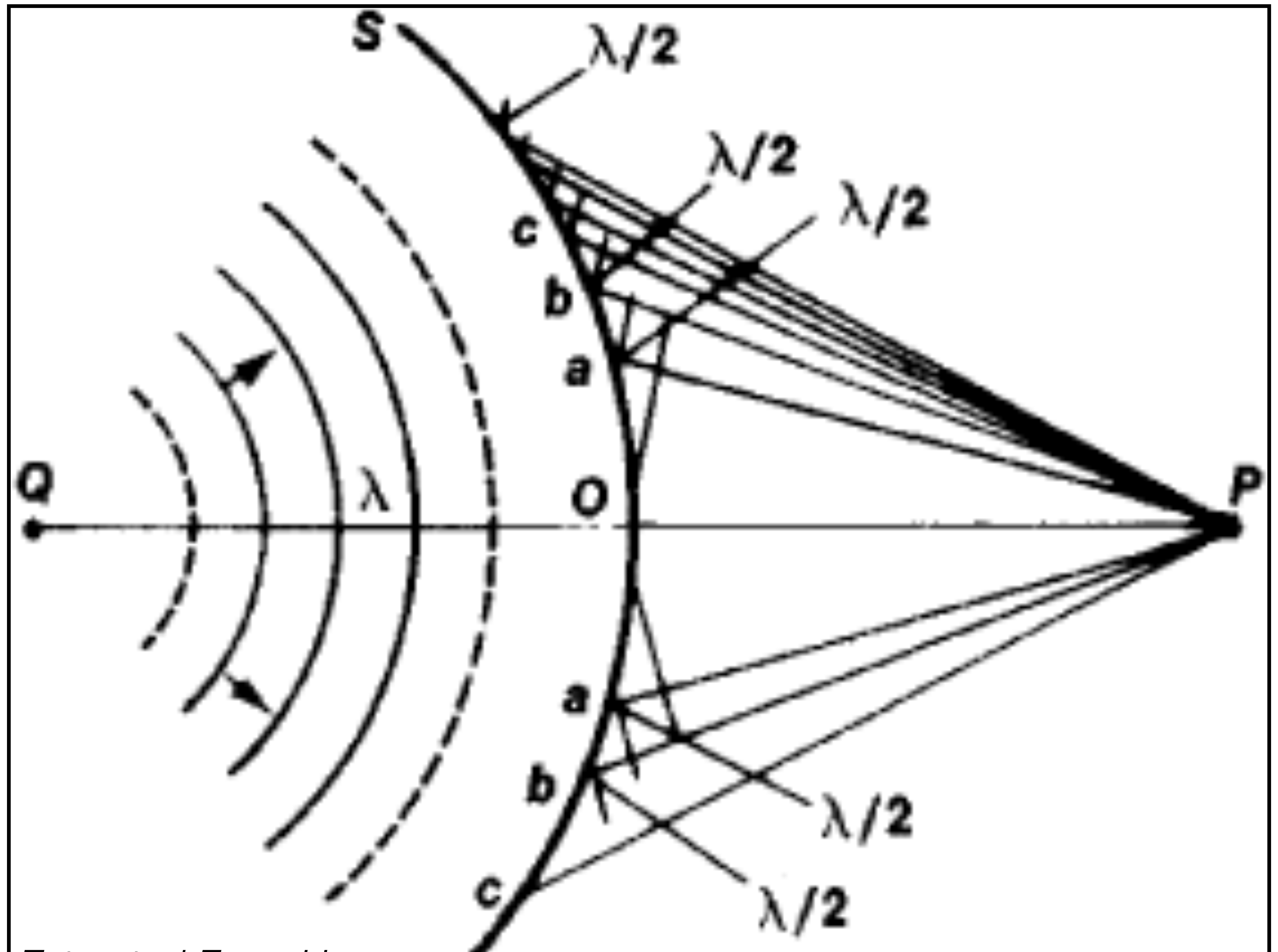
$$dS = 2\pi\left(\frac{r}{(r+R_0)}\right) R dR$$

$$\psi(P) = \frac{2\pi\psi}{i\lambda(r+R_0)} \int_{R_0}^{R_{max}} A(\theta) e^{2\pi i k R} dR$$

Resolution using amplitude phase diagram (APD)

(V) We have to evaluate the following integral $$\int_{R_0}^{\infty} A(\theta) e^{2\pi i k R} dR$$

➡We separate the wave front in ***Fresnel zones***

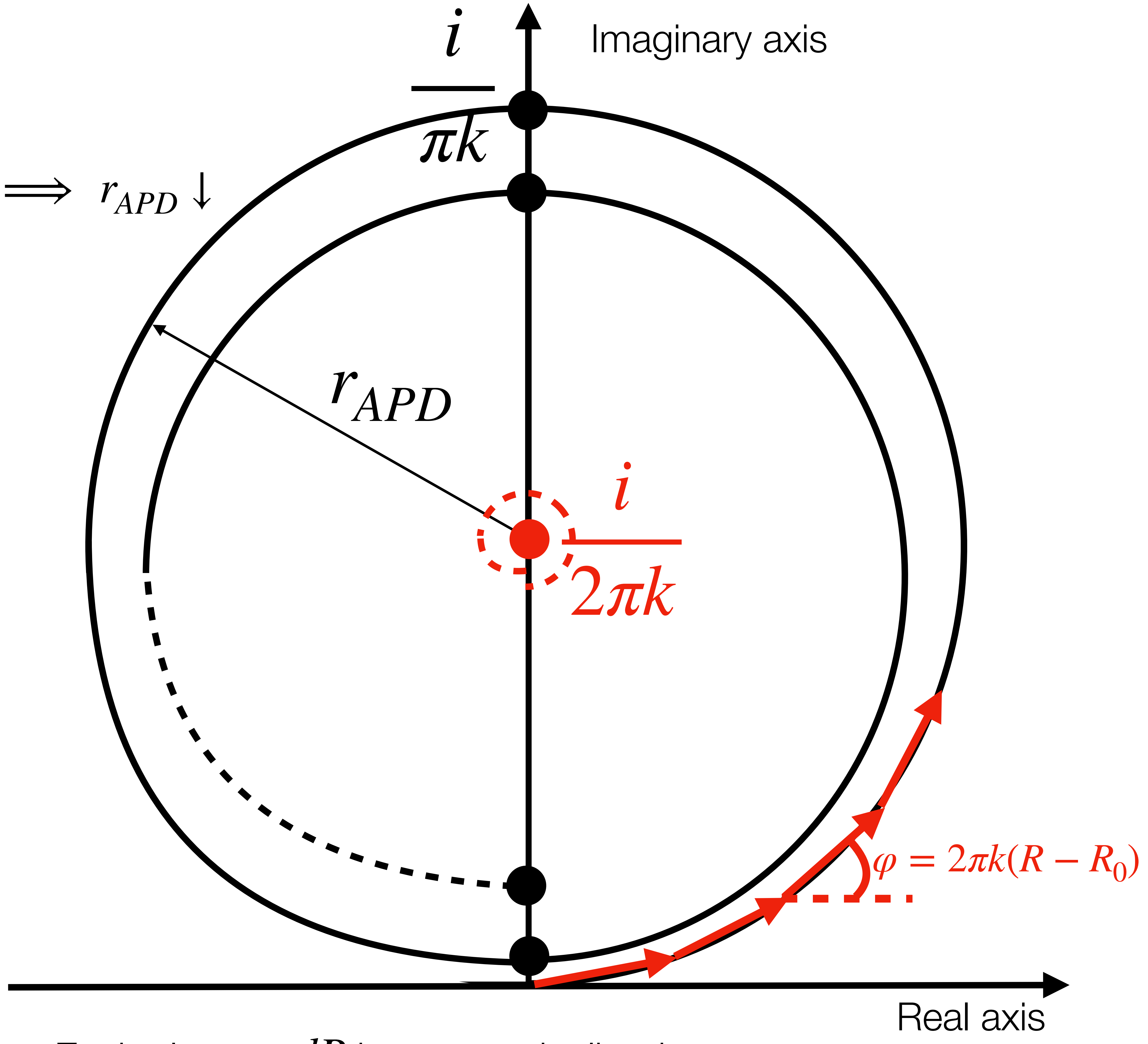


Extracted From Huygens

First Fresnel zone contribution

$$\varphi = 2\pi k (R - R_0) = \pi \implies R - R_0 = \frac{\lambda}{2}$$

$$R \uparrow \implies A(\theta) \downarrow \implies r_{APD} \downarrow$$



(VI) The integral can be obtained using the first Fresnel zone only :

$$\int_{R_0}^{\infty} A(\theta) e^{2\pi i k R} dR = \frac{1}{2} \int_{R_0}^{R_0 + \frac{\lambda}{2}} e^{2\pi i k R} dR = \frac{i}{2\pi k} e^{2\pi i k R_0} \Rightarrow \psi(P) = A_Q \frac{e^{2\pi i k (r + R_0)}}{r + R_0}$$

The secondary wavefront in P is indeed *a spherical wave*

Each element dR is a vector inclined by an angle φ in the complex plane $\rightarrow e^{i\varphi} dR$

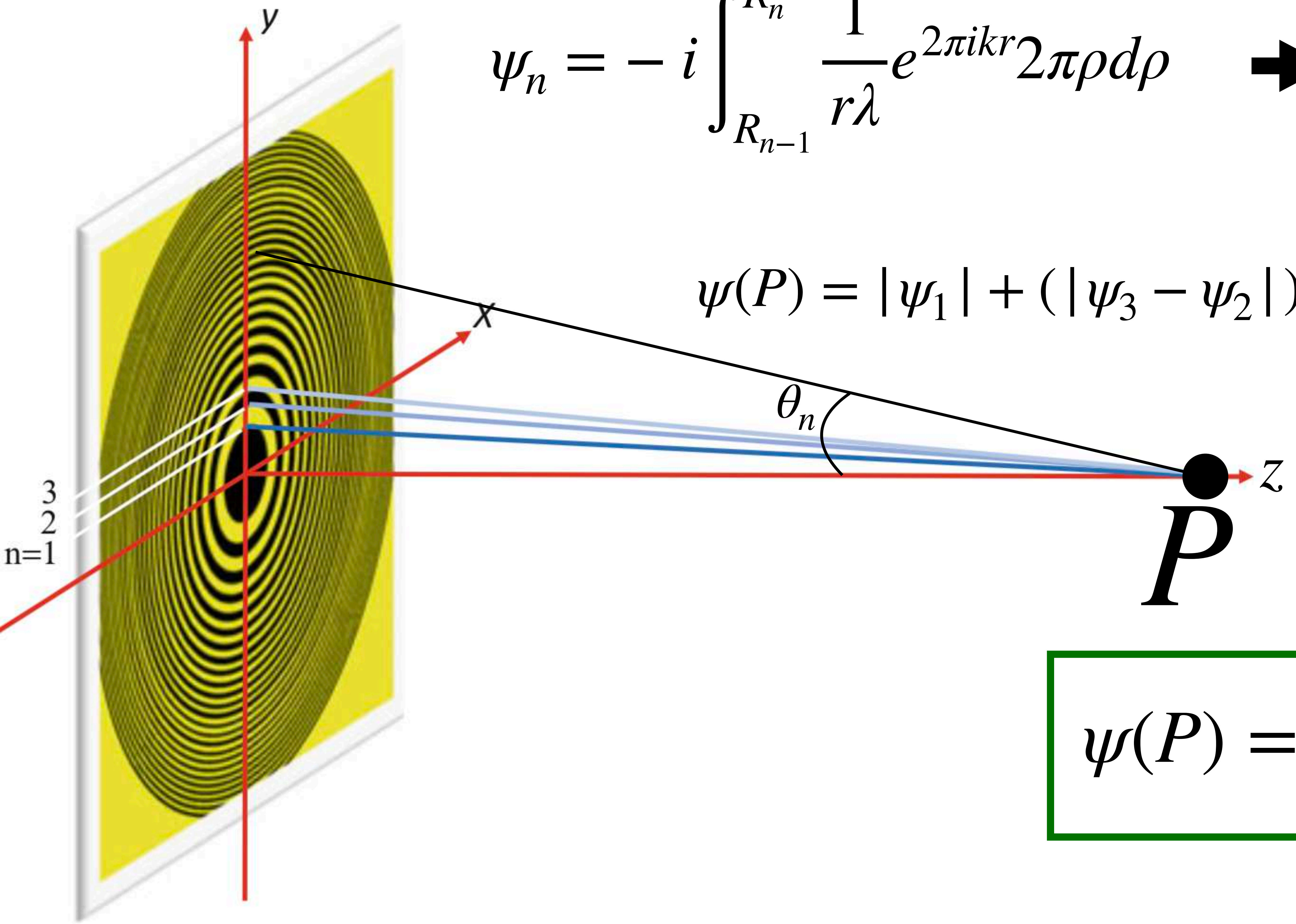
Fresnel zones rules : plane wave propagation

Let's consider the propagation of a plane wave written $e^{2\pi i \vec{k}.\vec{r}}$.

Using Huygens Fresnel principle we can show that each Fresnel zone will contribute through the following integral :

$$\psi_n = -i \int_{R_{n-1}}^{R_n} \frac{1}{r\lambda} e^{2\pi i k r} 2\pi\rho d\rho \quad \Rightarrow \quad \psi_n = (1 + \cos(\theta_n))(-1)^{n+1} e^{2\pi i k z}$$

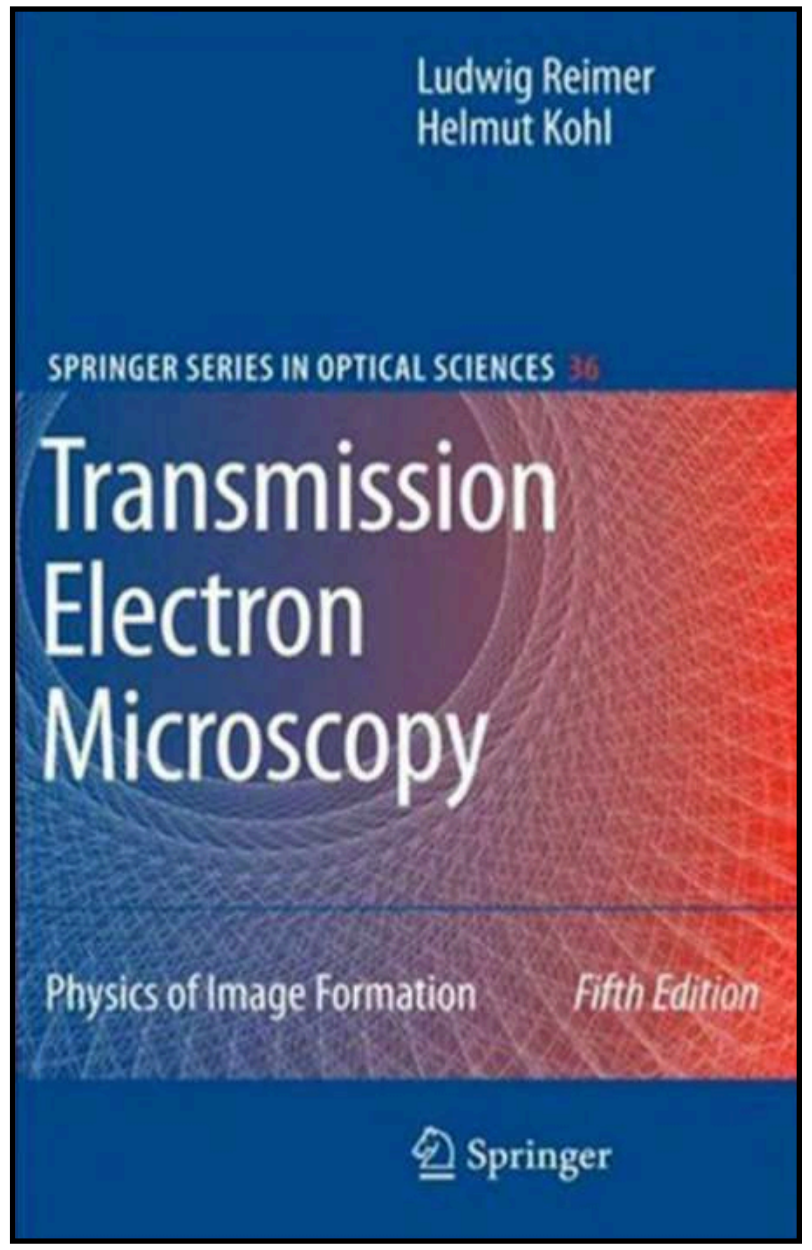
$$\psi(P) = |\psi_1| + (|\psi_3 - \psi_2|) + \ldots + (|\psi_{N-1} - \psi_{N-2}|) - |\psi_N|$$

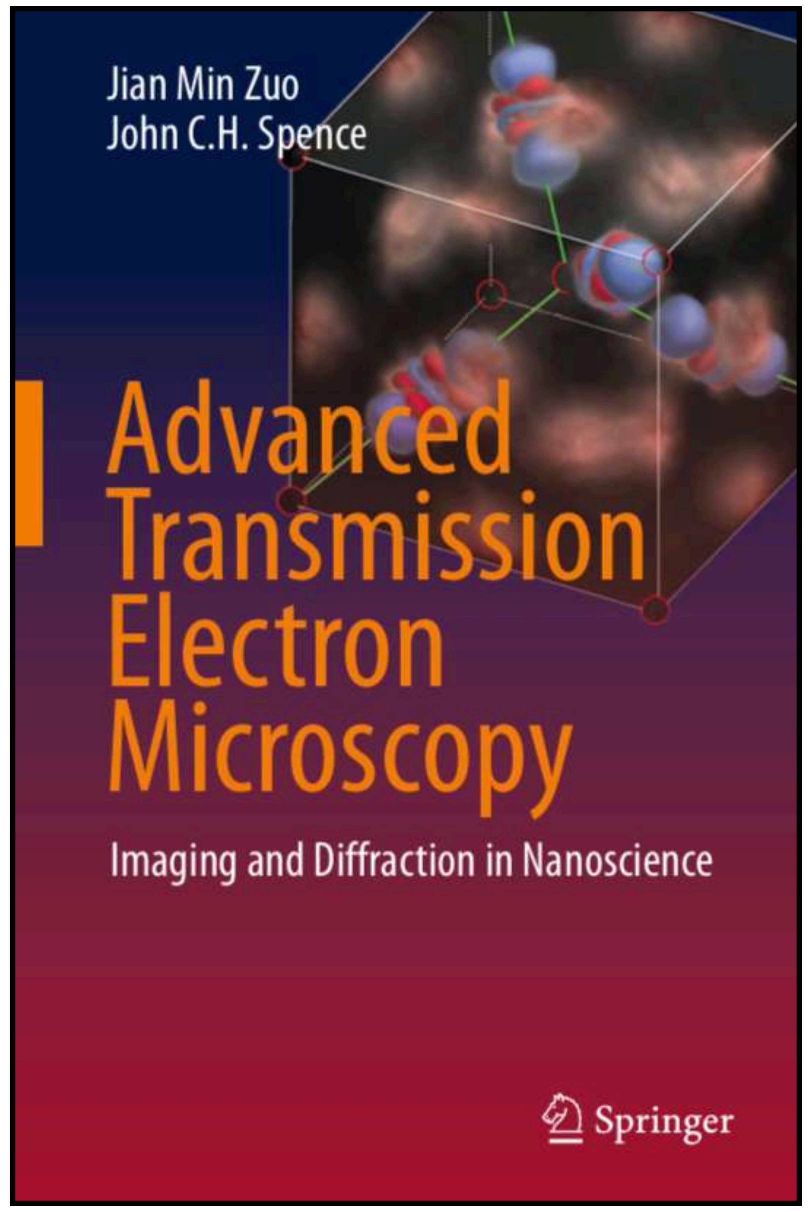


$$\boxed{\psi(P) = |\psi_1| - |\psi_N|}$$

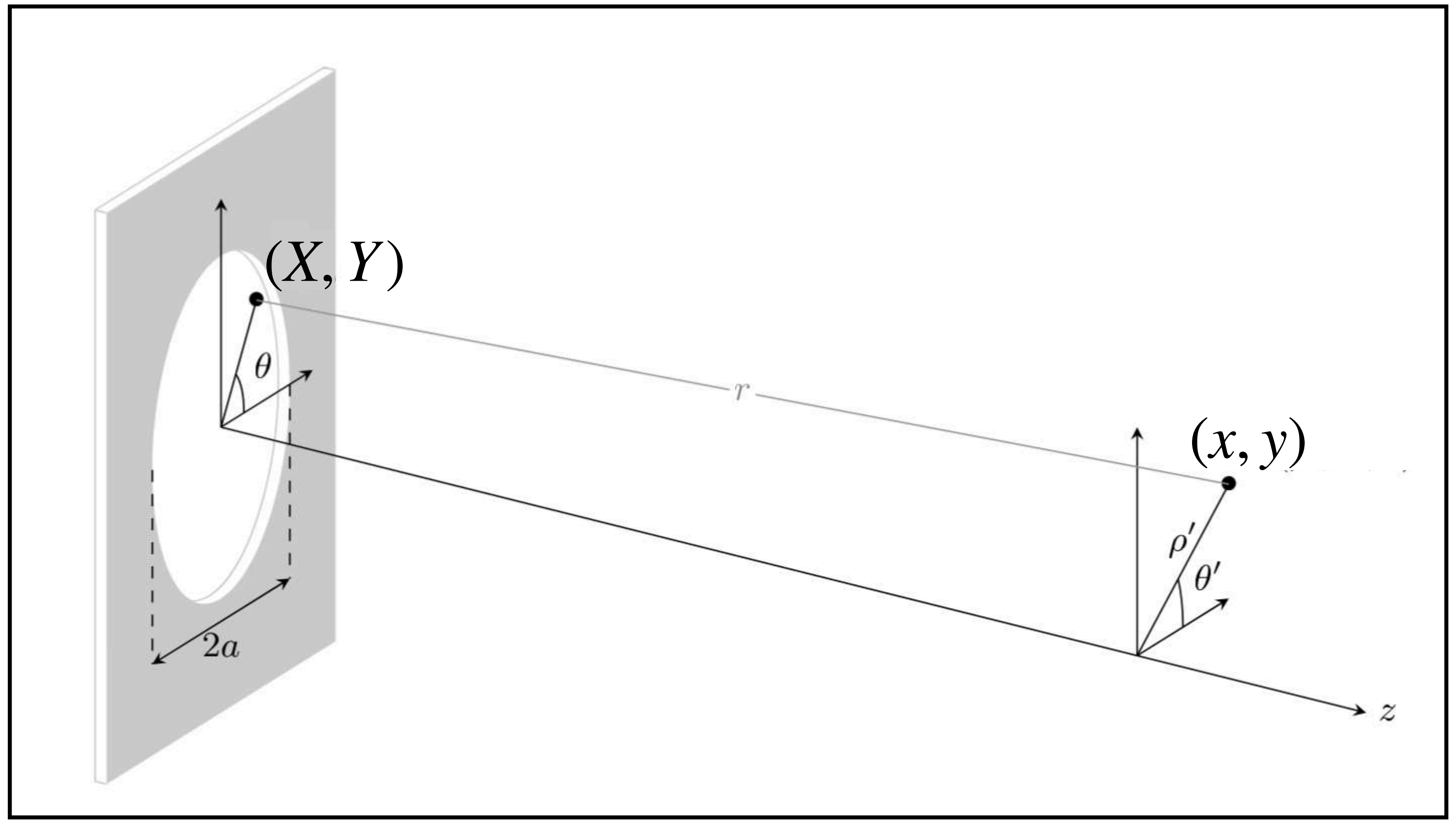
For an incoming plane wave we have ($n \to \infty \implies \theta_n \to 90°$) : $\psi(P) = 2e^{2\pi i k z} - e^{2\pi i k z} = e^{2\pi i k z}$

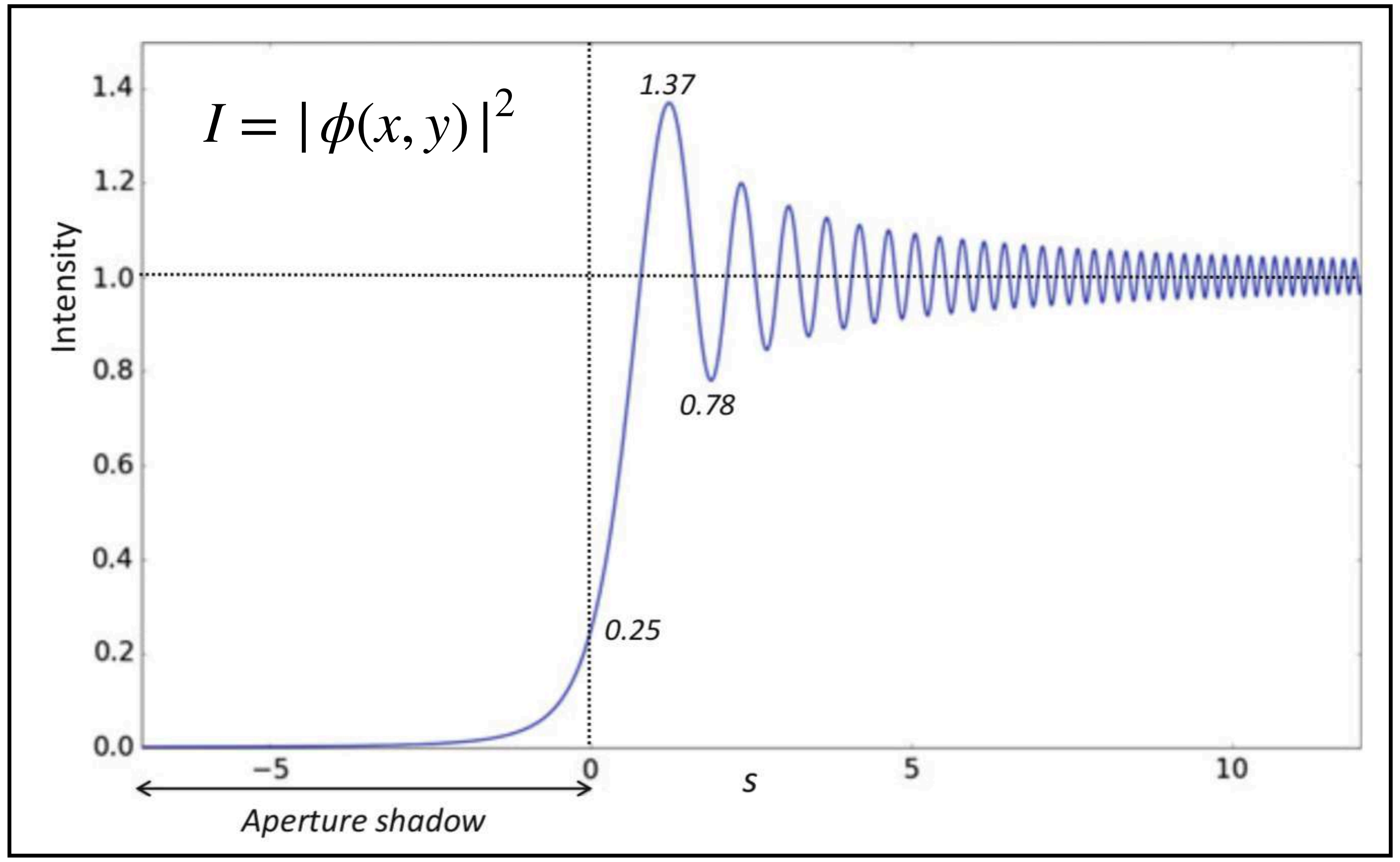
The secondary wavefront in P remains *a plane wave*





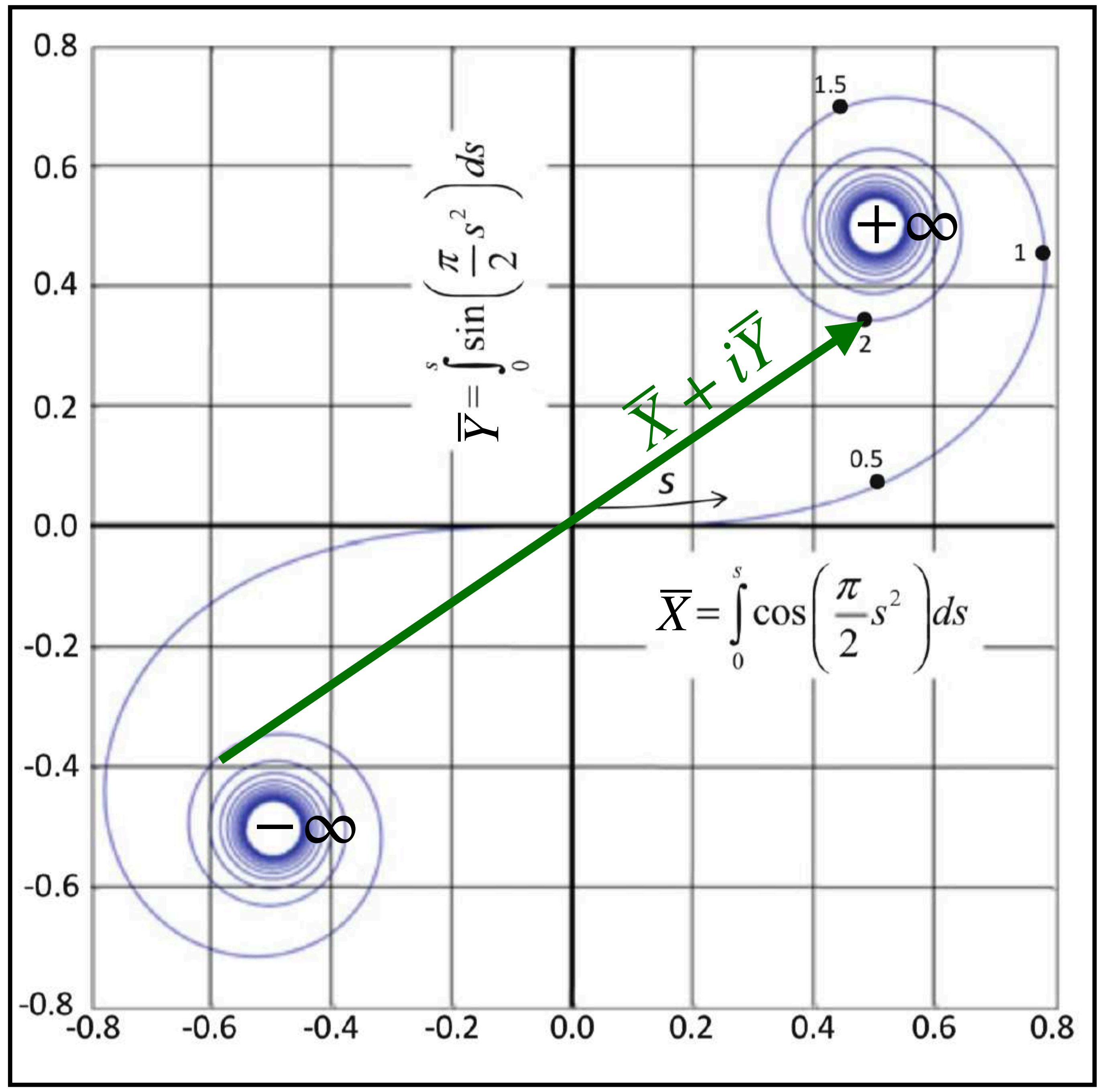
Application of Fresnel zones : Fresnel fringes





Evaluation of Fresnel integral using APD (Cornu's spiral)

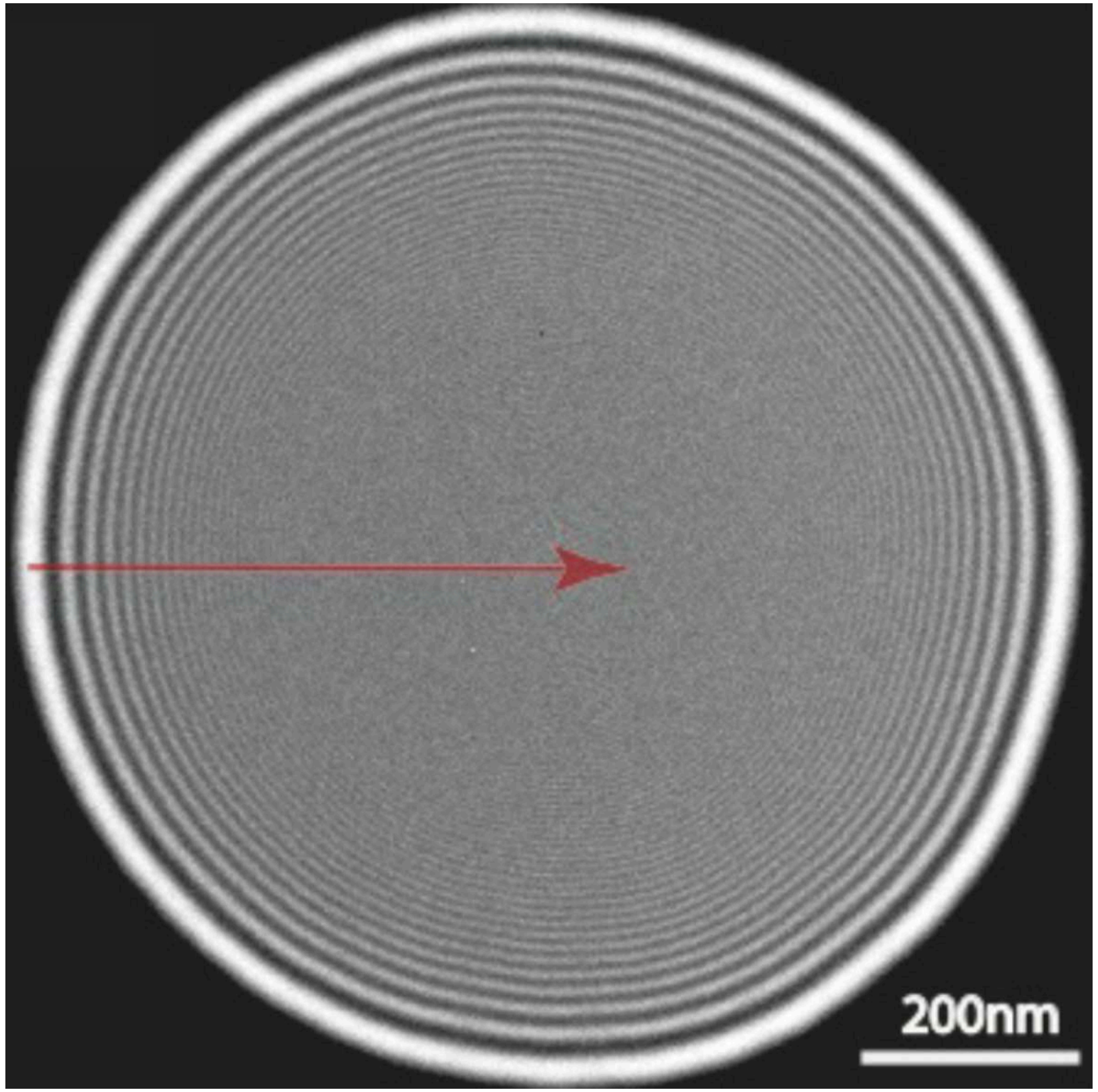
Converge to $\pm\left(\frac{1}{2}, \frac{1}{2}\right)$ as s moves from $0 \rightarrow \pm\infty$

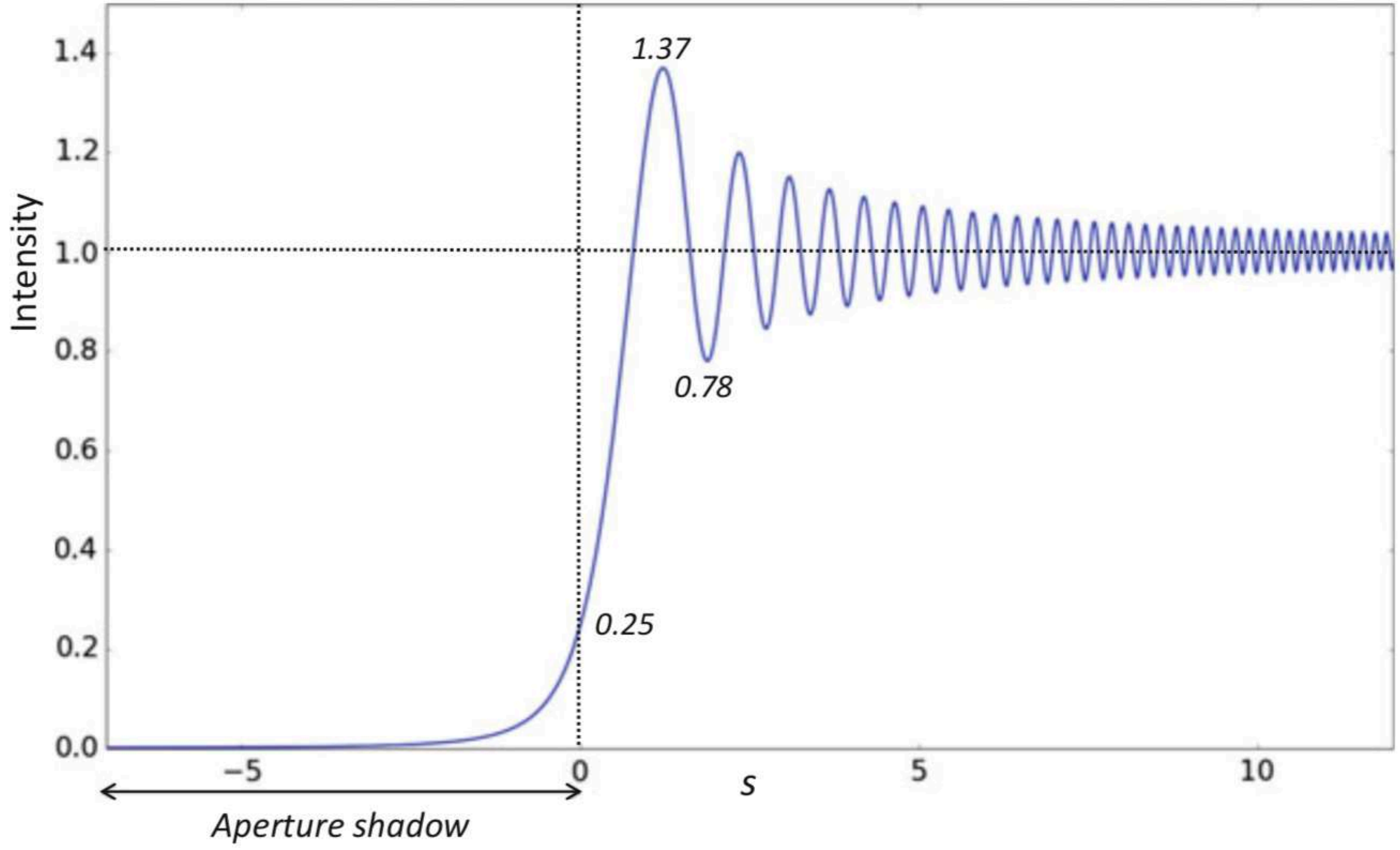


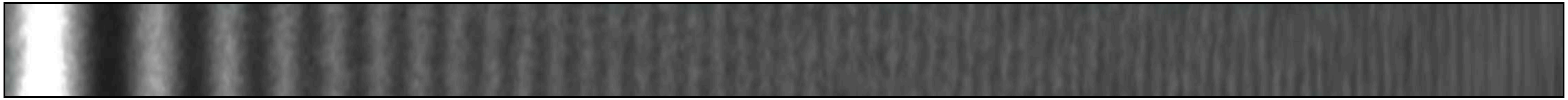
$$s = \sqrt{\frac{2(x - X)^2}{\lambda z}}$$

$$\phi(x, y) \propto \left[\left(\frac{1}{2} + i\frac{1}{2}\right) - (\overline{X} + i\overline{Y})\right]$$

Application of Fresnel zones : Fresnel Fringes







Kinematical theory of diffraction: the column approximation

- Let's consider an incoming plane wave ψ_0 on a crystal define by a thickness t
- Electrons wavelength : $\lambda = 3{,}7\text{pm} \rightarrow (100\text{keV})$
- Radius of the first Fresnel zone : $\rho_1 = \sqrt{R_0 \lambda}$

➡ *We want to estimate the contribution of the slice dz to $\psi_{\vec{g}}(P)$*

(I) For $R_0 = 100$nm we have $\rho_1 = 0{,}6$nm

This means that only a column with a diameter of $1 - 2nm$ is contributing to the amplitude at the point P. We will only consider this first Fresnel zone.

- The method is therefore called the **column approximation**.

(II)There are $\frac{dz}{V_e}$ unit cells per unit area in an element of thickness dz.

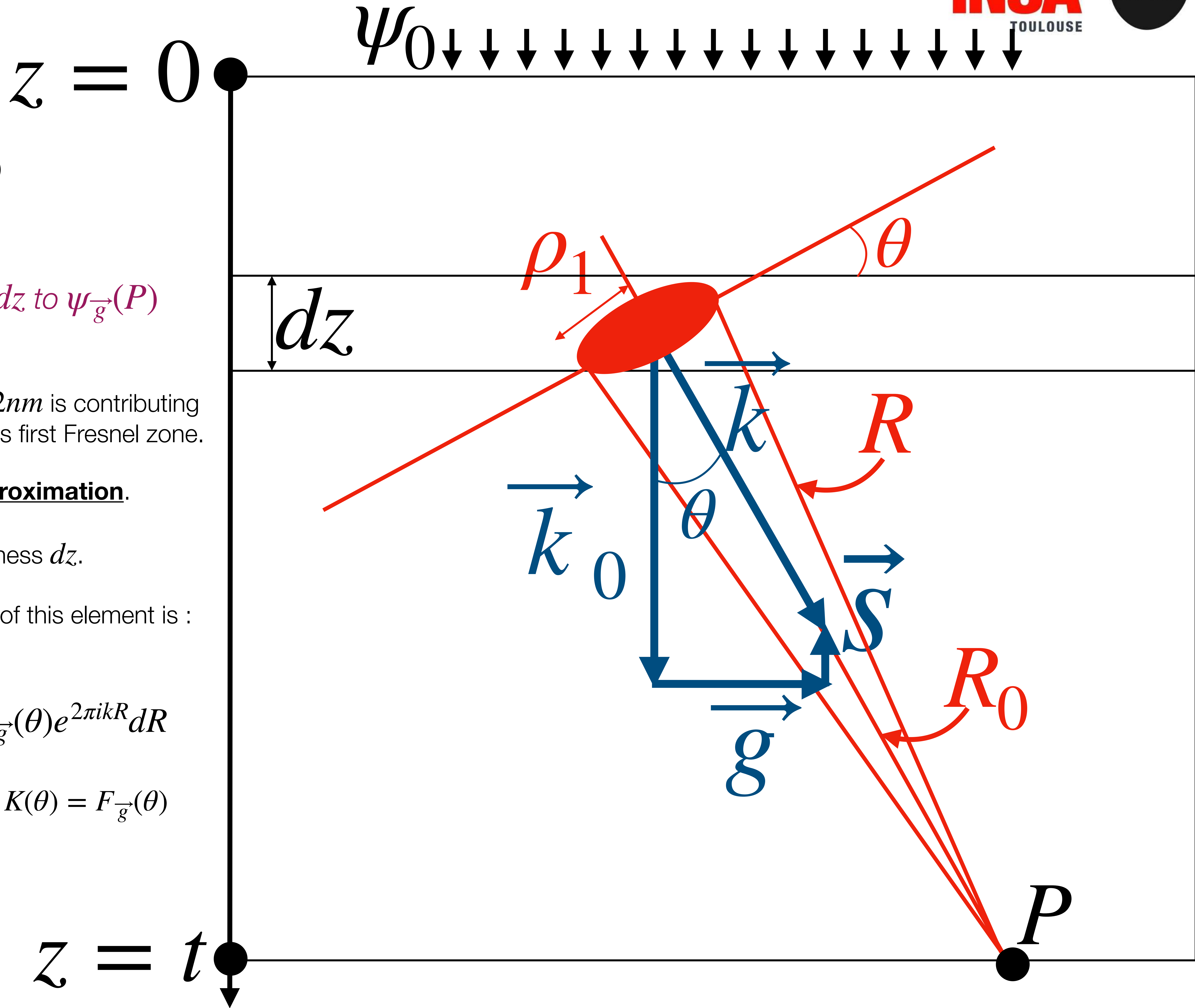
Thanks to Huygens-Fresnel Principe the contribution $d\psi_{\vec{g}}$ of this element is :

$$d\psi_{\vec{g}} = \psi_0 \frac{dz}{V_e} \iint F_{\vec{g}}(\theta) \frac{e^{2\pi ikR}}{R} dS = \psi_0 \frac{2\pi dz}{V_e} \int_{R_0}^{R} F_{\vec{g}}(\theta) e^{2\pi ikR} dR$$

$$K(\theta) = F_{\vec{g}}(\theta)$$

$$d\psi_{\vec{g}} = \frac{i\pi}{\xi_{\vec{g}}} \psi_0 e^{2\pi ikR_0} dz$$

$$\xi_{\vec{g}} = \frac{\pi V_e}{\lambda F_{\vec{g}}(\theta)}$$



Diffraction intensity under column approximation

(III) To estimate the diffracted intensity $I_{\vec{g}}$ we simply have to integrate $d\psi_{\vec{g}}$ over the thickness

$R_0 = t - z$

$|\psi_0| = 1$

$\vec{k} = \vec{k}_0 + \vec{g} + \vec{s}$

$$\psi_{\vec{g}} = \frac{i\pi}{\xi_{\vec{g}}} e^{2\pi i k_0 t} \int_0^t e^{-2\pi i(\vec{g}+\vec{s}).\vec{r}} dz$$

Because of column approx $\vec{s}$ and $\vec{r}$ are collinear : $\vec{s}\,.\,\vec{r} = sz$

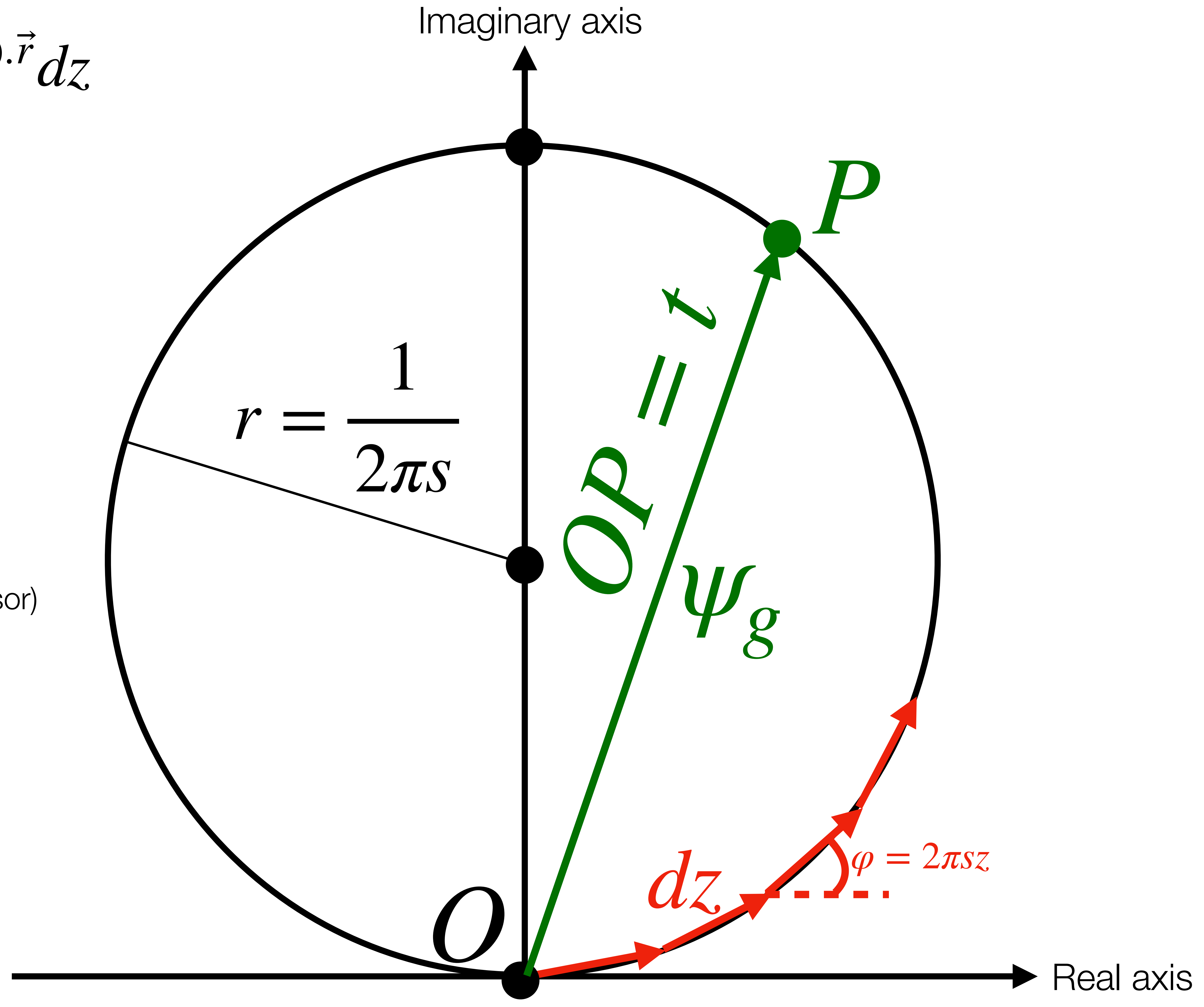
$$\psi_{\vec{g}} = \frac{i\pi}{\xi_{\vec{g}}} e^{2\pi i k_0 t} \int_0^t e^{-2\pi i s z} dz$$

(IV) We will now always estimate this Fresnel integral, and $I_{\vec{g}}$, using APD (phasor)

$$I_{\vec{g}} = \psi_{\vec{g}}\psi^*_{\vec{g}} = \frac{\pi^2}{\xi^2_{\vec{g}}} \frac{\sin^2(\pi t s)}{(\pi s)^2}$$

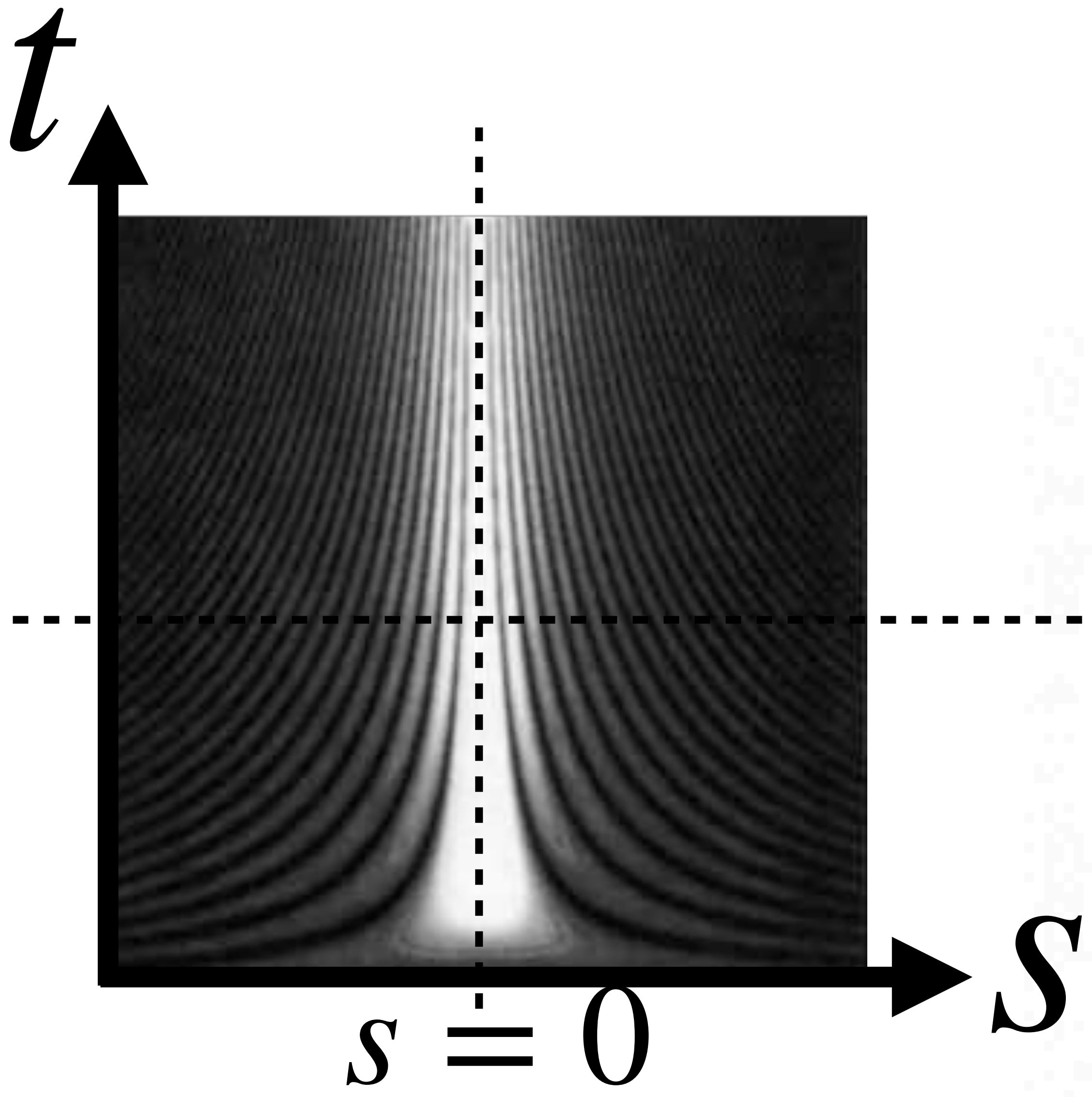
$$I_0 = 1 - I_{\vec{g}}$$

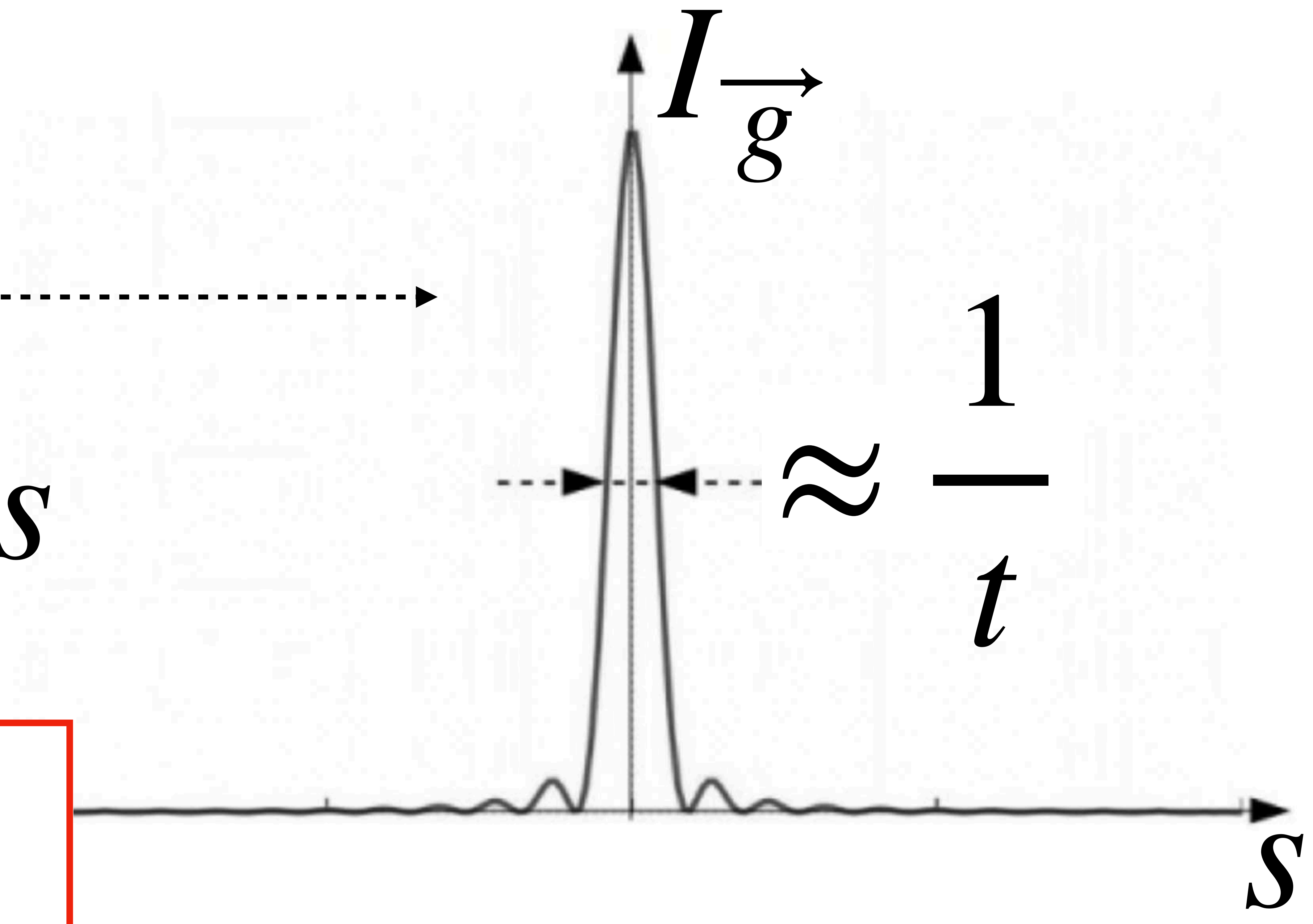
Integral estimation using APD :



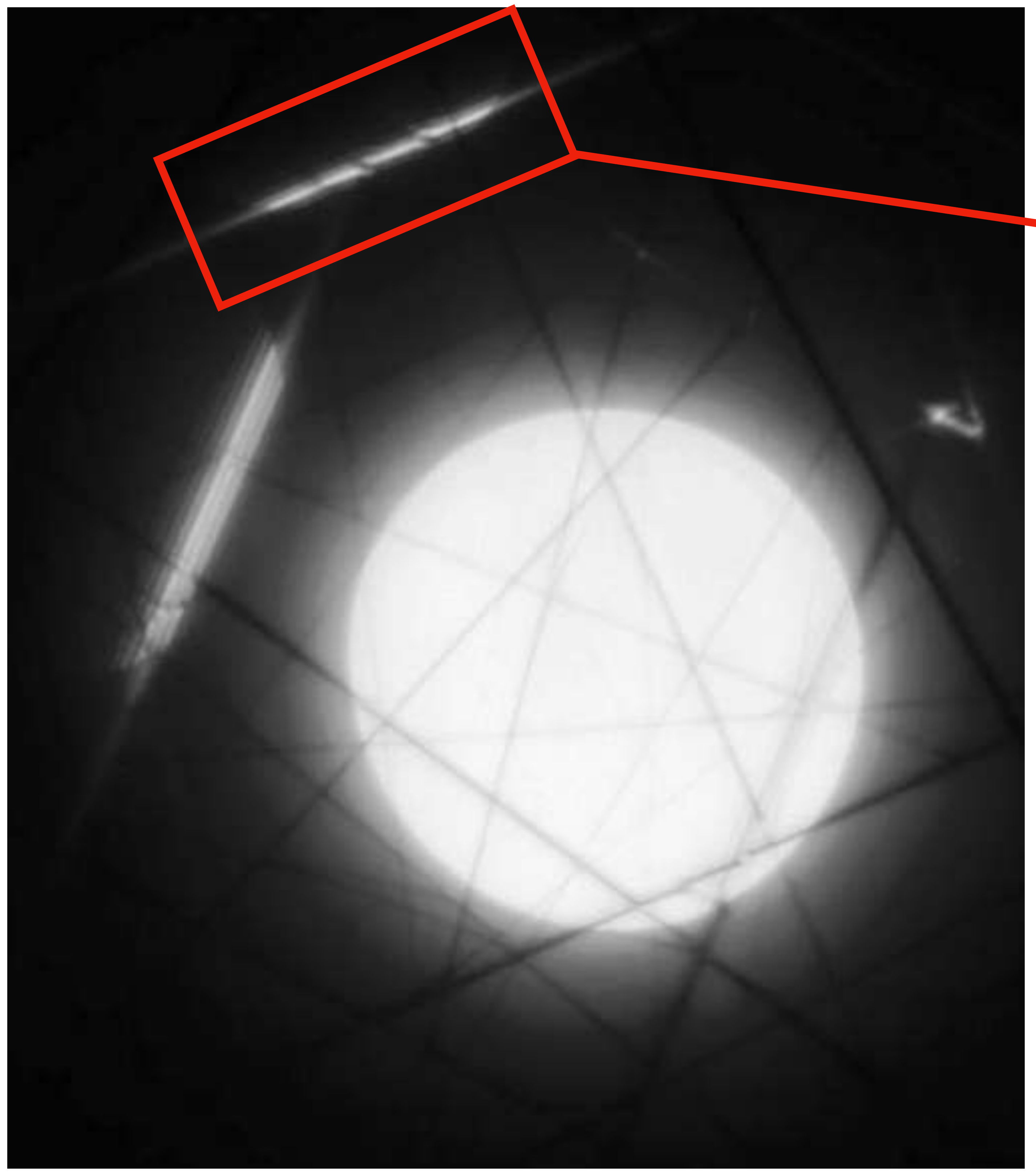
Going back to the diffracted intensities : the kinematical rocking curve

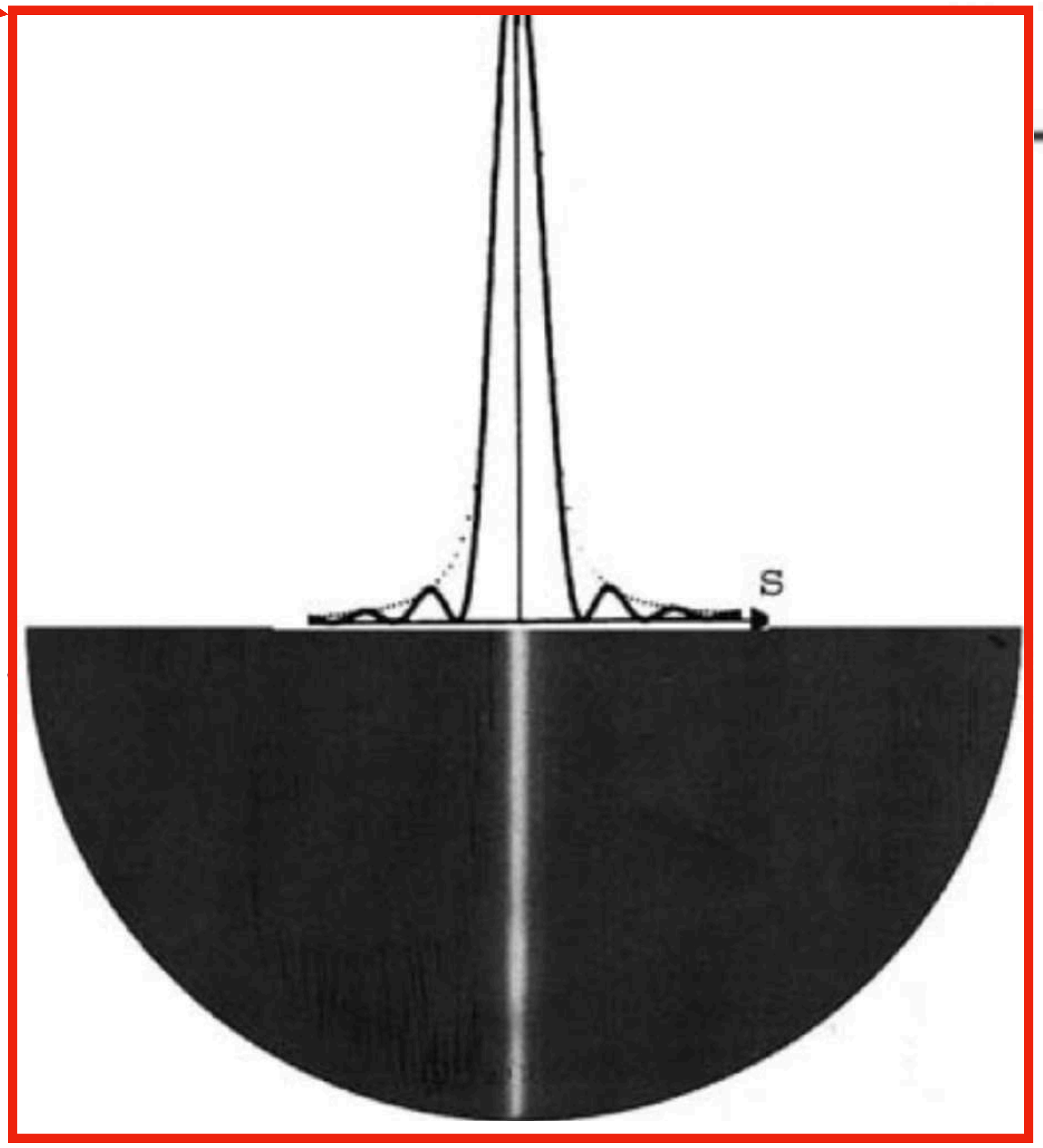
$$I_{\vec{g}} = \psi_{\vec{g}}\psi^*_{\vec{g}} = \frac{\pi^2}{\xi^2_{\vec{g}}}\frac{\sin^2(\pi t s)}{(\pi s)^2}$$





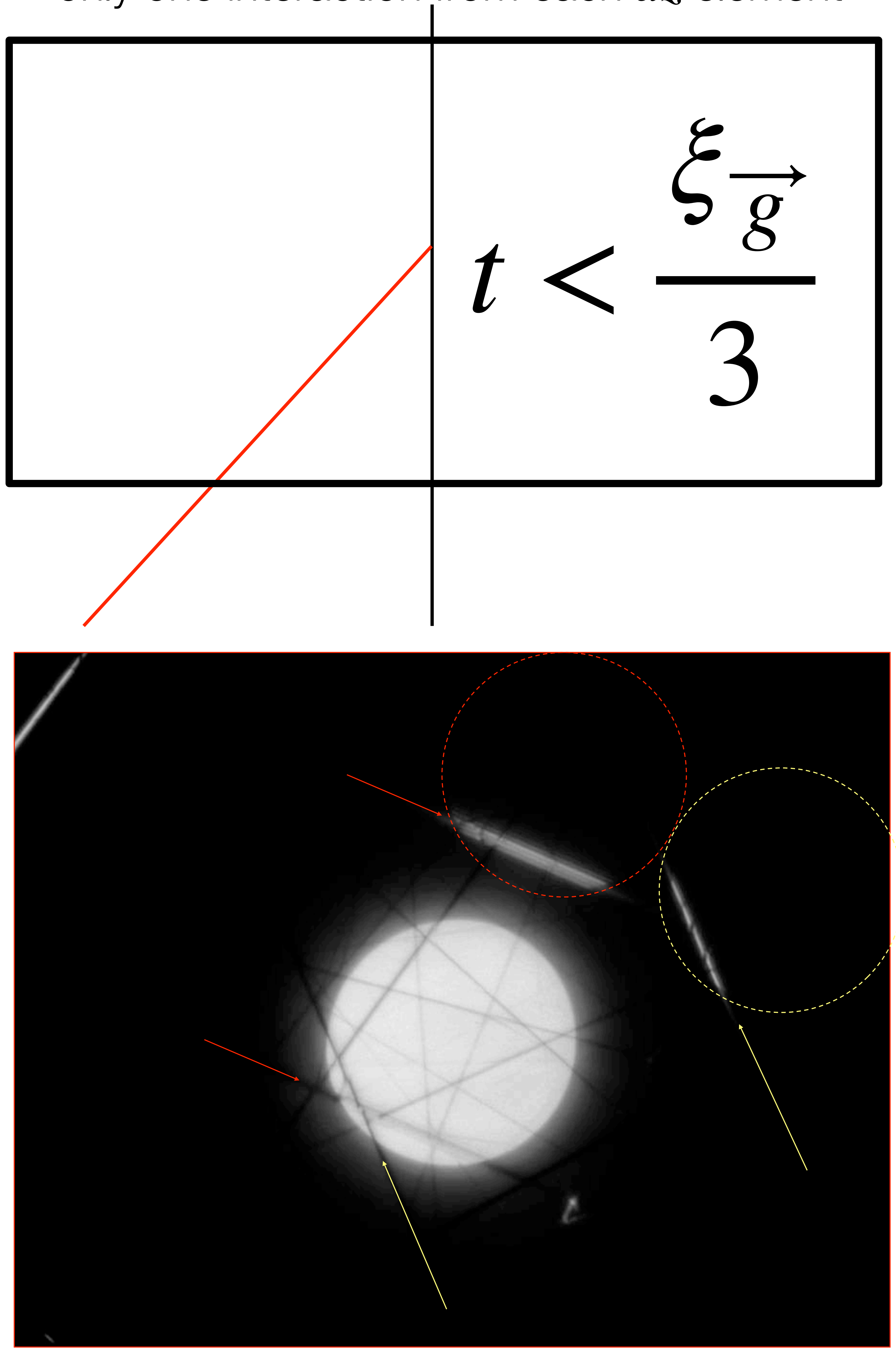
CBED kinematical lines profile = rocking curve





Introduction to dynamical theory

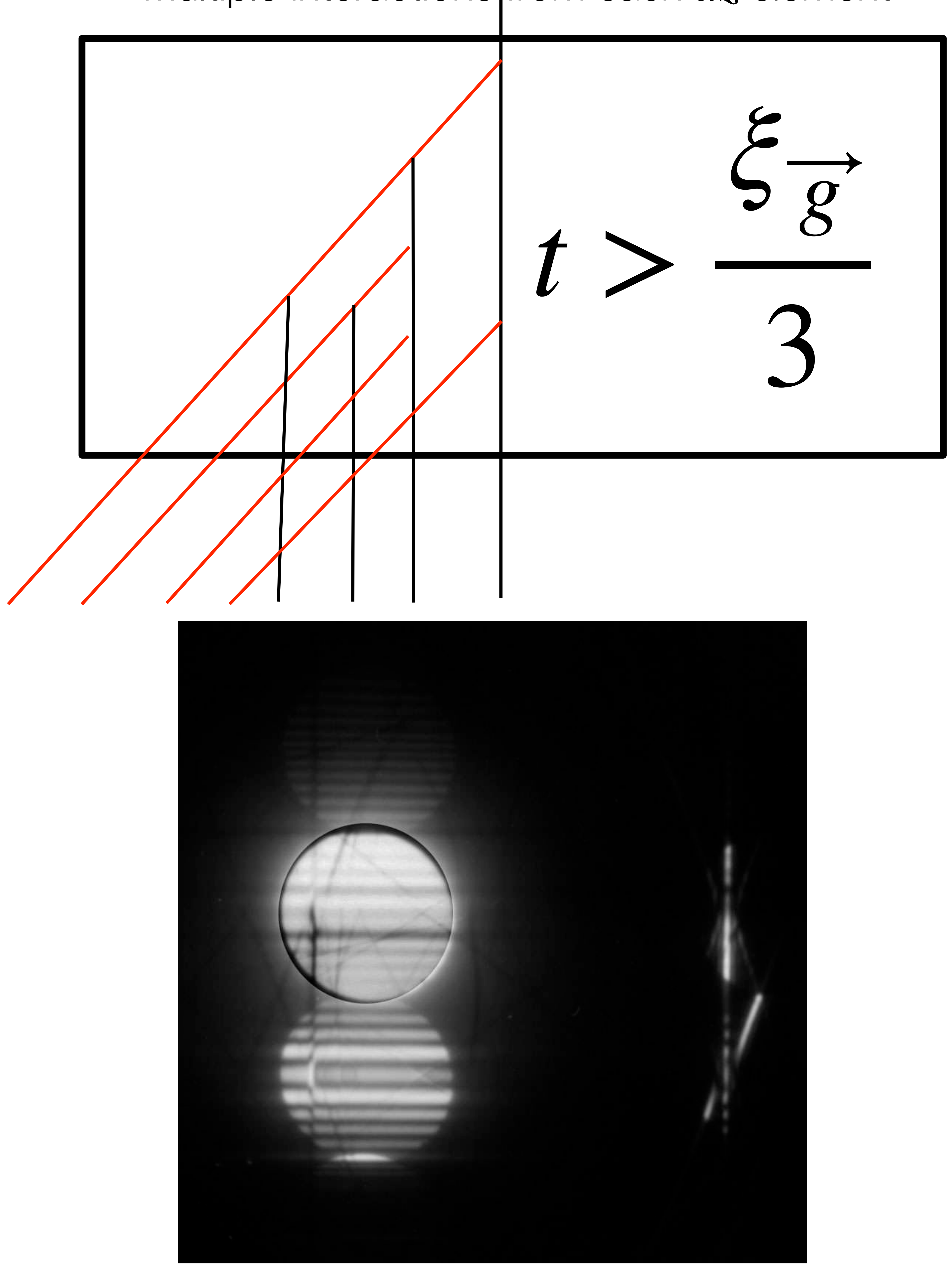
Kinematical approach :
only one interaction from each dz element

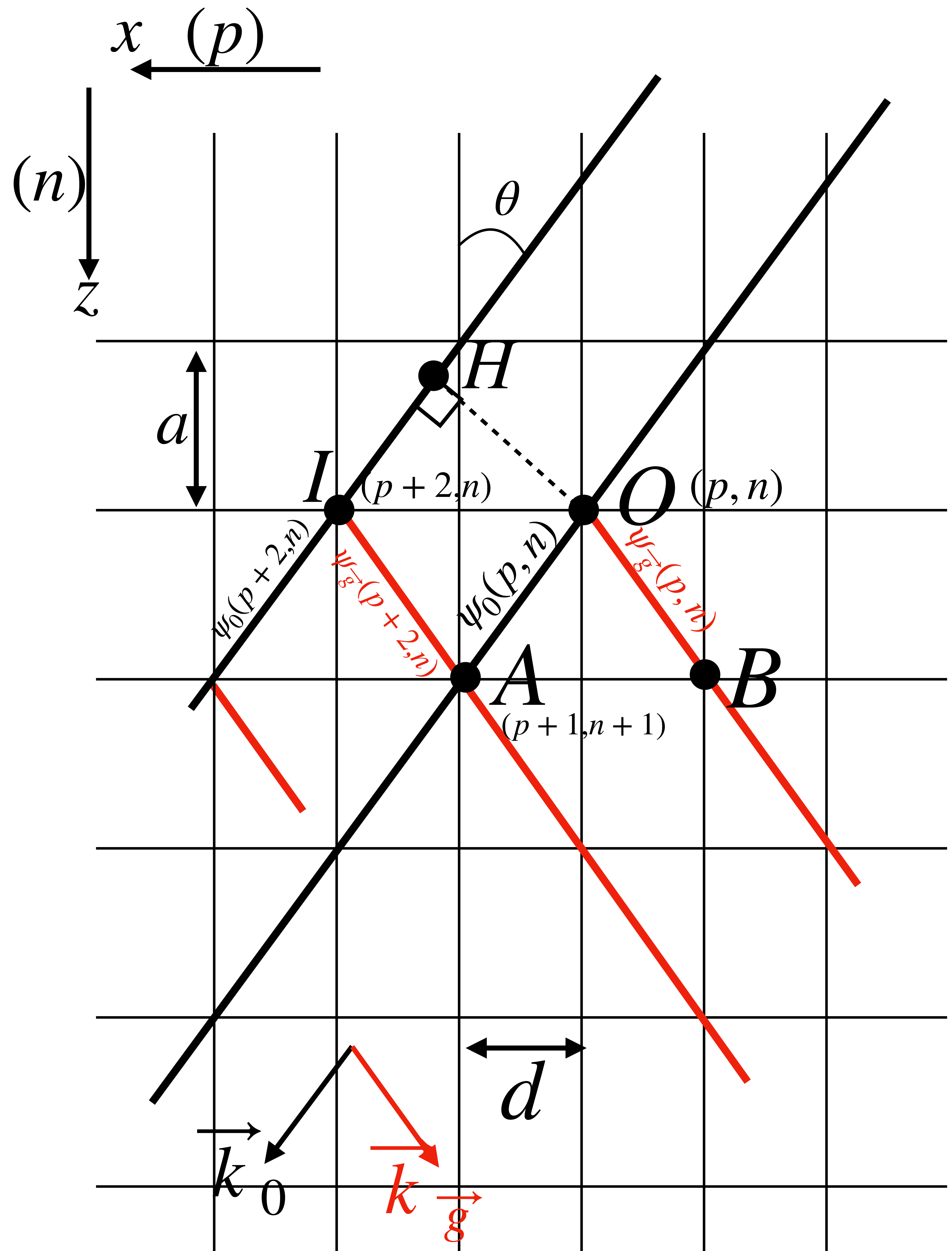


Dynamical extinction distance :

$$\xi_{\vec{g}}$$

Dynamical approach :
Multiple interactions from each dz element





Let's consider the phase origin located in O.
All the demonstration will previous results obtained using Huygens-Fresnel approach

➡ A point will affect the incoming wave ψ_0 by the value $d\psi_{\vec{g}} = \dfrac{i\pi}{\xi_{\vec{g}}}\psi_0 e^{2\pi i k R_0} dz$

➡ Let's note $q = \dfrac{a\pi}{\xi_{\vec{g}}}$.

➡ The ratio between the wave amplitude coming from O

After crossing B is $\dfrac{|\psi(B)|}{|\psi(O)|} = iq$. Then after crossing A we have $\dfrac{|\psi(A)|}{|\psi(O)|} = iq_0$

(I) The wave starting from O and arriving in A is : $\psi_0(p,n)e^{2\pi i(\vec{k}_0.\overrightarrow{OA})}$

After crossing A we add the point contribution the wavefront becomes :

$$\psi_0(p,n)(1+iq_0)e^{2\pi i(\vec{k}_0.\overrightarrow{OA})}$$

(II) The wave diffracted from I and arriving in A is : $\psi_{\vec{g}}(p+2,n)e^{2\pi i\vec{k}_{\vec{g}}.(\overrightarrow{HI}+\overrightarrow{IA})}$

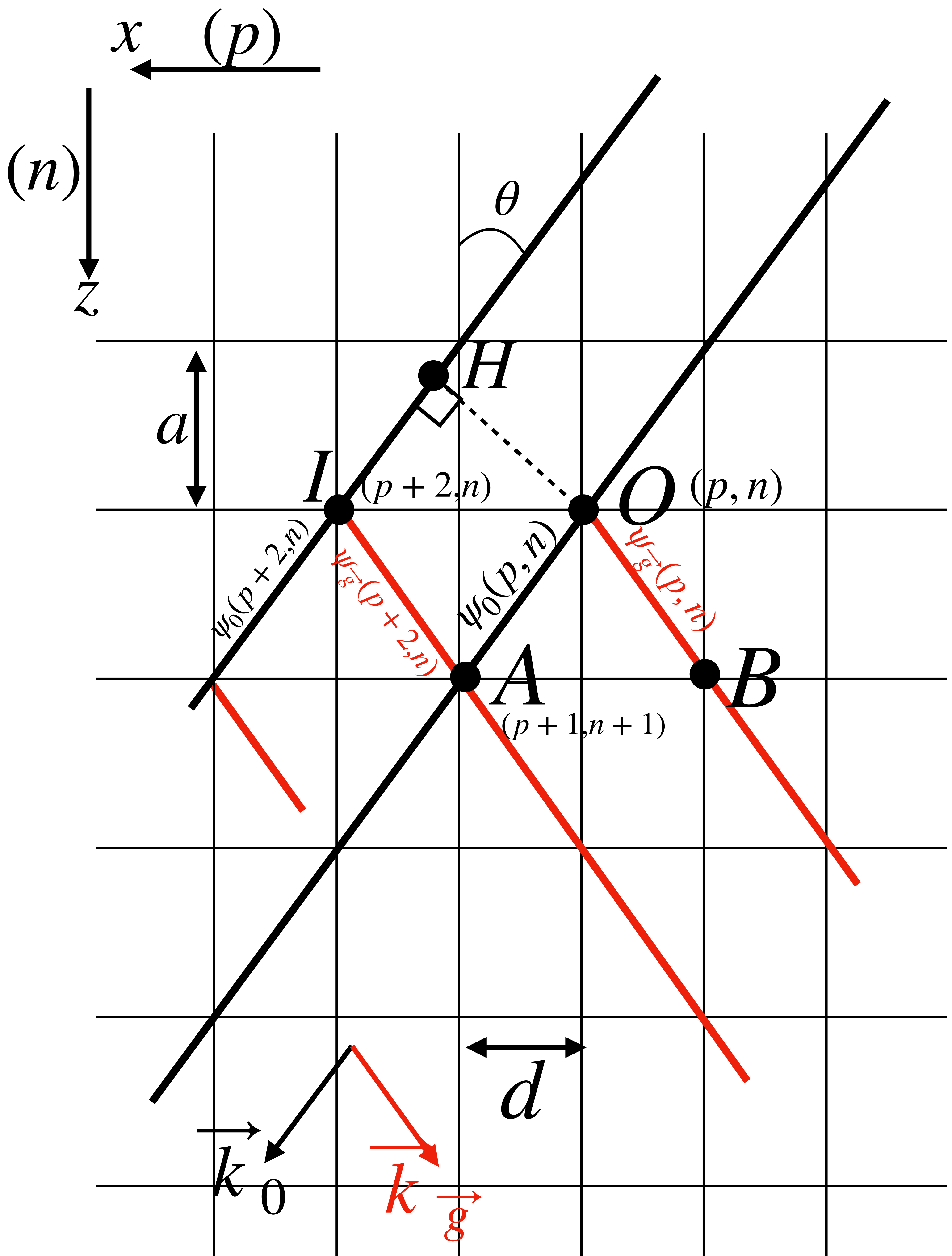
After crossing A the wavefront along $\vec{k}_0$ becomes :

$$iq\psi_{\vec{g}}(p+2,n)e^{2\pi i\vec{k}_{\vec{g}}.(\overrightarrow{HI}+\overrightarrow{IA})}$$

And along $\vec{k}$ we add the point contribution to the incoming wavefront

$$(1+iq_0)\psi_{\vec{g}}(p+2,n)e^{2\pi i\vec{k}_{\vec{g}}.(\overrightarrow{HI}+\overrightarrow{IA})}$$

Step by step derivation of Howie-Whelan equations : 2-beams dynamical theory



$$2\pi \vec{k}_0 . \overrightarrow{OA} = 2\pi \bar{k}_0 . \overline{OA} = \frac{2\pi \bar{k}_0 d}{\sin(\theta)} = \delta$$

$$2\pi \vec{k}_{\vec{g}} . (\overrightarrow{HI} + \overrightarrow{IA}) = 2\pi \vec{k}_{\vec{g}} . \overrightarrow{HI} + 2\pi \vec{k}_{\vec{g}} . \overrightarrow{IA} = 2\pi \bar{k}_{\vec{g}} . \overline{HI} + 2\pi \bar{k}_{\vec{g}} . \overline{IA} = \phi + \delta$$

$$\begin{cases} \psi_0(p+1,n+1)e^{-i\delta} = (1+iq_0)\psi_0(p,n) + iq\psi_{\vec{g}}(p+2,n)e^{i\phi} \\ \psi_{\vec{g}}(p+1,n+1)e^{-i\delta} = (1+iq_0)\psi_{\vec{g}}(p+2,n)e^{i\phi} + iq\psi_0(p,n)e^{i\phi} \end{cases}$$

(III) We will now consider the Friedel Law : $\psi_{\vec{g}} = \psi_{-\vec{g}}$ and some first geometrical simplifications

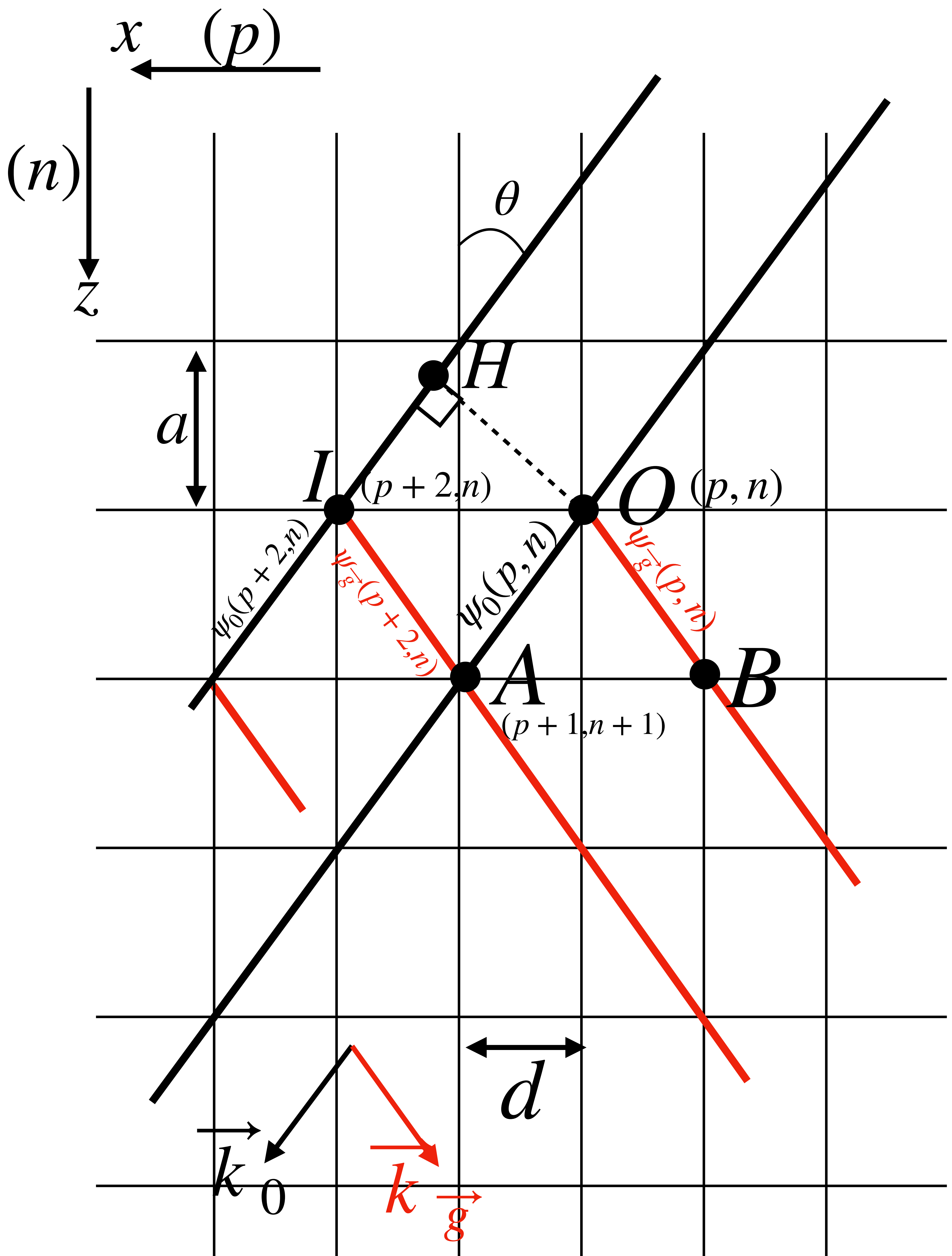
$\theta = \theta_B + \delta\theta \qquad \sin(\delta\theta) \approx 0, \cos(\delta\theta) \approx 1 \qquad e^{-i\delta} \approx 1$

$$HI = \lambda + 2d\delta\theta\cos(\theta_B) = \lambda + 2ds\frac{\cos(\theta_B)}{g} = \lambda + 2d^2 s\cos(\theta_B)$$

$$\tan(\theta_B) = \frac{d}{a_B} \;\Rightarrow\; \phi = \frac{2\pi}{\lambda}\left(\lambda + 2d^2 s \sin(\theta_B)\frac{a_B}{d}\right) = 2\pi(1 + a_B s) \;\Rightarrow\; e^{i\phi} = e^{2\pi a_B s}$$

(IV) We will now do first linear approximation : $e^{i\phi} iq \approx iq \qquad e^{i\phi}(1+iq_0) \approx 1 + iq_0$

$$\Rightarrow \begin{cases} \psi_0(p+1,n+1) - \psi_0(p,n) = iq_0\psi_0(p,n) + iq\psi_{\vec{g}}(p+2,n) \\ \psi_{\vec{g}}(p+1,n+1) - \psi_{\vec{g}}(p+2,n) = i(q_0+\phi)\psi_{\vec{g}}(p+2,n) + iq\psi_0(p,n) \end{cases}$$



(V) Introducing differential operators thanks to Taylor development :

$$\begin{cases} \psi_0(p+1, n+1) - \psi_0(p,n) = \dfrac{\partial \psi_0}{\partial x}(x_{p+1} - x_p) + \dfrac{\partial \psi_0}{\partial z}(z_{n+1} - z_n) \\ \psi_{\vec{g}}(p+1, n+1) - \psi_{\vec{g}}(p+2,n) = \dfrac{\partial \psi_{\vec{g}}}{\partial x}(x_{p+1} - x_{p+2}) + \dfrac{\partial \psi_{\vec{g}}}{\partial z}(z_{n+1} - z_n) \end{cases}$$

(VI) Considering that the amplitude depends only on the variable z, the two equations become a first system of differential equations (column approximation) :

$$\Rightarrow \begin{cases} \dfrac{d\psi_0}{dz}(z_{n+1} - z_n) = iq_0\psi_0(p,n) + iq\psi_{\vec{g}}(p+2,n) \\ \dfrac{d\psi_{\vec{g}}}{dz}(z_{n+1} - z_n) = i(q_0 + \phi)\psi_{\vec{g}}(p+2,n) + iq\psi_0(p,n) \end{cases}$$

(VI) Remembering : $z_{n+1} - z_n = a \qquad \dfrac{q_0}{a} = \dfrac{\pi}{\xi_0} \qquad \dfrac{q}{a} = \dfrac{\pi}{\xi_{\vec{g}}}$

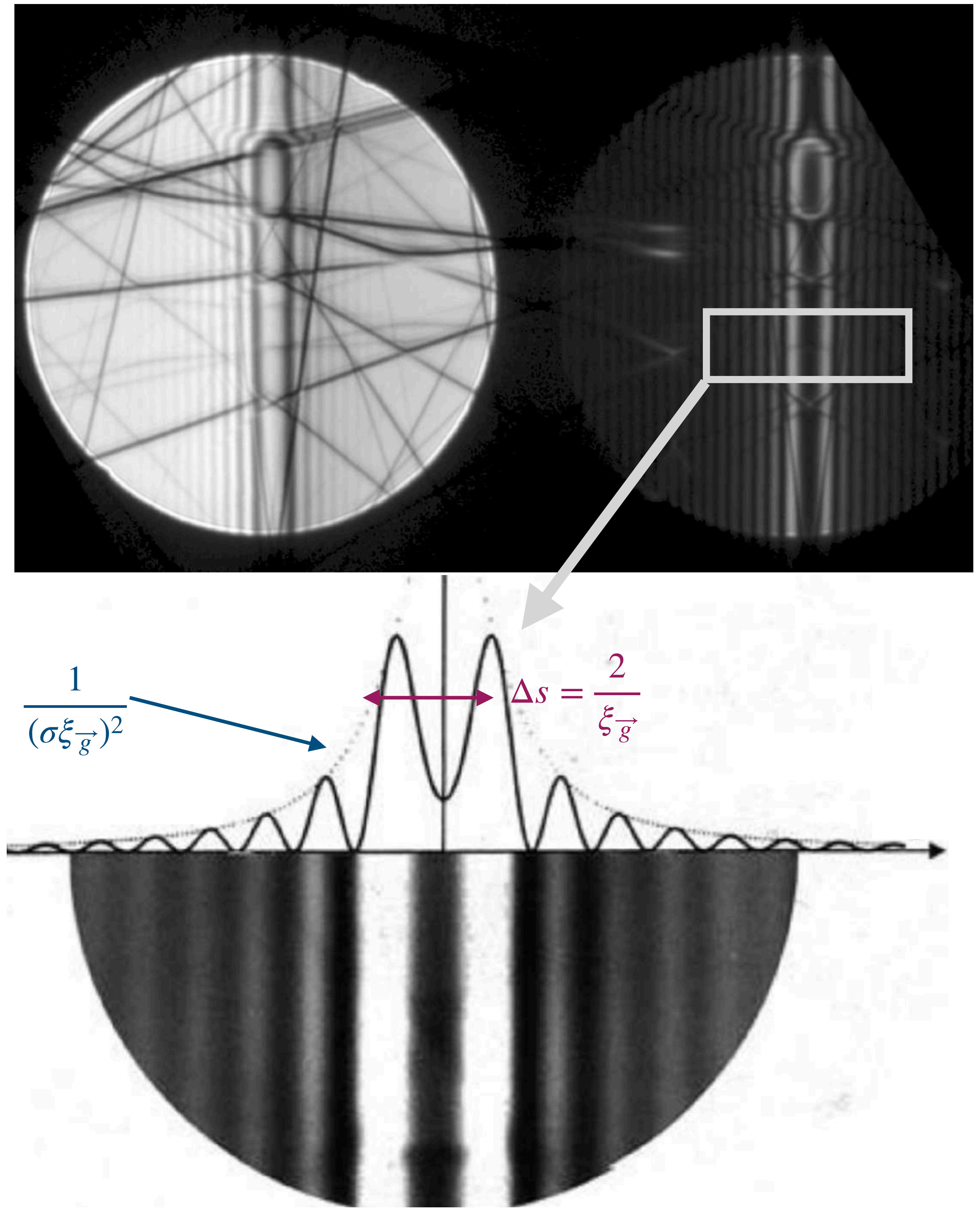
We find the 2-beams Howie-Whelan equations

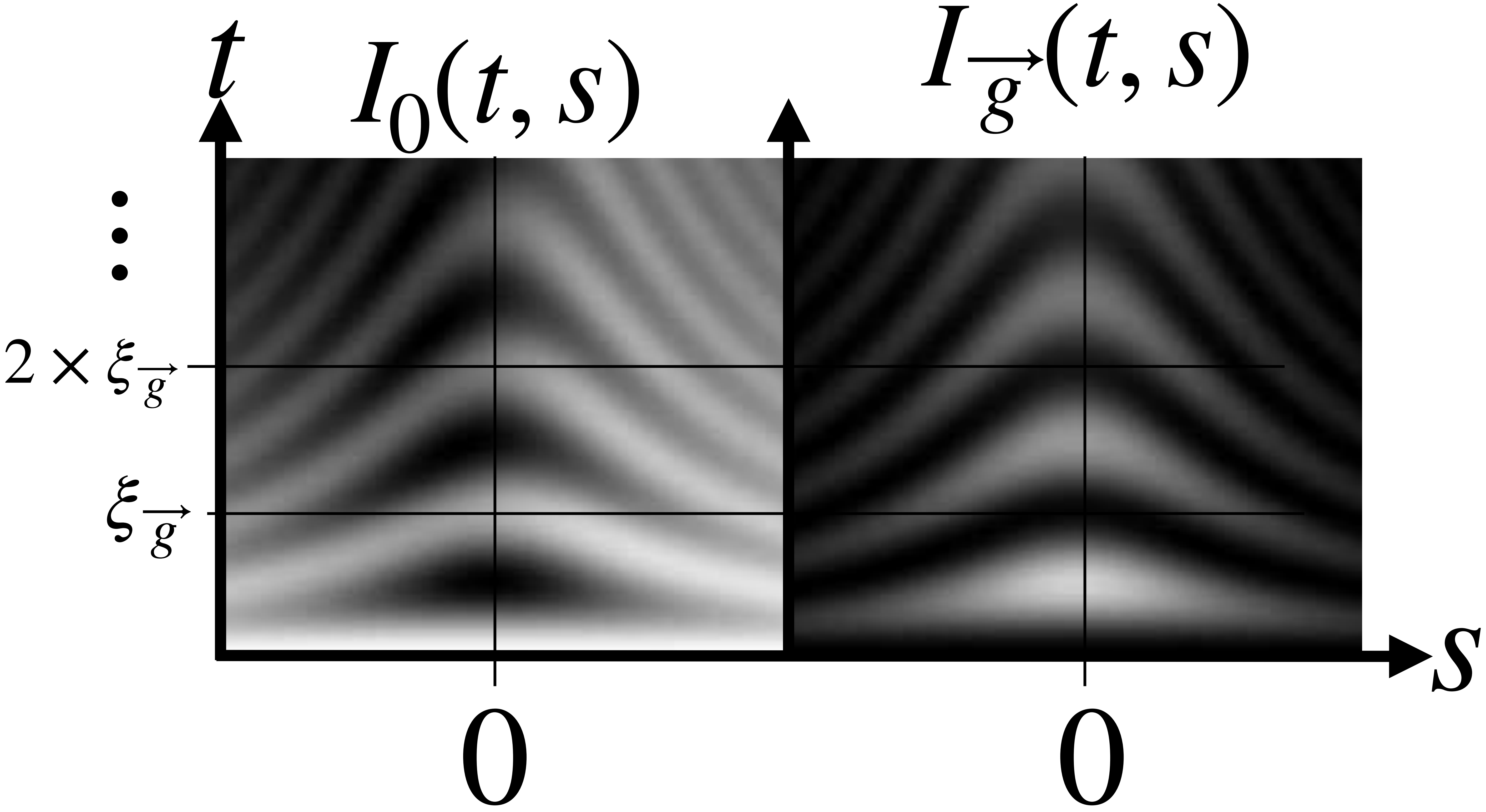
$$\Rightarrow \begin{cases} \dfrac{d\psi_0}{dz} = i\dfrac{\pi}{\xi_0}\psi_0 + i\dfrac{\pi}{\xi_{\vec{g}}}\psi_{\vec{g}} \\ \dfrac{d\psi_{\vec{g}}}{dz} = i\left(\dfrac{\pi}{\xi_0} + 2\pi s\right)\psi_{\vec{g}} + i\dfrac{\pi}{\xi_{\vec{g}}}\psi_0 \end{cases}$$

Diffracted intensities in 2-beams dynamical theory : the dynamical rocking curve

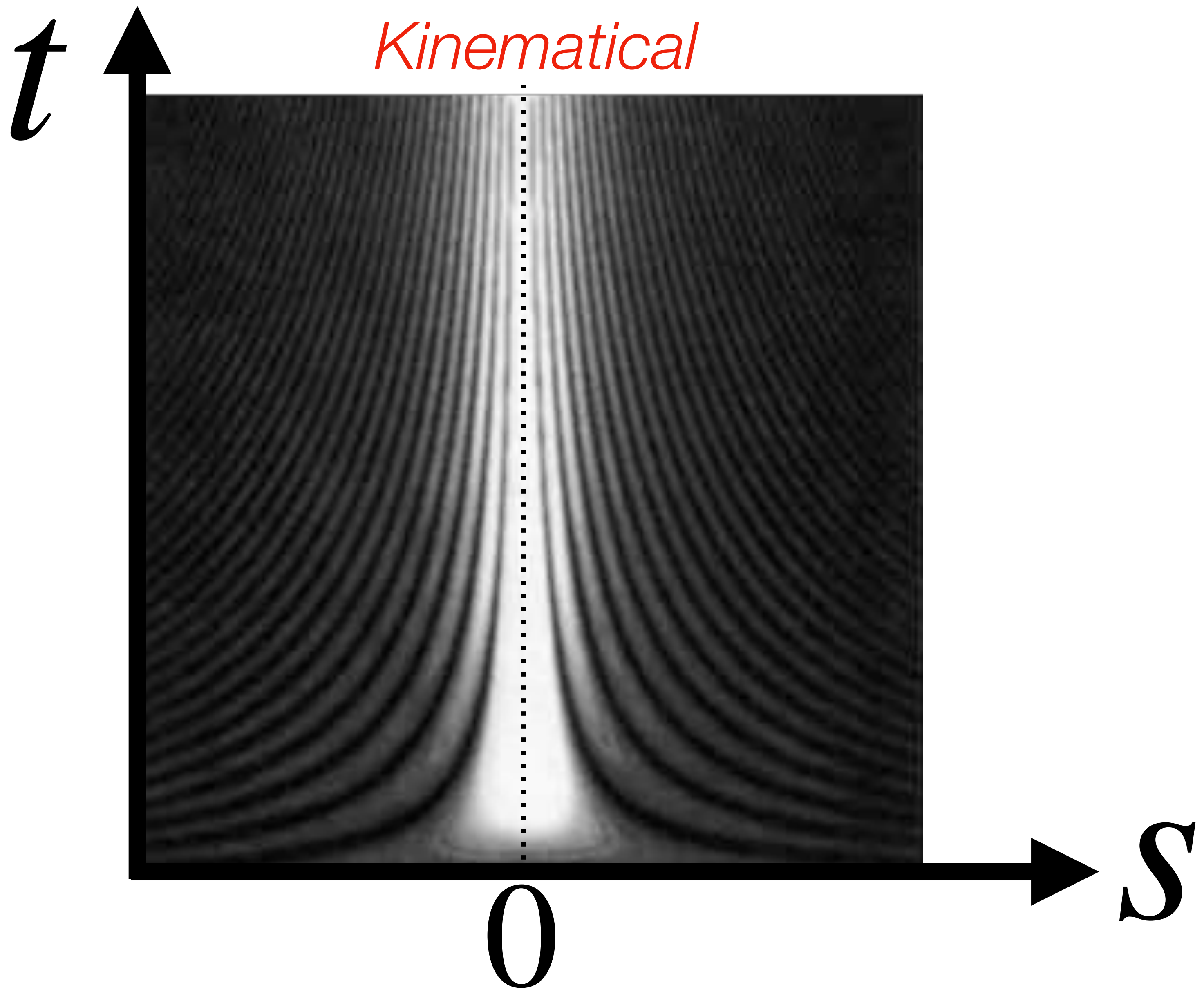
$$\begin{cases} \dfrac{d\psi_0}{dz} = i\dfrac{\pi}{\xi_0}\psi_0 + i\dfrac{\pi}{\xi_{\vec{g}}}\psi_{\vec{g}} \\ \dfrac{d\psi_{\vec{g}}}{dz} = i\left(\dfrac{\pi}{\xi_0} + 2\pi s\right)\psi_{\vec{g}} + i\dfrac{\pi}{\xi_{\vec{g}}}\psi_0 \end{cases}$$

$$\sigma = \frac{\sqrt{1 + (s\xi_{\vec{g}})^2}}{\xi_{\vec{g}}} \Rightarrow \begin{cases} I_0 = \cos^2(\pi\sigma t) + \left(\dfrac{s}{\sigma}\right)^2 \sin^2(\pi\sigma t) \\ I_{\vec{g}} = \dfrac{\sin^2(\pi\sigma t)}{(\sigma\xi_{\vec{g}})^2} \end{cases}$$

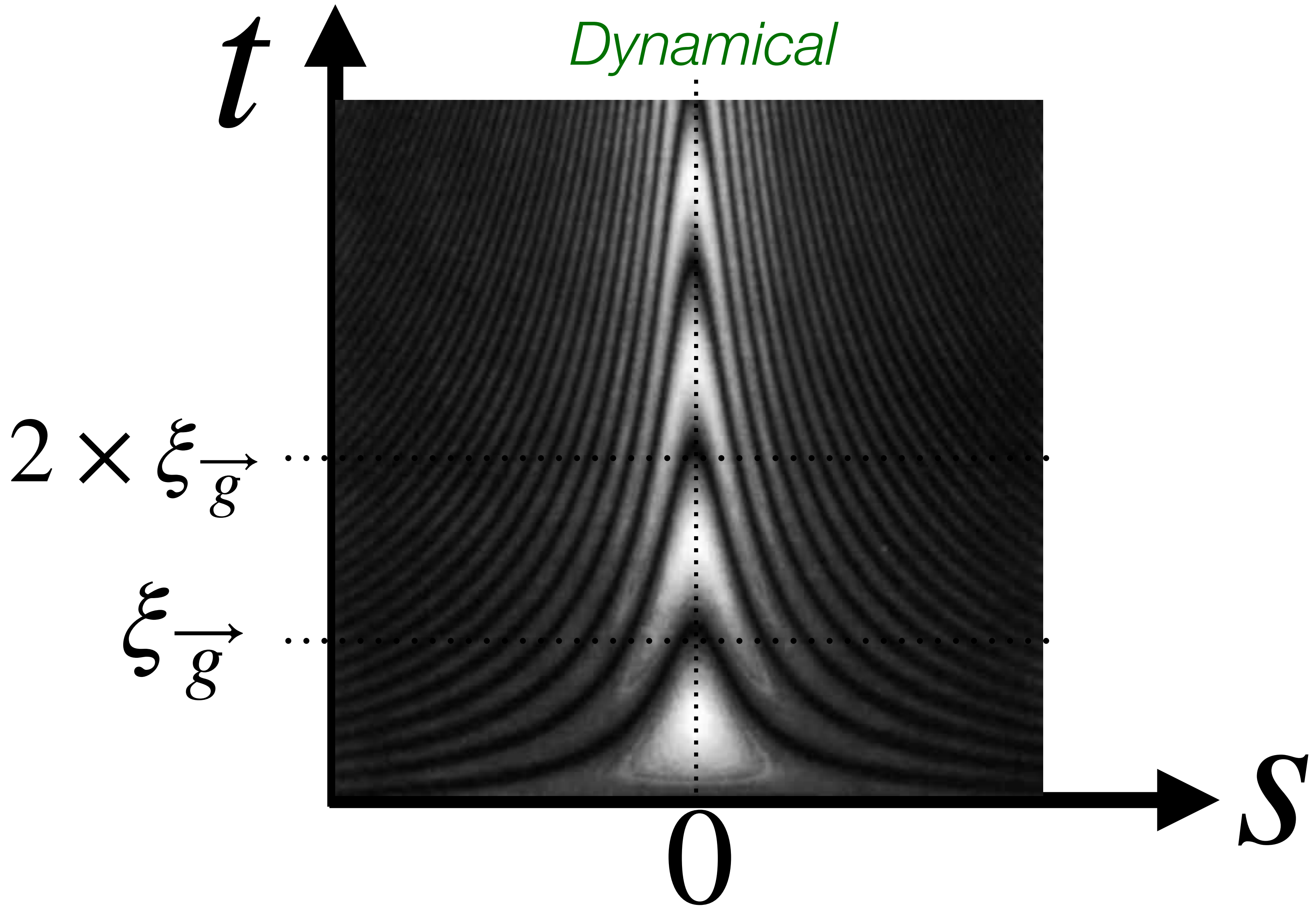


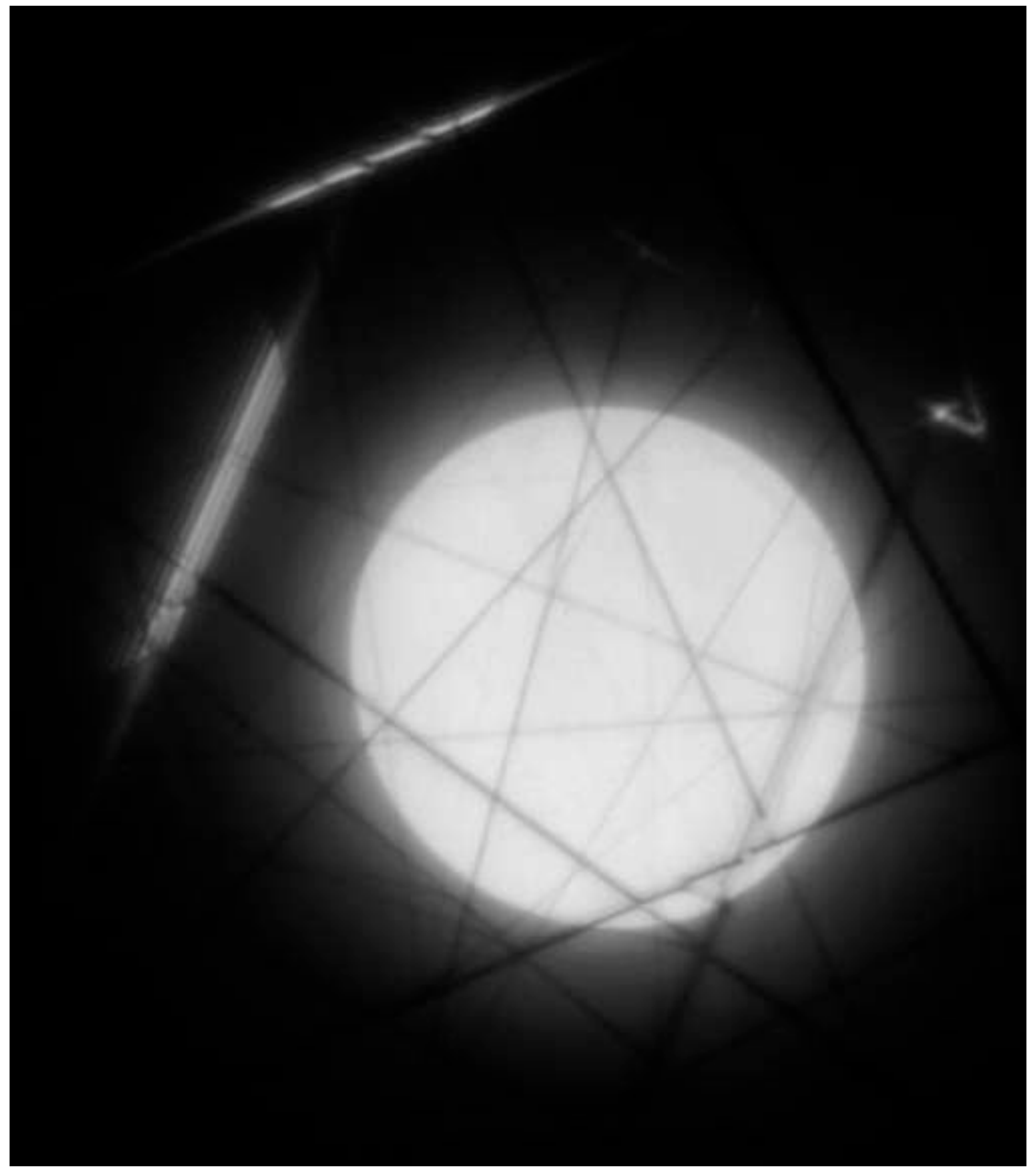


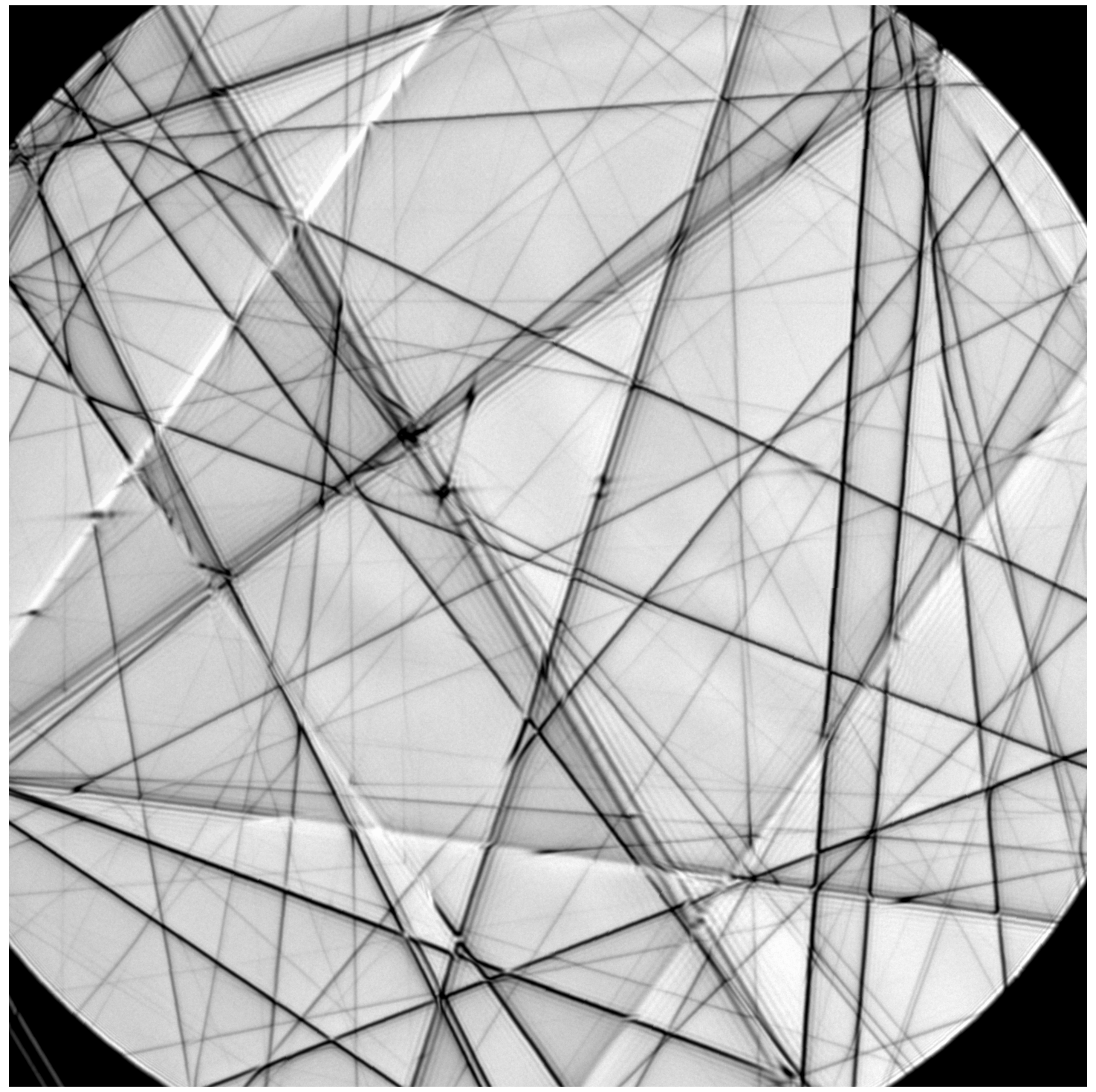
Comparison (again) between kinematical and dynamical rocking curves

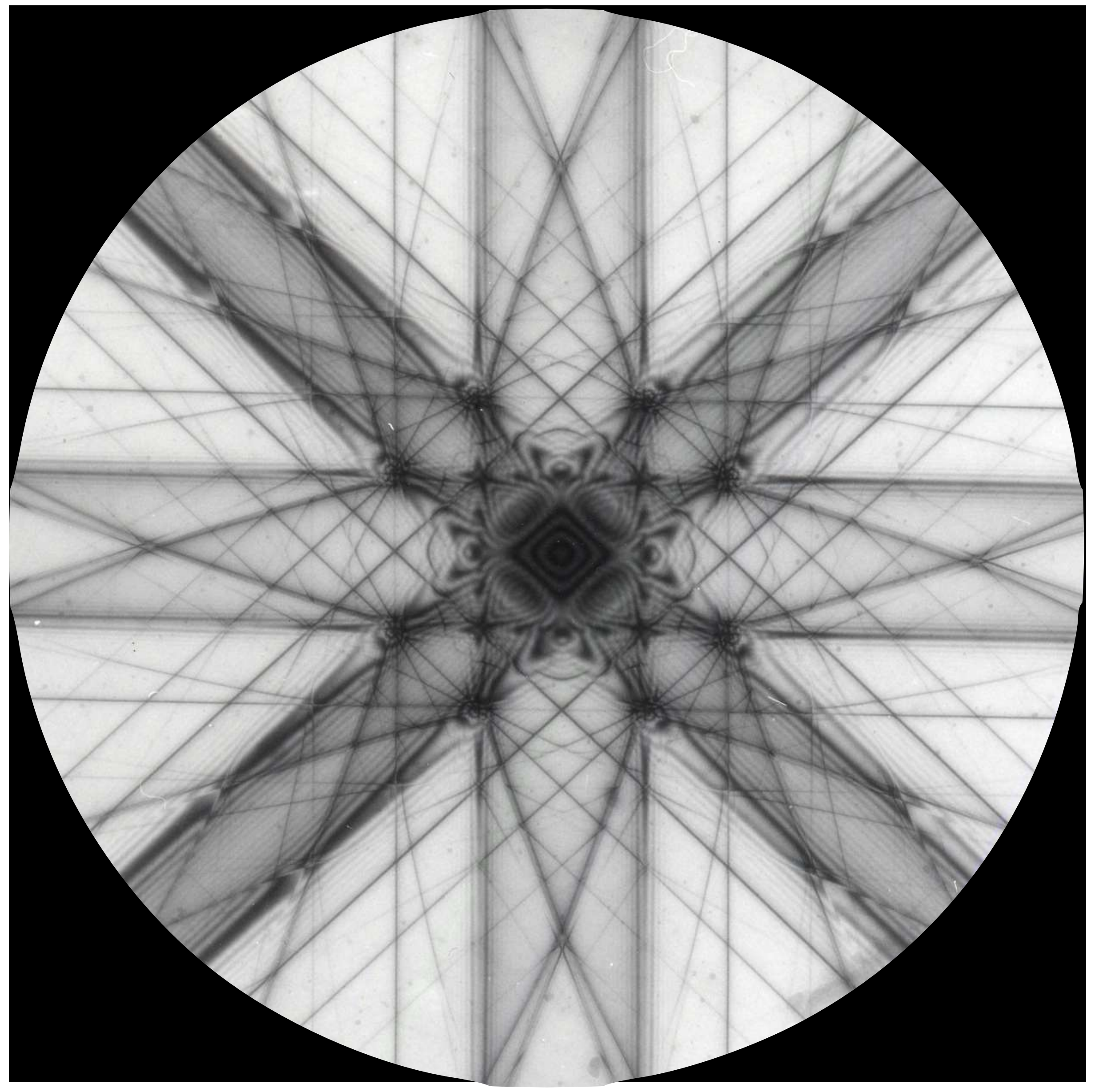


VS







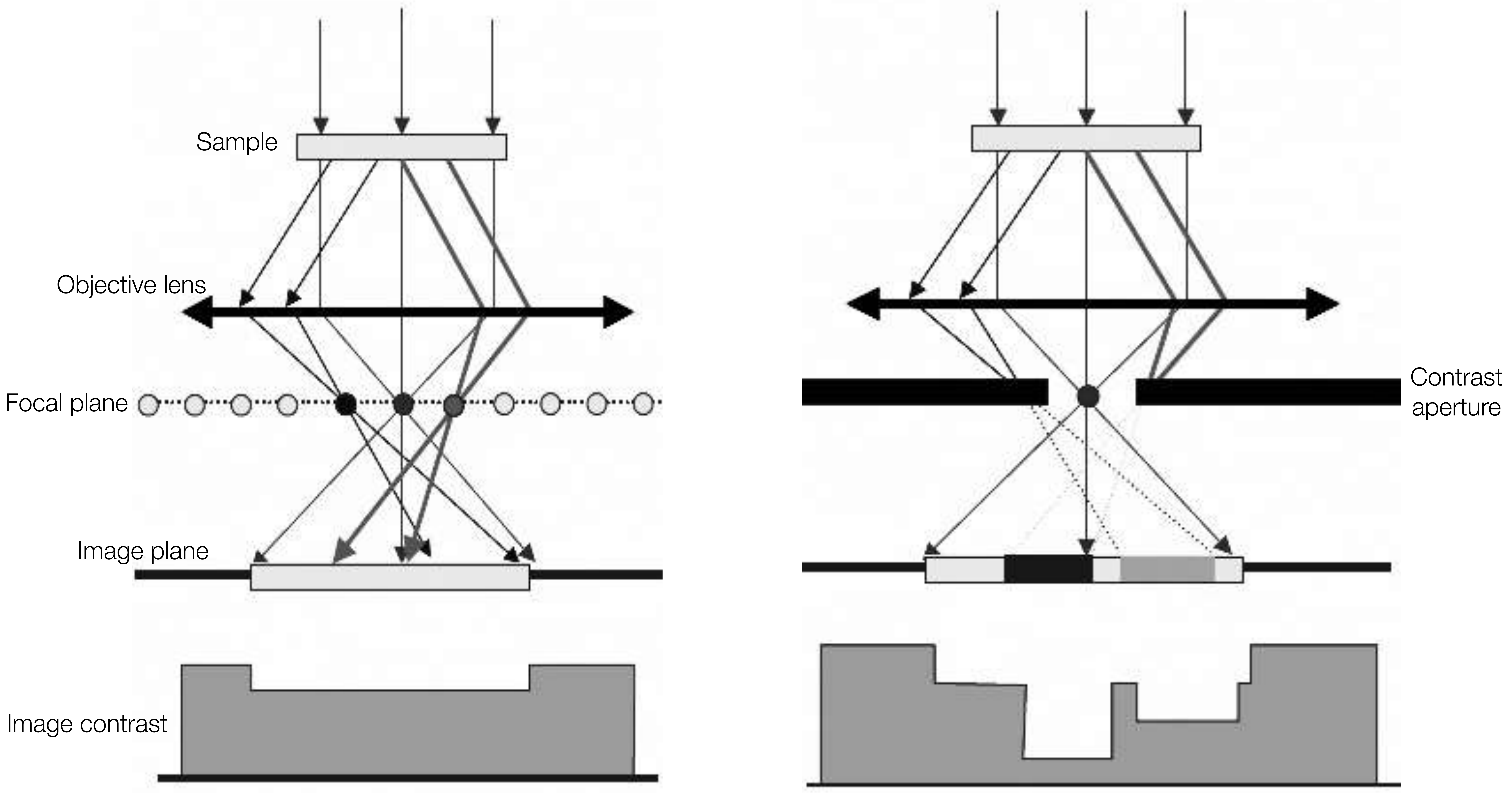


2
Diffraction contrast imaging in perfect crystal

DF-DF imaging
Bend contours
Thickness fringes

Bright field vs dark field imaging : diffraction contrast

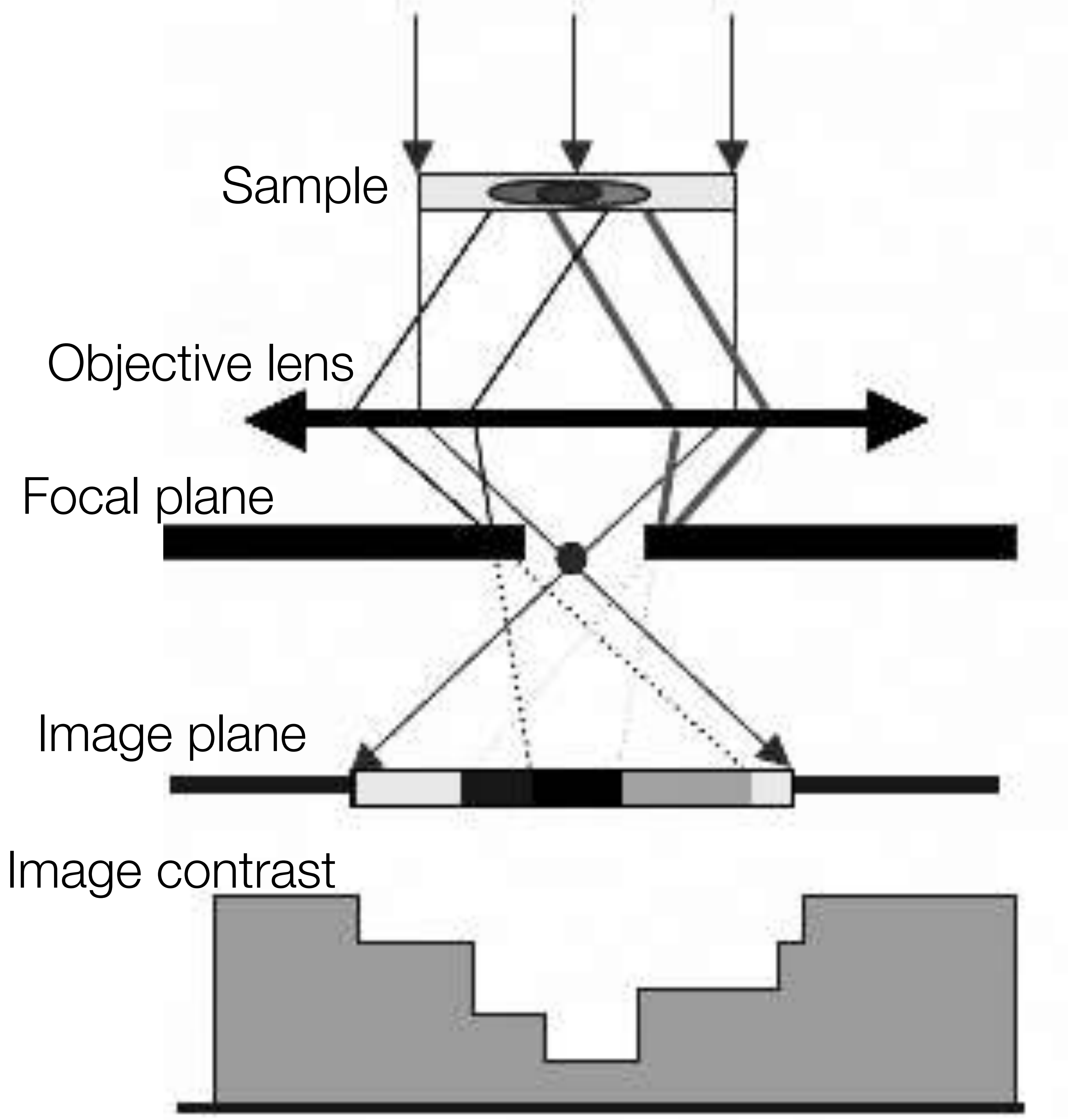
A. *Without* contrast aperture | B. *With* contrast aperture (bright field)



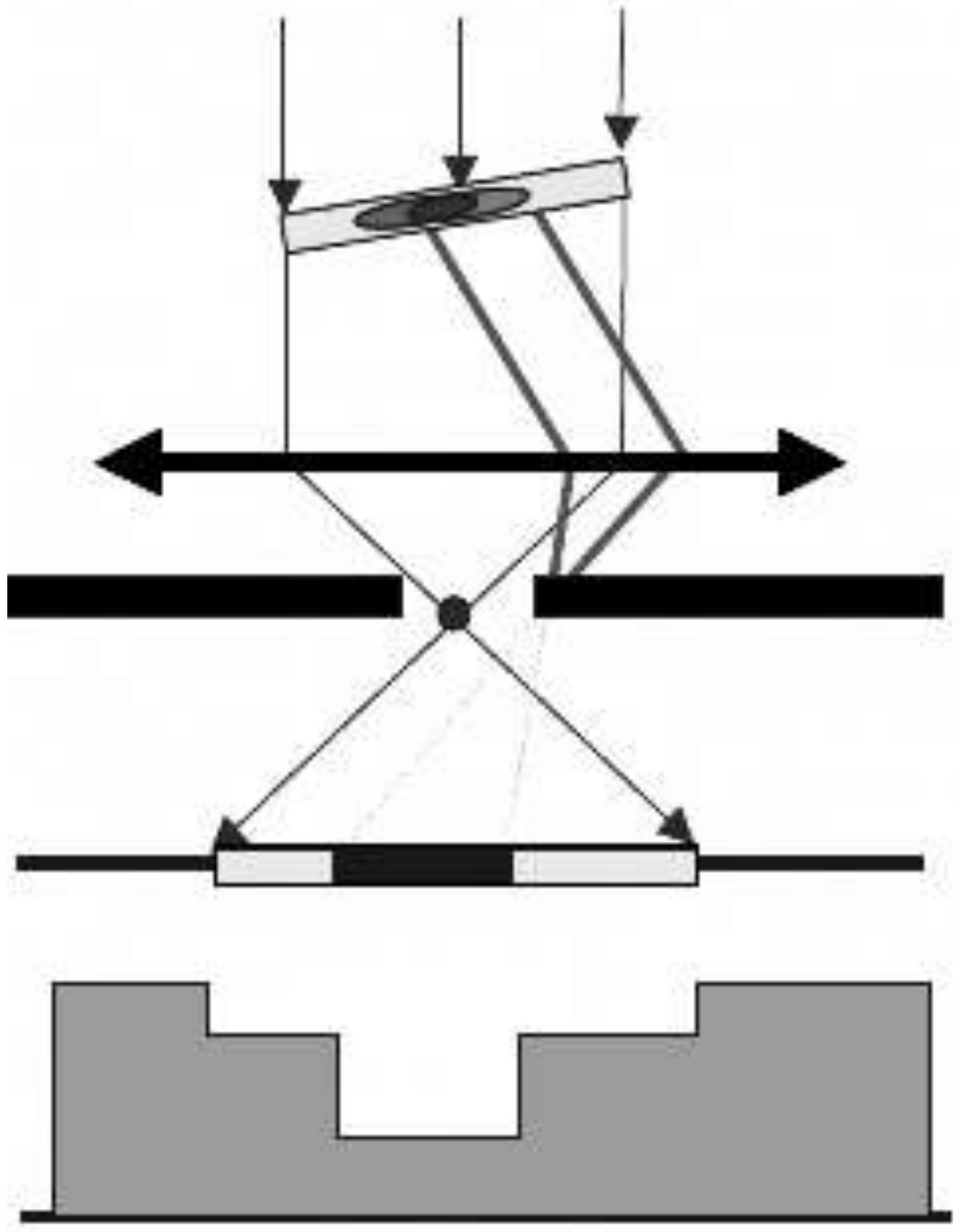
Bright field vs dark field imaging : diffraction contrast

C. ***With*** contrast aperture (bright field)

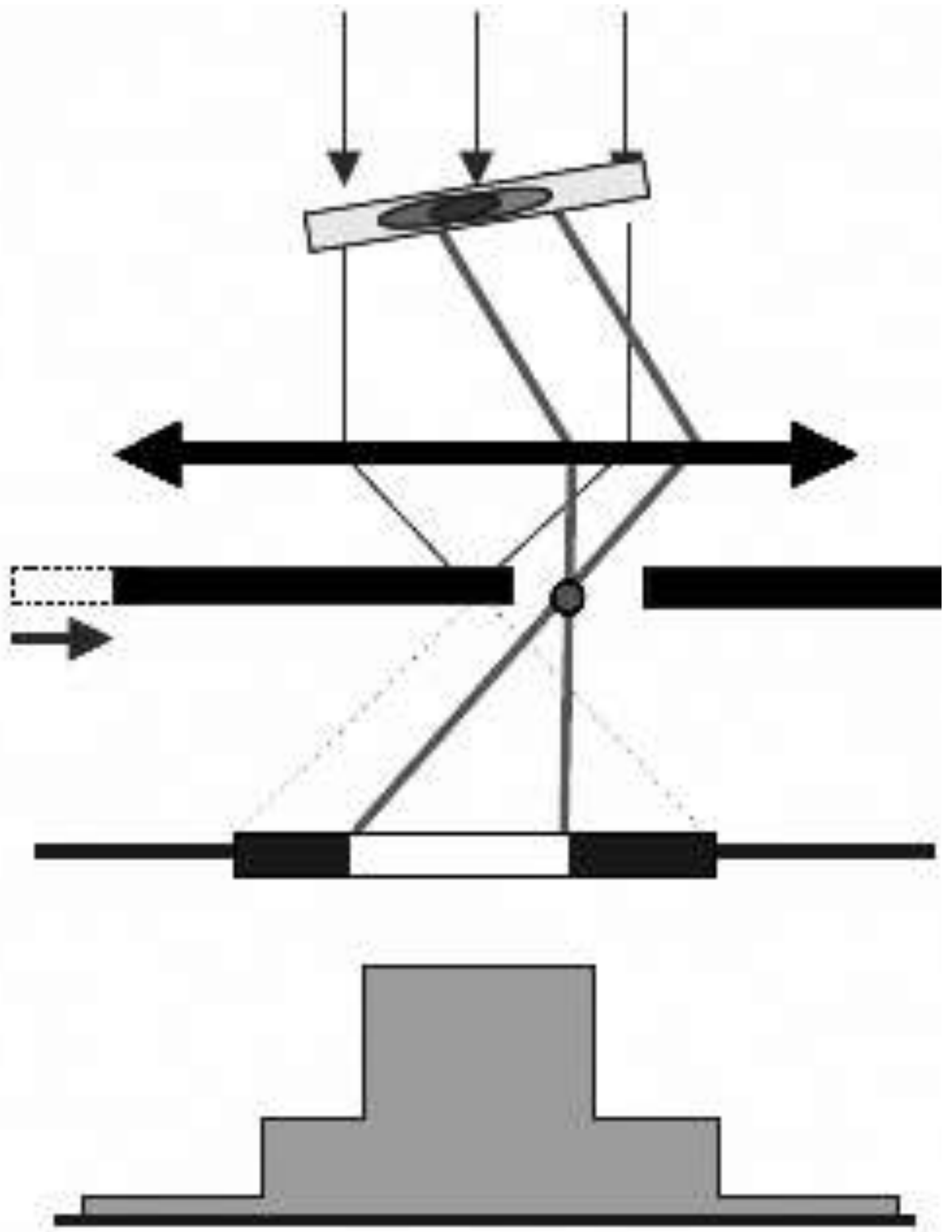
D. ***With*** contrast aperture (Dark field)



2 separate areas have different diffraction conditions
We can distinctly observed these 2 zones in the image

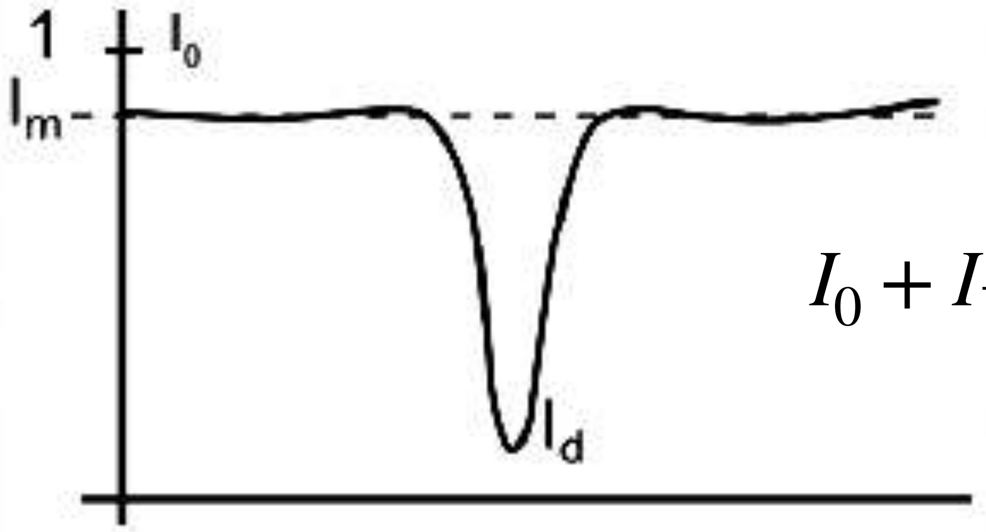


After tilting the sample, we can choose to observe only one area

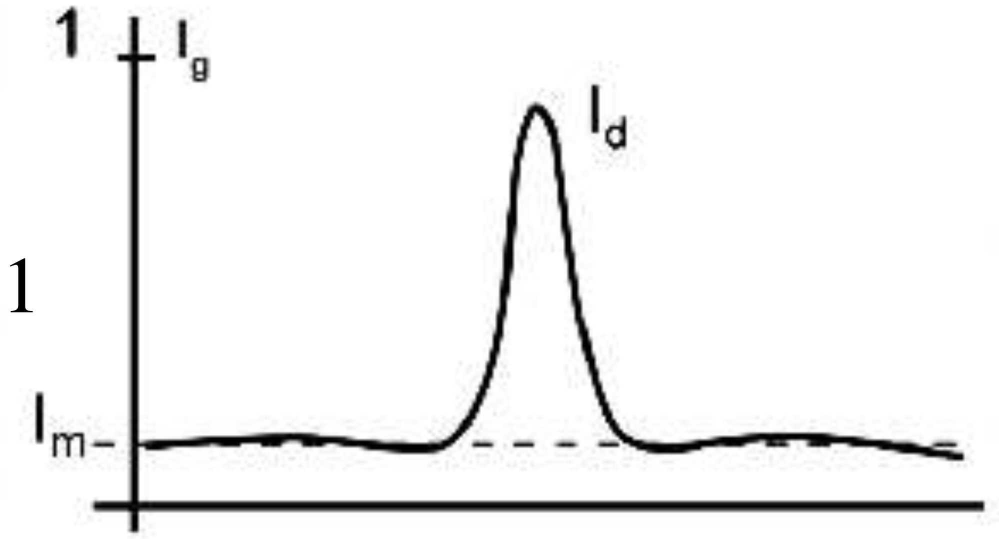


By selecting the diffracted beam we can observed A dark field contrast reversed from the BF.

BF vs DF imaging ?



$$I_0 + I_{\vec{g}} = 1$$

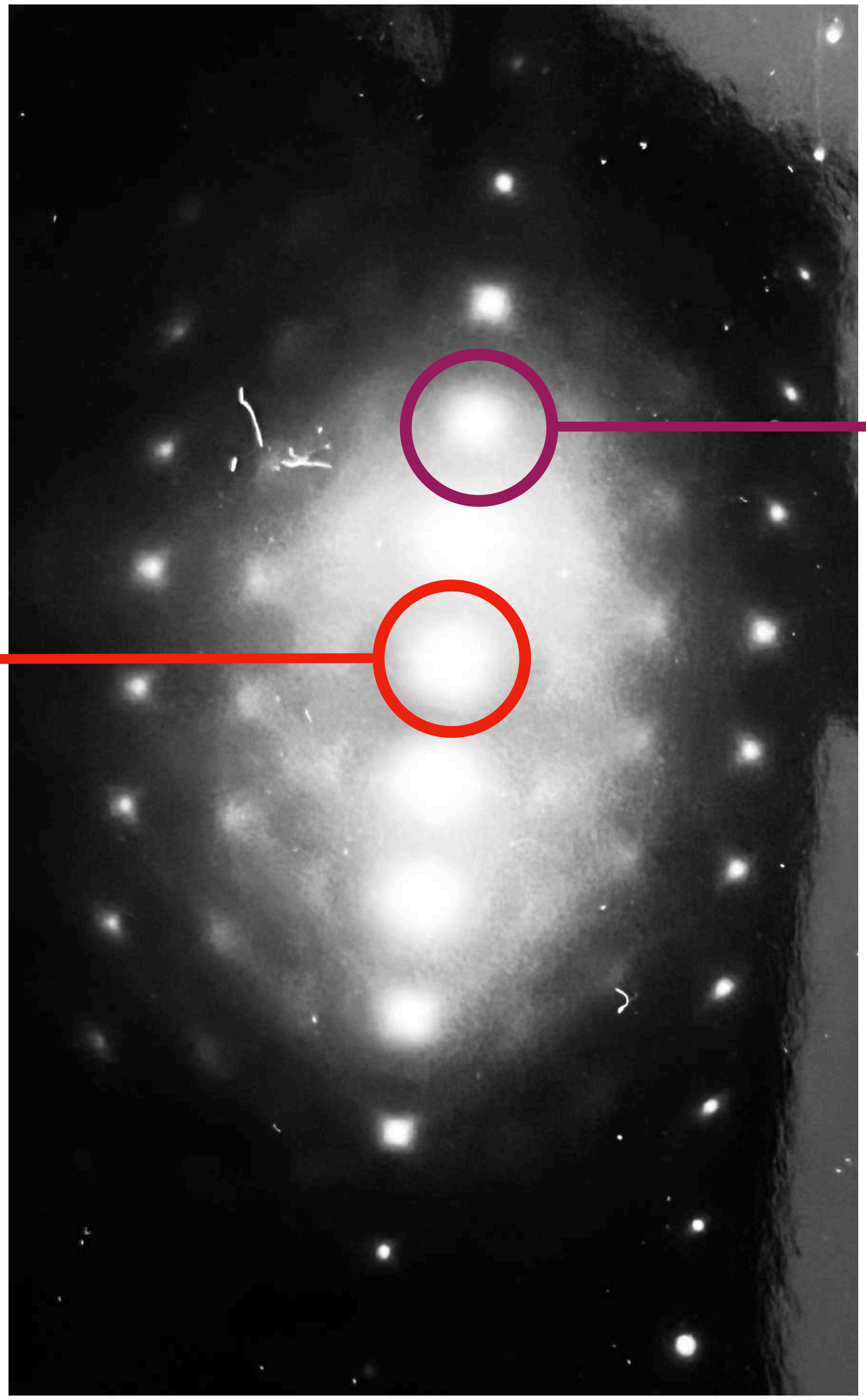


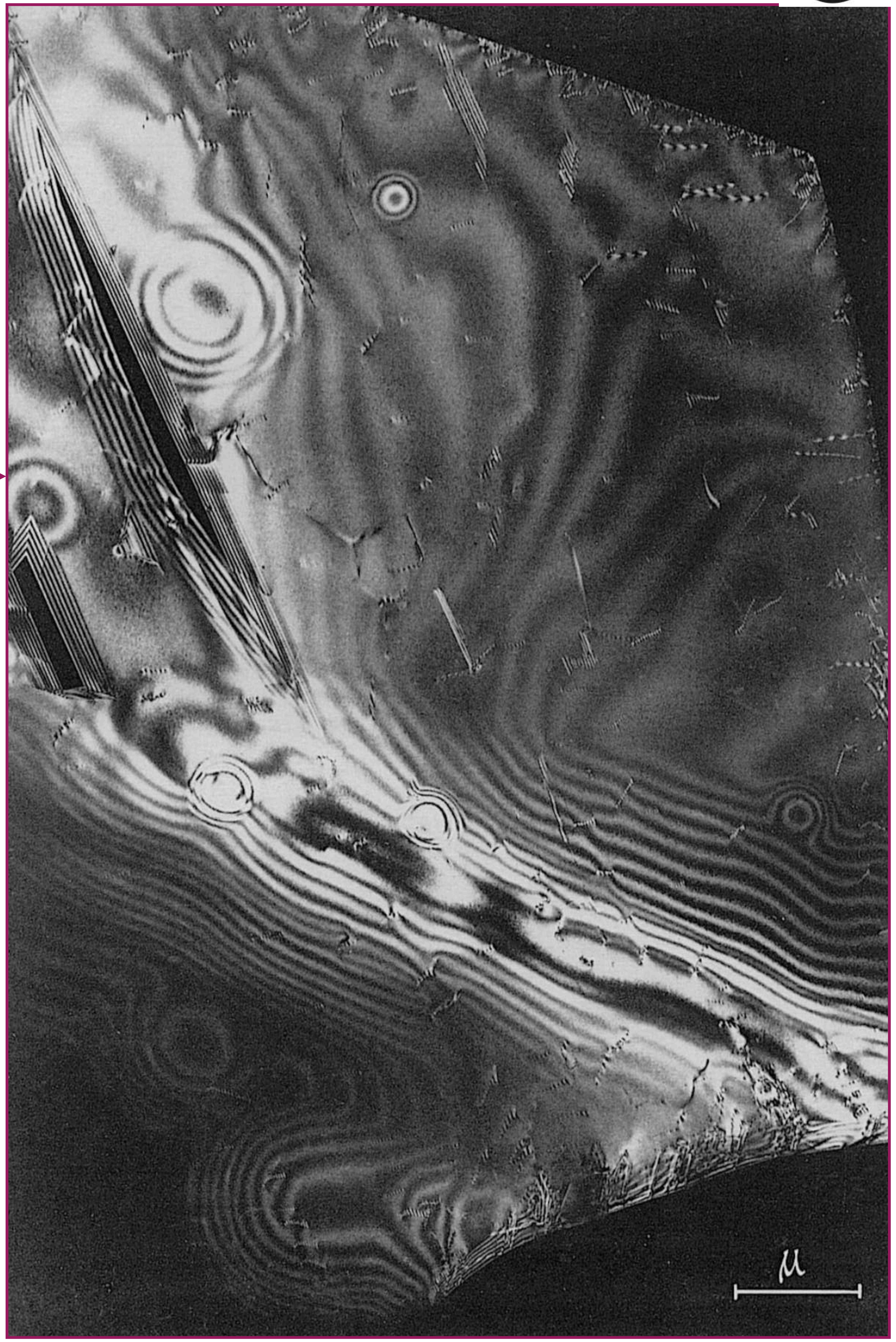
$$C = \frac{|I_d - I_m|}{I_m}$$

Contrast C is optimum in dark field

Bright field vs dark field imaging : diffraction contrast

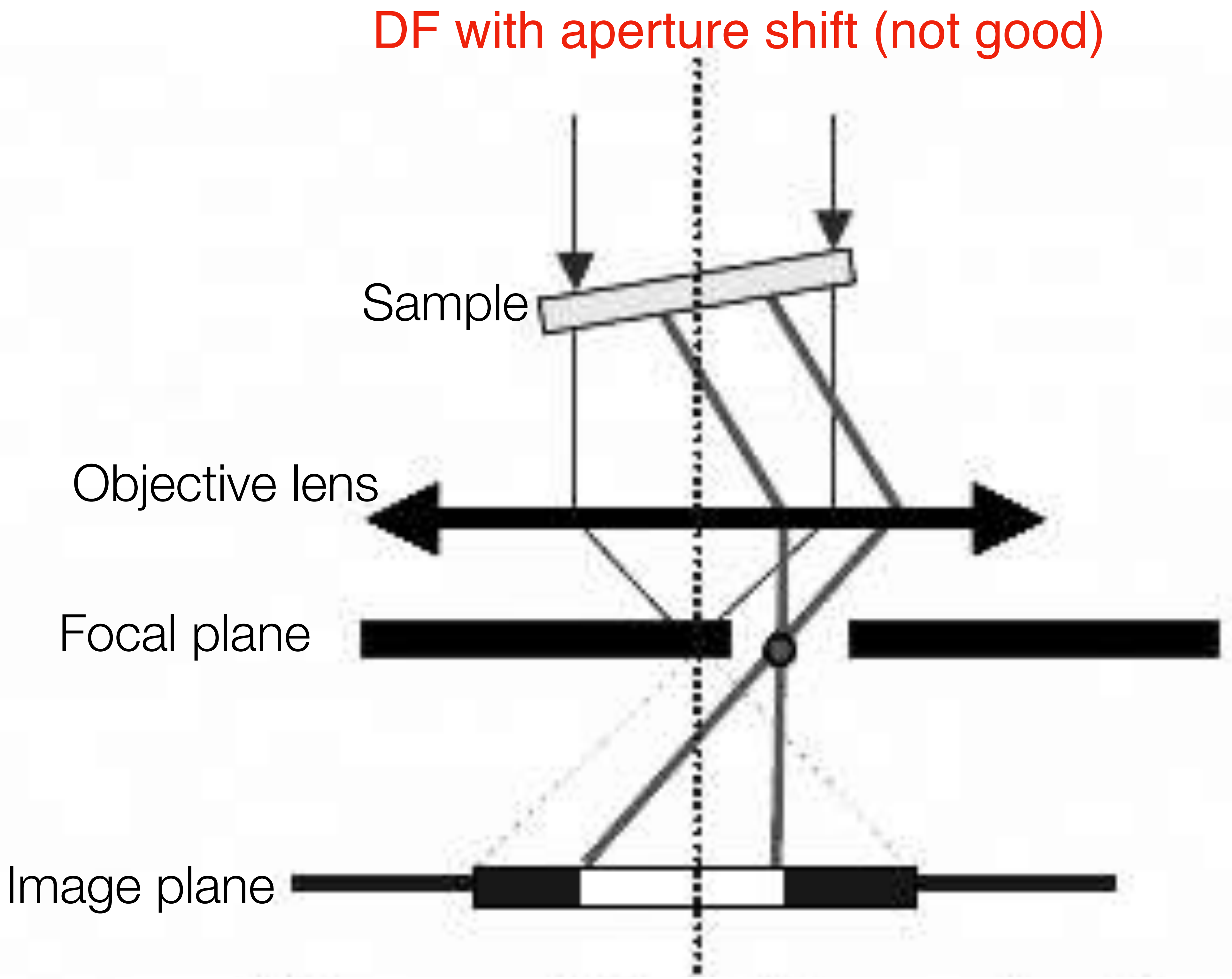
Bright Field (BF)

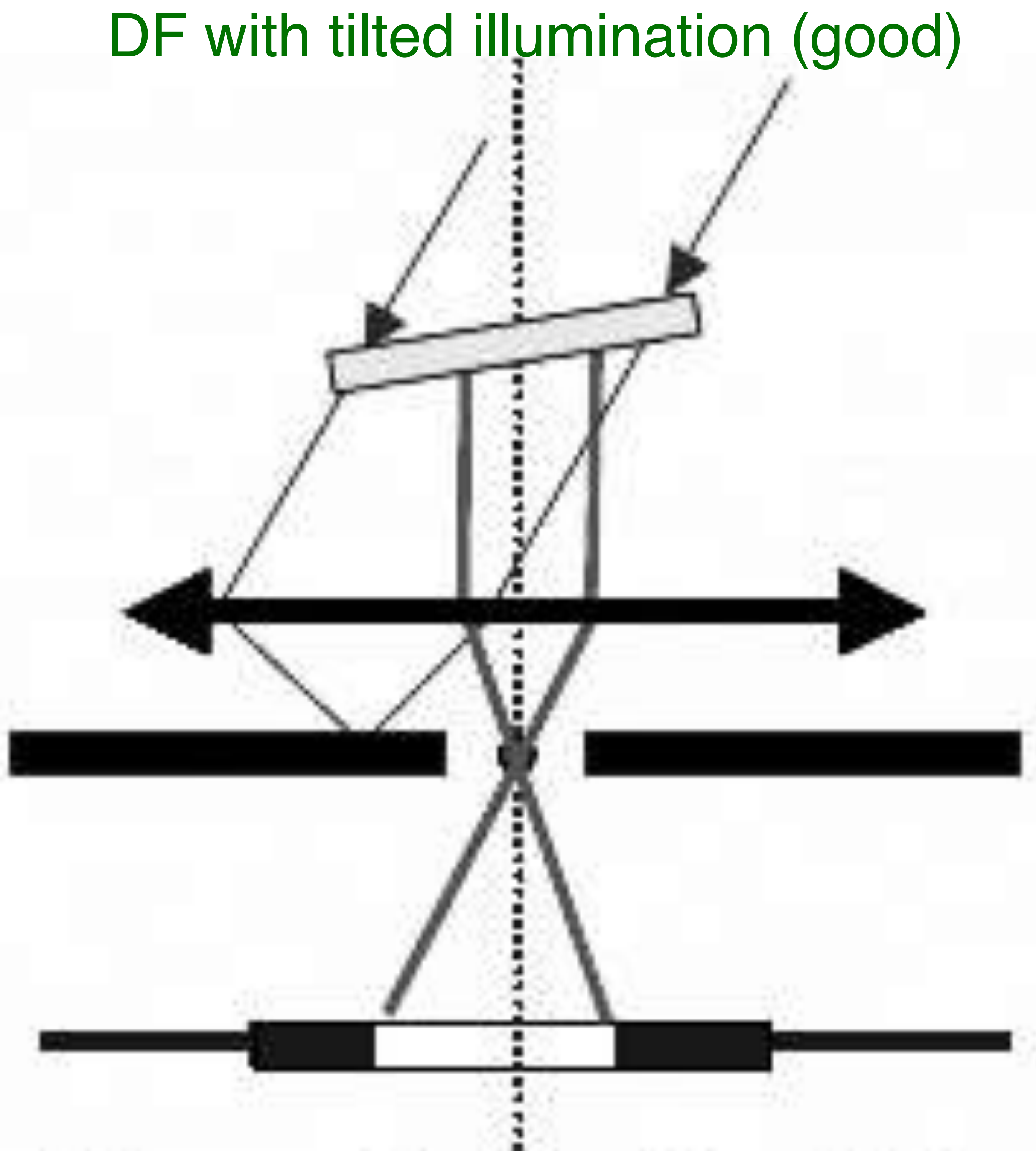


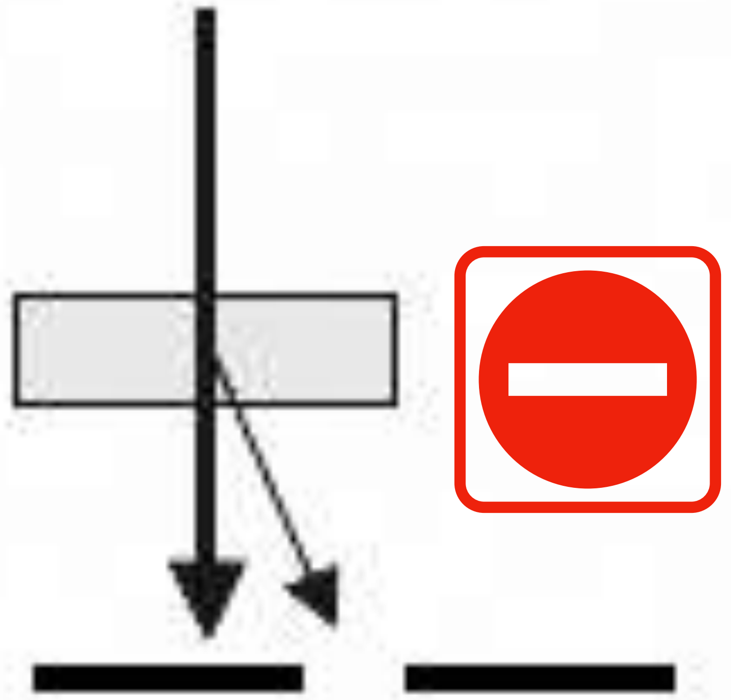


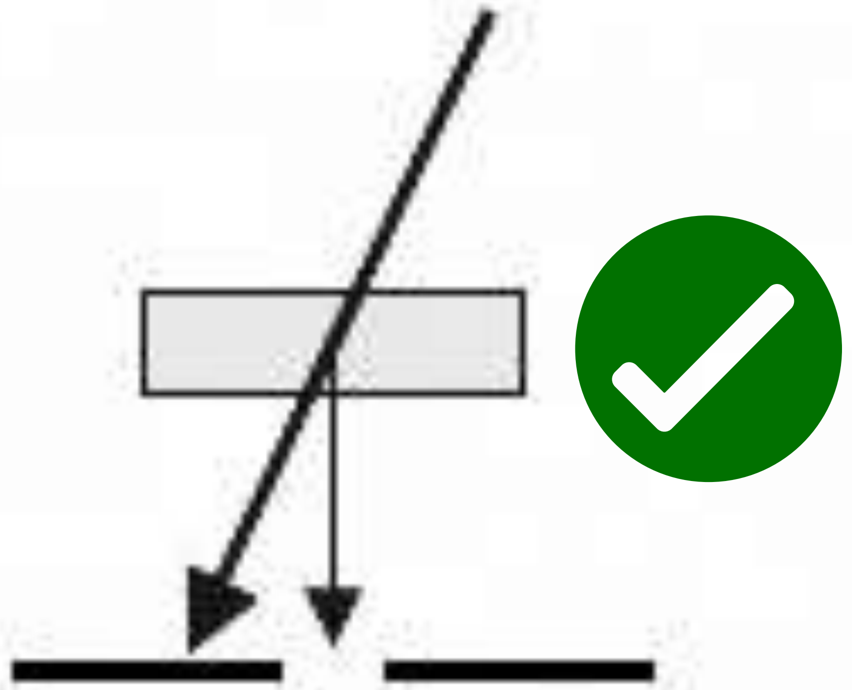
Dark Field (DF)

Dark field imaging : important practical consideration

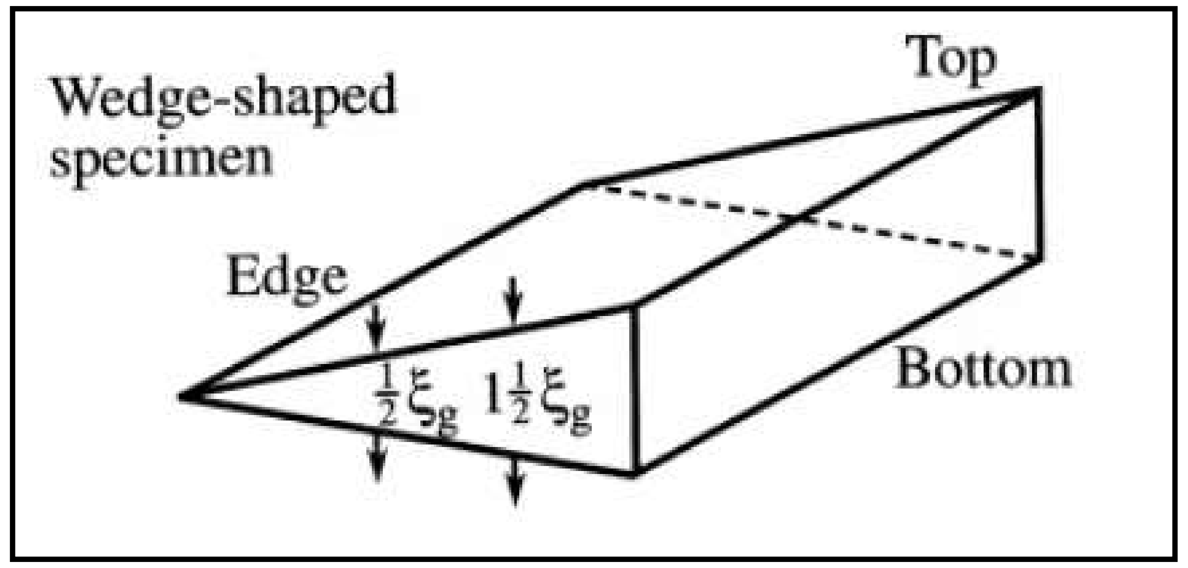




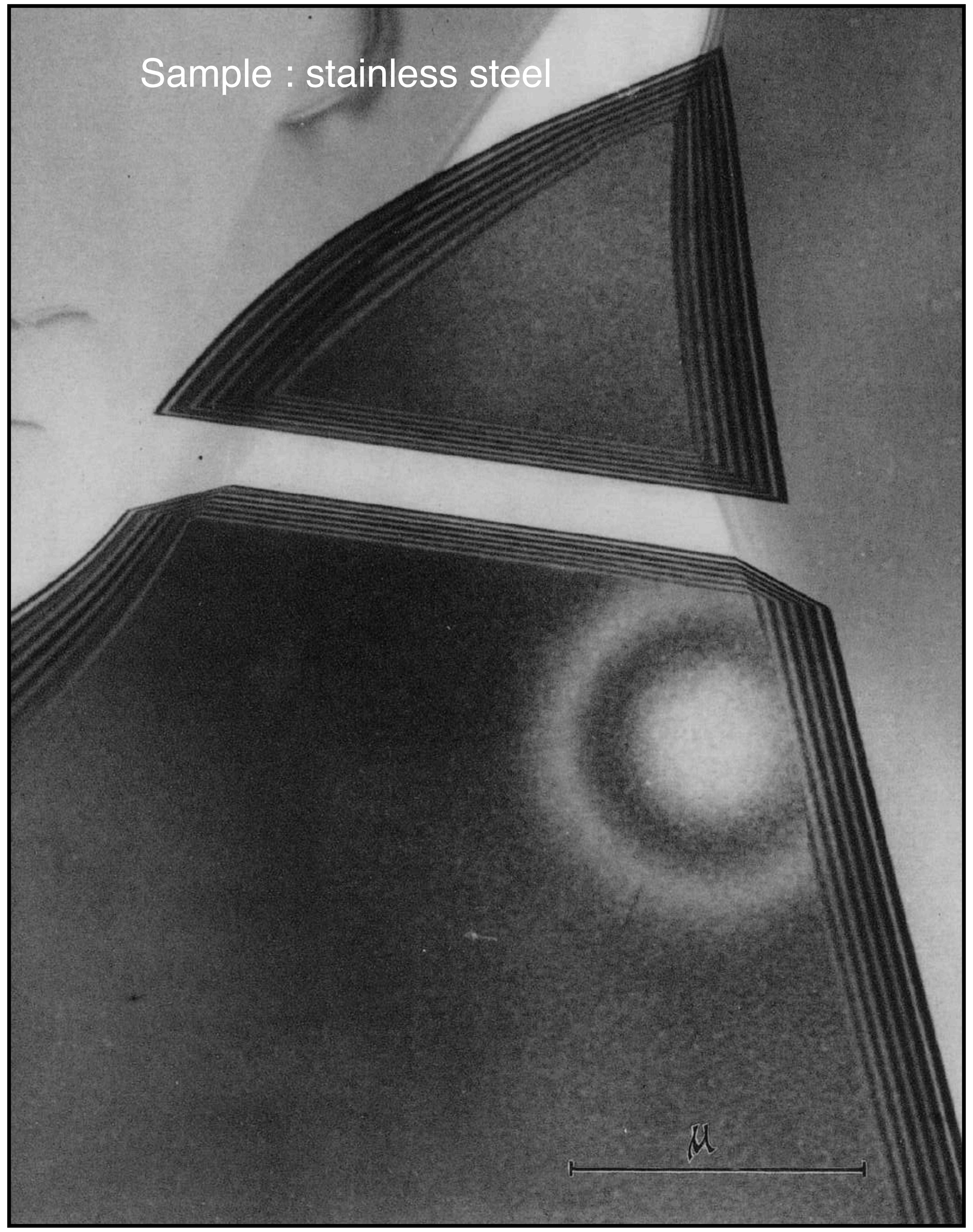


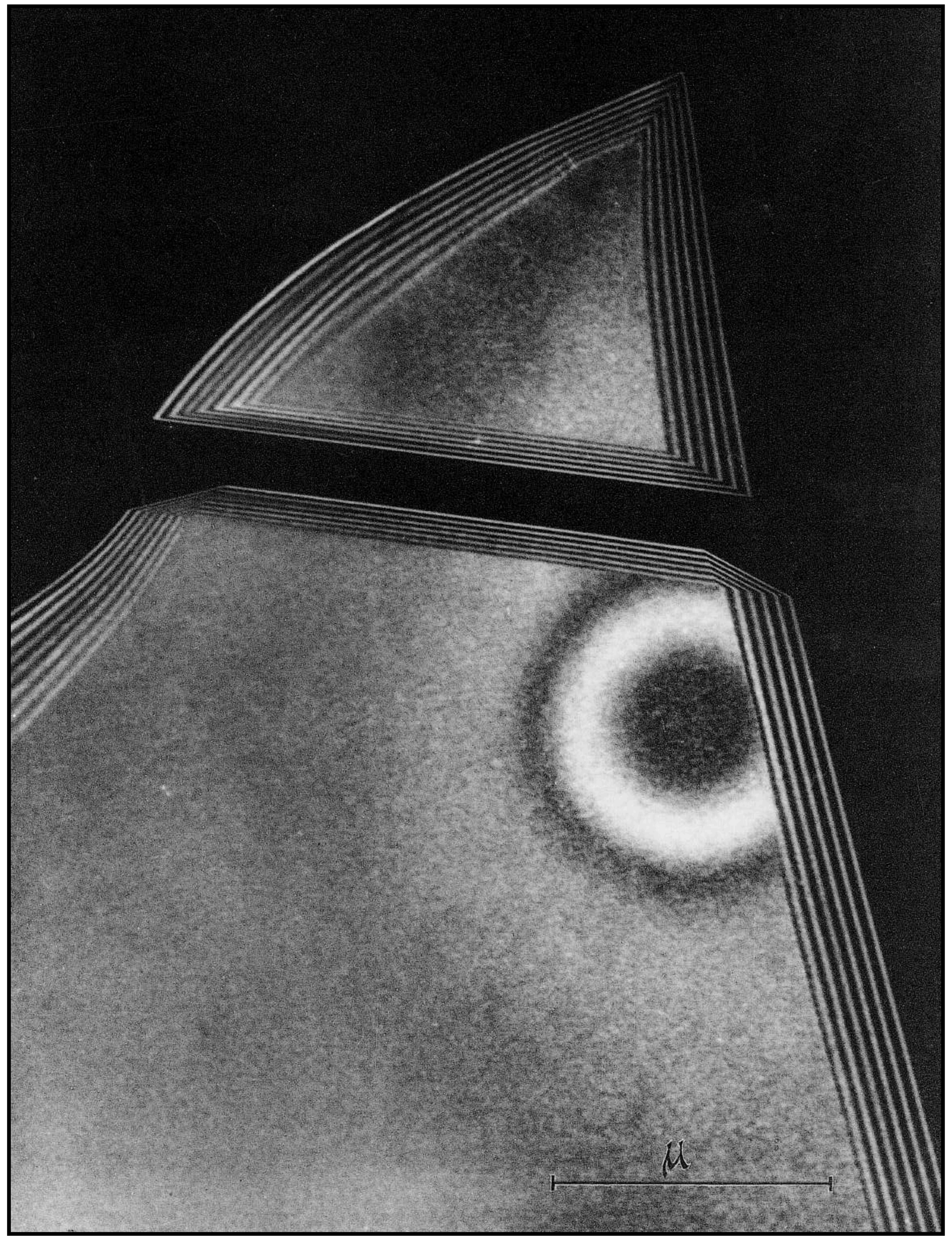


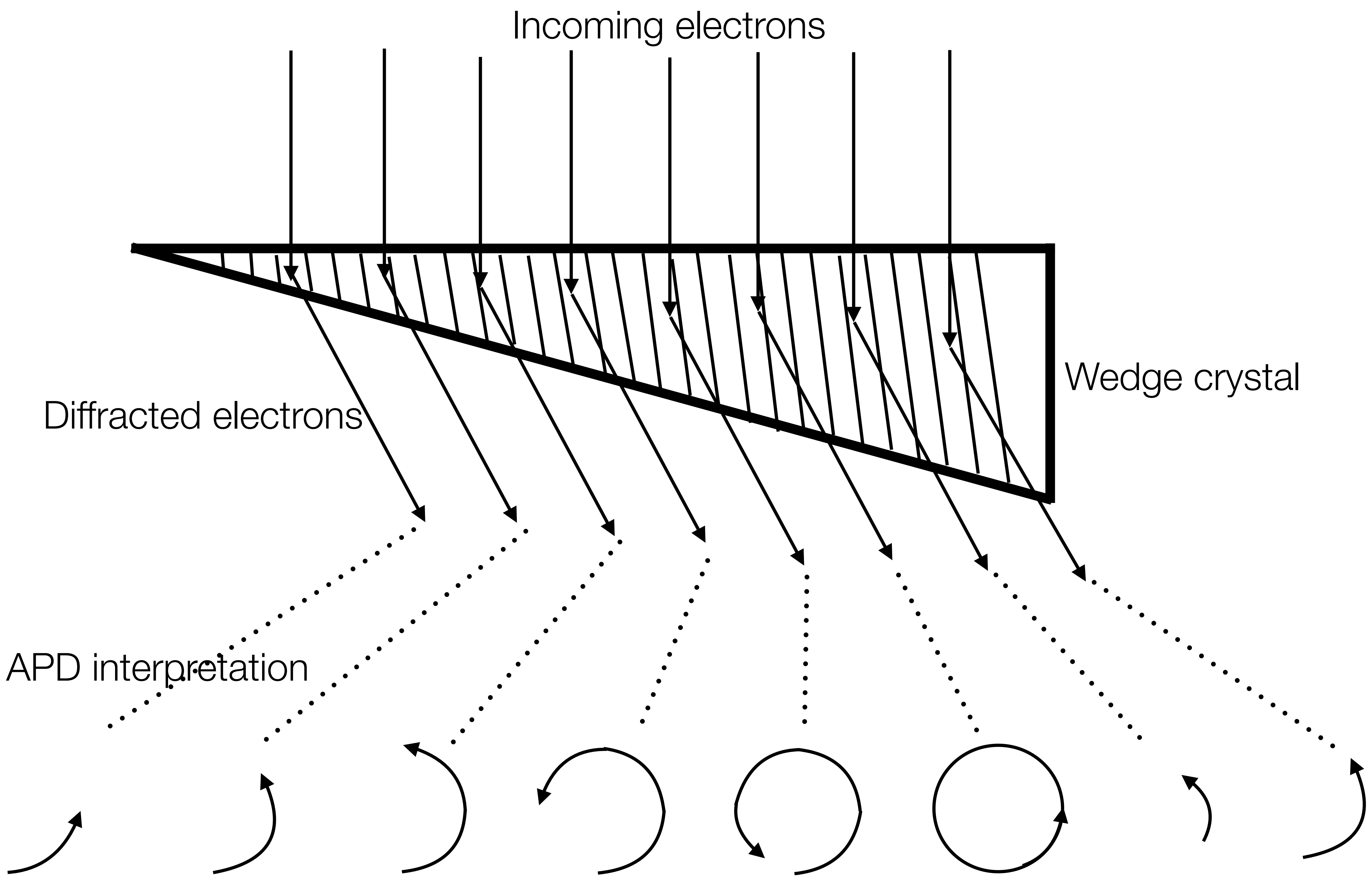
Thickness fringes : kinematical approach



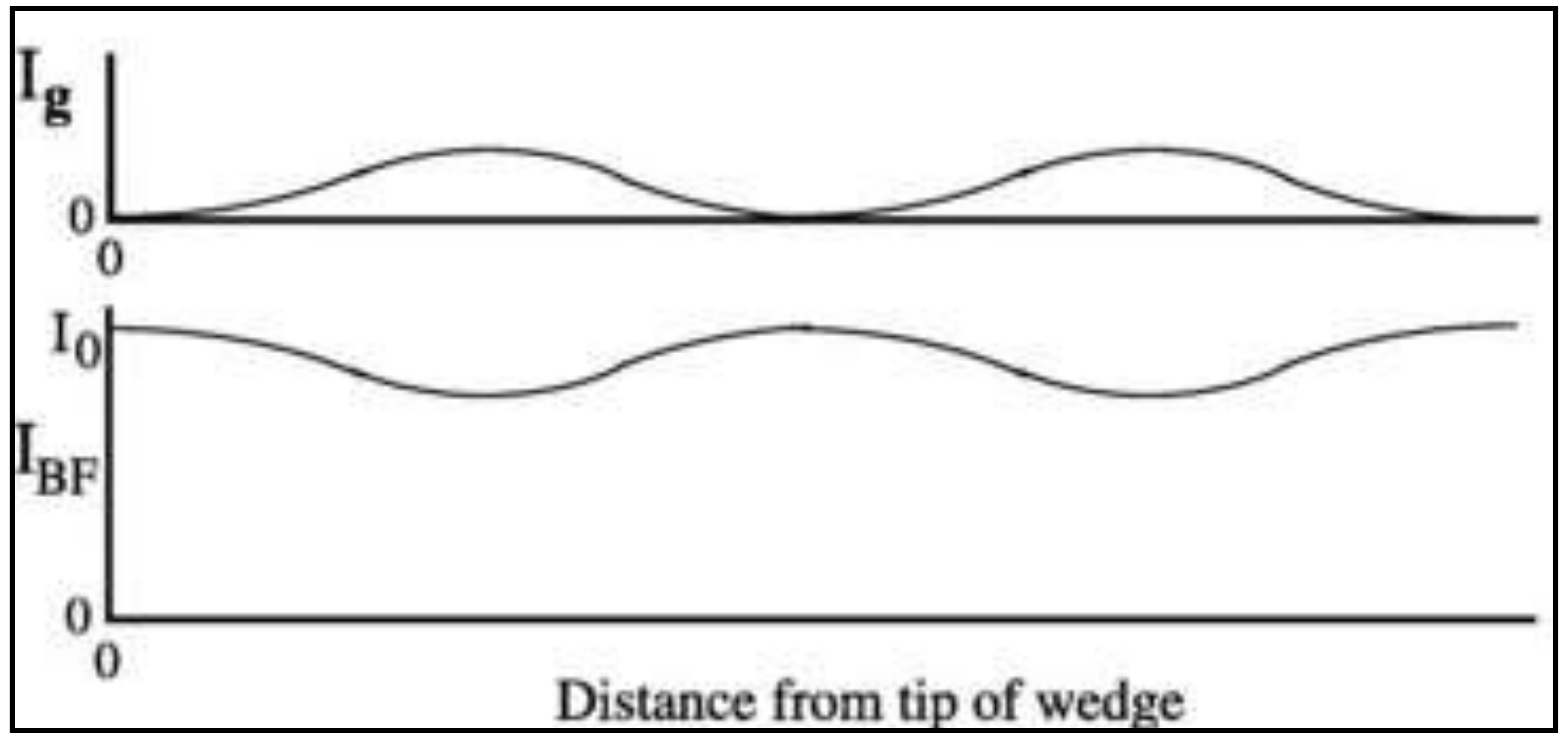
$$I_{\vec{g}} = \psi_{\vec{g}}\psi^*_{\vec{g}} = \frac{\pi^2}{\xi^2_{\vec{g}}}\frac{\sin^2(\pi t s)}{(\pi s)^2}$$



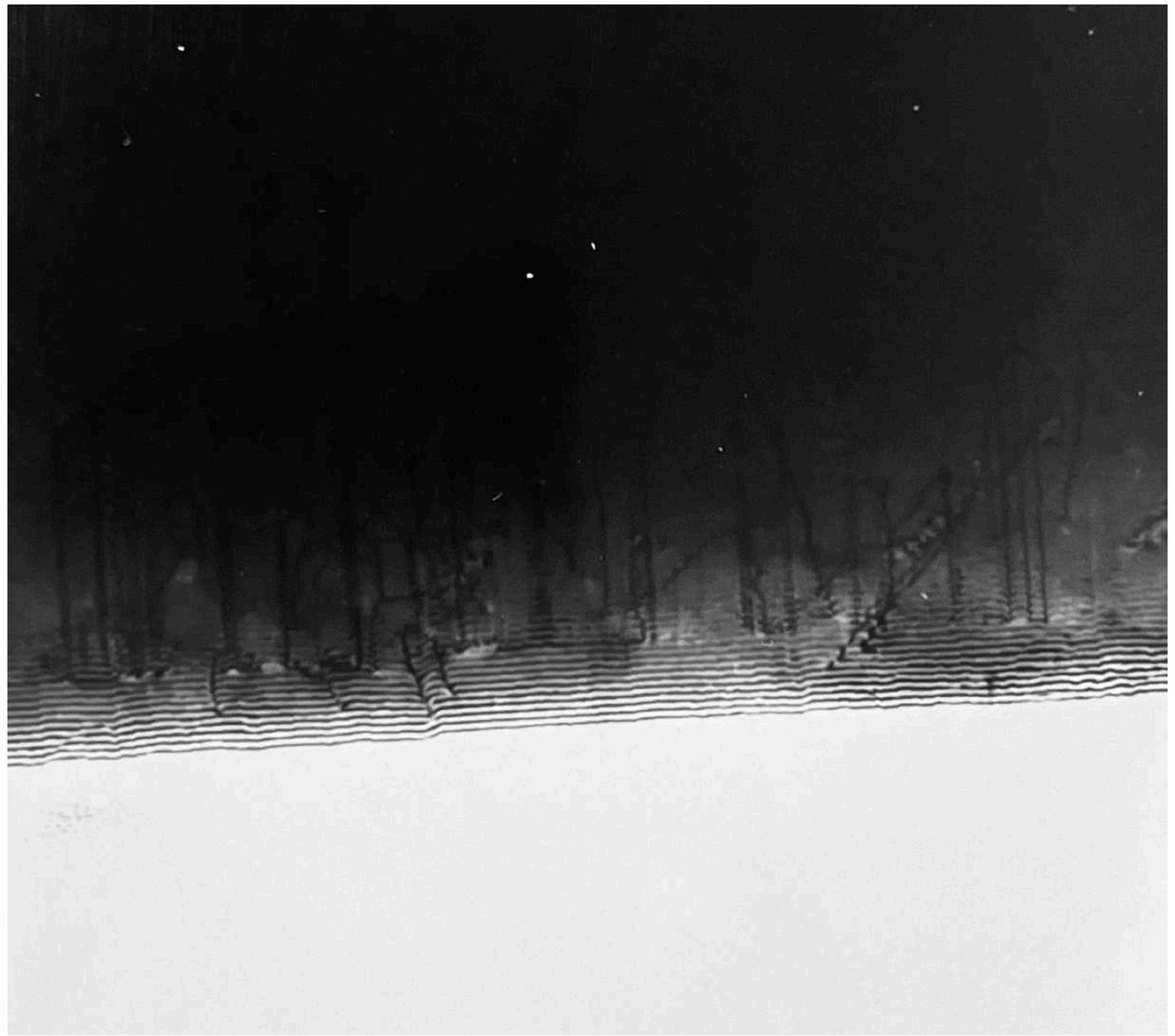


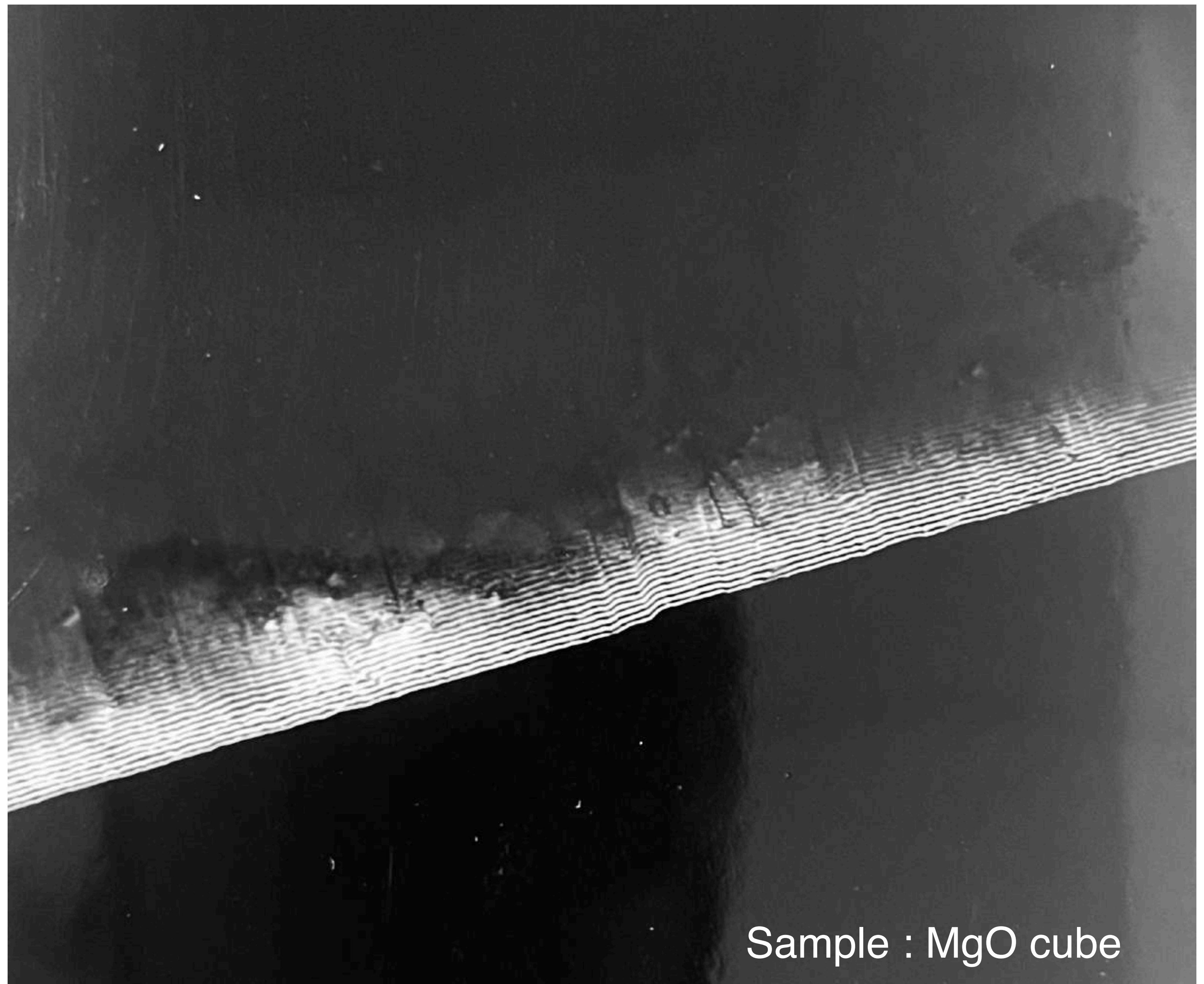


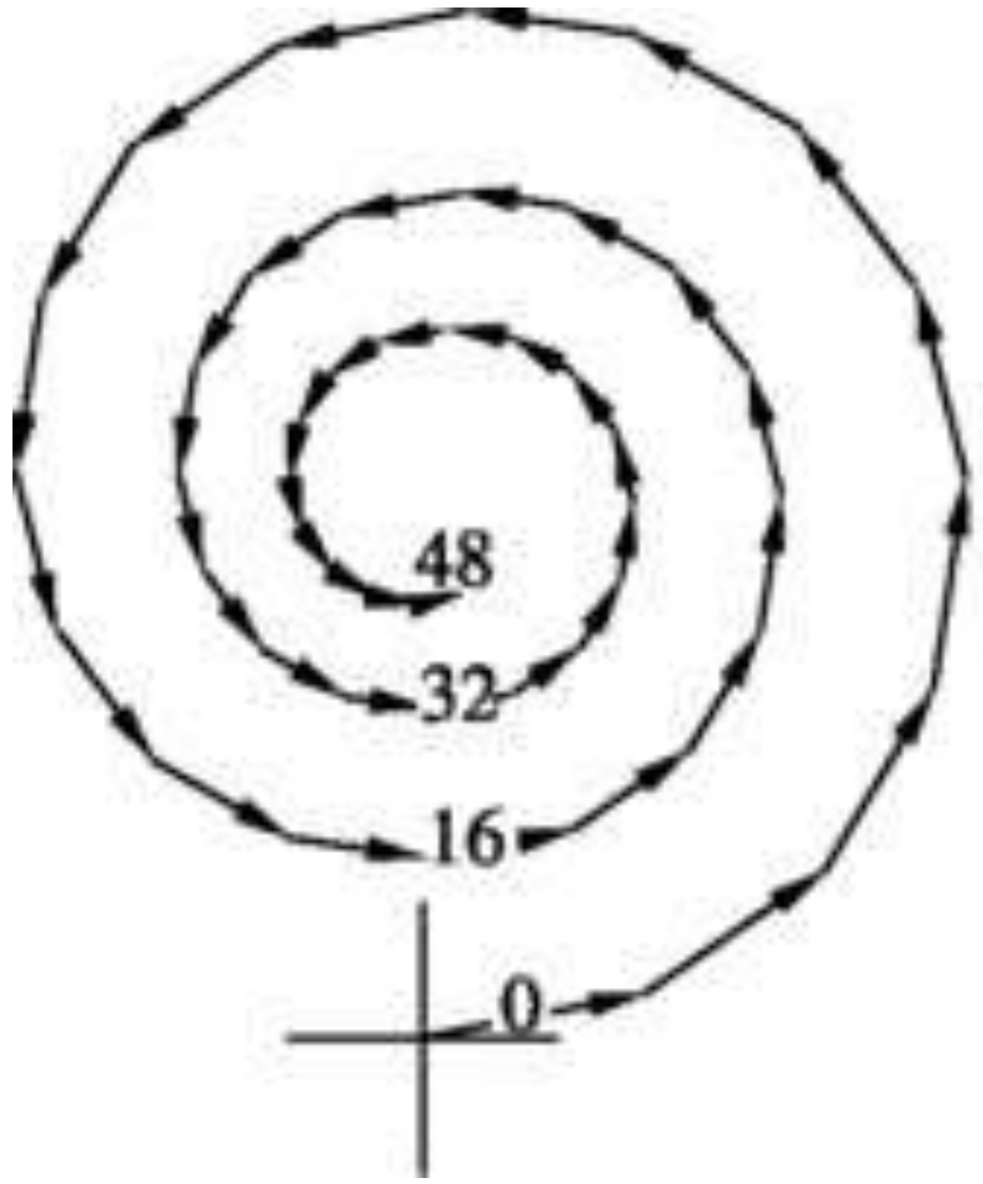
Resulted intensities after APD estimation

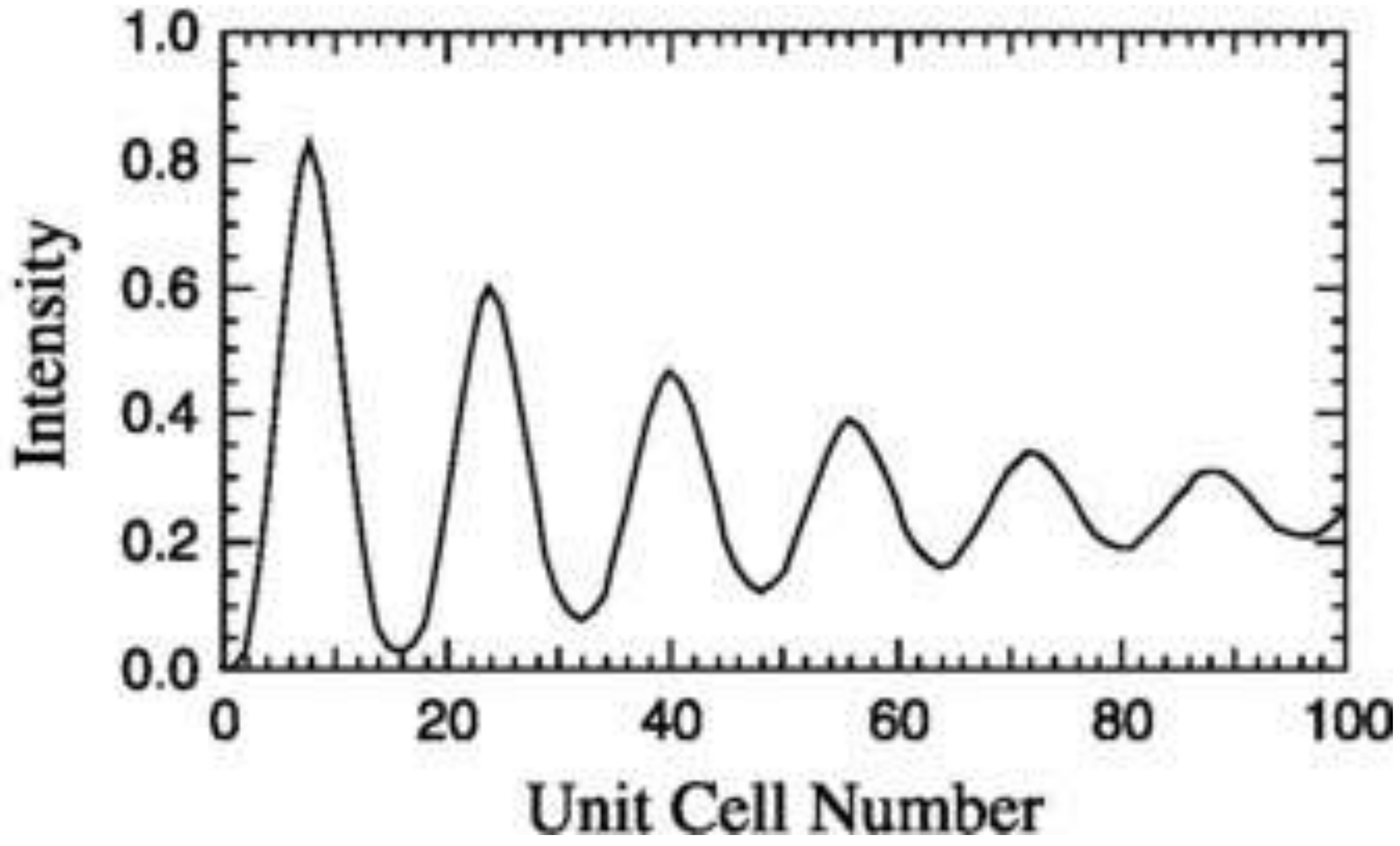


Thickness fringes : kinematical approach including absorption

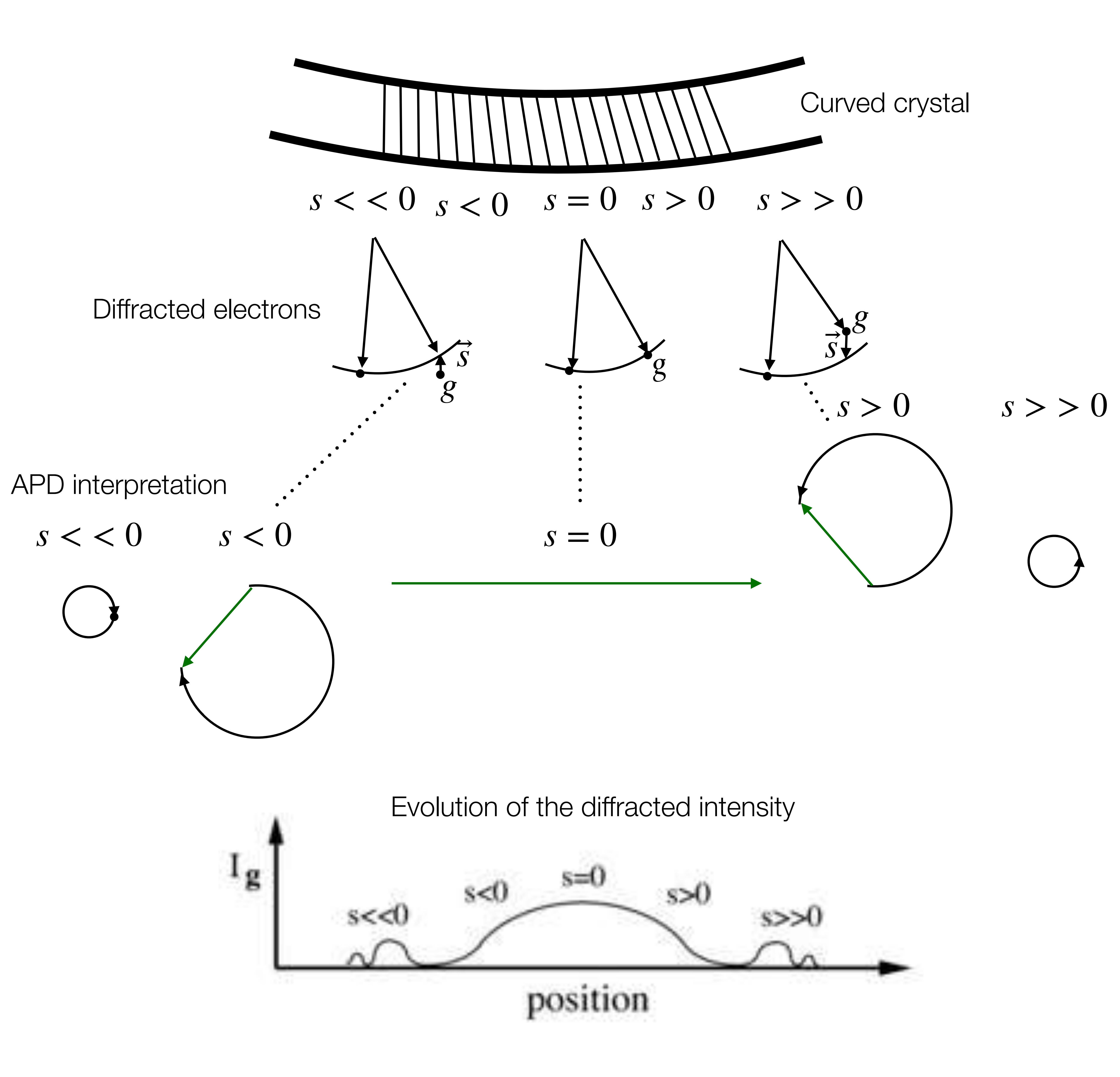


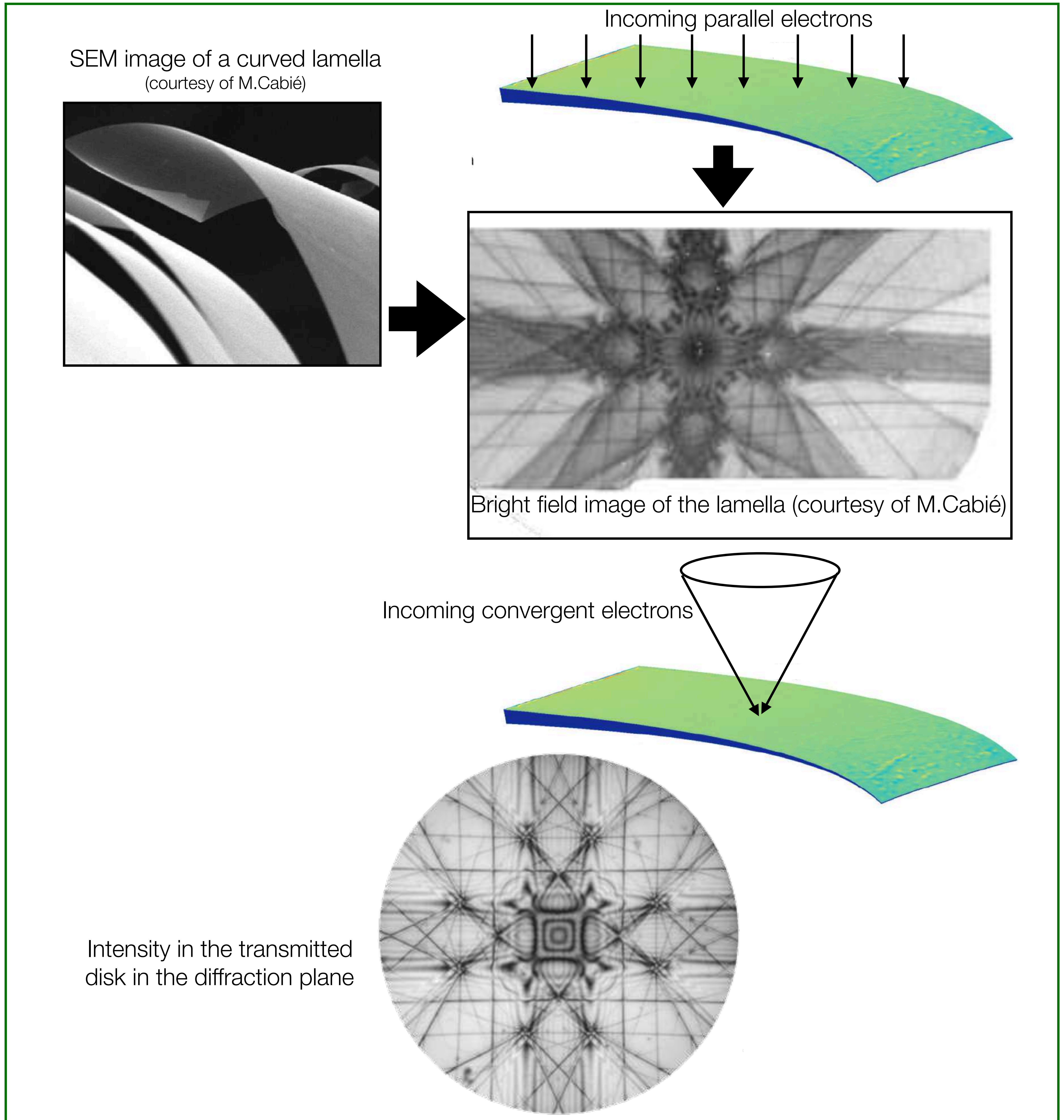

Sample : MgO cube





Bend contours : a kinematical approach





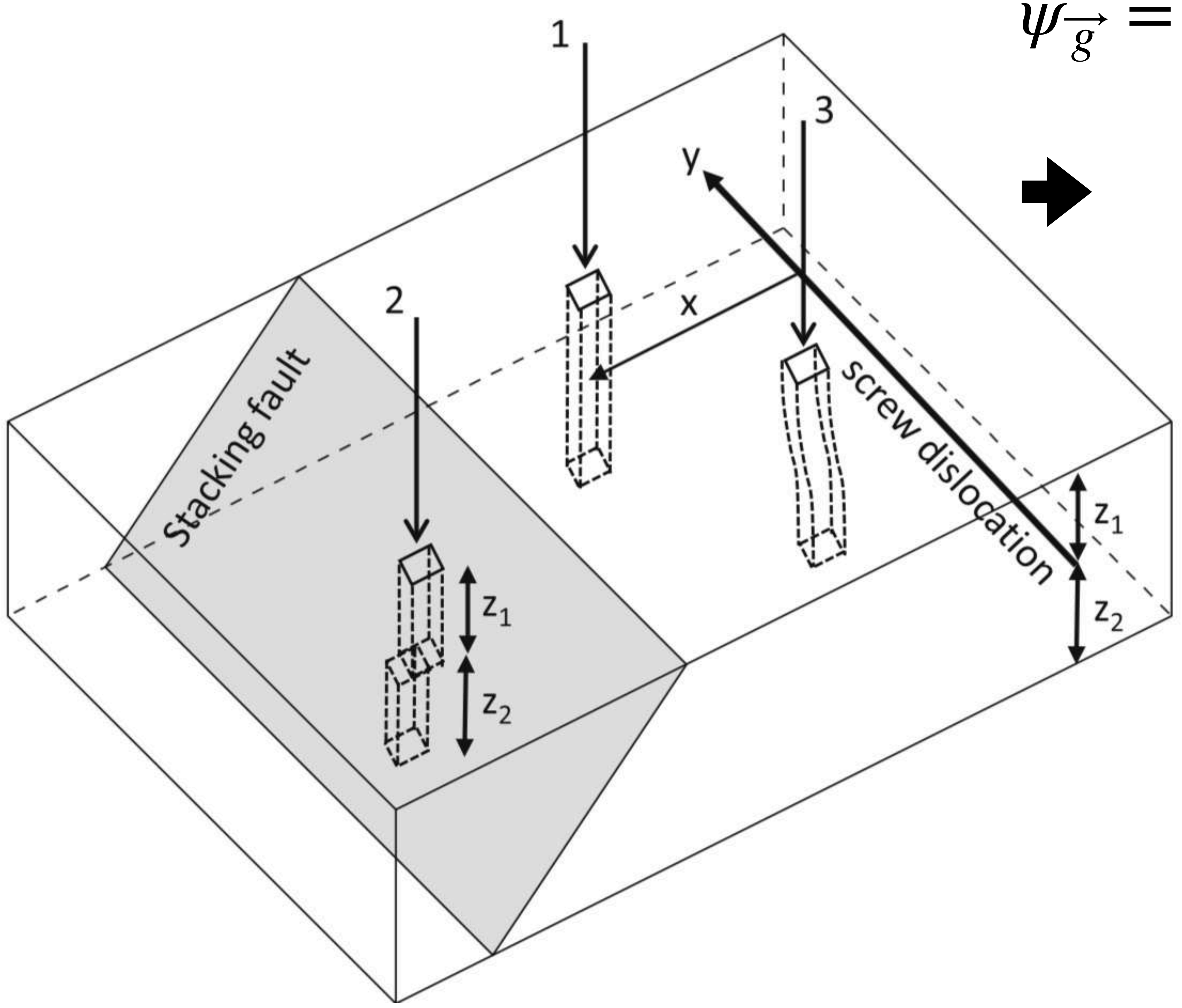
3

Diffraction theory of faulted crystal

Kinematical simulation of faulted crystal

- Let's consider an incoming plane wave $|\psi_0| = 1$ on a crystal define by a thickness t
- In the kinematical approximation, after Huygens-Fresnel, the diffracted wave will be given by :

$$\psi_{\vec{g}} = \frac{i\pi}{\xi_{\vec{g}}} \int_0^t e^{-2\pi i(\vec{g}+\vec{s}).\vec{r}'} dz$$



- Consider now any crystal defect which shift the atomic sites by a fault vector

$$\vec{r}' \rightarrow \vec{r} + \overrightarrow{R}(z)$$

Because of column approximation : $\vec{s}\,.\,\vec{r} = sz$ and $\vec{s}\,.\,\overrightarrow{R} \approx 0$

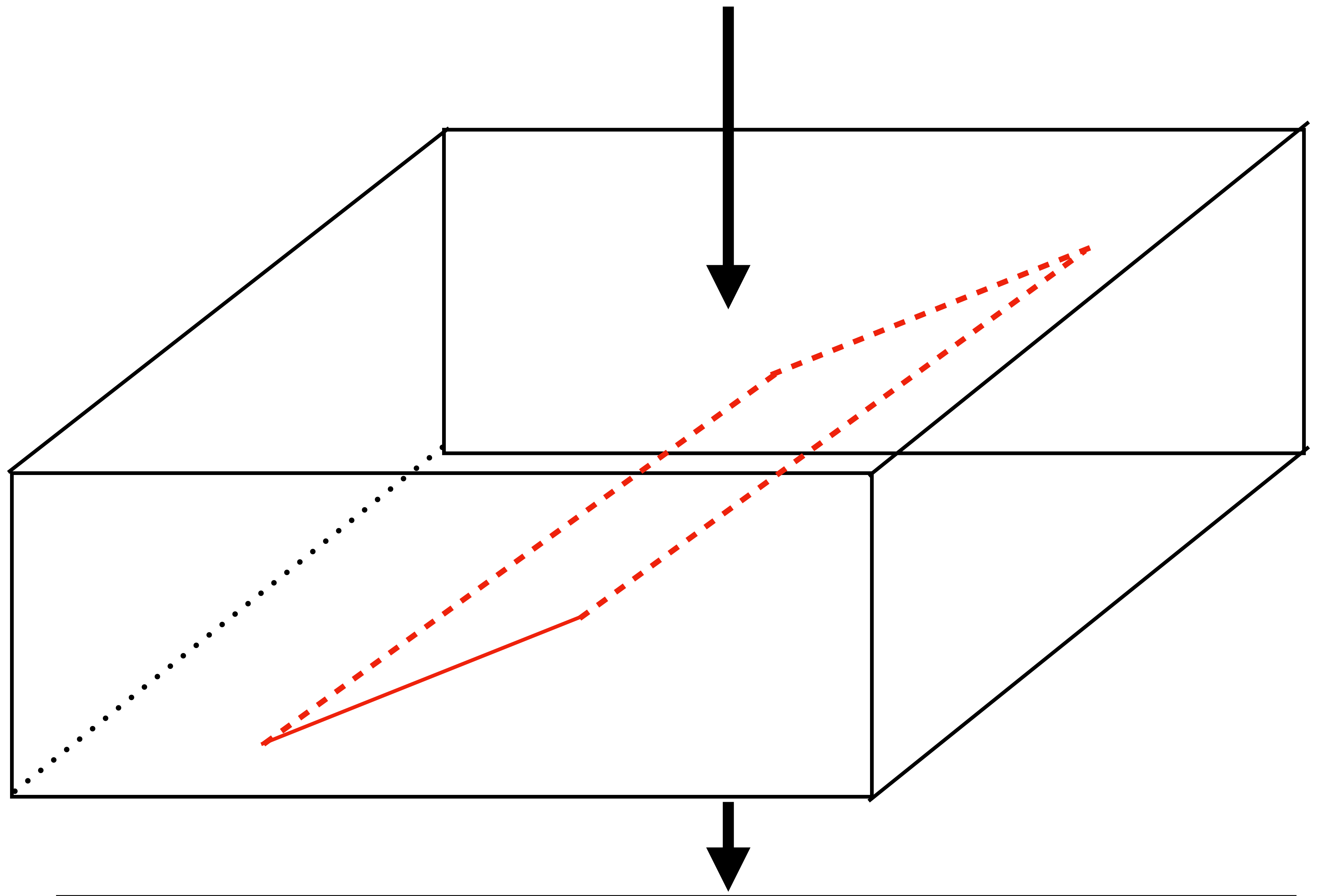
$$\psi_{\vec{g}} = \frac{i\pi}{\xi_{\vec{g}}} \int_0^t e^{-2\pi i(sz+\vec{g}.\overrightarrow{R(z)})} dz$$

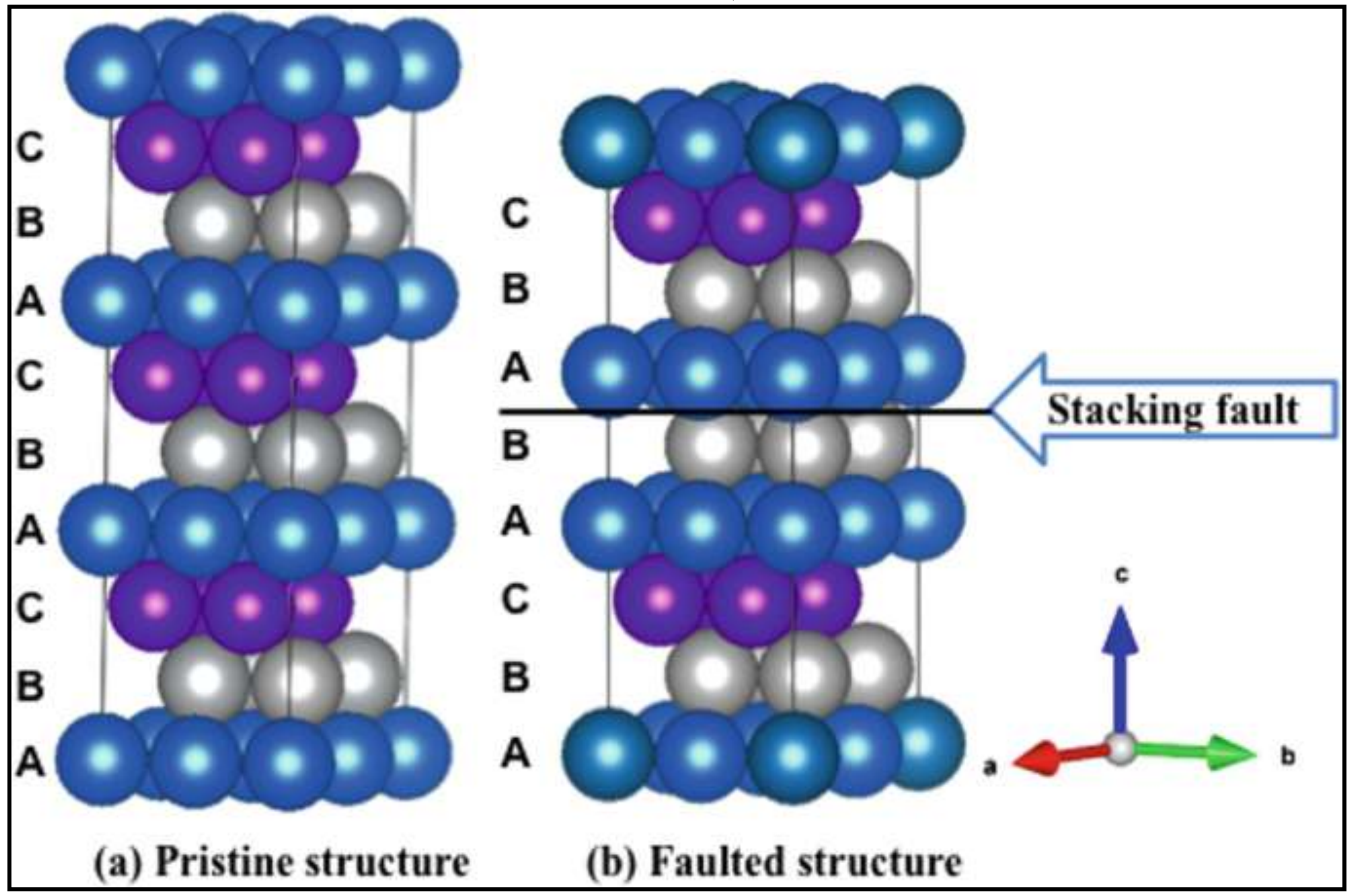
- Usually we define the phase term : $\alpha(z) = 2\pi \vec{g}\,.\,\overrightarrow{R}(z)$

$$= \frac{i\pi}{\xi_{\vec{g}}} \int_0^t e^{-2\pi isz} e^{-2\pi i\vec{g}.\overrightarrow{R(z)}} dz$$

$$\Psi_{\vec{g}} = \frac{i\pi}{\xi_{\vec{g}}} \int_0^t e^{-2\pi isz} e^{-i\alpha(z)} dz$$

Application to stacking fault





(a) Pristine structure (b) Faulted structure

In FCC for instance a stacking fault moves :

- B layer in the C position by applying the displacement vector :
$$\vec{R}(z) = \frac{a\{112\}}{6}$$
- The stacking becomes : $ABCA\,|\,CABC$

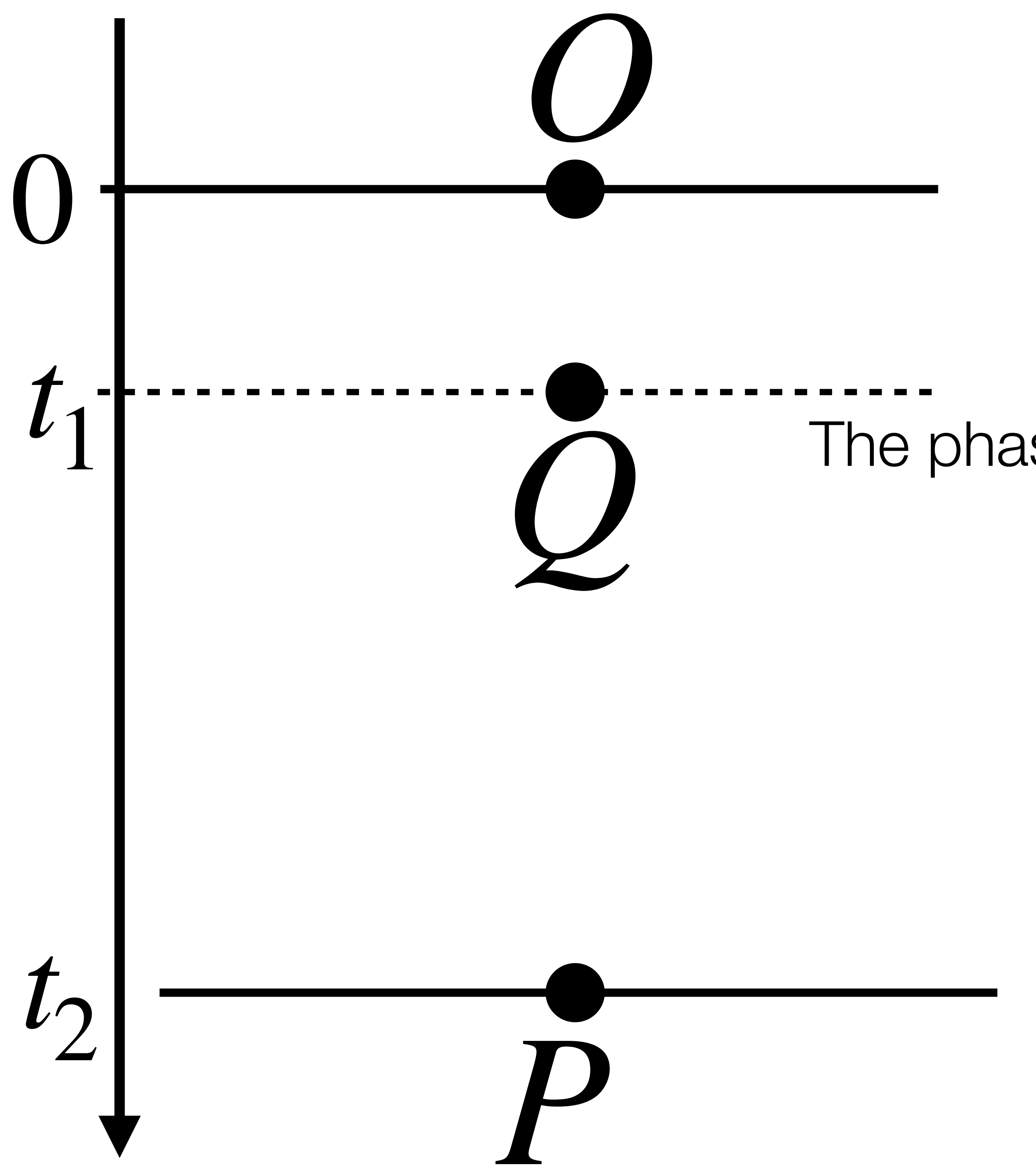
$$2\pi\vec{g}\,.\,\vec{R}(z) = 0 \Leftrightarrow -\frac{t}{2} \leq z \leq z_1$$
$$= \alpha \Leftrightarrow z_1 \leq z \leq \frac{t}{2}$$

Estimation of stacking fault diffraction contrast using APD

- Let's go back to the kinematical expression of the diffracted wave :

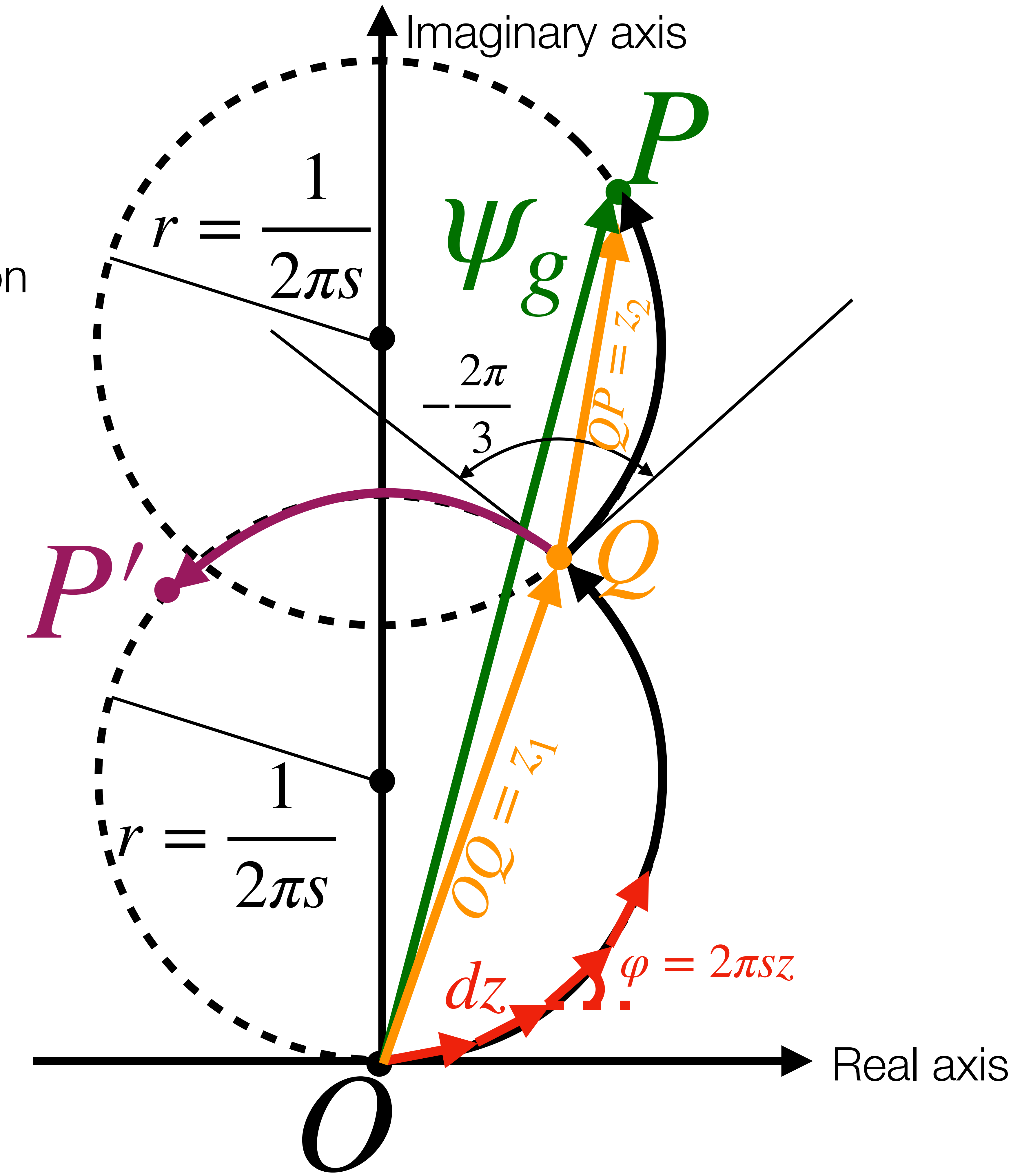
$$\psi_{\vec{g}} = \frac{i\pi}{\xi_{\vec{g}}}\int_0^{t_1} e^{-2\pi i s z}dz + \frac{i\pi}{\xi_{\vec{g}}}e^{-i\alpha}\int_{t_1}^{t_2} e^{-2\pi i s z}dz$$

The stacking fault is horizontal and located at $t = t_1$



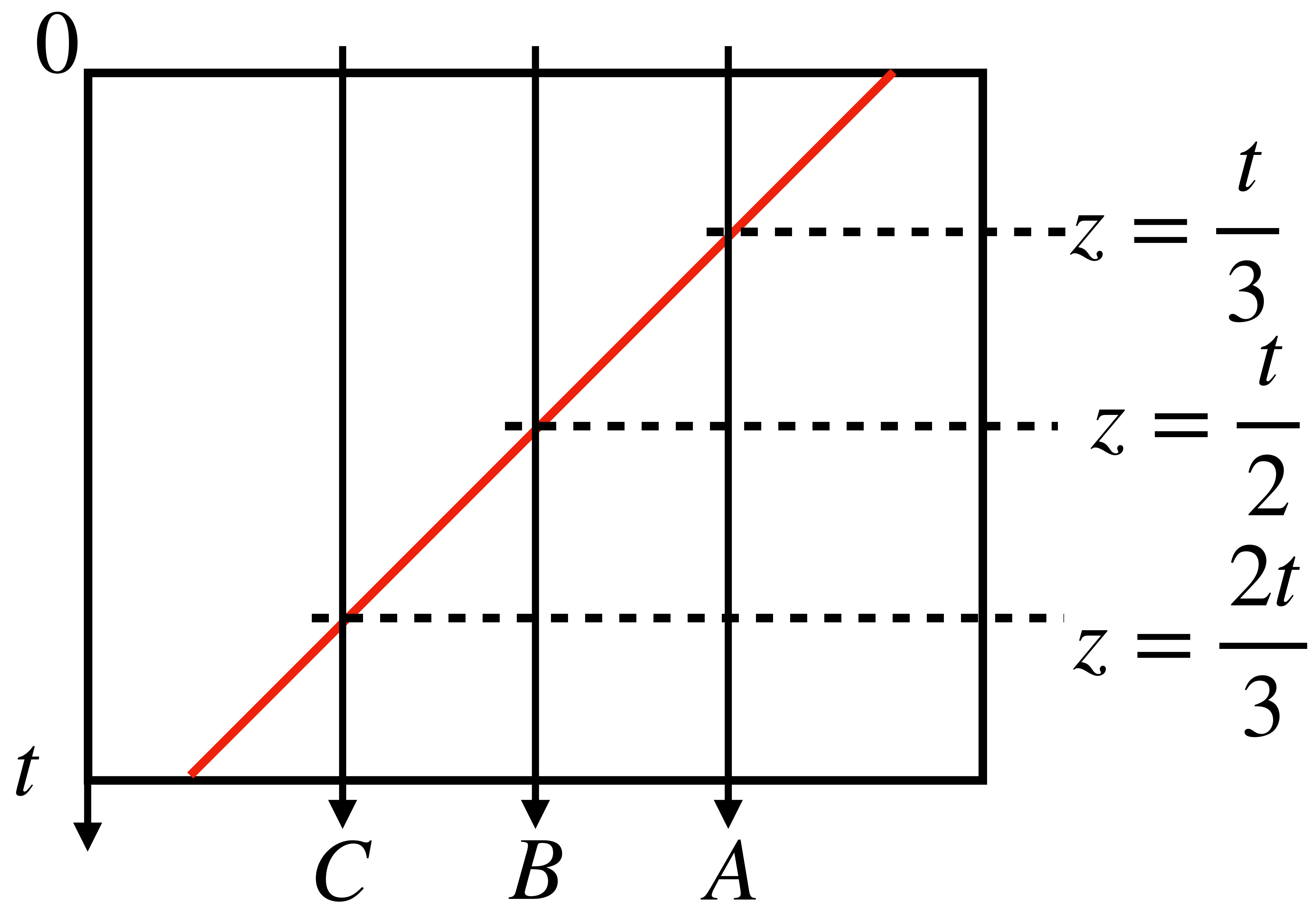
The phase shift is $\alpha = -\frac{2\pi}{3}$

APD interprétation



Estimation of stacking fault diffraction contrast using APD : exercice

Consider sample thickness $t = 30$nm, with inclined stacking fault with SF vector $\overrightarrow{R} = \frac{1}{4}[110]$



DF image with $\overrightarrow{g} = 200$ and $s = 0.05$nm^{-1}

Use amplitude phase diagram (APD) to determine ratio of DF image intensities for the columns.
Assume kinematical approximation valid.

Possible answers:

$1 \rightarrow I(A) > I(B) > I(C)$

$2 \rightarrow I(A) = I(C) > I(B)$

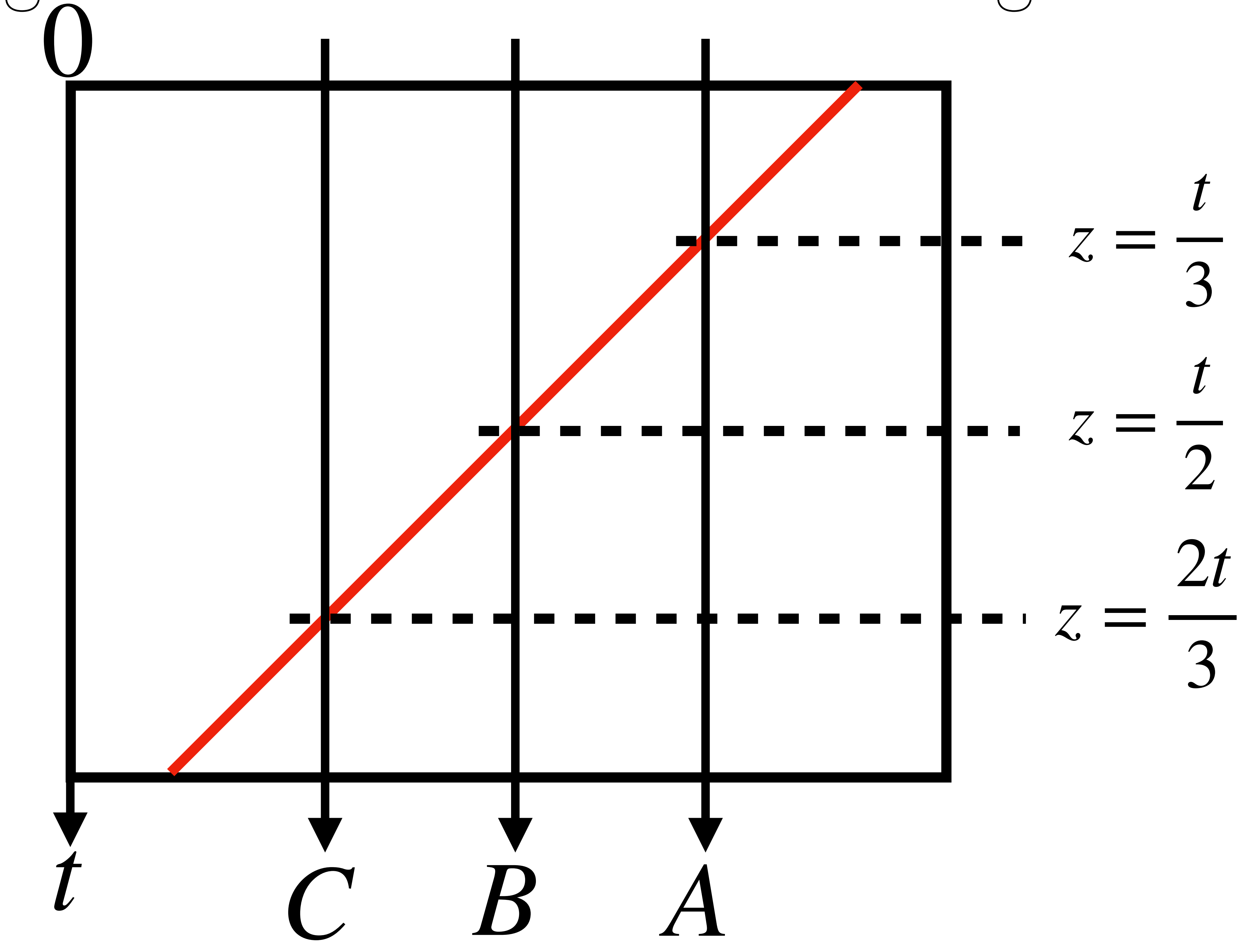
$3 \rightarrow I(A) = I(C) = I(B)$

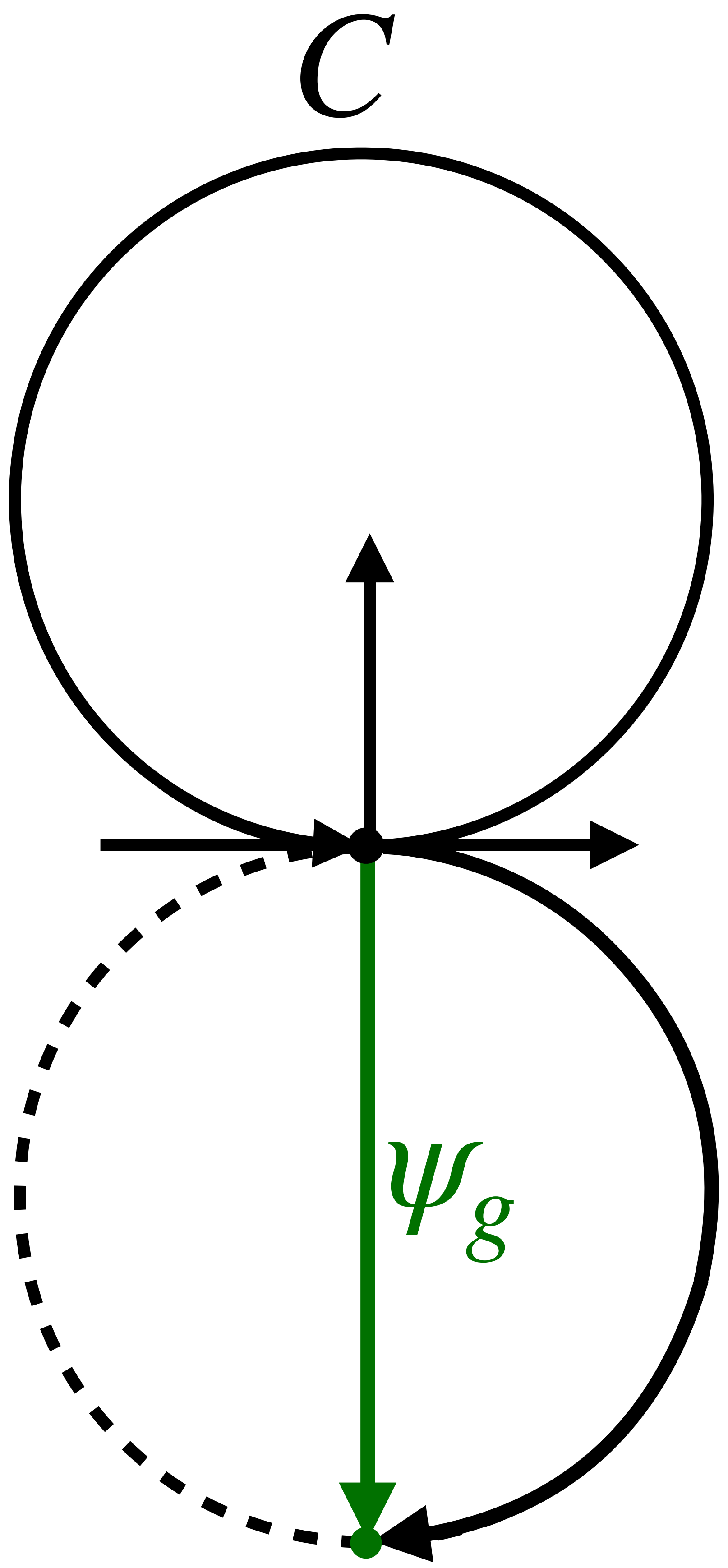
Hint: convert thickness change to fraction of circumference $= \frac{1}{s}$

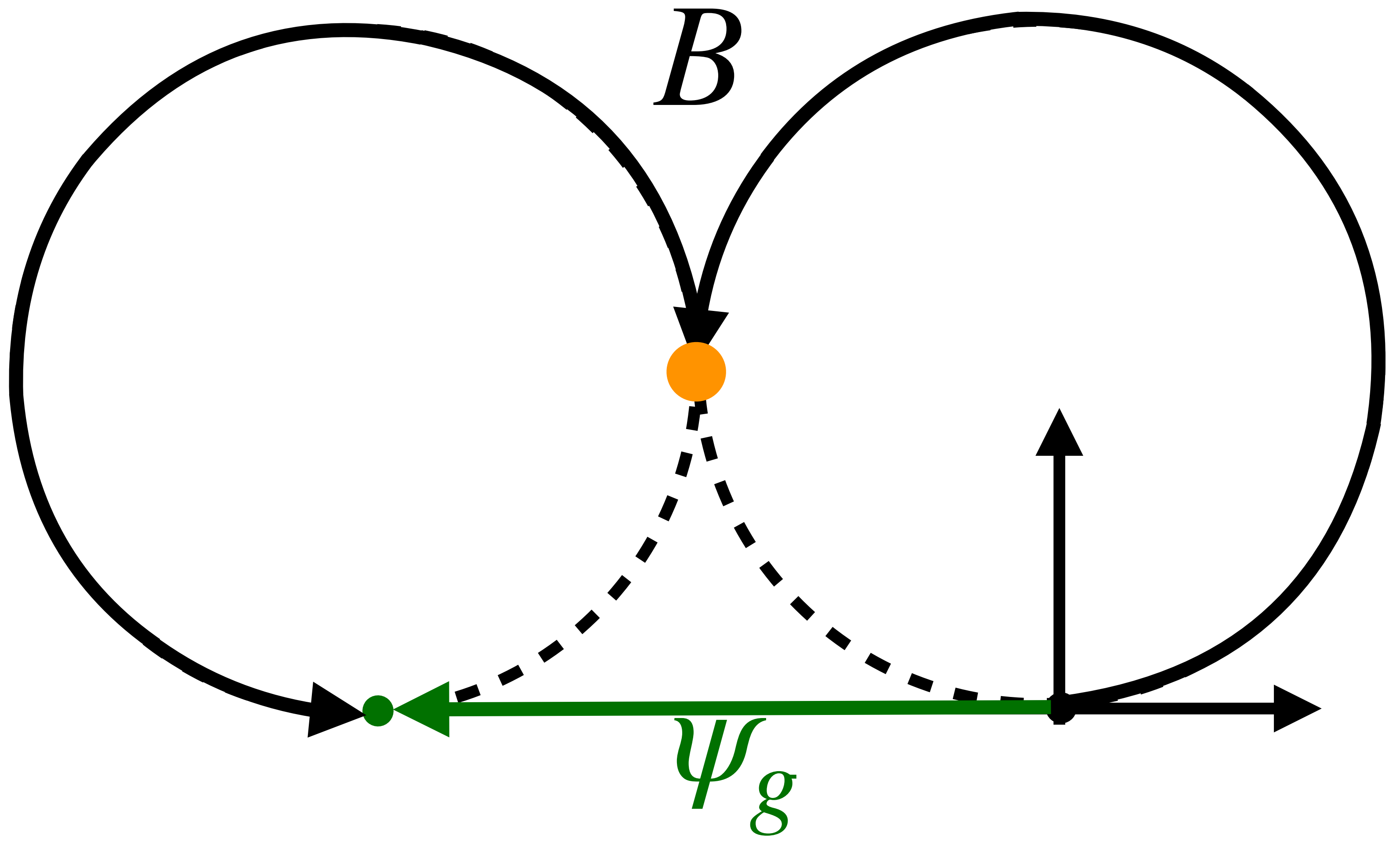
1.Radius : $\frac{1}{2\pi s}$

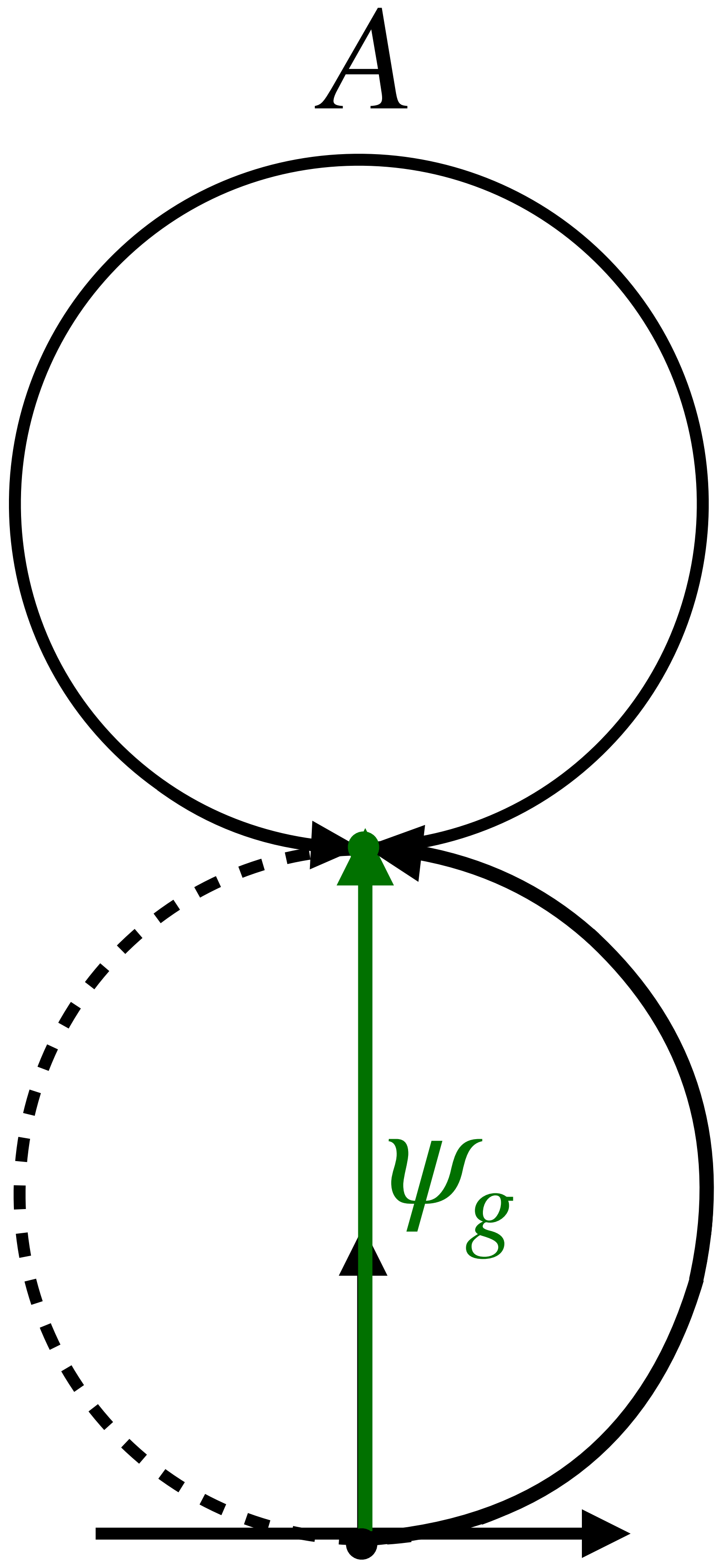
2. Circumference $= 2\pi\frac{1}{2\pi s} = \frac{1}{s} = 20\text{nm} \implies \frac{2}{3} \times t$

3. Phase shift $\alpha = 2\pi\,.\,\vec{g}\,.\,\vec{R} = \pi$





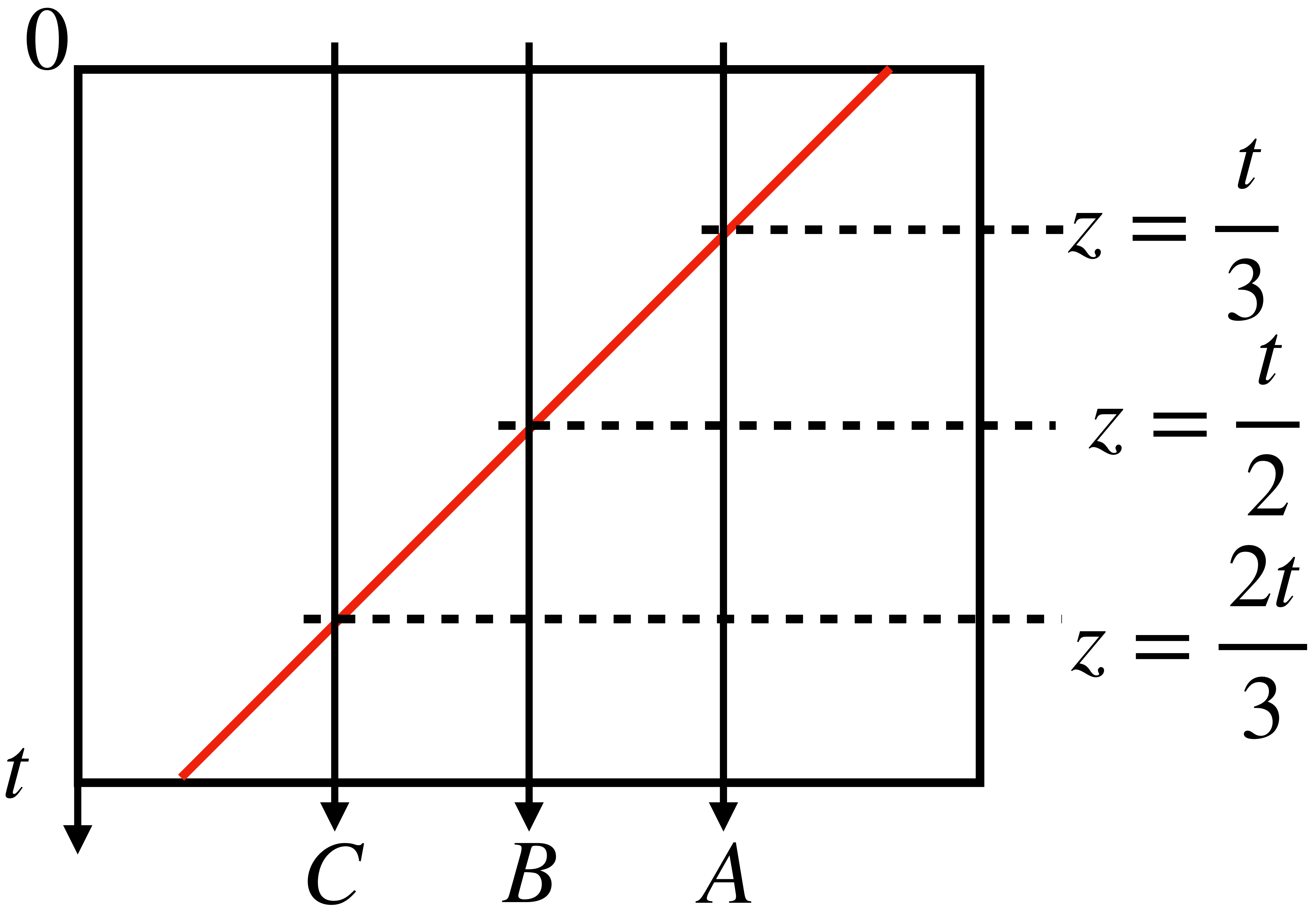




$\implies 2 \rightarrow I(A) = I(C) = I(B)$

Estimation of stacking fault diffraction contrast using APD : exercice

Consider sample thickness $t = 30$nm, with inclined stacking fault with SF vector $\overrightarrow{R} = \frac{1}{4}[110]$



DF image with $\overrightarrow{g} = -100$ and $s = 0.05$nm^{-1}

Possible answers:

$1 \rightarrow I(C) > I(A); I(B) = 0$

$2 \rightarrow I(A) = I(C); I(B) = 0$

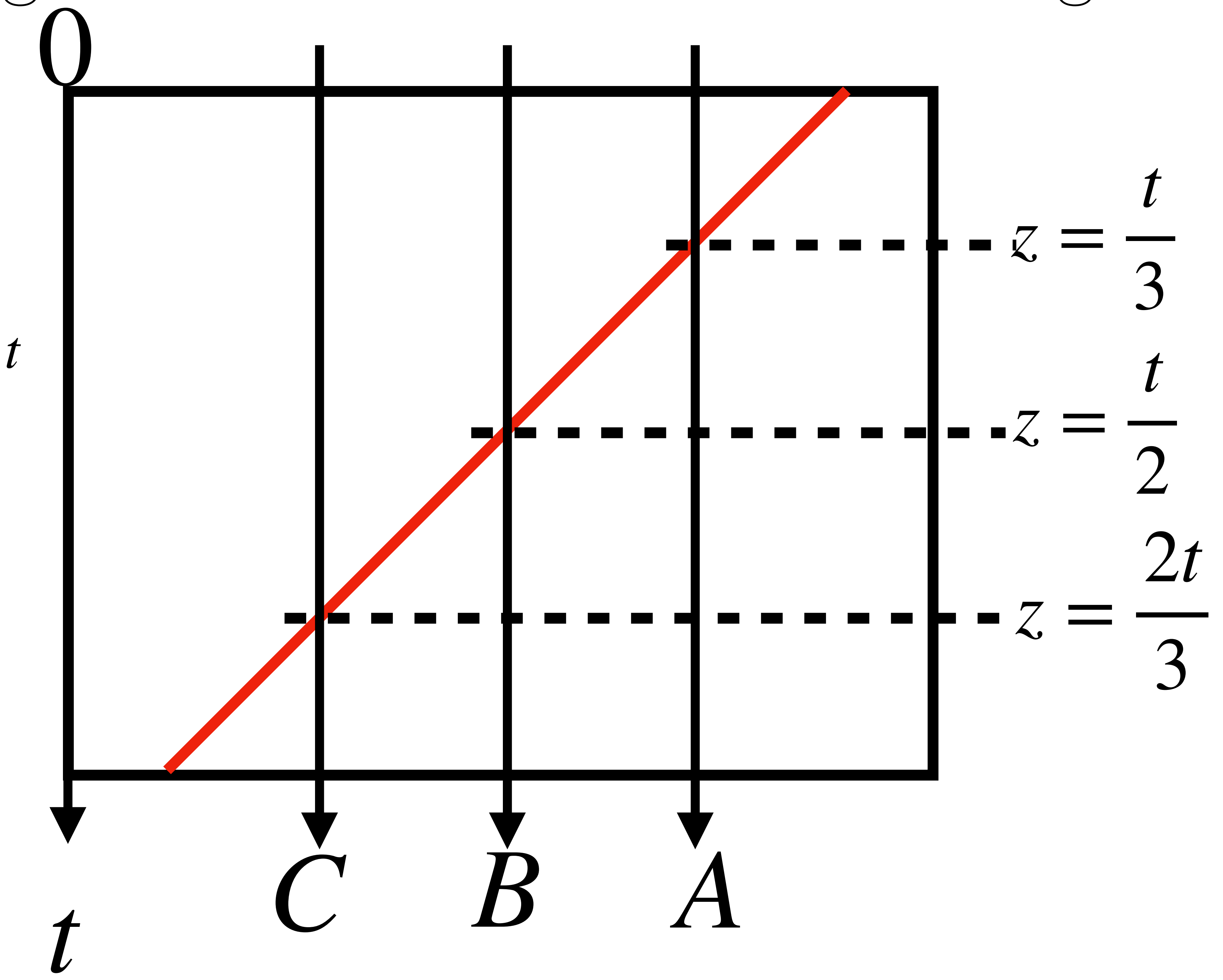
$3 \rightarrow I(C) > I(B); I(A) = 0$

Estimation of stacking fault diffraction contrast using APD : exercice

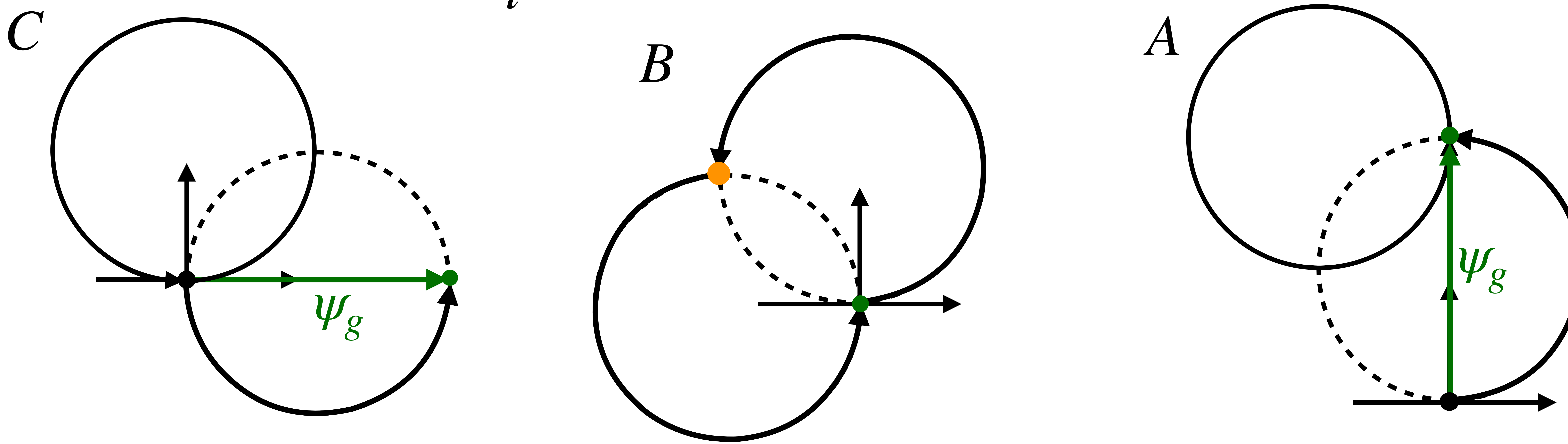
1. Radius $\frac{1}{2\pi s}$

2. Circumference $= 2\pi\frac{1}{2\pi s} = \frac{1}{s} = 20\text{nm} \implies \frac{2}{3} \times t$

3. Phase shift $\alpha = 2\pi \,.\, \vec{g} \,.\, \vec{R} = \frac{\pi}{2}$

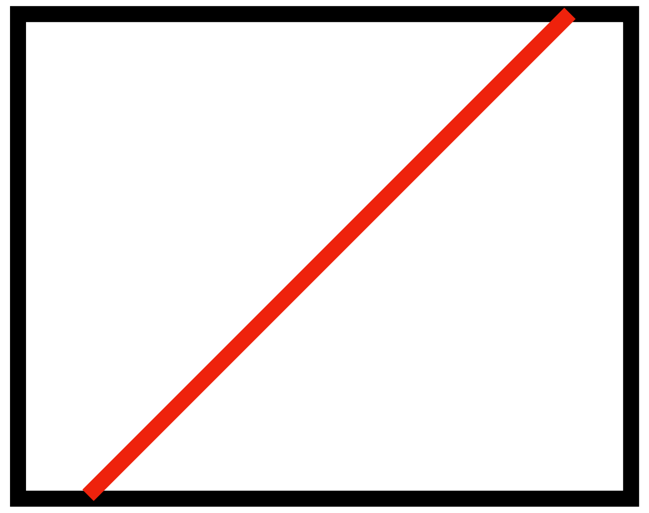


$\implies I(A) = I(C); I(B) = 0$

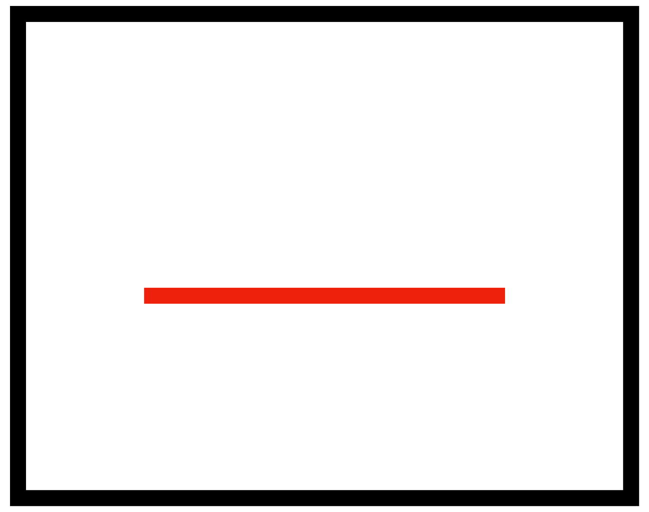


Estimation of stacking fault diffraction contrast using APD : real cases

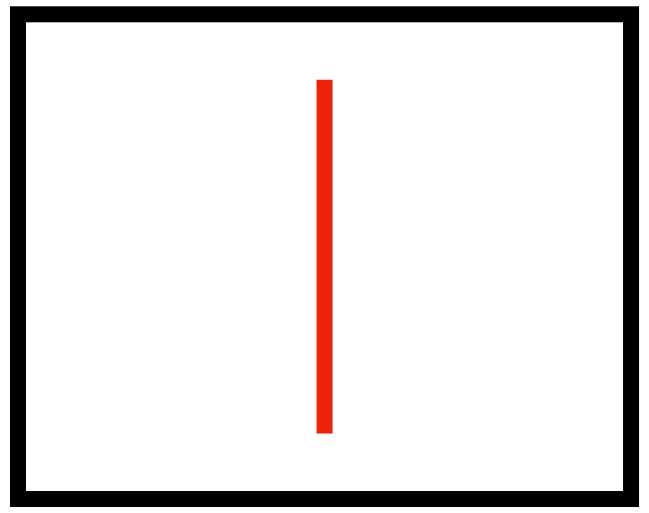
Various configuration :



Fringes

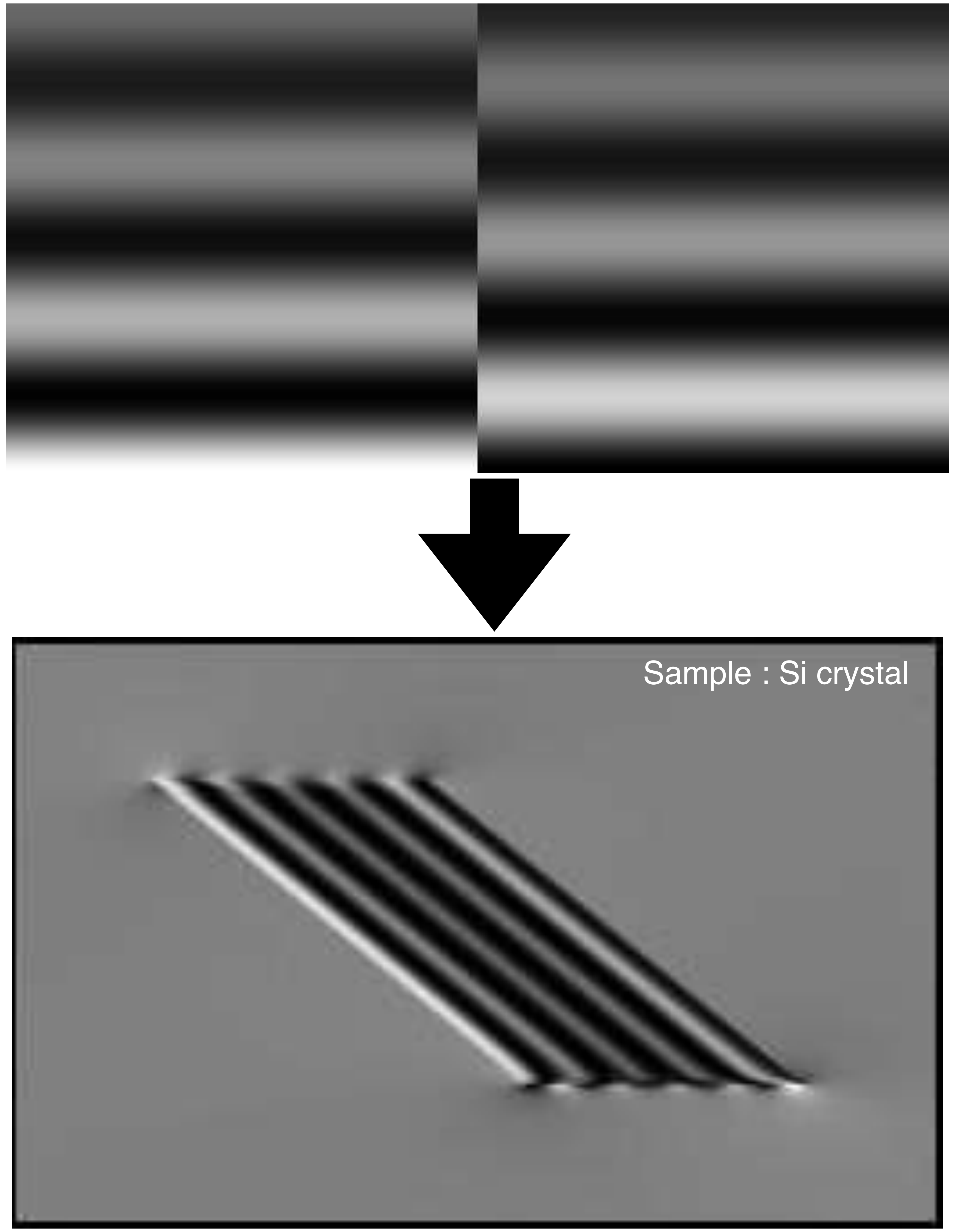


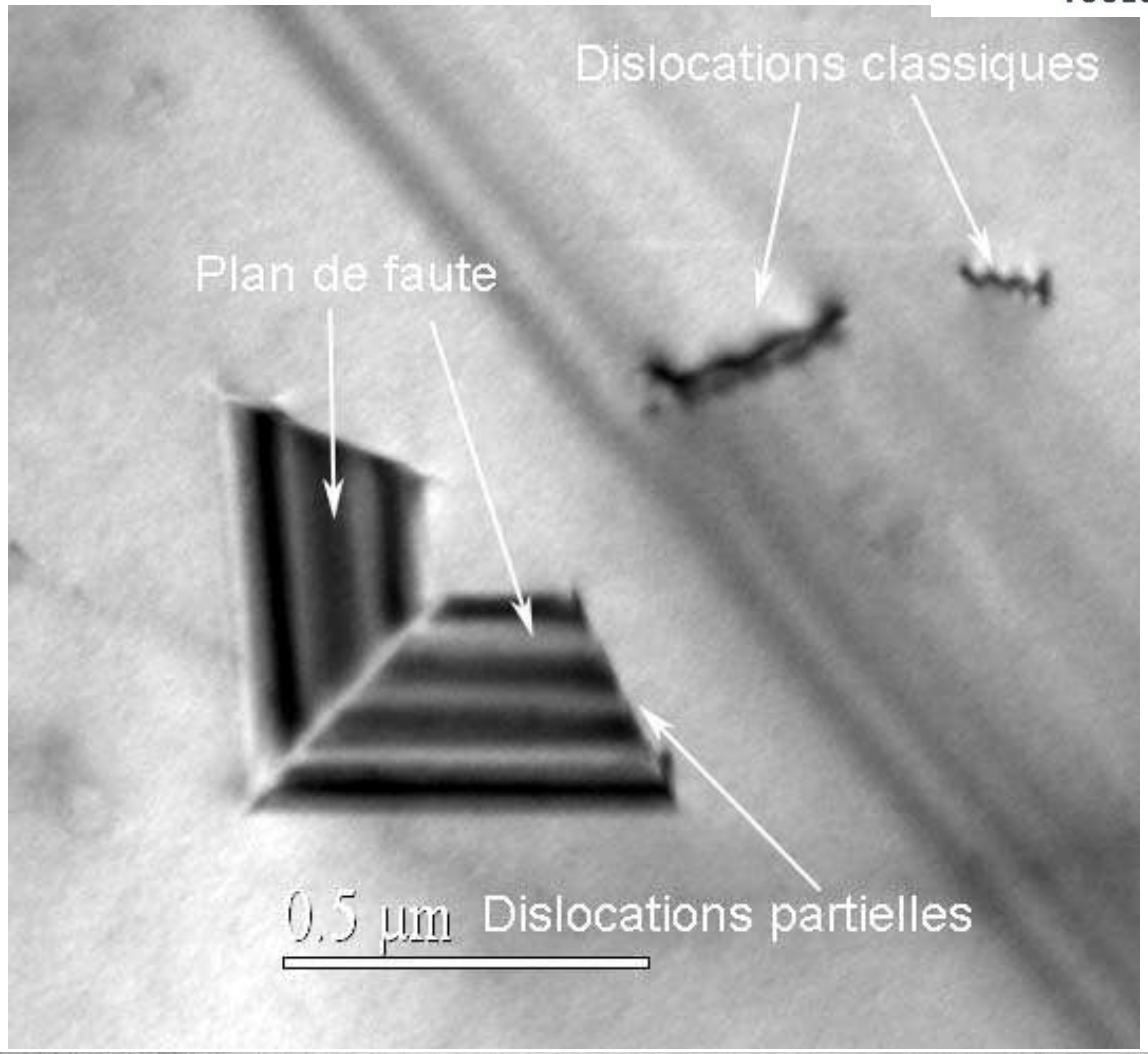
Constant contrast

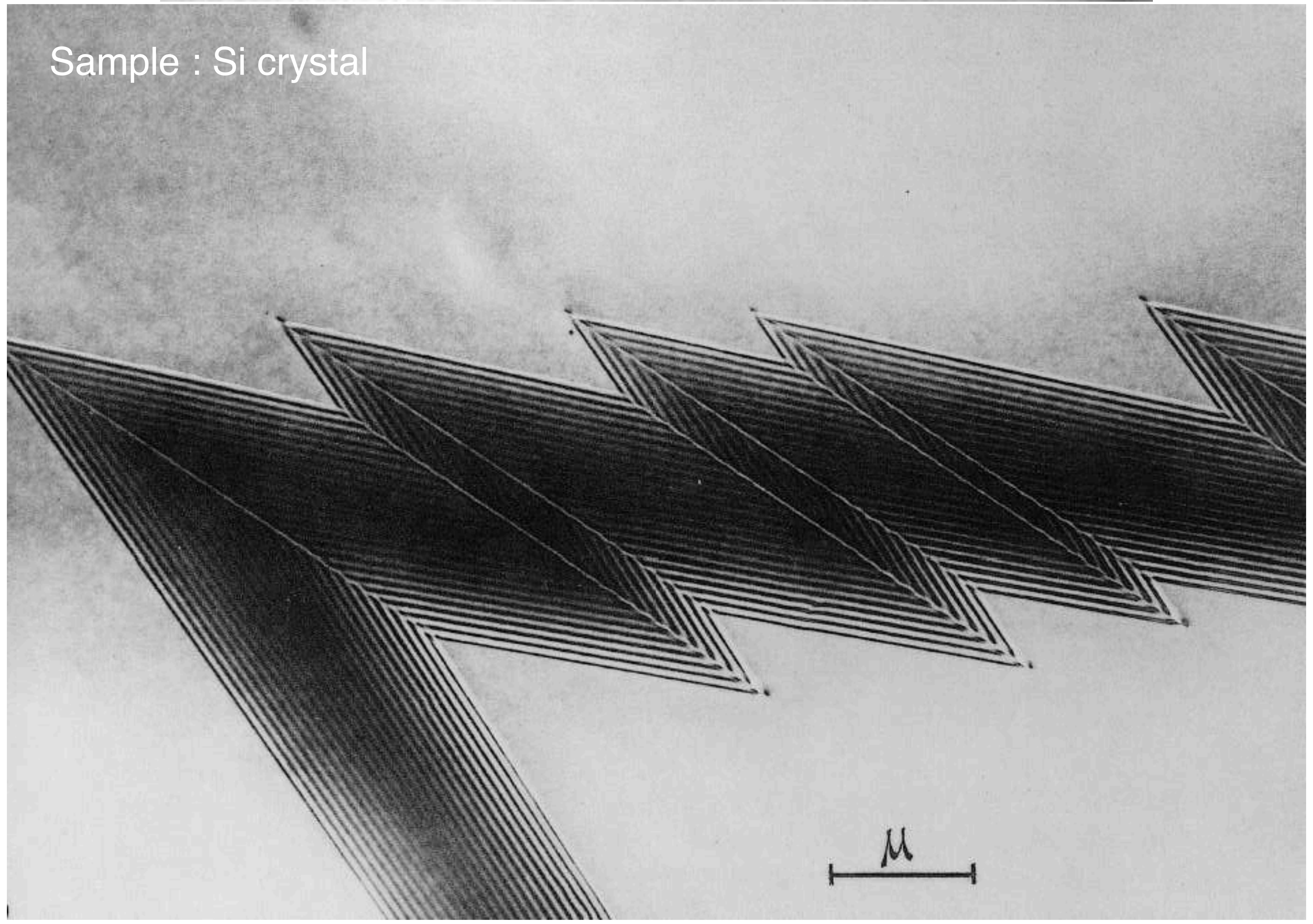


Invisible

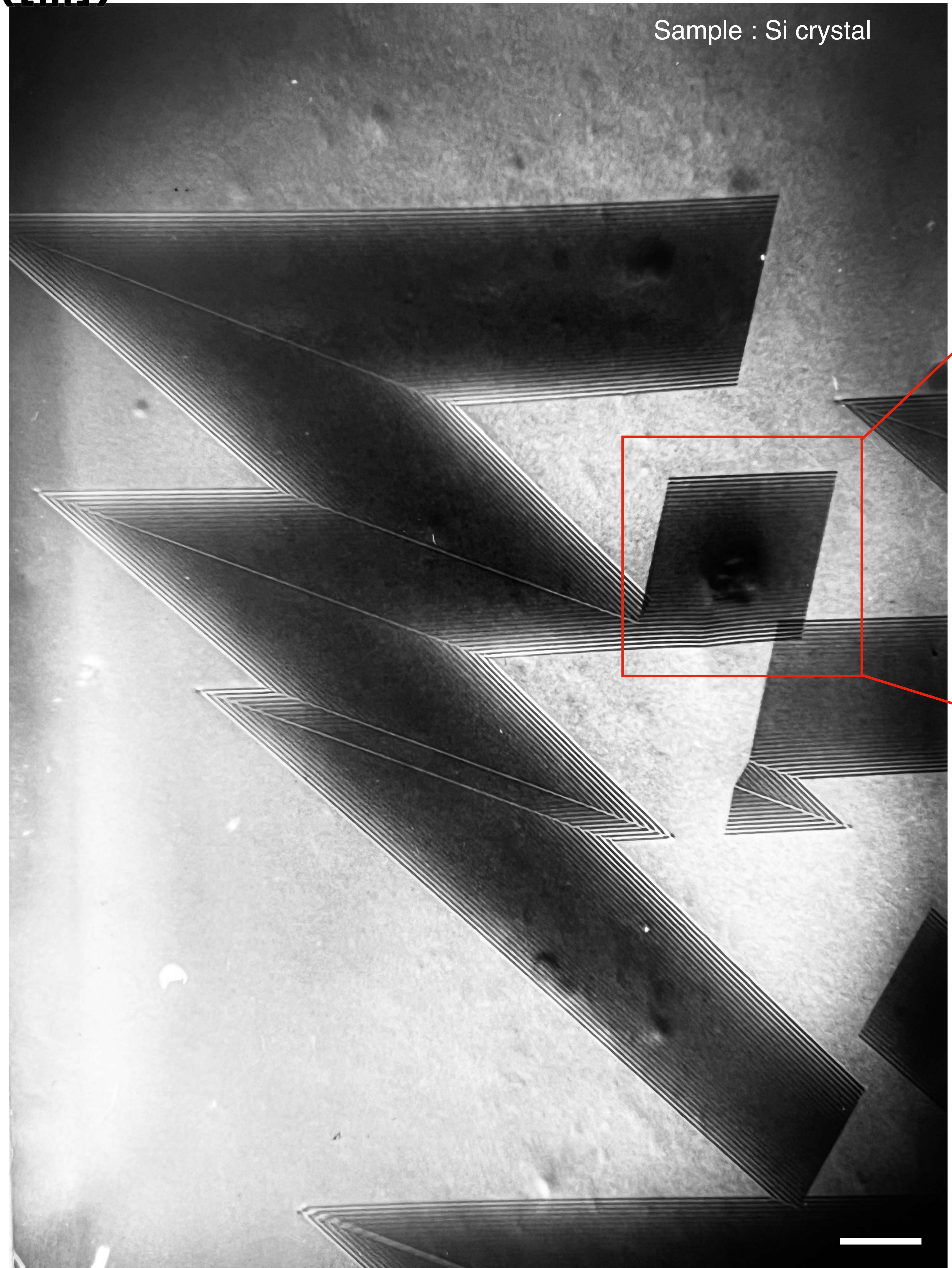
Kinematical simulation of an inclined stacking fault

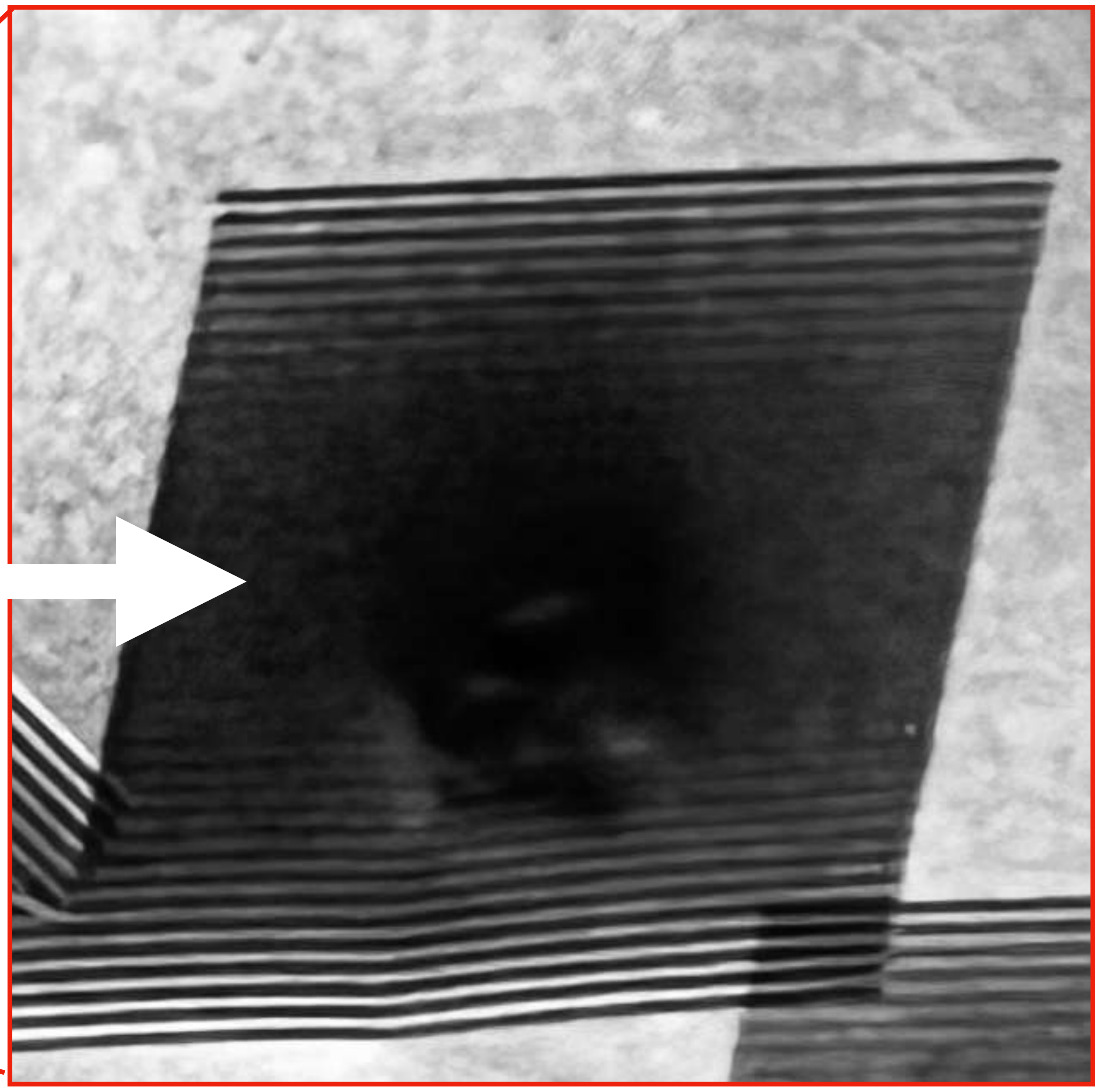


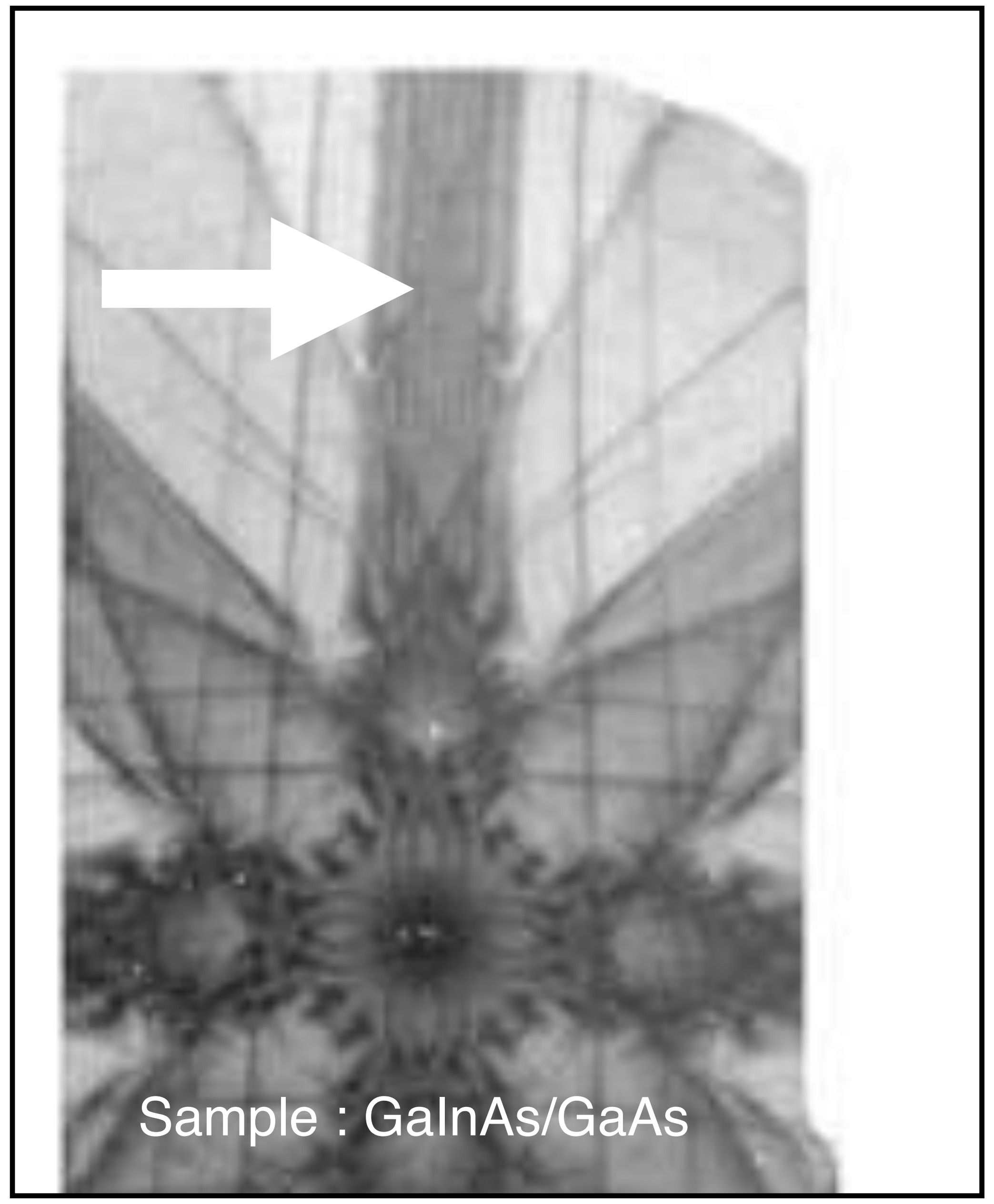




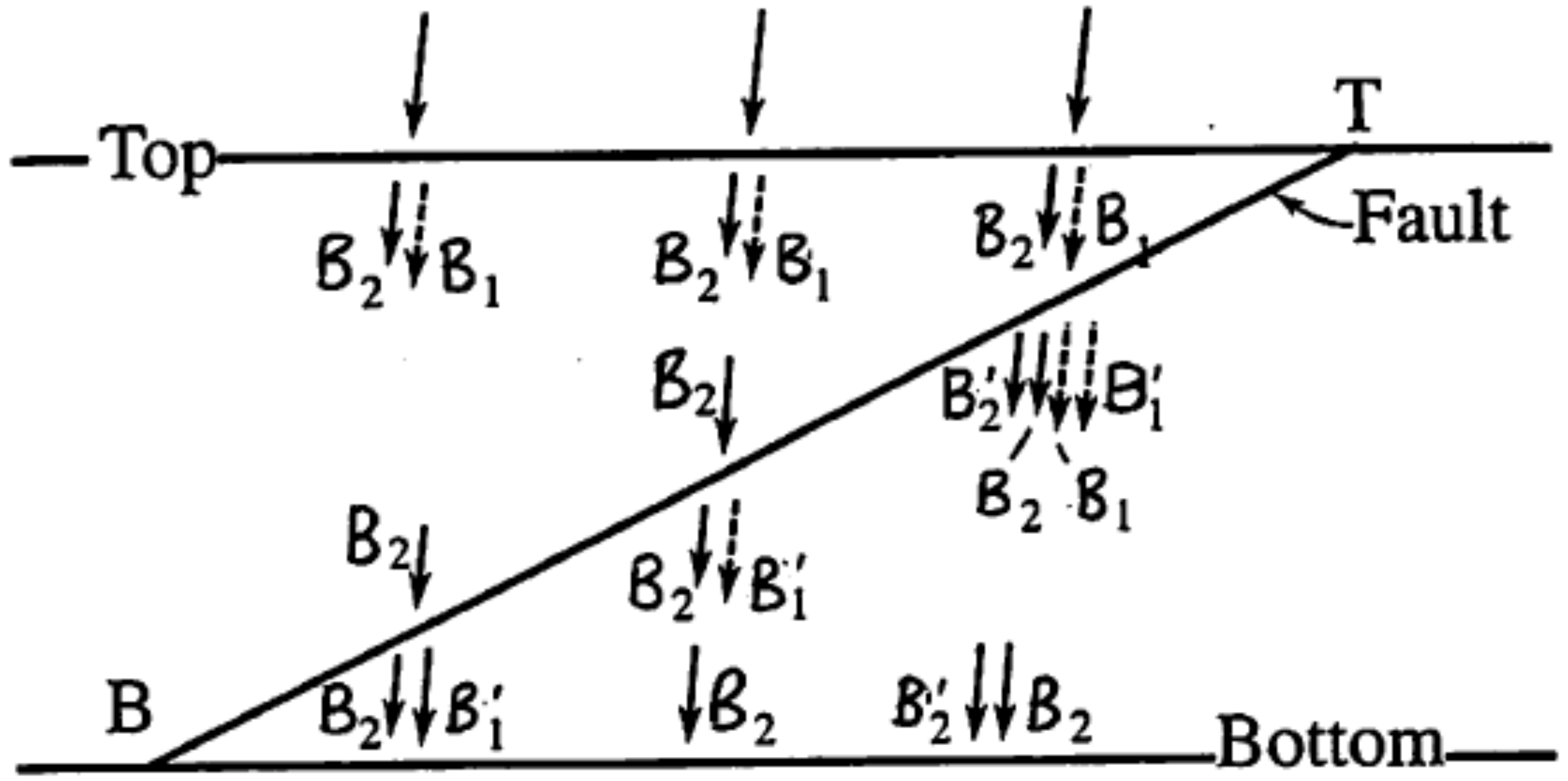
Kinematical vs dynamical theory in diffraction contrast







Interpreted as anomalous absorption of Bloch waves in dynamical 2 beams theory

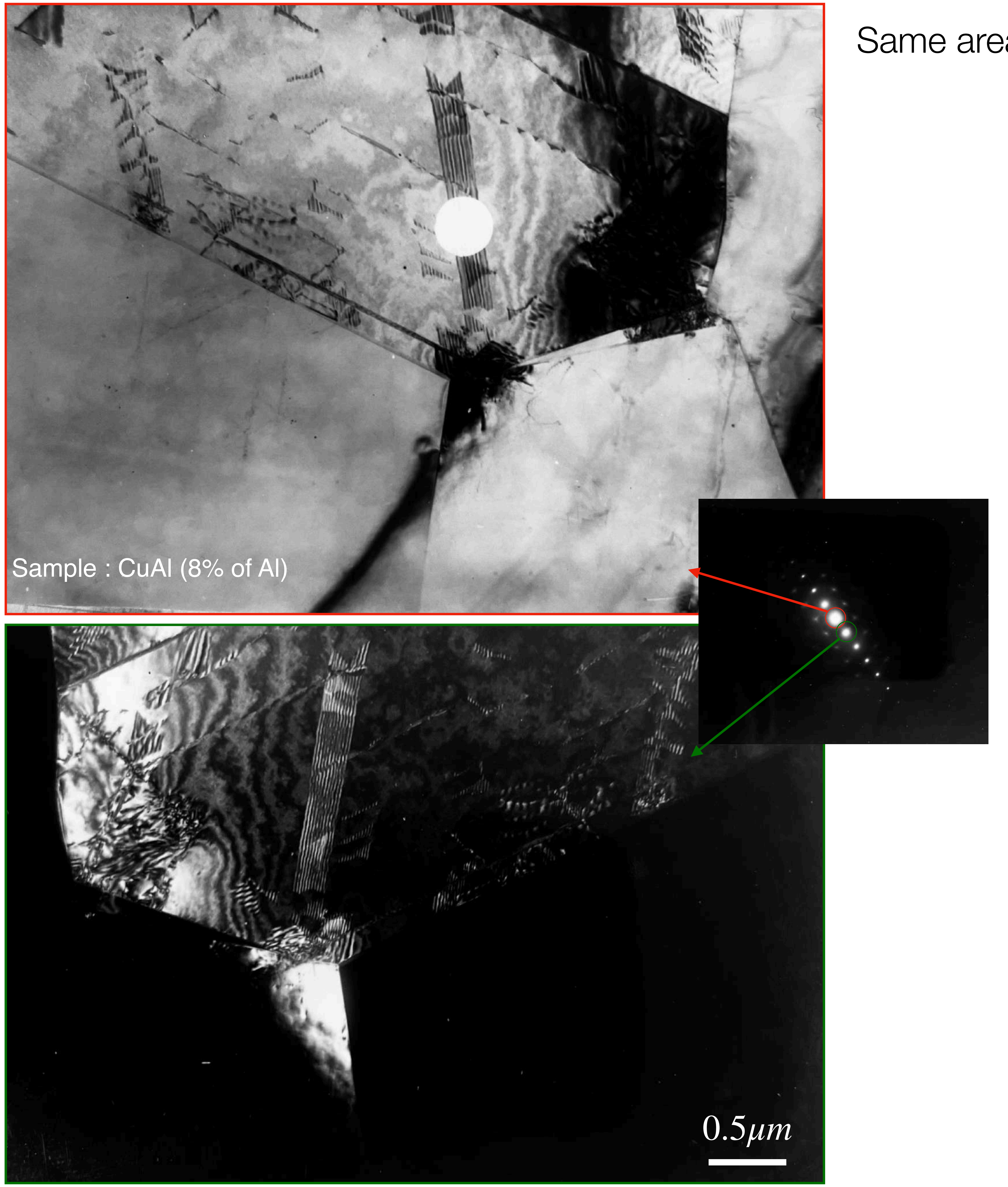


4

Diffraction contrast Imaging in faulted crystal

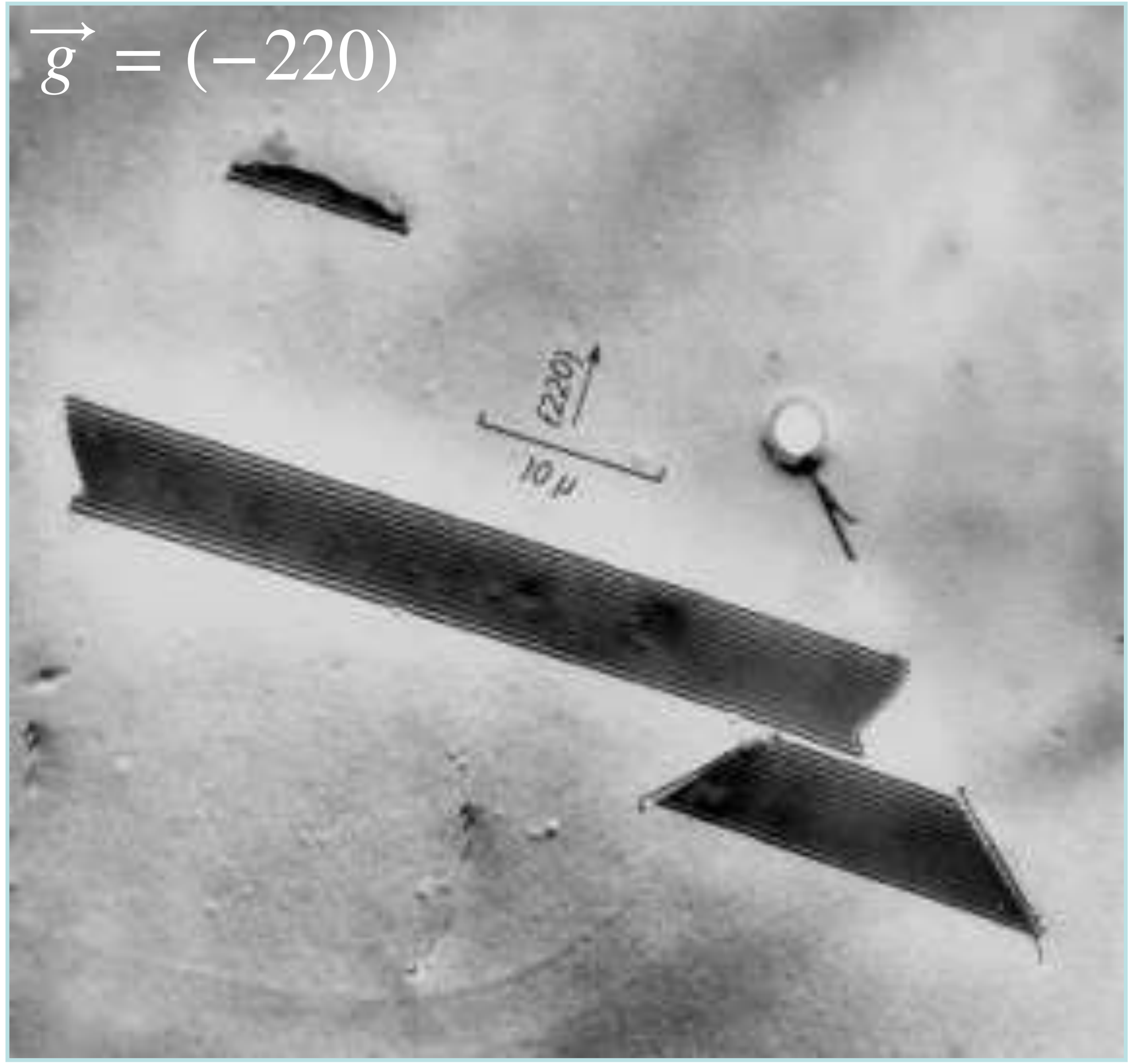
BF-DF imaging vs weak beam
Dislocations
Stacking fault
Antiphrase boundaries
Grain boundaries
etc.

Conventional observation of stacking fault

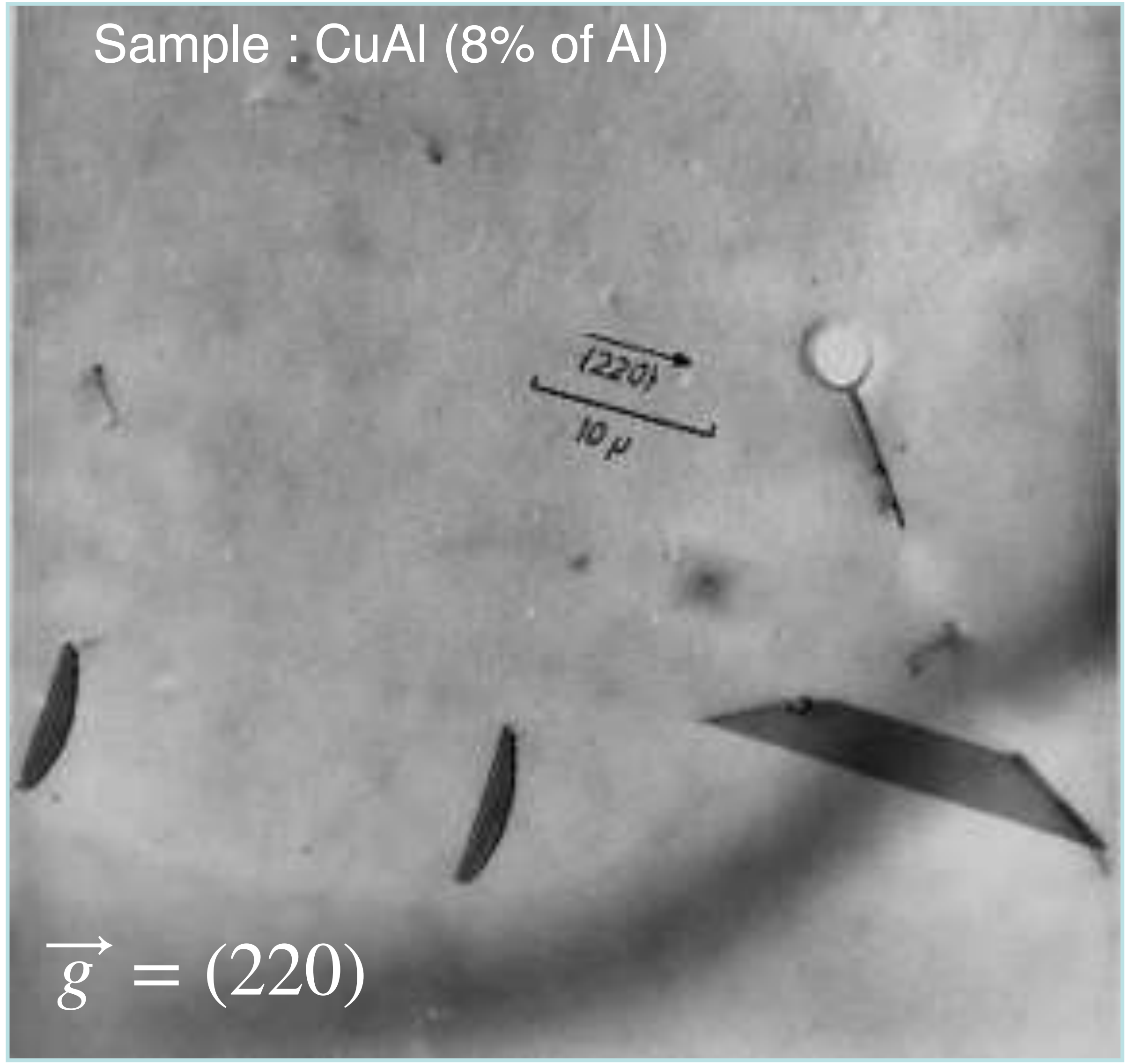


Same area of a Si sample imaged with two different diffraction vectors of the $\{220\}$ type

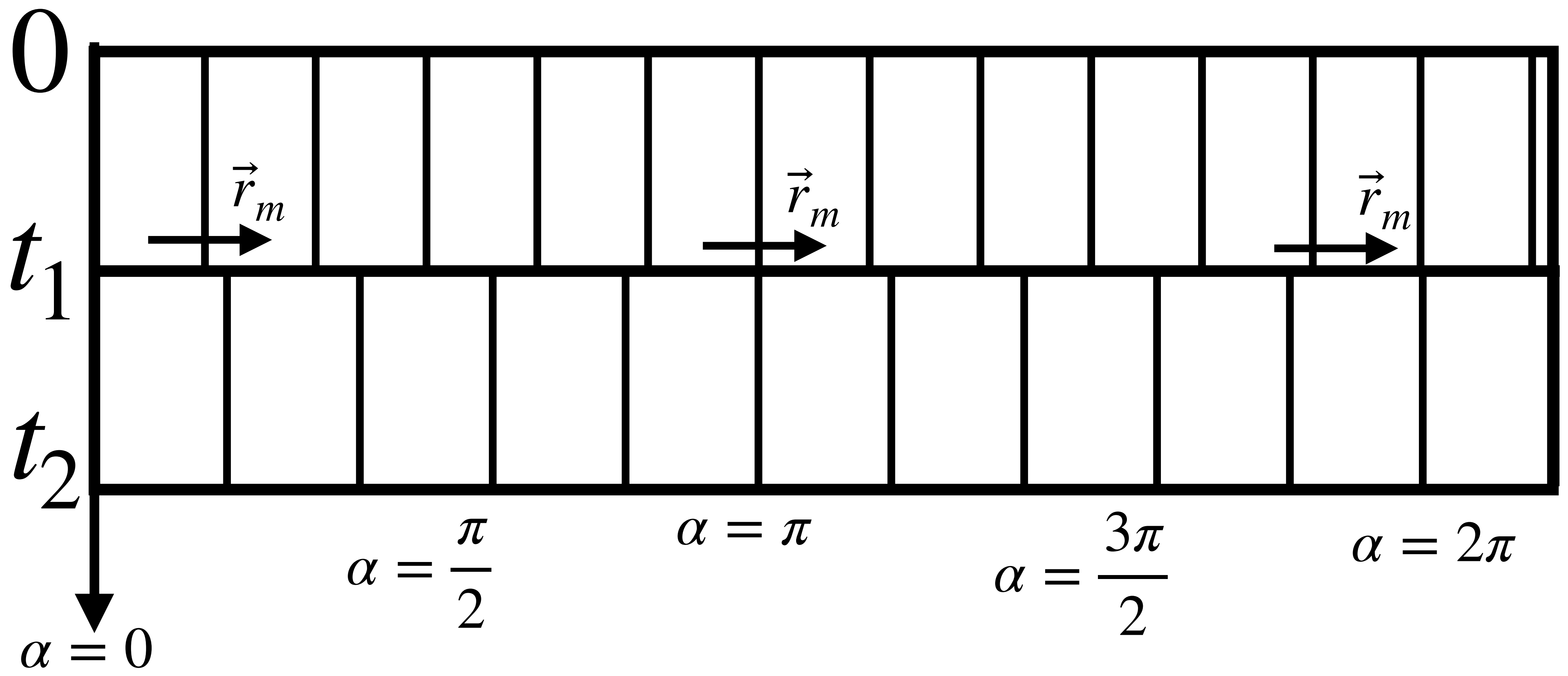
Phase shift $\alpha = 2\pi \,.\, \vec{g} \,.\, \vec{R} \neq 0$

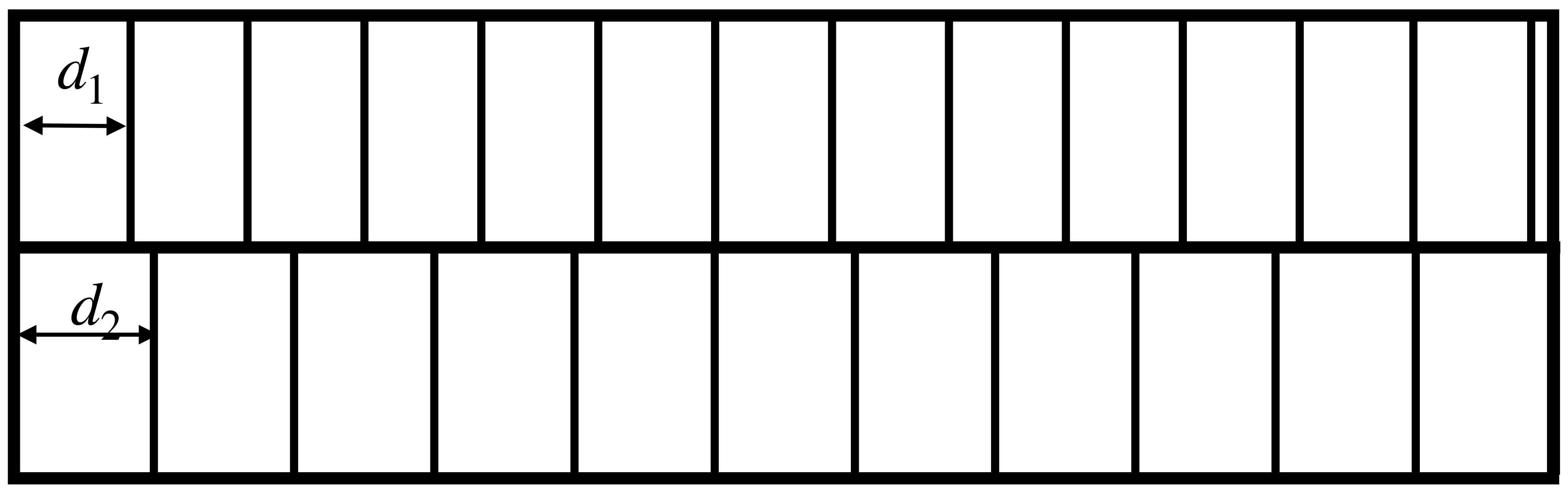


Phase shift $\alpha = 2\pi \,.\, \vec{g} \,.\, \vec{R} = 0$



Conventional observation of Moiré fringes

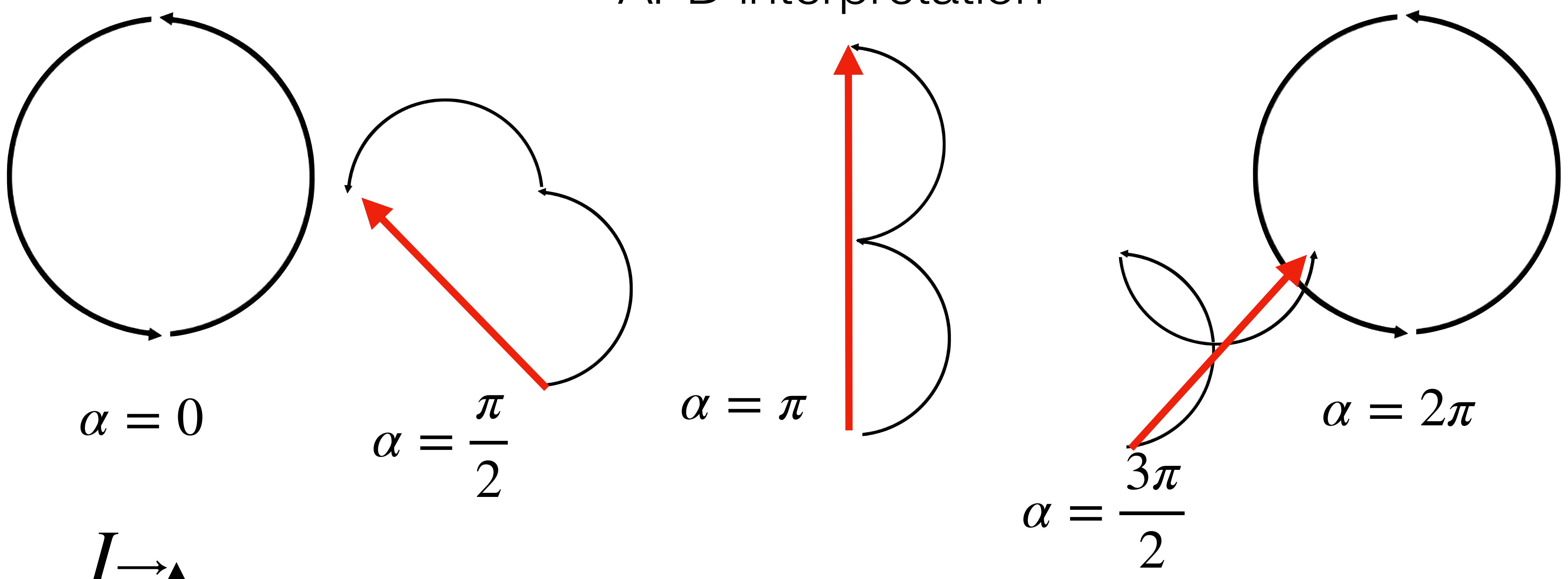




Phase shift : $\alpha = 2\pi\Delta\vec{g}\,.\,\vec{r}_m$

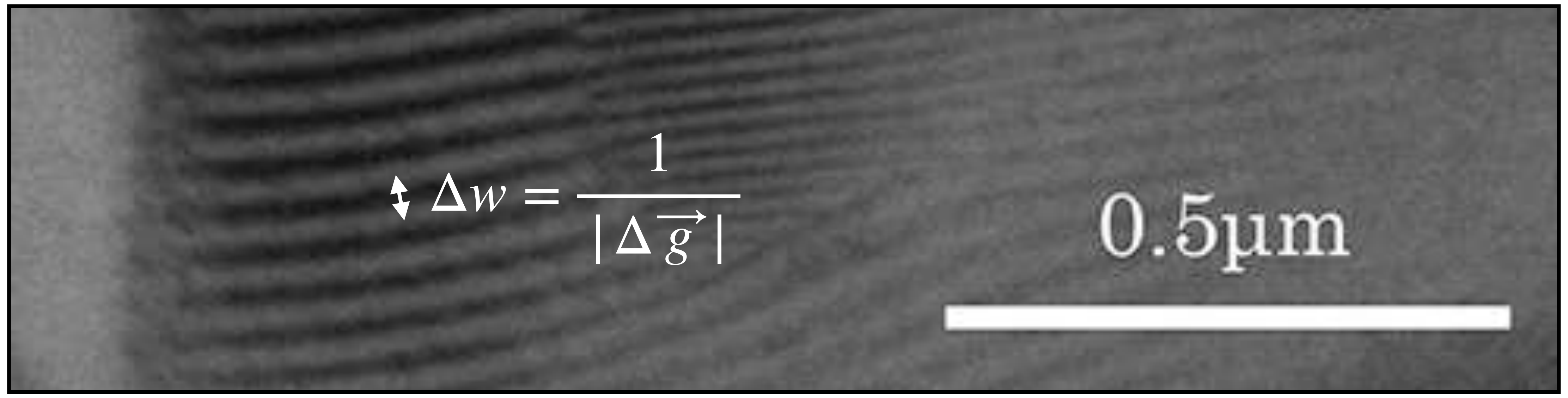
where $\vec{r}_m$ is a vector in the interface that describes the mis-orientation

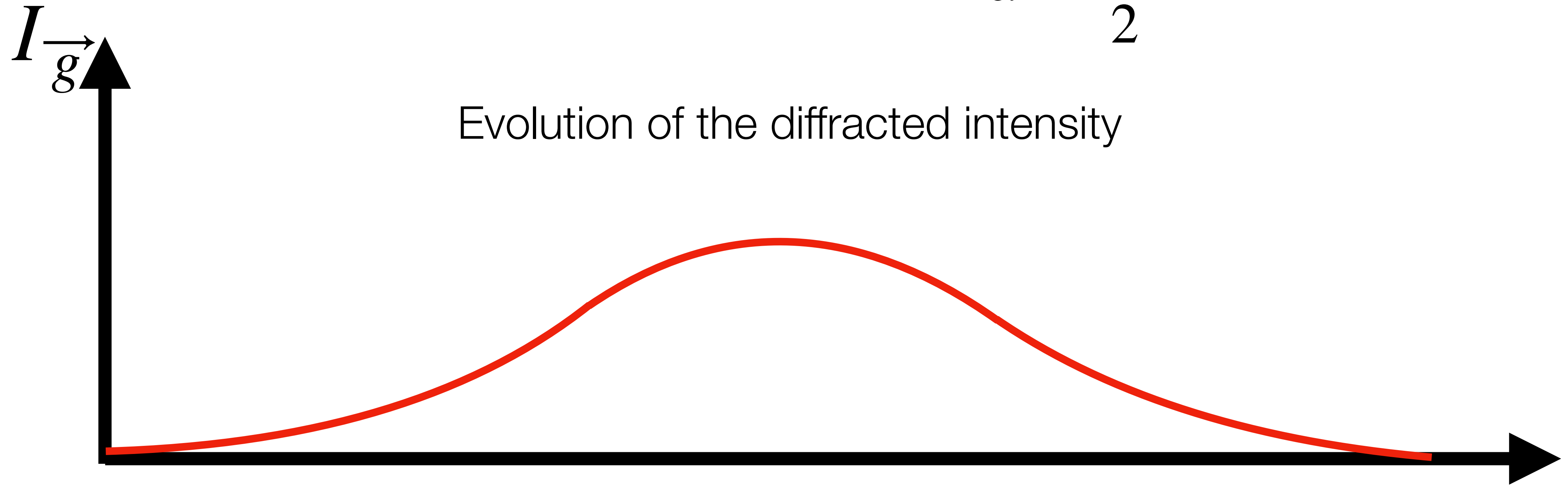
APD interprétation



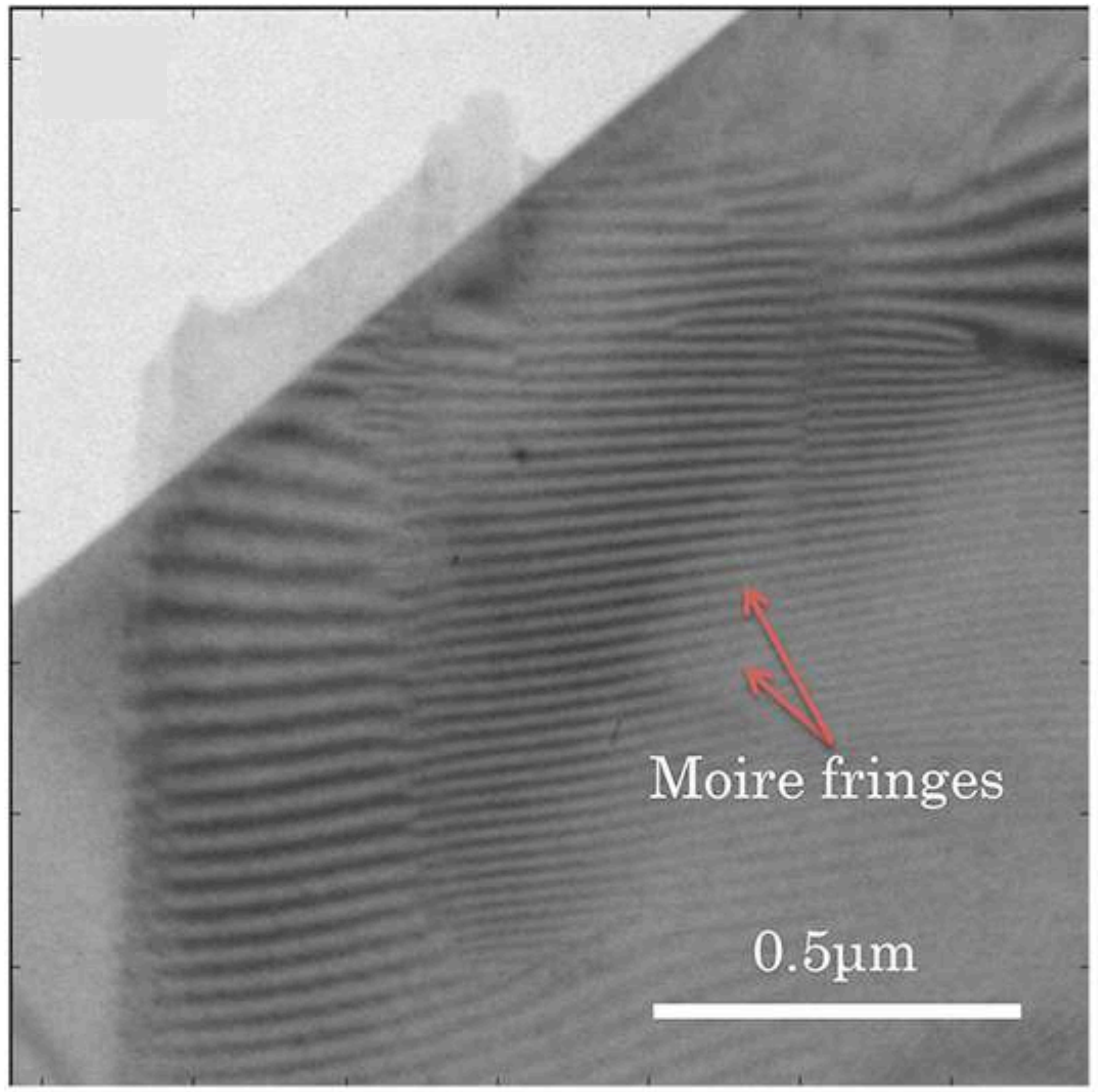
$$\Rightarrow \quad \psi_{\vec{g}} = \frac{i\pi}{\xi_{\vec{g}}}\int_0^{t_1} e^{-2\pi isz}dz + \frac{i\pi}{\xi_{\vec{g}}}\int_{t_1}^{t_2} e^{-2\pi isz}e^{-i\alpha(z)}dz \quad \Rightarrow$$

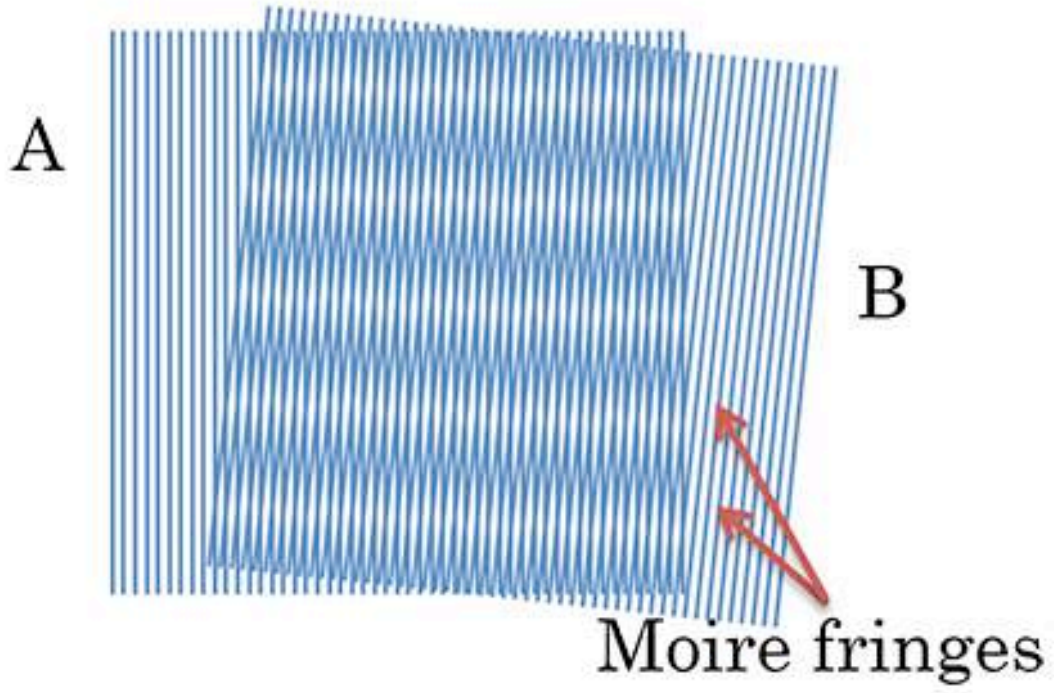
Fringes are observed with inter fringes distance $\Delta w = \frac{1}{|\Delta\vec{g}|}$



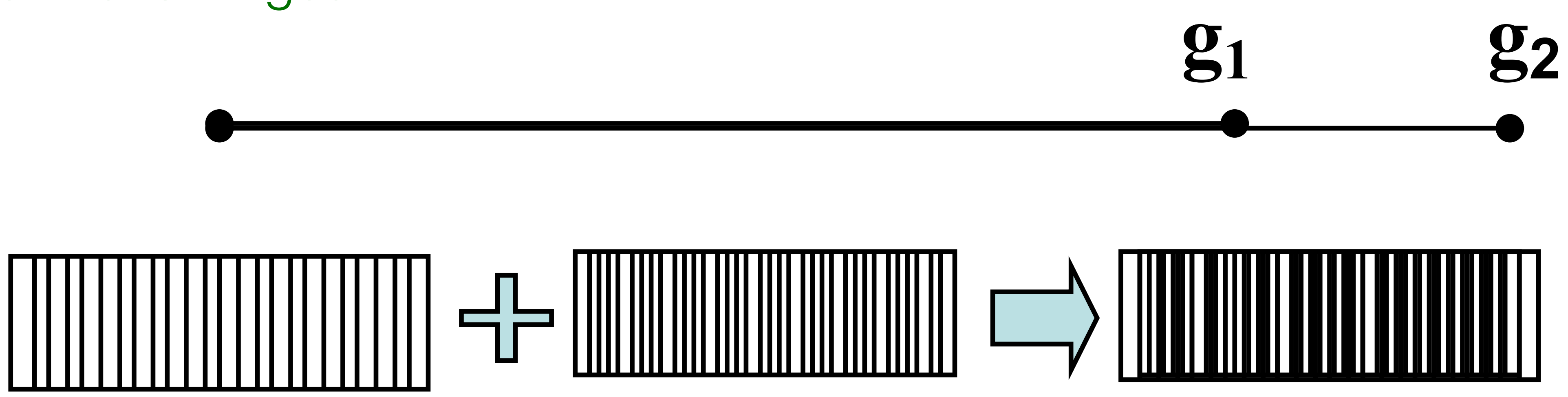


Conventional observation of Moiré fringes



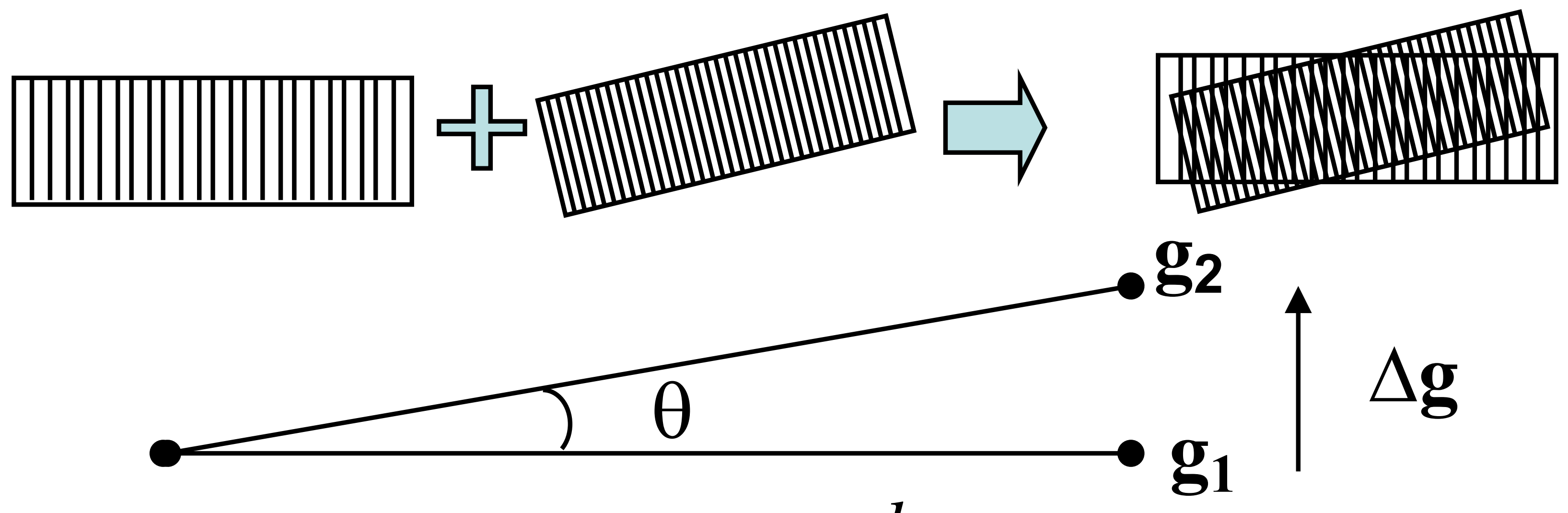


A. Parallel moiré fringes



$$\Delta w = \frac{1}{|\Delta \vec{g}|}\frac{1}{g_1 - g_2} = \frac{1}{1/d_1 - 1/d_2} = \frac{d_1 d_2}{d_1 - d_2}$$

B. Rotational moiré fringes

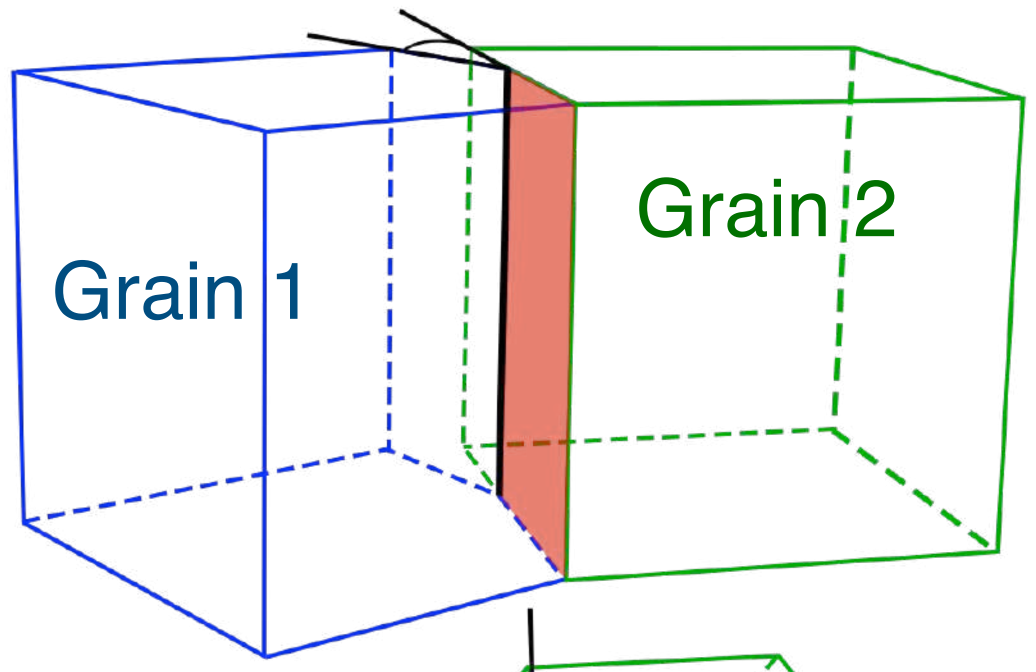


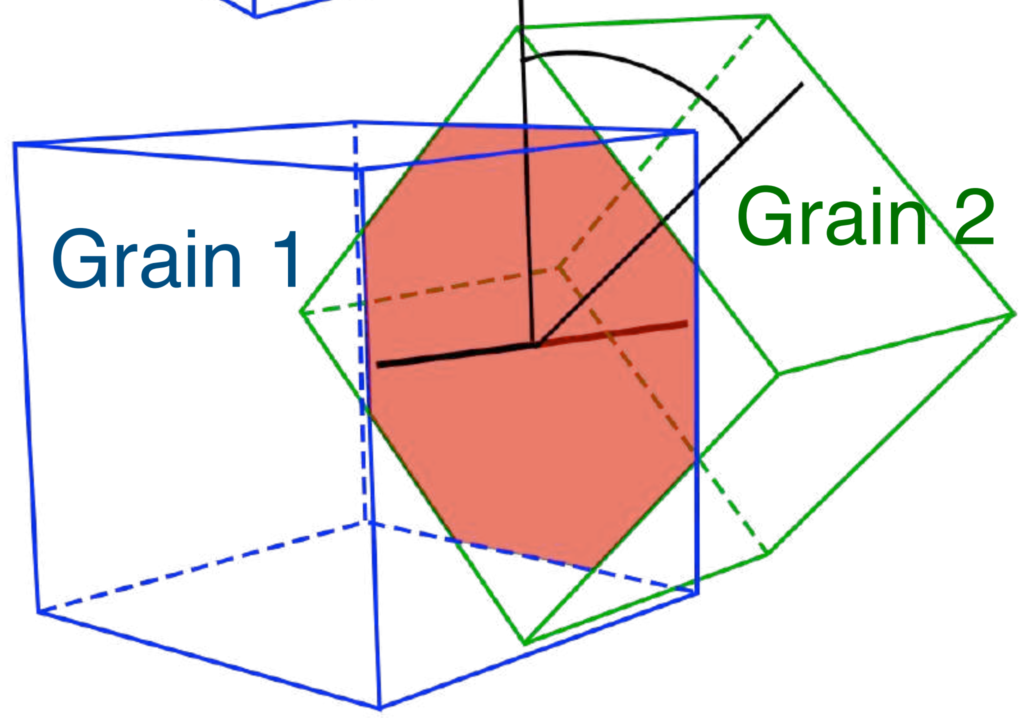
$$\Delta w = \frac{d}{\theta}$$

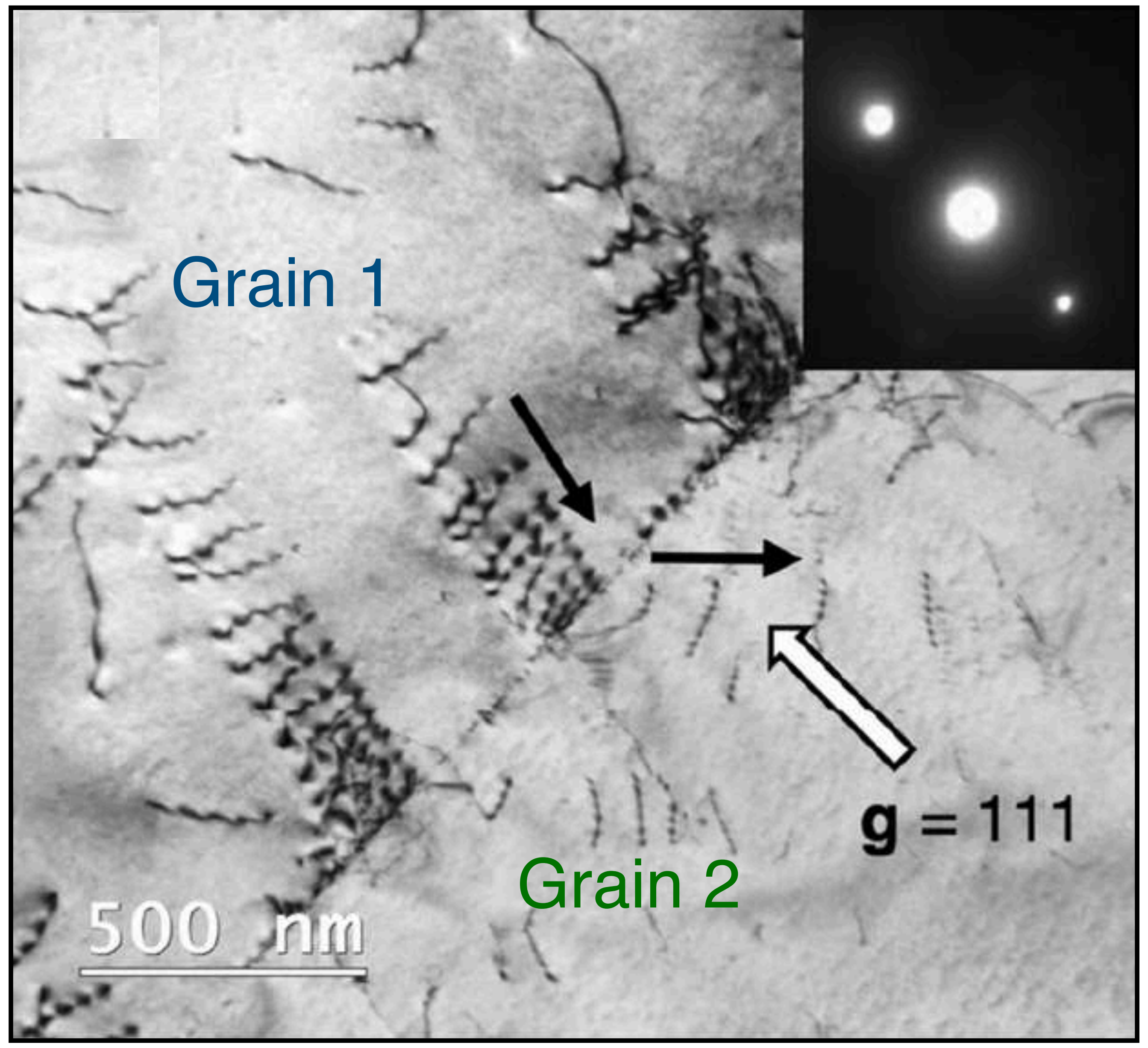
C. Mixed moiré fringes

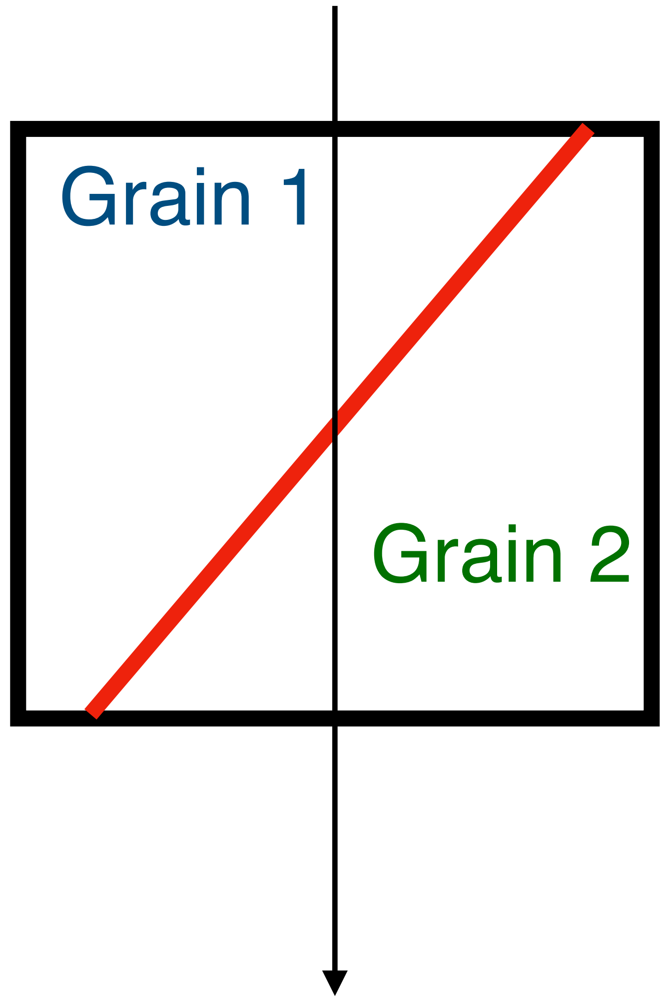
$$\Delta w = \frac{d_1 d_2}{\sqrt{(d_2 - d_2)^2 + d_1 d_2 \theta^2}}$$

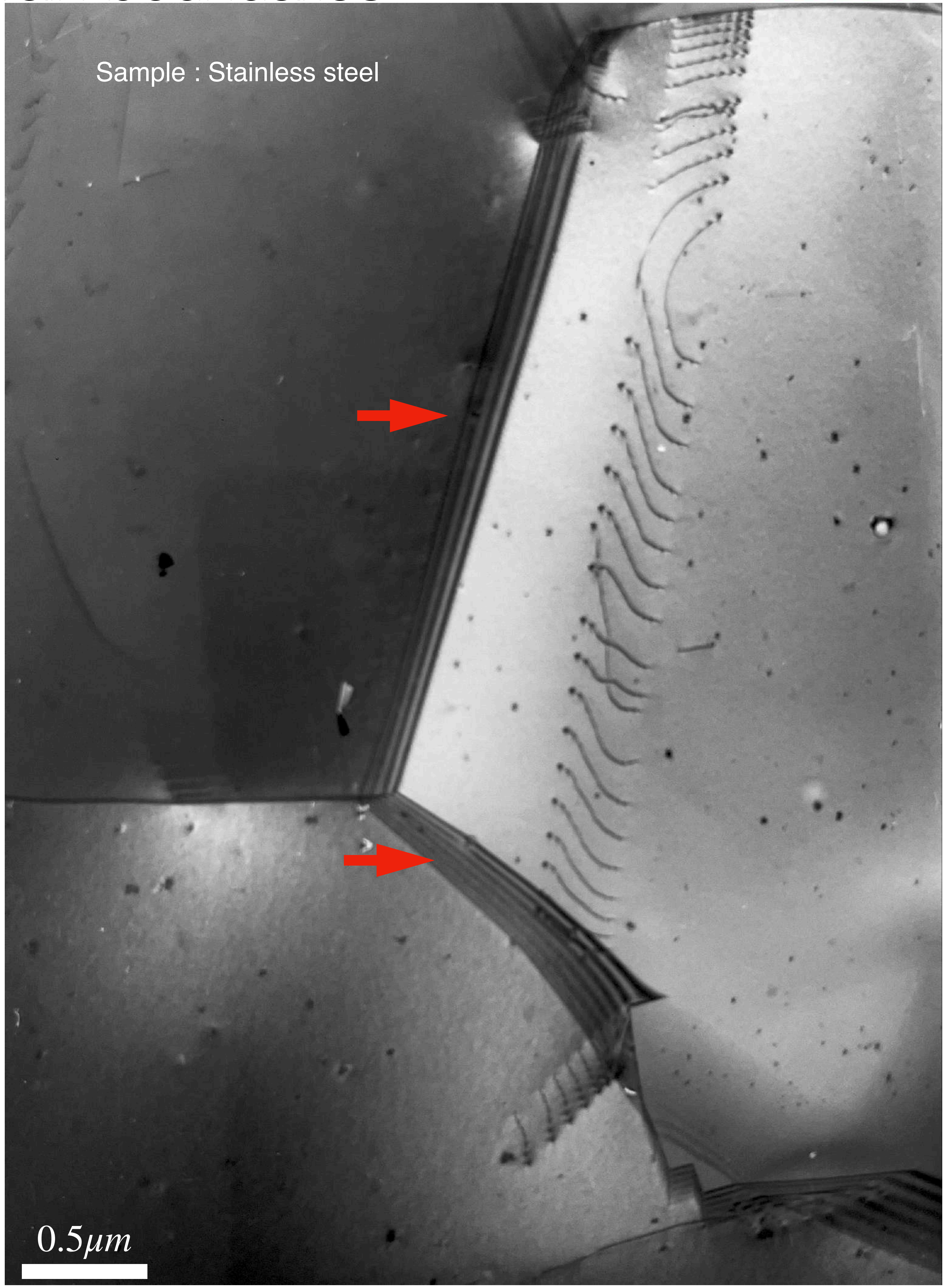
Conventional observation of grain boundaries



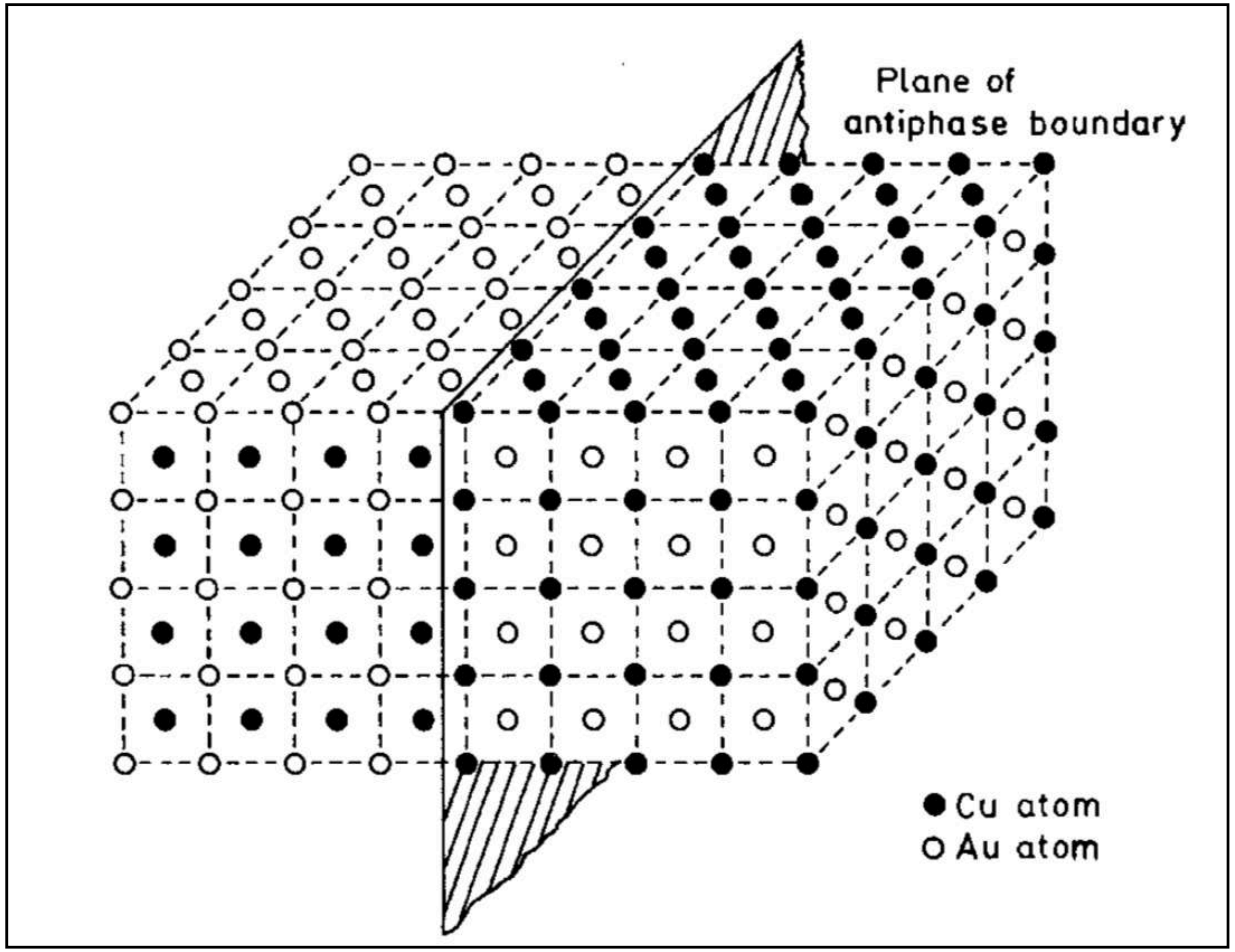




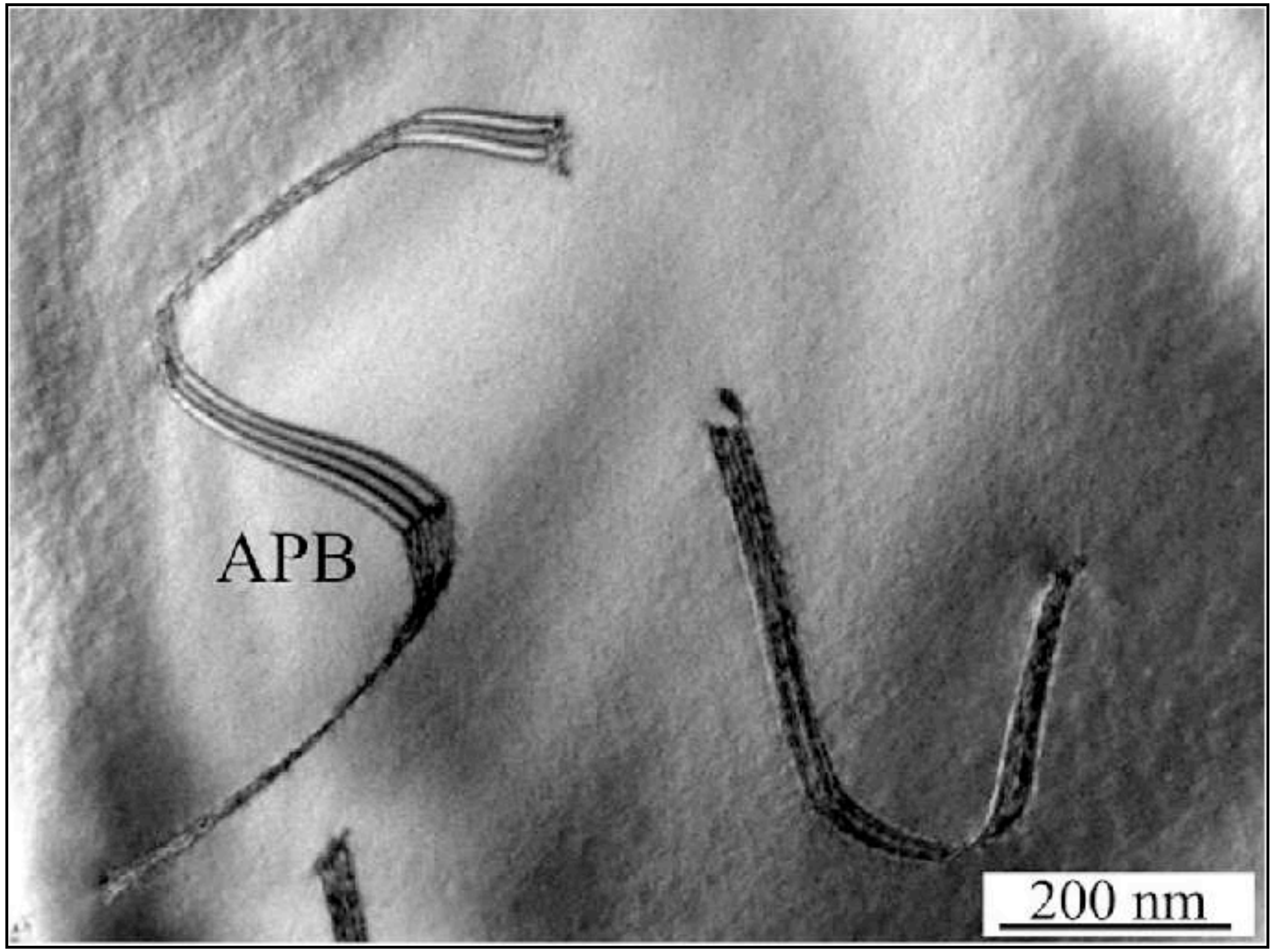




Conventional observation of antiphase boundaries

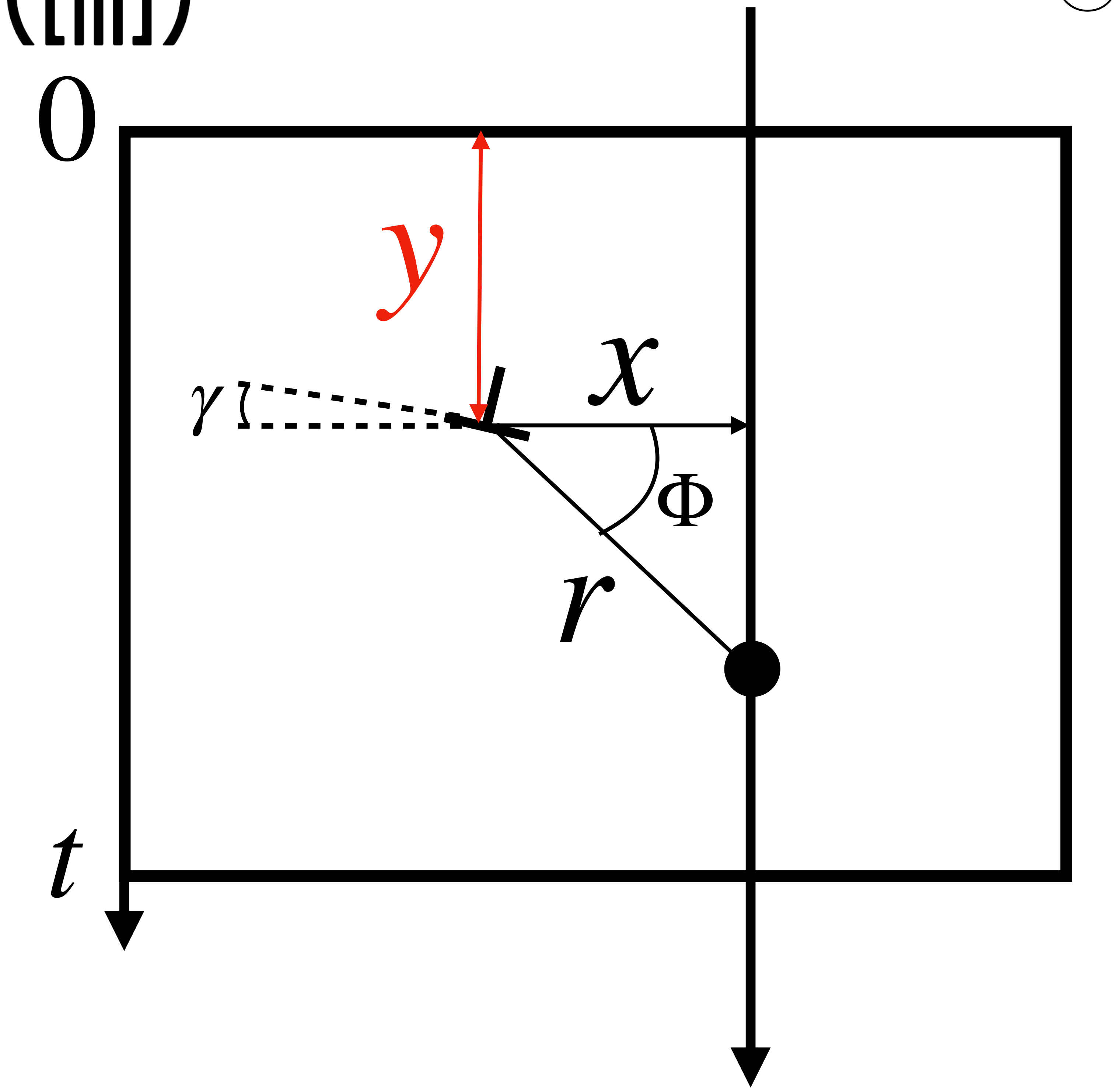


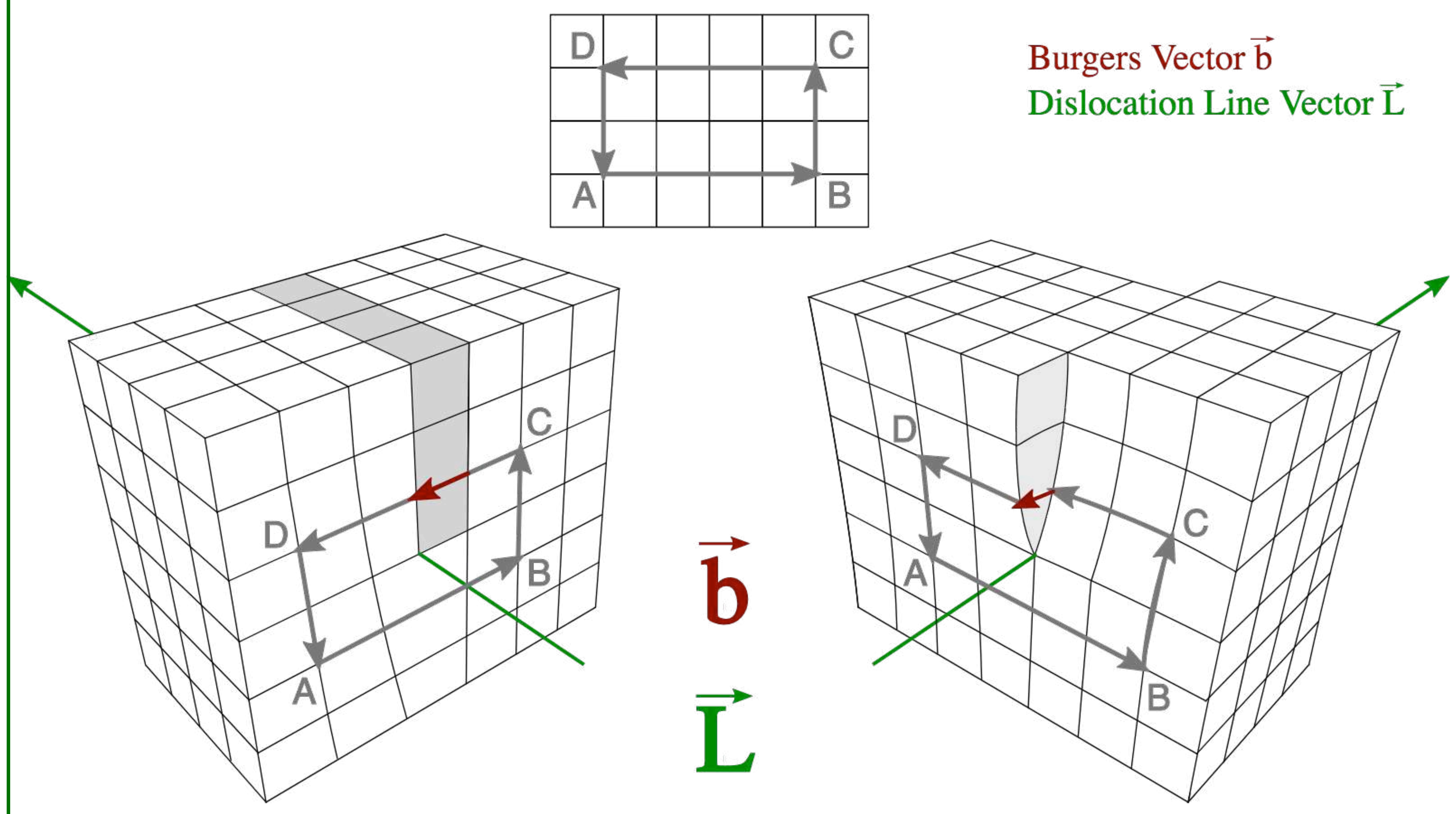
Fringes contrast : same interpretation as for stacking fault



Extracted from : P. Zhao, L. Feng, K. Nielsch, T.G. Woodcock, Microstructural defects in hot deformed and as-transformed τ-MnAl-C, Journal of Alloys and Compounds,Volume 852, 2021,156998, https://doi.org/10.1016/j.jallcom.2020.156998.

Conventional observation of dislocations





- Screw dislocations : $\vec{b} \,//\, \vec{L}$
- Edge dislocations : $\vec{b} \perp \vec{L}$
- Mixed dislocation

- Perfect dislocation : $\vec{b}$ is a unit lattice vector and therefore $\vec{g}\,.\,\vec{b} = 0,1,2,...$
- Partial dislocations : $\vec{g}\,.\,\vec{b}$ can take fractional values

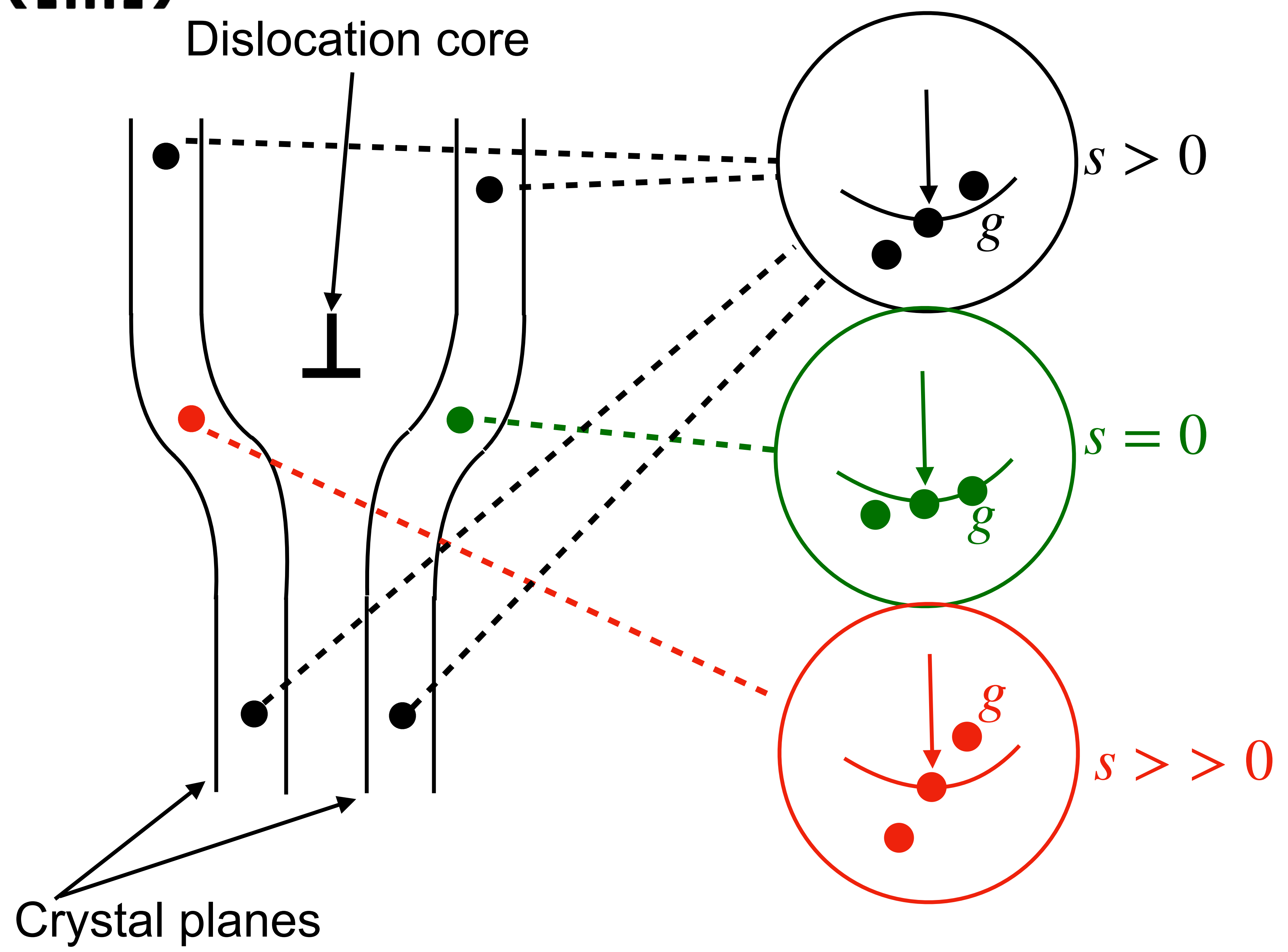
1.Screw dislocations : $\vec{R} = \vec{b}\dfrac{\Phi}{2\pi} = \dfrac{\vec{b}}{2\pi}\arctan\left(\dfrac{z-y}{x}\right)$

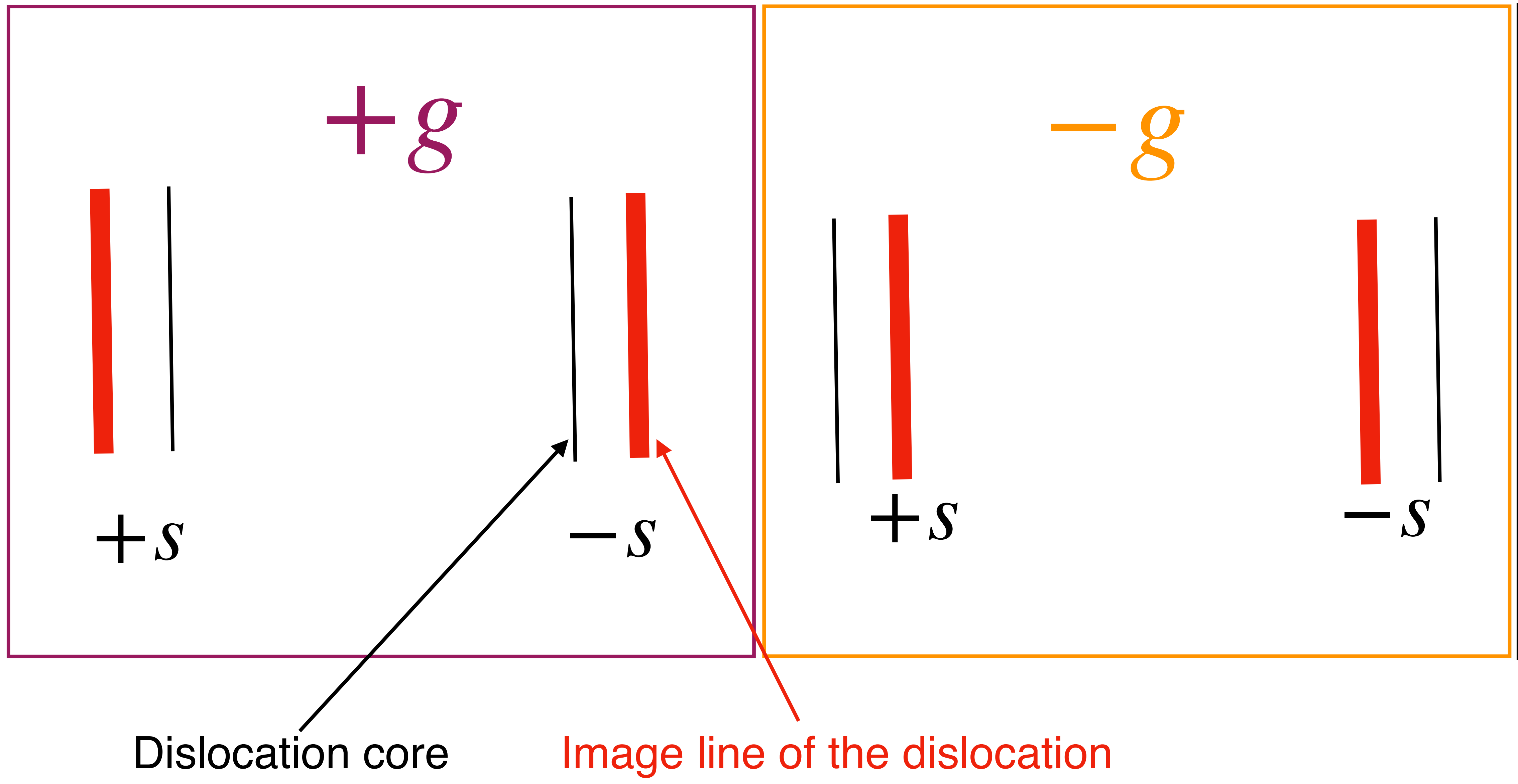
2.General dislocations : $\vec{R} = \dfrac{1}{2\pi}\left(\vec{b}(\Phi-\gamma) + b_e\dfrac{\sin(2(\Phi-\gamma))}{4(1-\nu)} + \vec{b}\wedge\vec{L}\left[\dfrac{1-2\nu}{2(1-\nu)}\ln(r) + \dfrac{\cos(2(\Phi-\gamma))}{4(1-\nu)}\right]\right)$ ➡ b_e is the edge component of dislocation and ν is the Poisson coefficient of the material

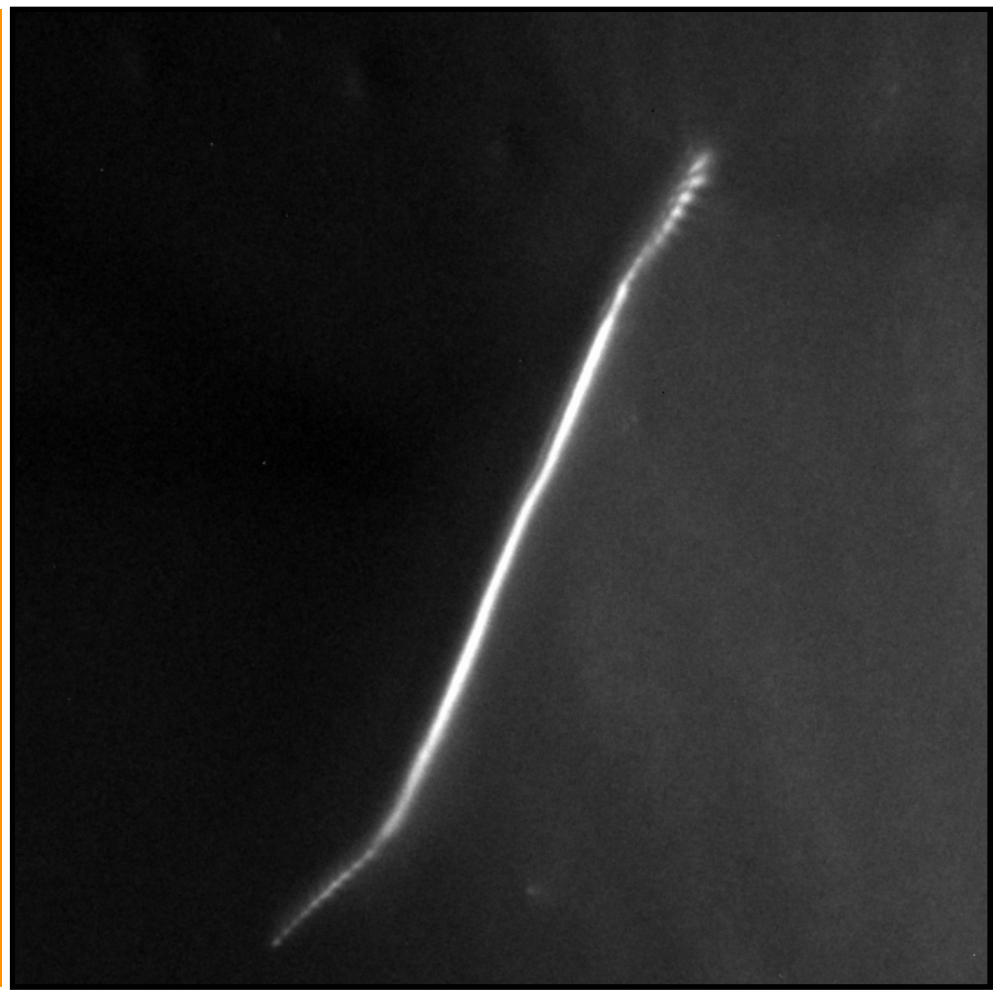
To estimate the diffracted intensity we go back once again to the kinematical expression of the diffracted wave :

$\psi_{\vec{g}} = \dfrac{i\pi}{\xi_{\vec{g}}}\displaystyle\int_0^t e^{-2\pi isz}e^{-2\pi i\vec{g}.\vec{R}(z)}dz$ with $\alpha(z) = 2\pi\vec{g}\,.\,\vec{R}(z)$ ➡ If $\vec{g}\,.\,\vec{b} = 0 \implies \alpha = 0$ and the dislocation is invisible

Conventional observation of dislocations

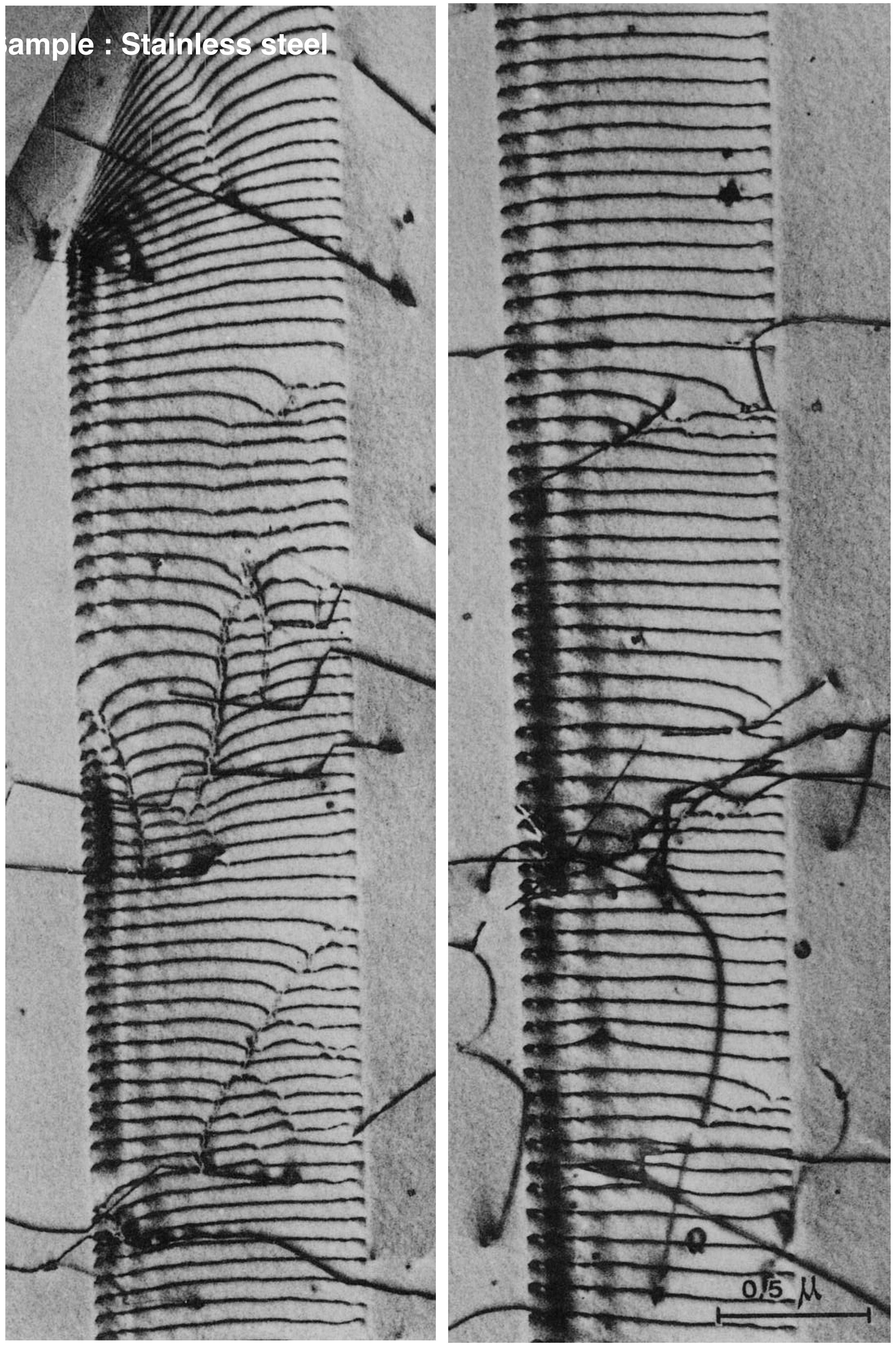


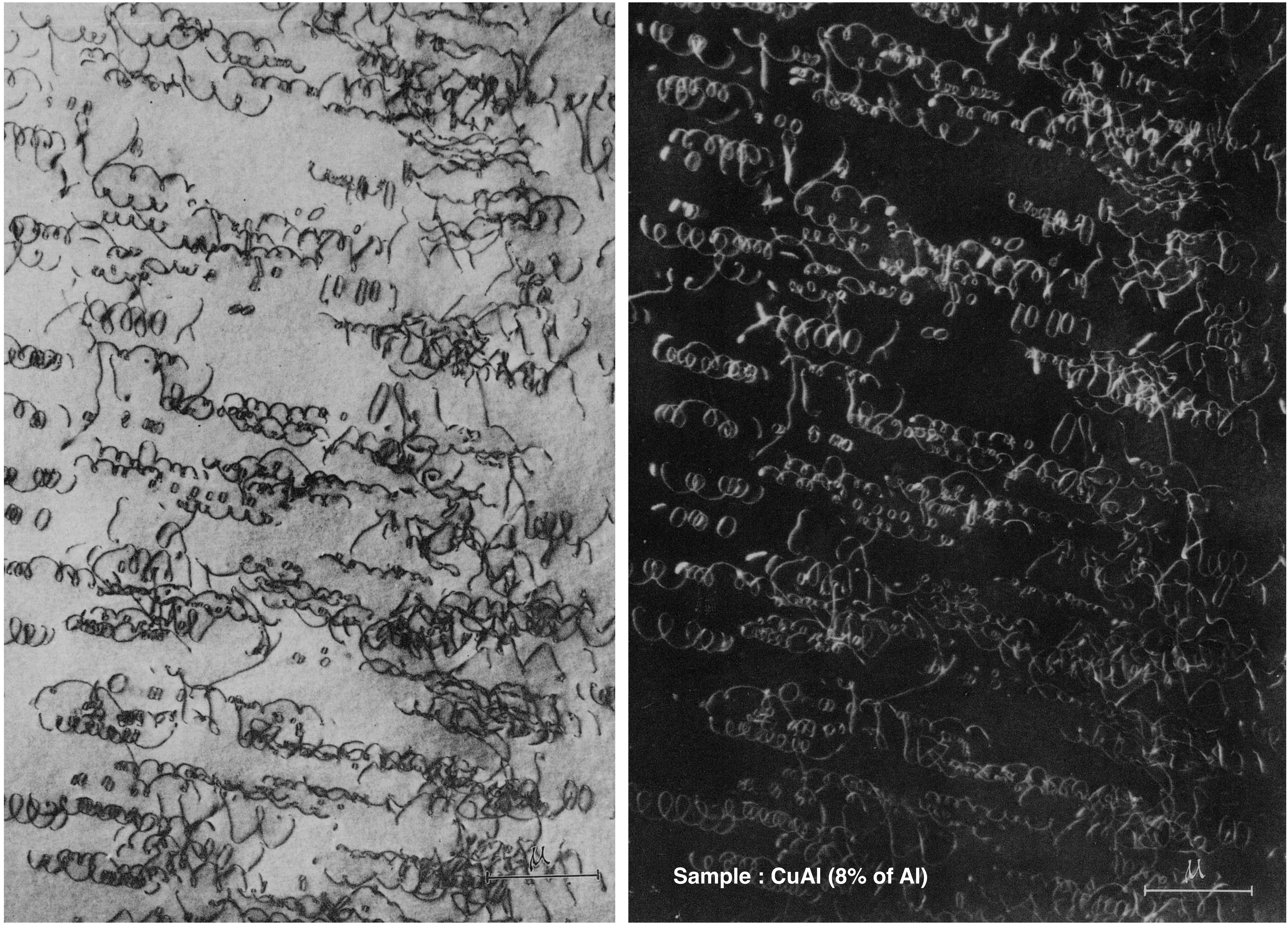




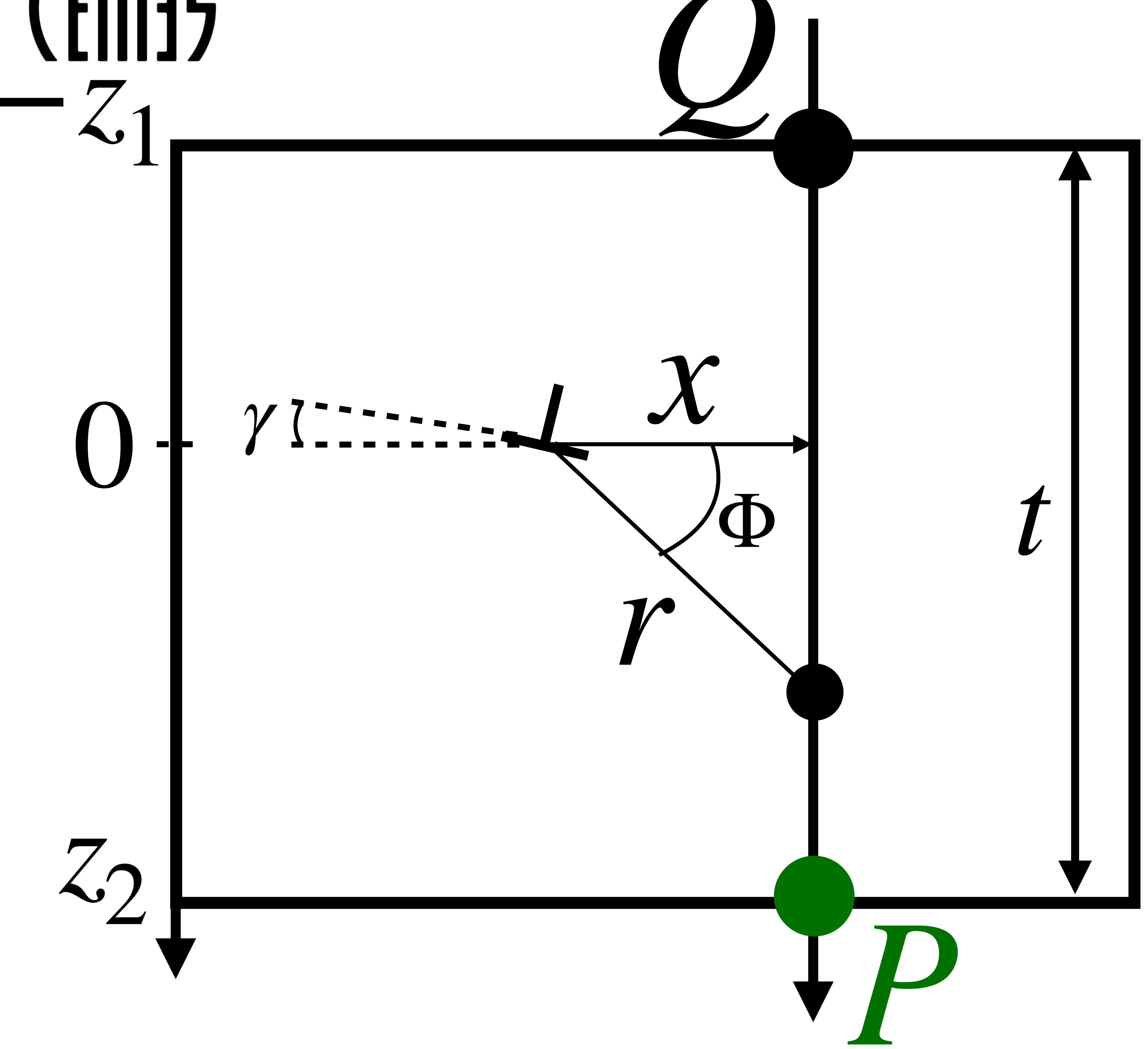
- The planes are bent around the core of the dislocation
- On one side of the dislocation, the planes might be bent closer to the Bragg condition ($\implies$ s is smaller) such that the intensity, $I_{\vec{g}}$ is higher than the background
- On the other side of the dislocation, the planes would be bent away from the Bragg condition, therefore $I_{\vec{g}} \approx I_{background}$
- Dislocations parallel to the surface show uniform contrast, inclined dislocations alternating contrast
- Reversing either $\vec{g}$ or $\vec{s}$ will reverse the position of the image of the dislocation relative to the dislocation core

Conventional observation of dislocations





Diffraction contrast of dislocation : APD interpretation



- Simpler case : the screw dislocation

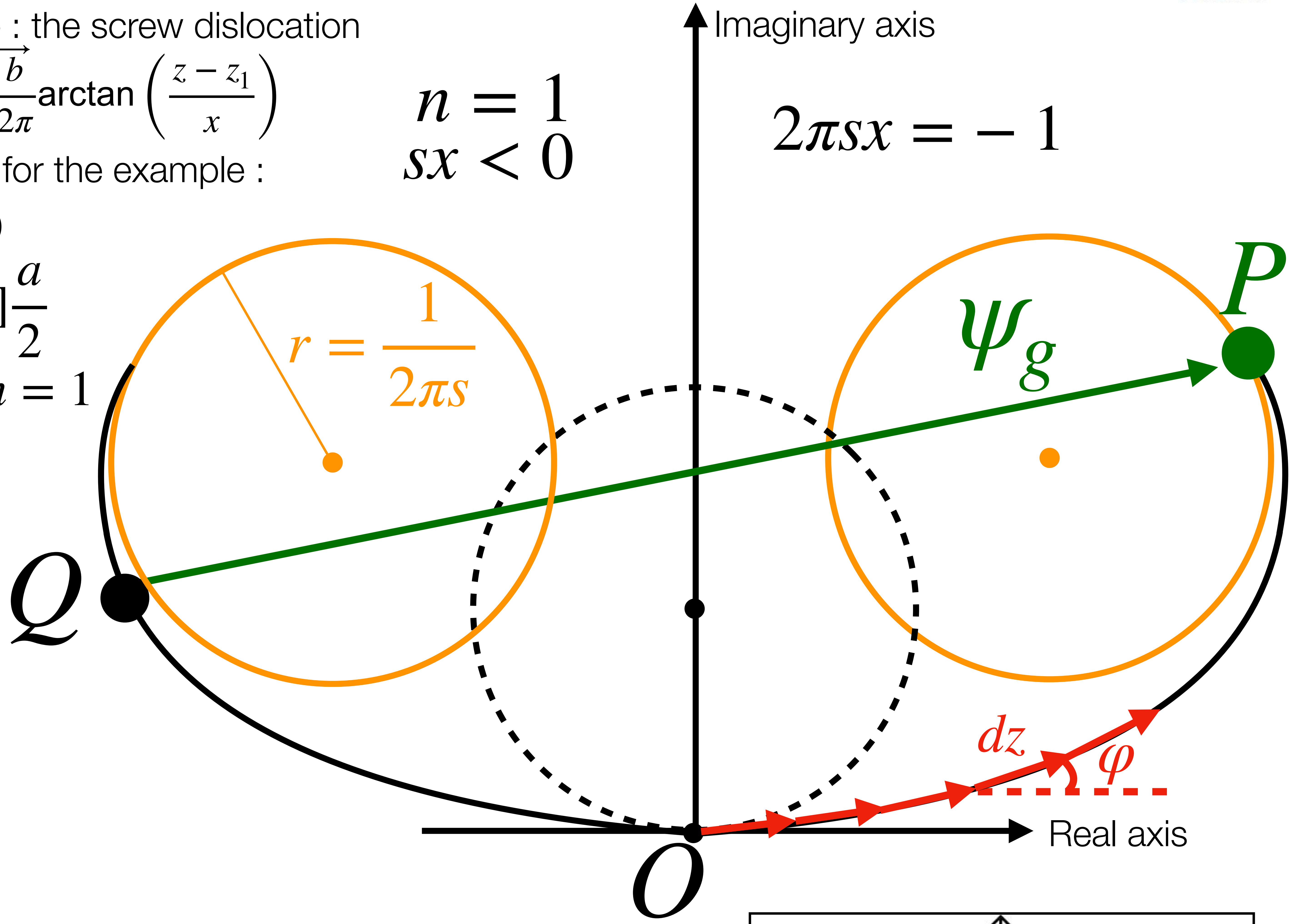
$$\vec{R} = \vec{b}\,\frac{\Phi}{2\pi} = \frac{\vec{b}}{2\pi}\arctan\left(\frac{z - z_1}{x}\right)$$

- We choose for the example :

$$\vec{g} = (111)$$

$$\vec{b} = [110]\frac{a}{2}$$

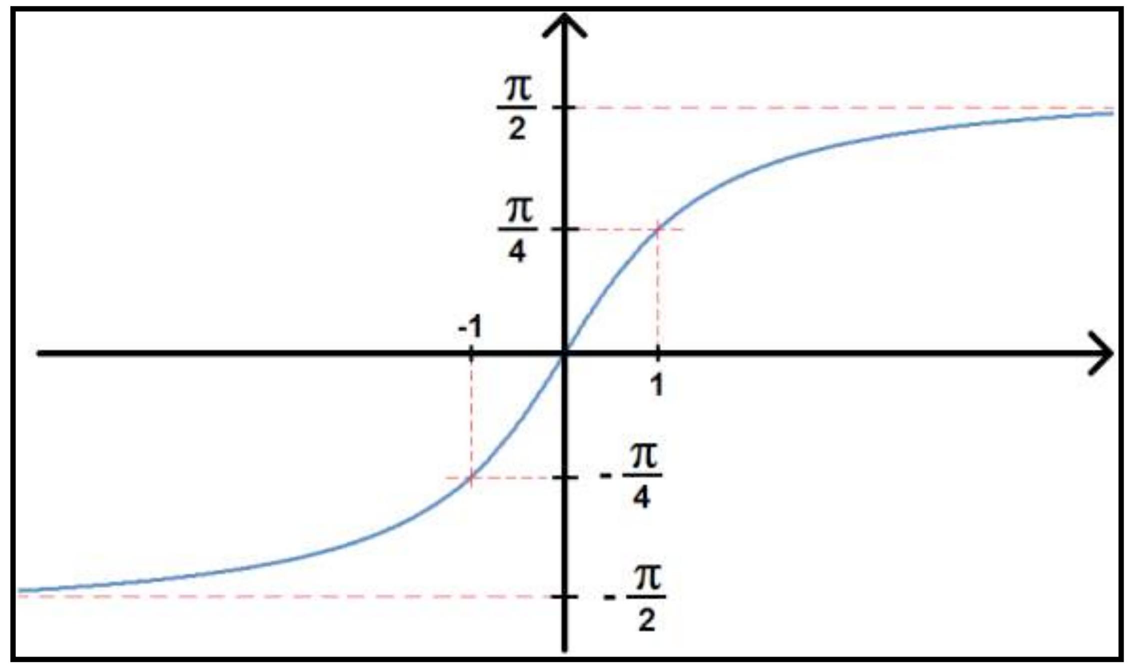
$$\vec{g}\,.\,\vec{b} = n = 1$$



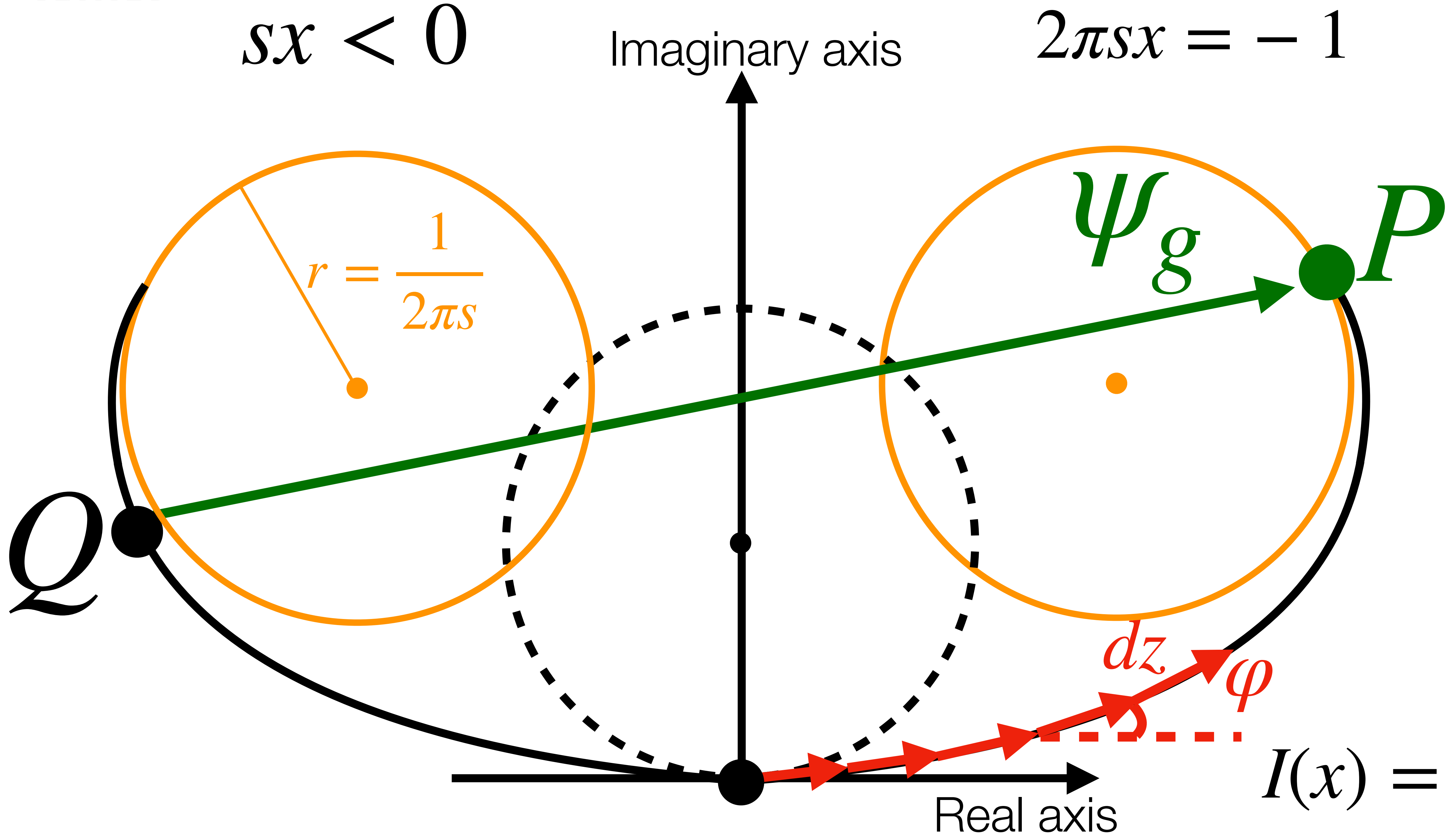
- The diffracted wave is given by :

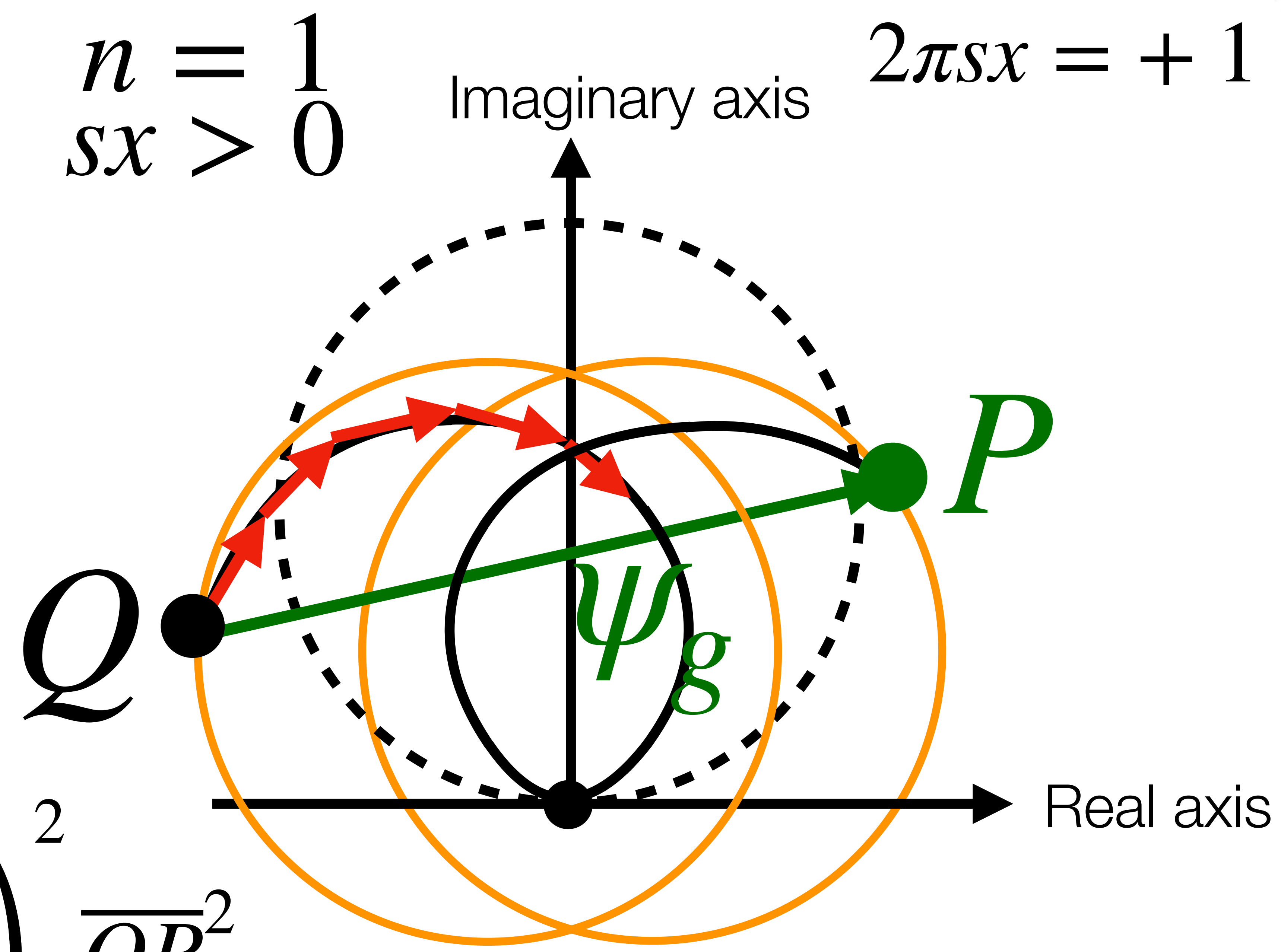
$$\psi_{\vec{g}} = \frac{i\pi}{\xi_{\vec{g}}}\int_0^t e^{-2\pi i s z} e^{-2\pi i \vec{g}.\vec{R(z)}} dz$$

$$\psi_{\vec{g}} = \frac{i\pi}{\xi_{\vec{g}}}\int_{-z_1}^{z_2} \mathbf{exp}\left(-i\left(2\pi s z + n\,\mathbf{arctan}\left(\frac{z}{x}\right)\right)\right) dz = \frac{i\pi}{\xi_{\vec{g}}}\int_{-z_1}^{z_2} \mathbf{exp}\left(i\varphi\right) dz$$

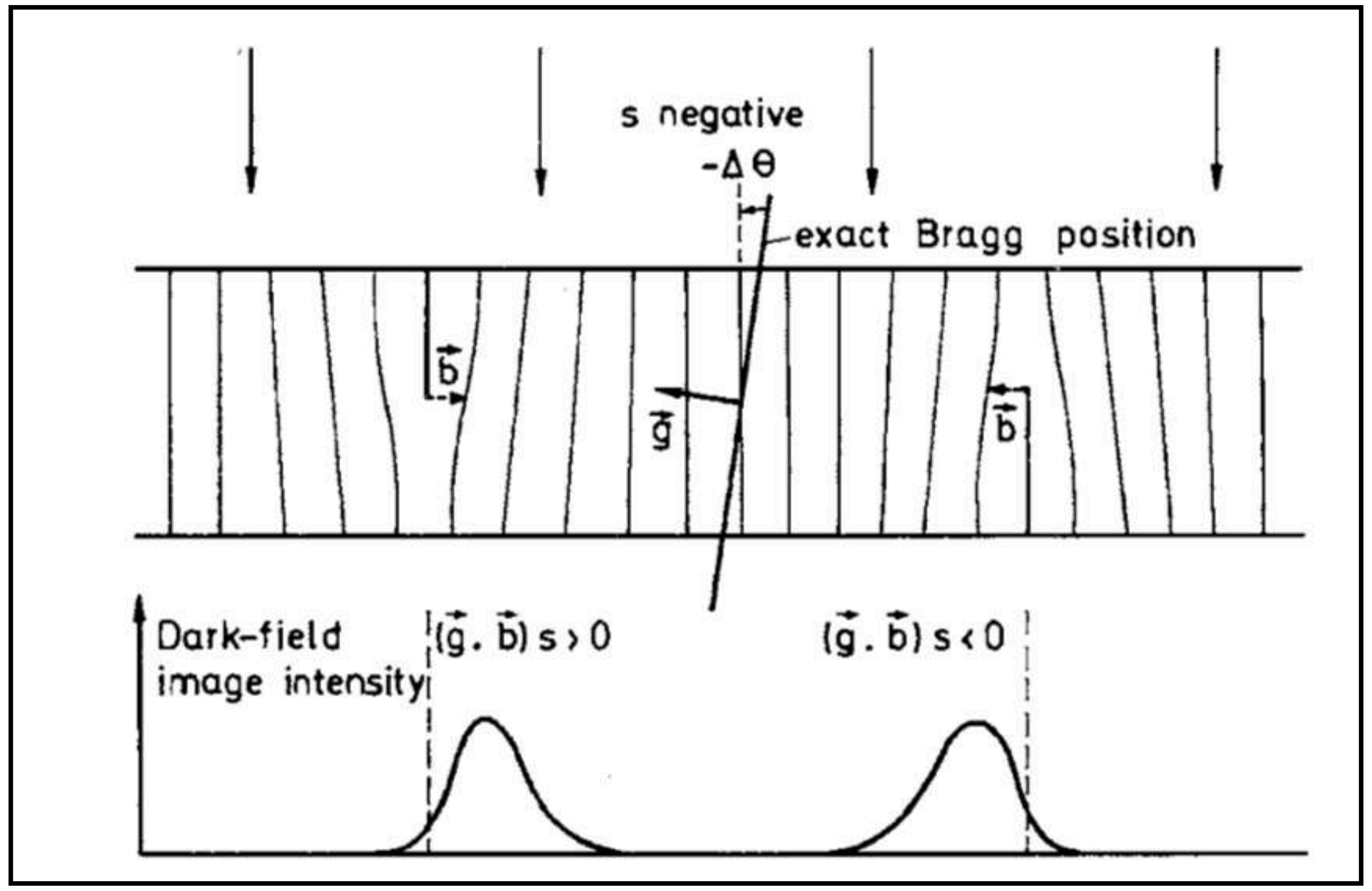


Diffraction contrast of dislocation : APD interpretation



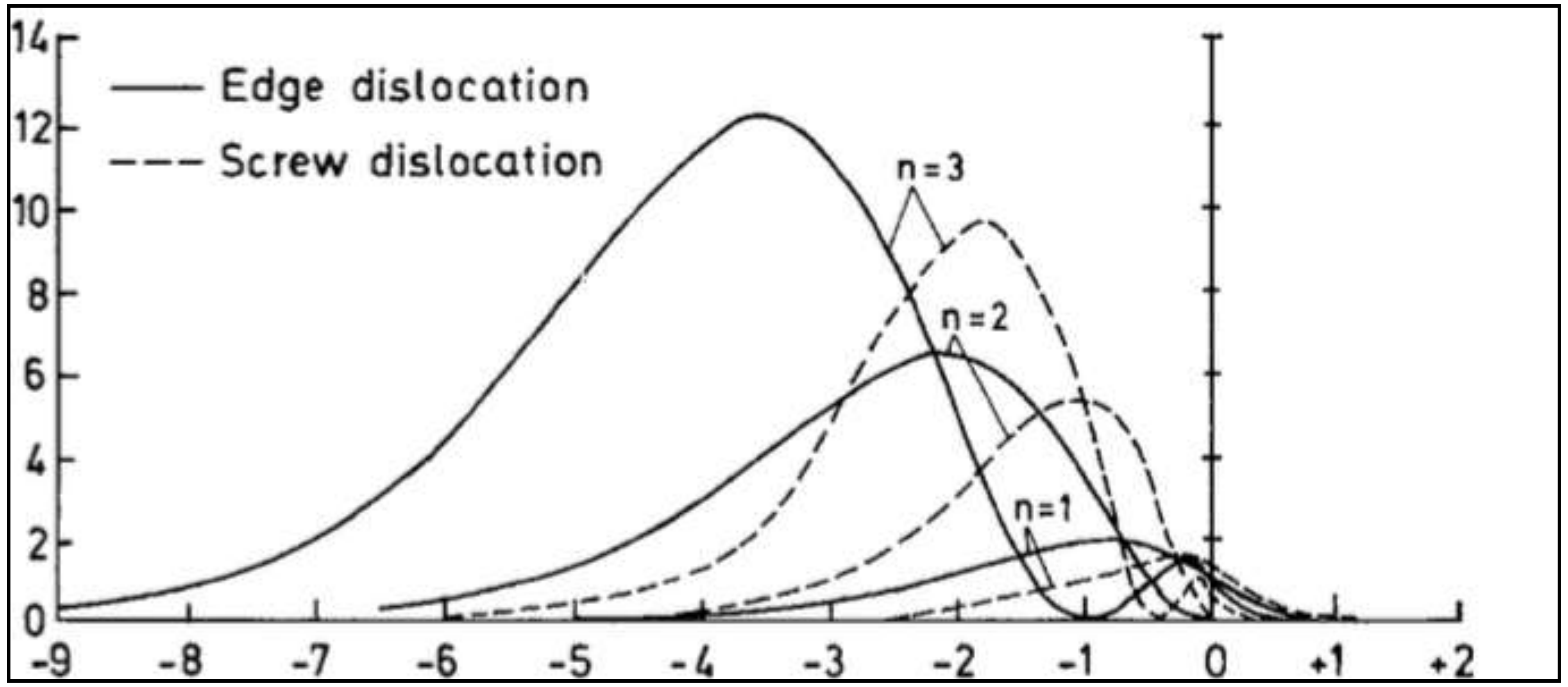


$$I(x) = \psi_{\vec{g}} \psi^*_{\vec{g}} = \left(\frac{\pi}{\xi_{\vec{g}}}\right)^2 \overline{QP}^2$$



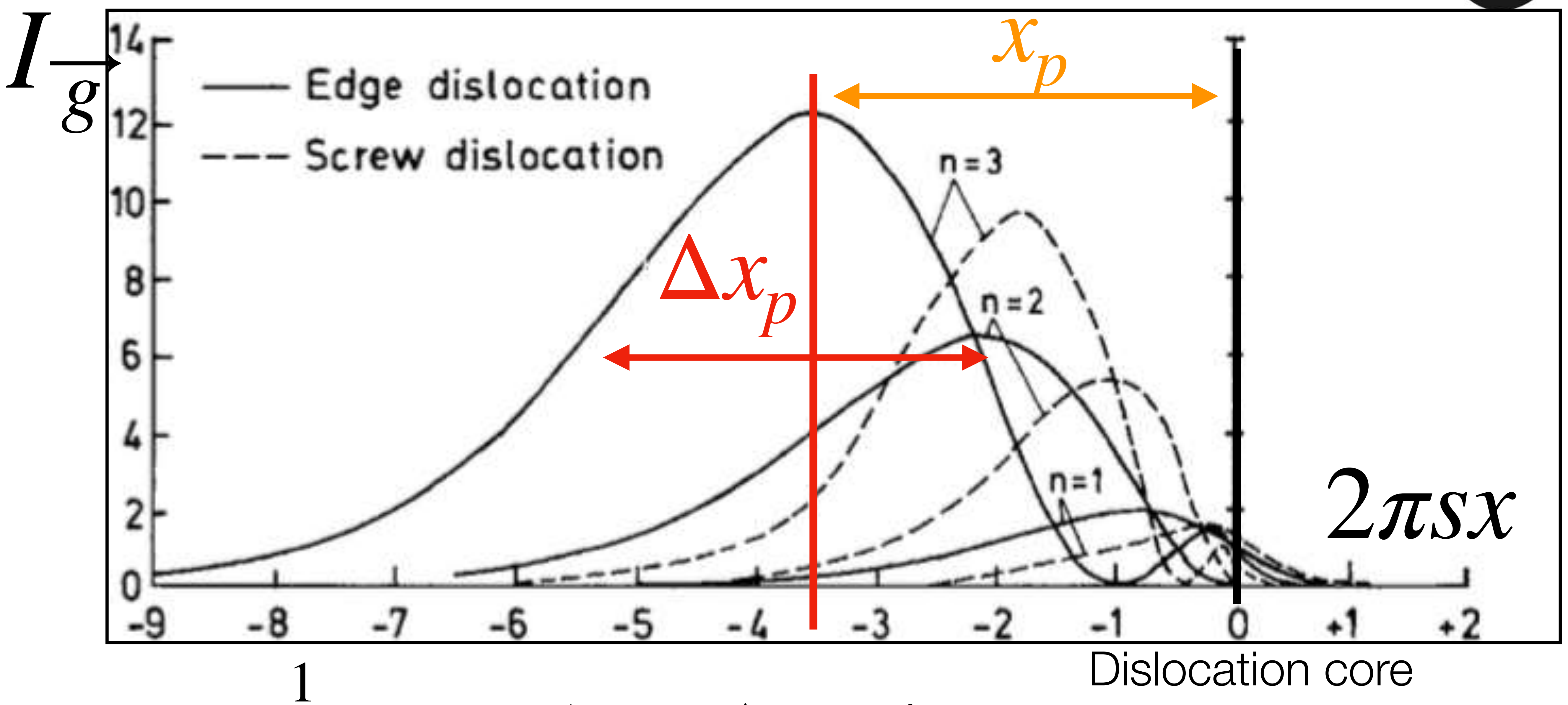
$I_{\vec{g}}$

Dislocation core $x = 0$



$2\pi sx$

Typical values for metals (fcc) with $\vec{b} = \frac{1}{2}[110]$



- If $\vec{g} = (1\bar{1}1) \implies \vec{g} \cdot \vec{b} = 0$ the dislocation is invisible

- If $\vec{g} = (111) \implies \vec{g} \cdot \vec{b} = 1$

For $\vec{g} \cdot \vec{b} = 1$ the image width is $\Delta x_p \approx \frac{1}{2\pi s}$ and the image position $x_p = -\frac{1}{4\pi s}$ so that $s \uparrow \implies \Delta x_p, x_p \downarrow$

For example, if $s = 1 \times 10^{-3}\text{Å}^{-1} \implies x_p = 75\text{Å}, \Delta x_p = 150\text{Å}$

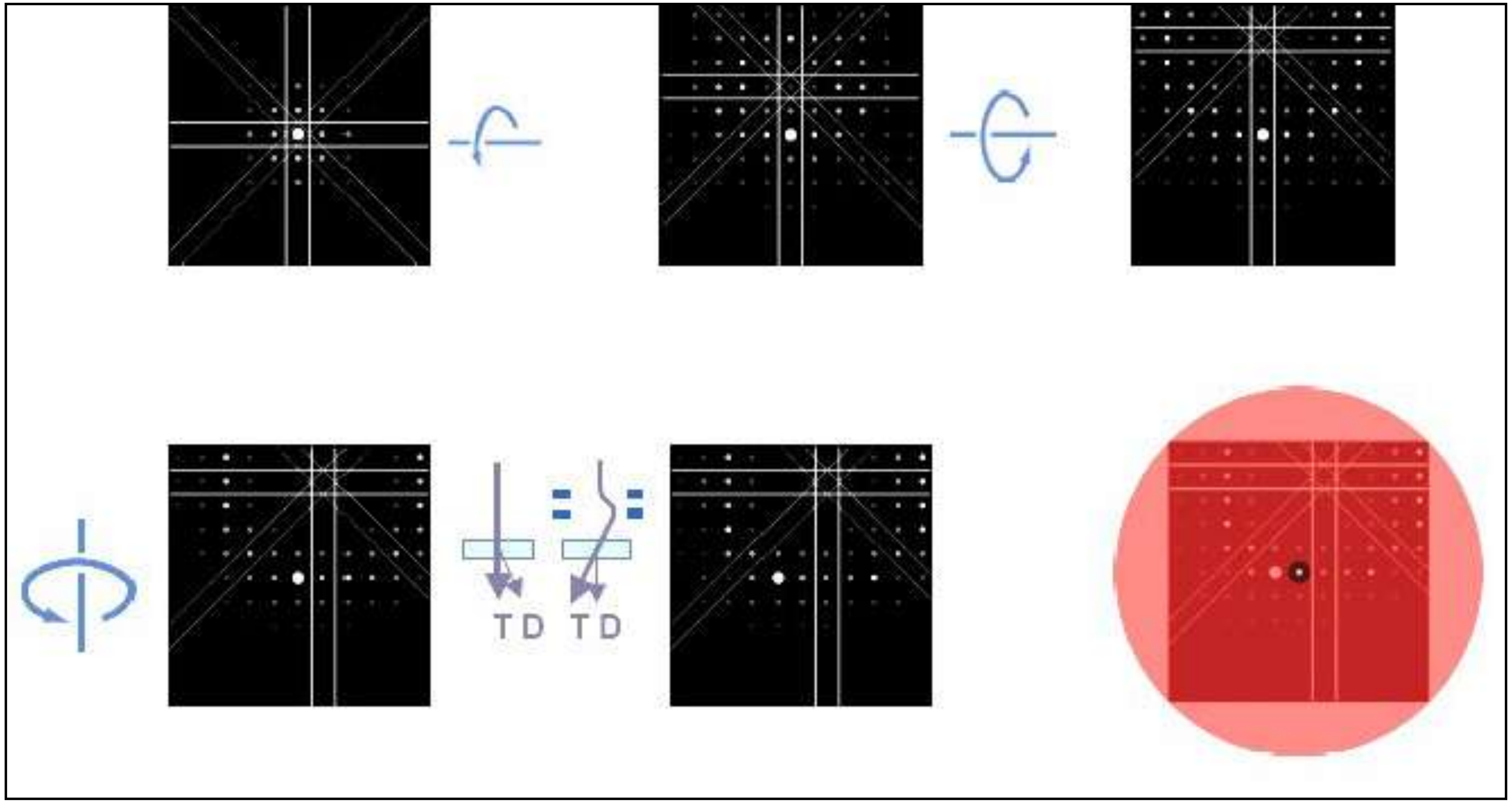
- If $\vec{g} \cdot \vec{b} = 2 \implies \Delta x_p \approx \frac{1}{\pi s}, x_p = -\frac{1}{2\pi s}$

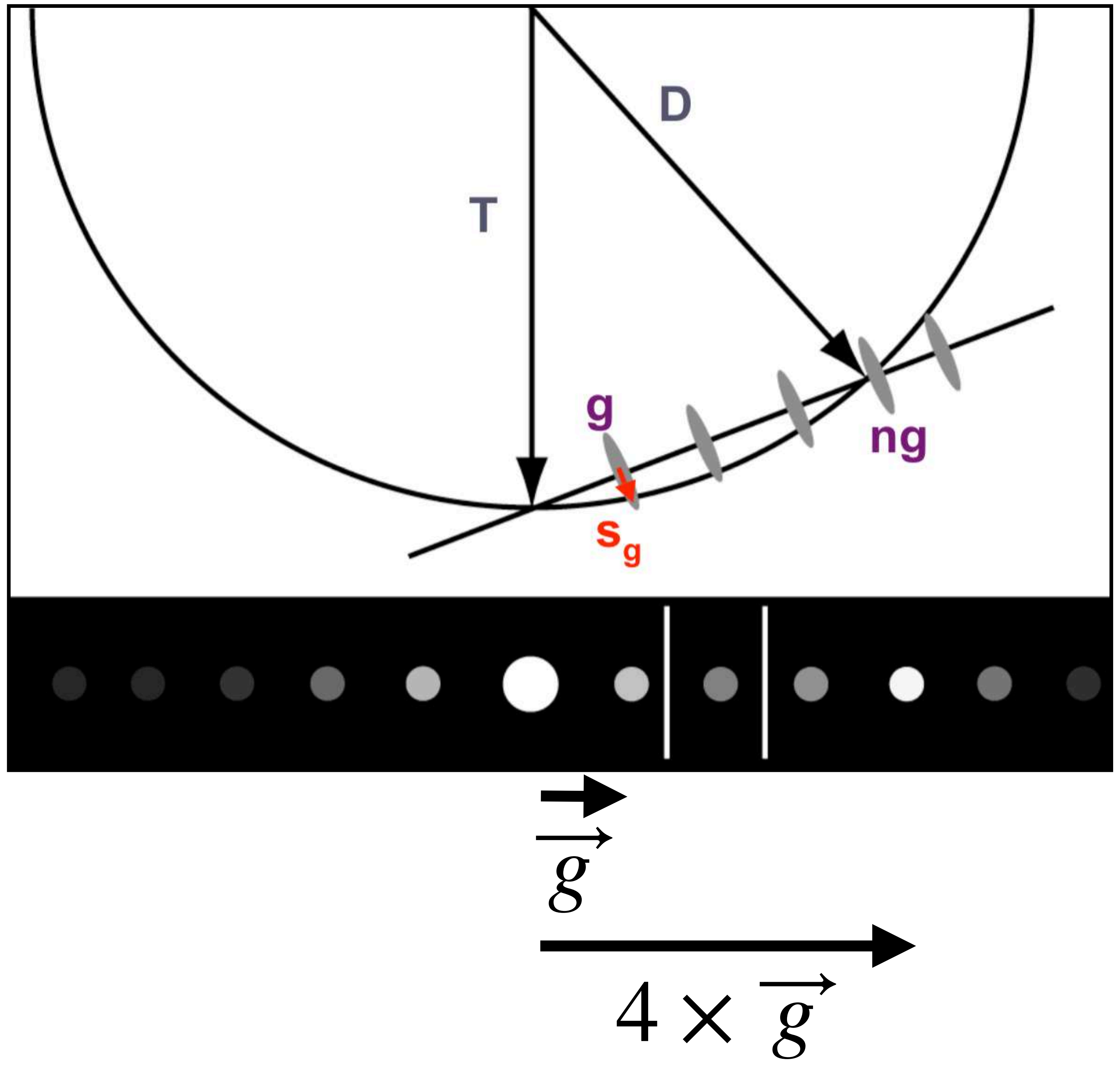
For $\vec{g} = 022$ and $s = 2 \times 10^{-2}\text{Å}^{-1} \implies x_p = 10\text{Å}, \Delta x_p = 15\text{Å}$

➡ Thus the pick width is such smaller and the peak much closer to the core for large values of s. This is the principe of weak beam imaging.

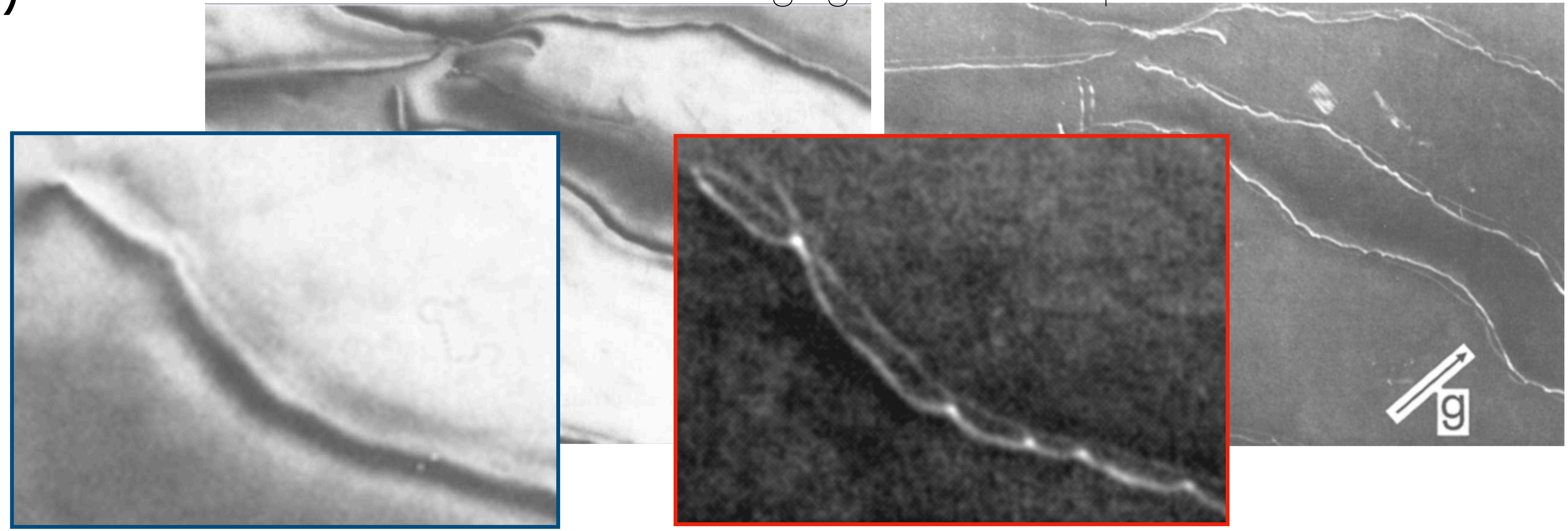
Caution : choose s carefully as $I_{\vec{g}} \propto \frac{1}{s^2}$. Furthermore kinematic approximation holds well for large s. When $s \rightarrow 0$ dynamical theory must be us

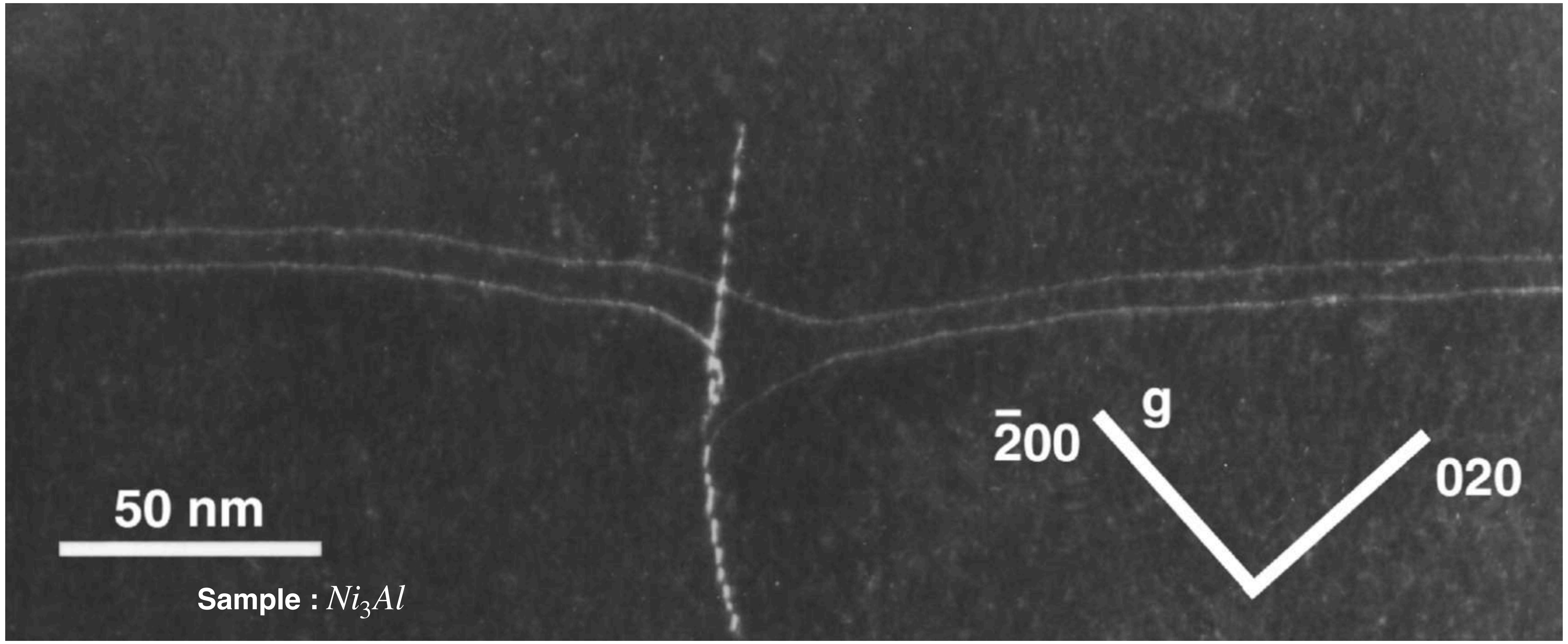
Weak beam imaging : $\overrightarrow{g} - n\overrightarrow{g}$ conditions



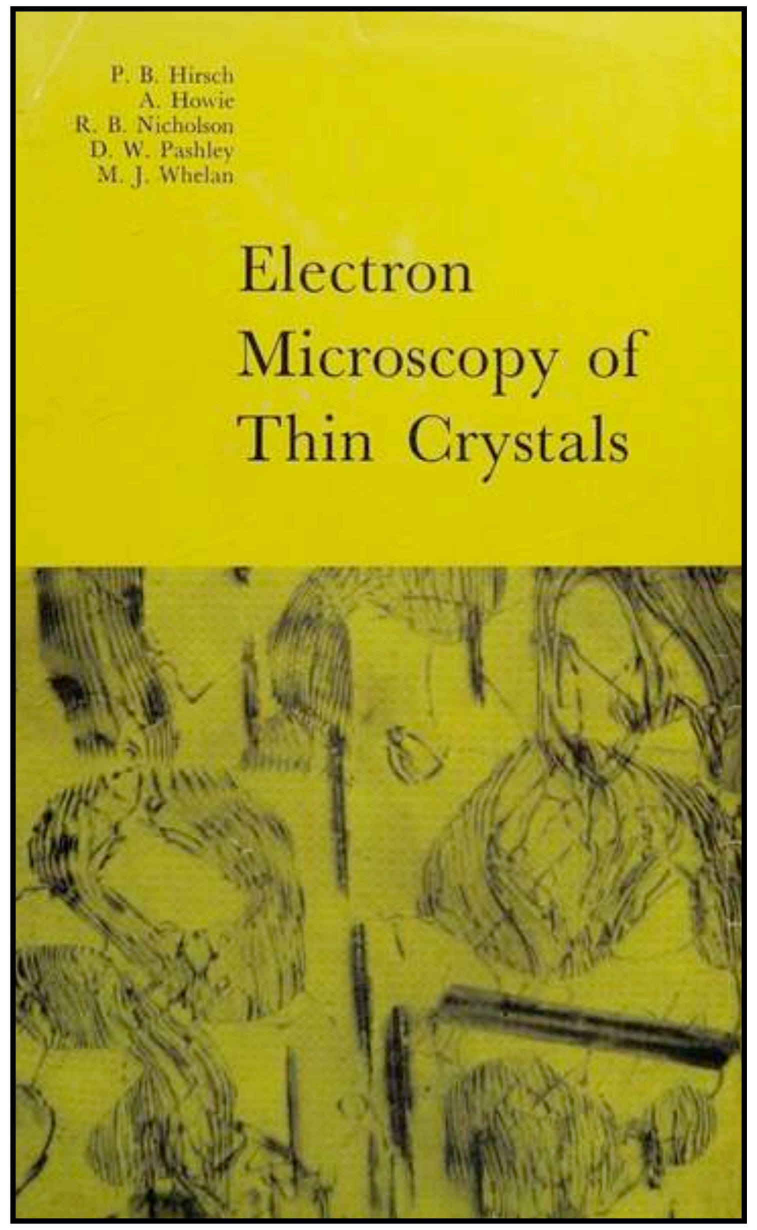


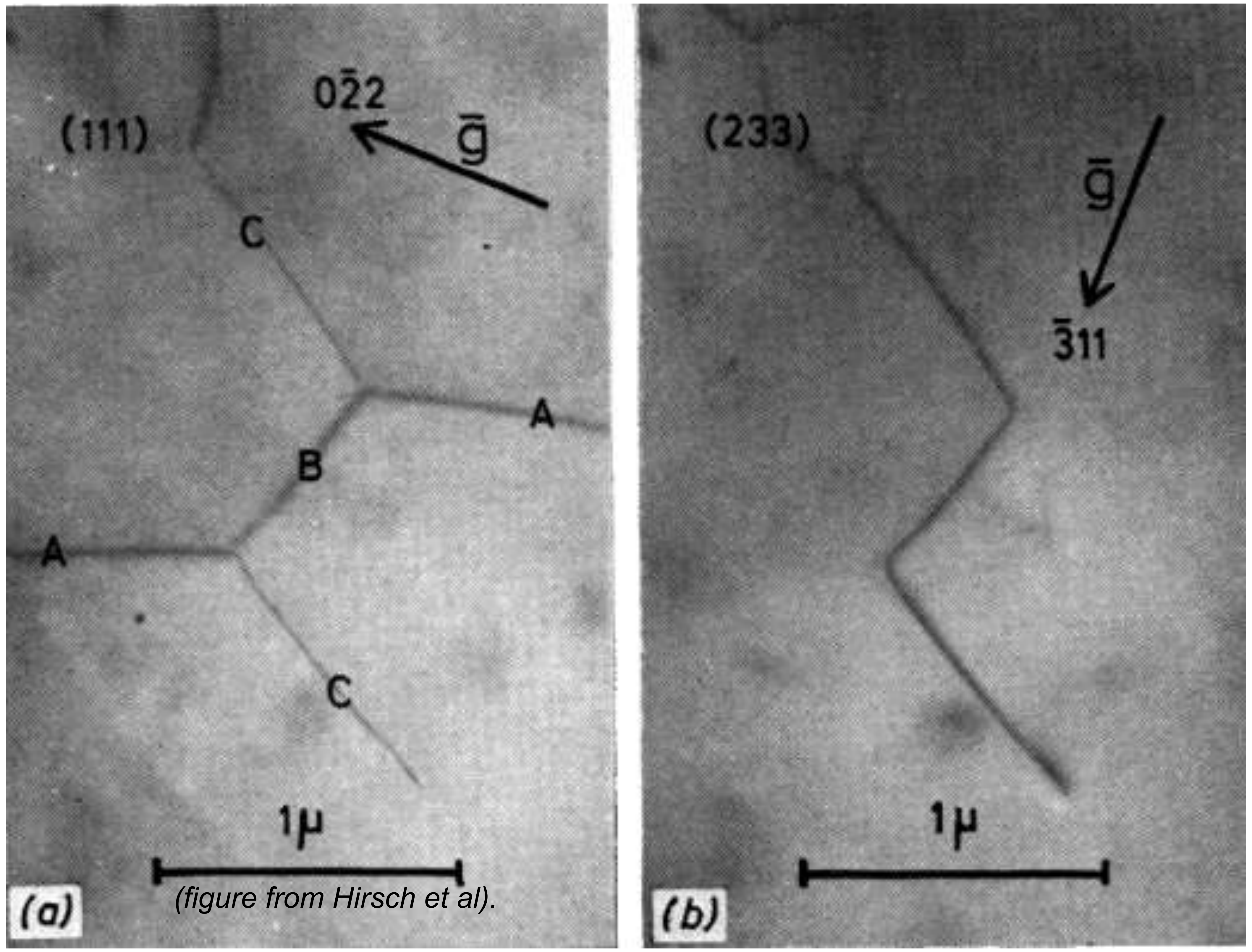
Weak beam imaging :some examples





Characterization of dislocation using CTEM





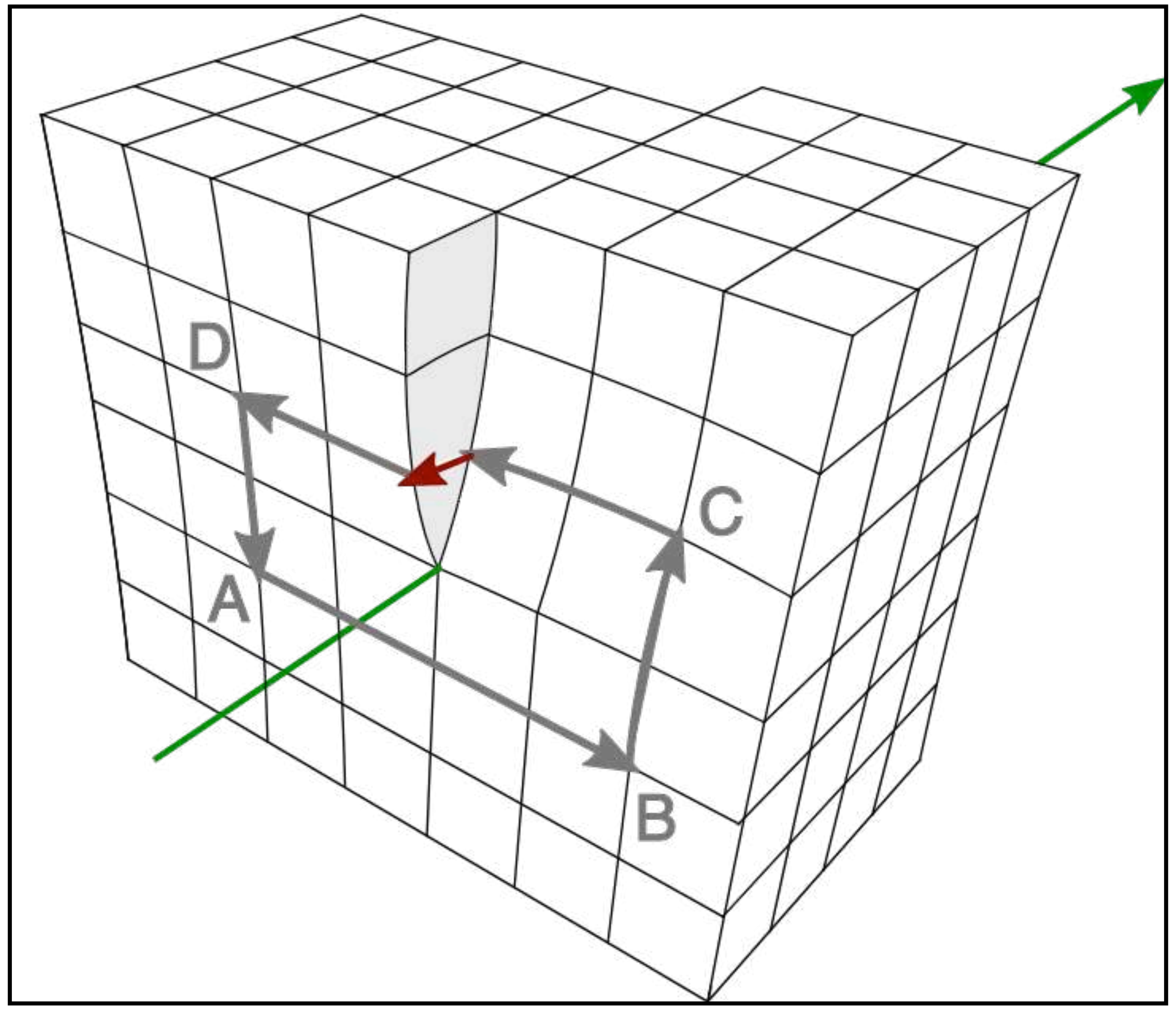
(figure from Hirsch et al).

Screw Dislocations in Si

Two BF images taken with different diffraction conditions

- $\vec{g} = 022$
- $\vec{g} = 311$

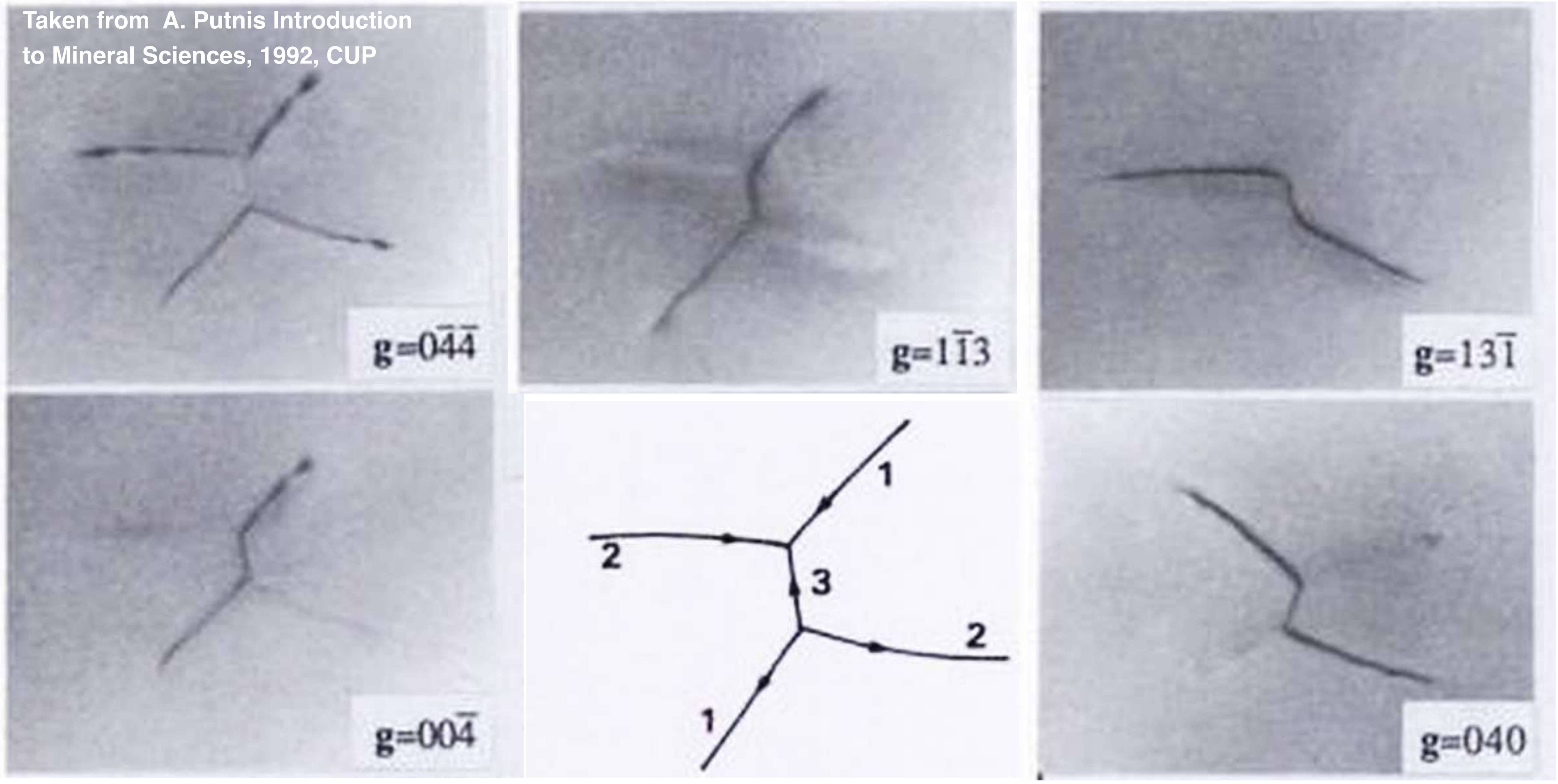
➡The dislocation marked A is invisible in (b)



Edge dislocations

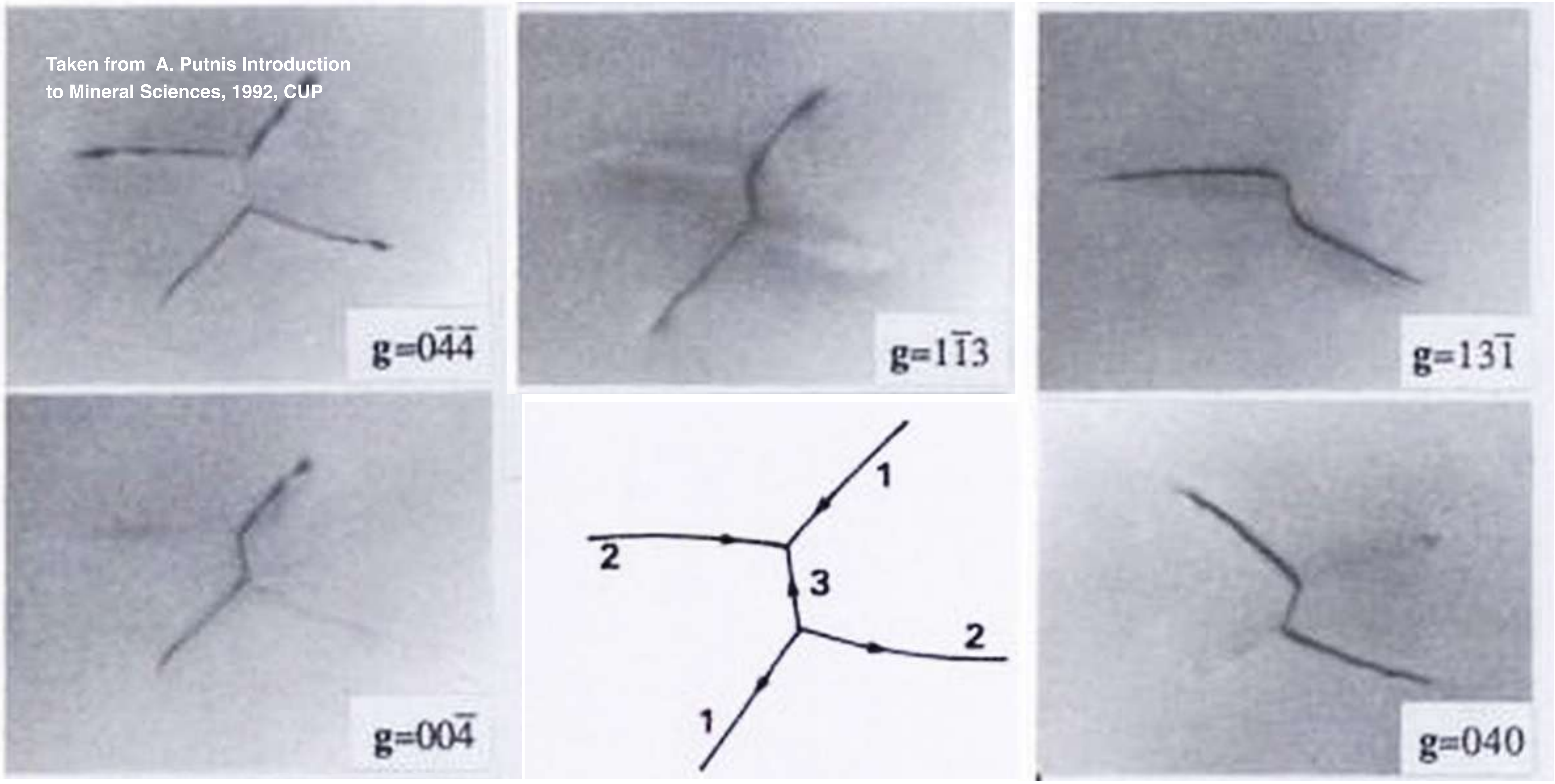
Edge dislocations are not as straightforward
Even if $\vec{g} \, . \, \vec{b} = 0$, then there might still be some component of displacement causing diffraction from the $\vec{g} \, . \, (\vec{b} \wedge \vec{L}) = 0$ term

Characterization of dislocation using CTEM : exercice



$\vec{g}$ \ $\vec{b}$	$\frac{1}{2}[110]$	$\frac{1}{2}[101]$	$\frac{1}{2}[011]$	$\frac{1}{2}[1\bar{1}0]$	$\frac{1}{2}[10\bar{1}]$	$\frac{1}{2}[01\bar{1}]$	1	2	3
$13\bar{1}$	2	0	1	-1	1	2	INVIS	VIS.	VIS
040	2	0	2	-2	0	2	INVIS	VIS.	VIS
$1\bar{1}3$	0	2	1	1	-1	-2	VIS.	INVIS	VIS
$00\bar{4}$	0	-2	-2	0	2	2	VIS.	INVIS	VIS
$0\bar{4}\bar{4}$	2	2	4	-2	-2	0	VIS.	VIS	INVIS

Characterization of dislocation using CTEM : solution



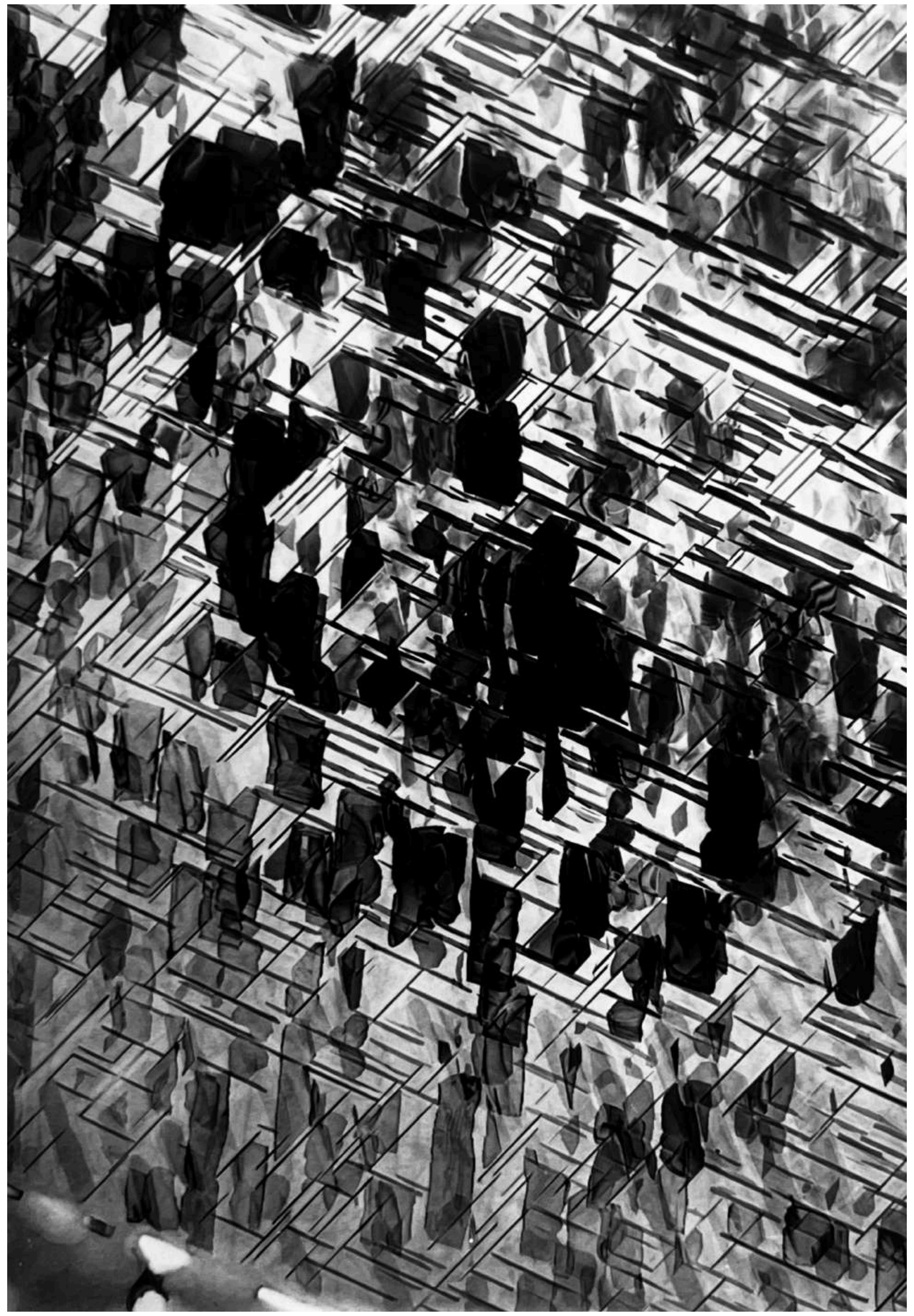
$$\vec{b}_1 = \frac{a}{2}[101]$$

$$\vec{b}_2 = \frac{a}{2}[110]$$

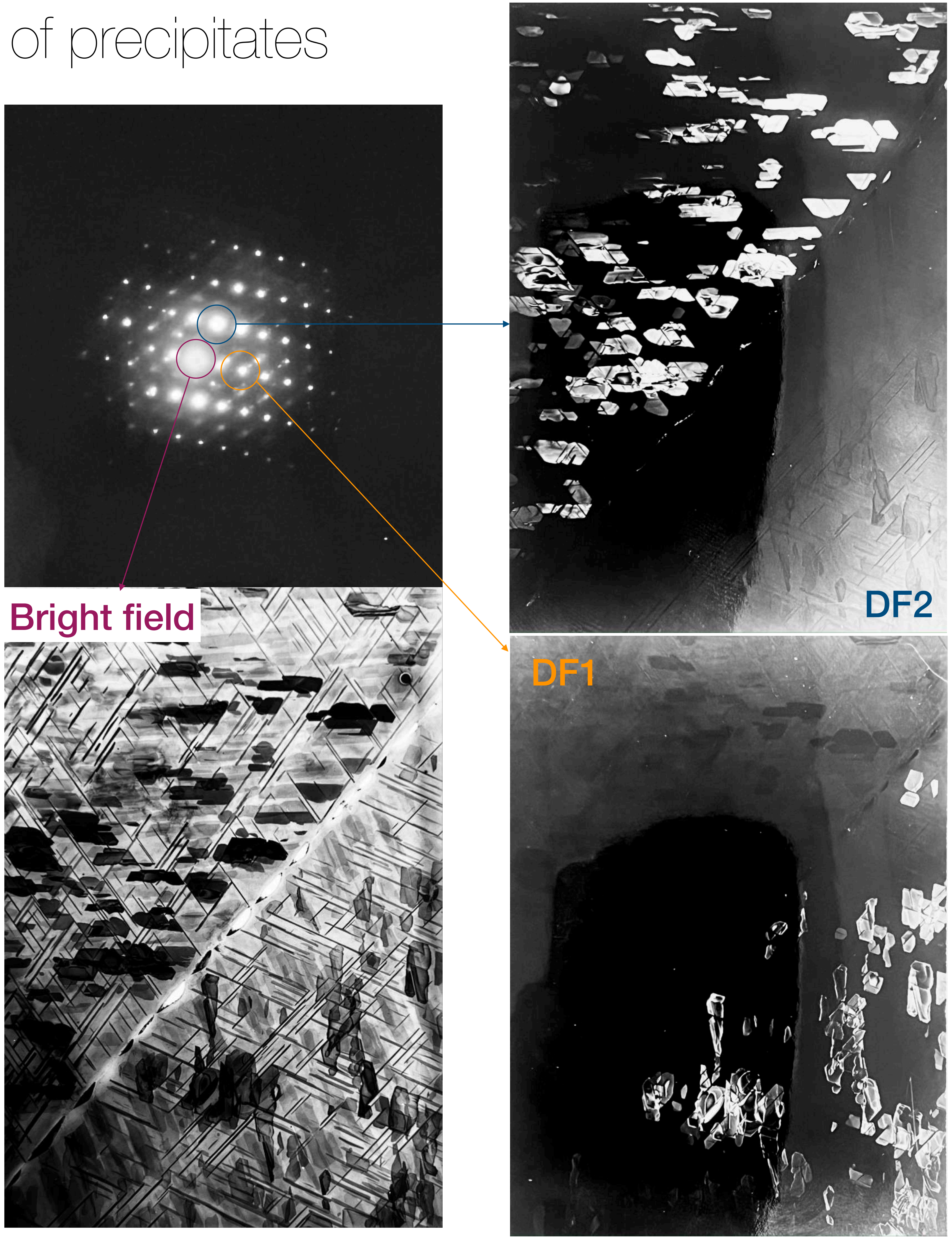
$$\vec{b}_3 = \frac{a}{2}[01\bar{1}]$$

Conventional observation of precipitates

Bright field



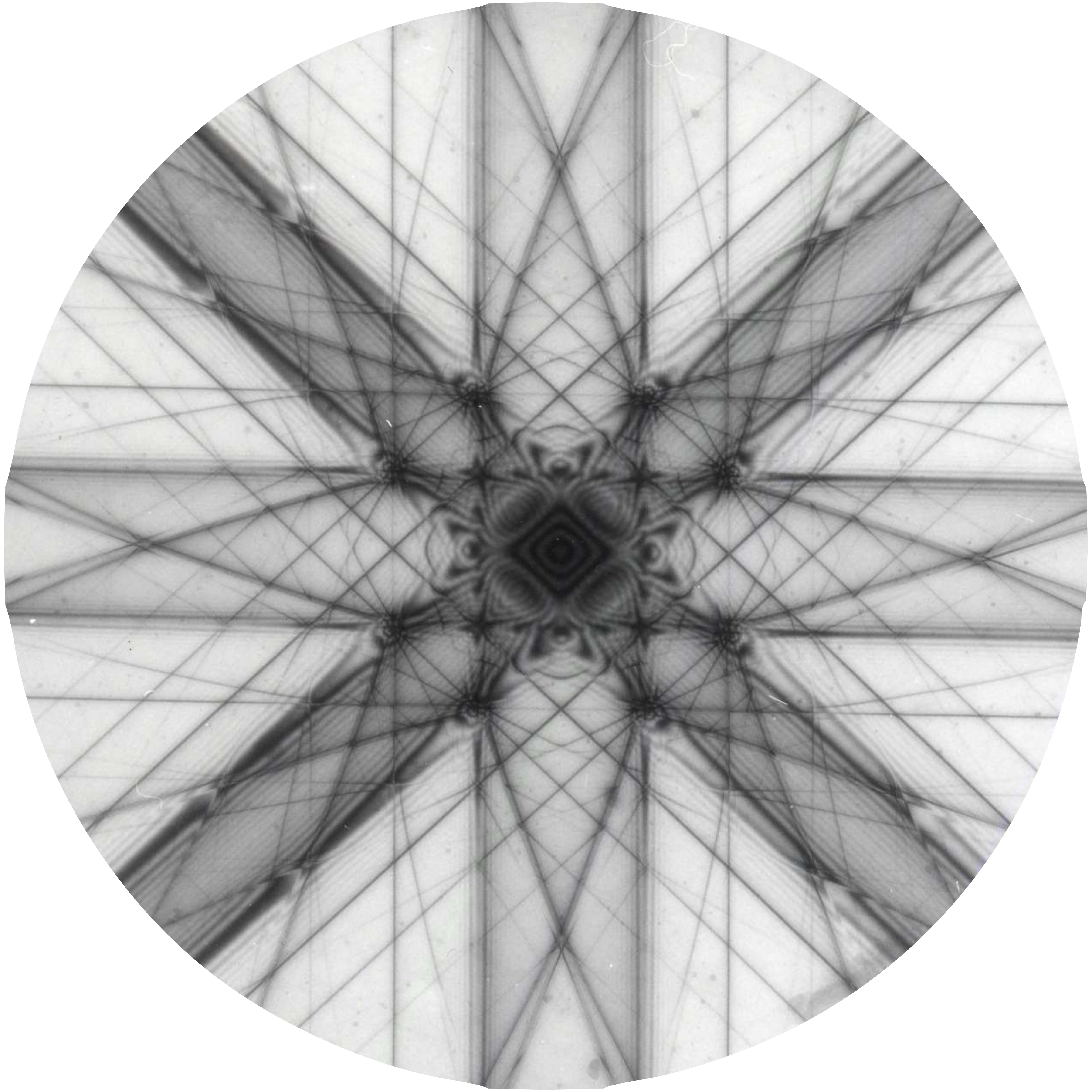
Dark Field



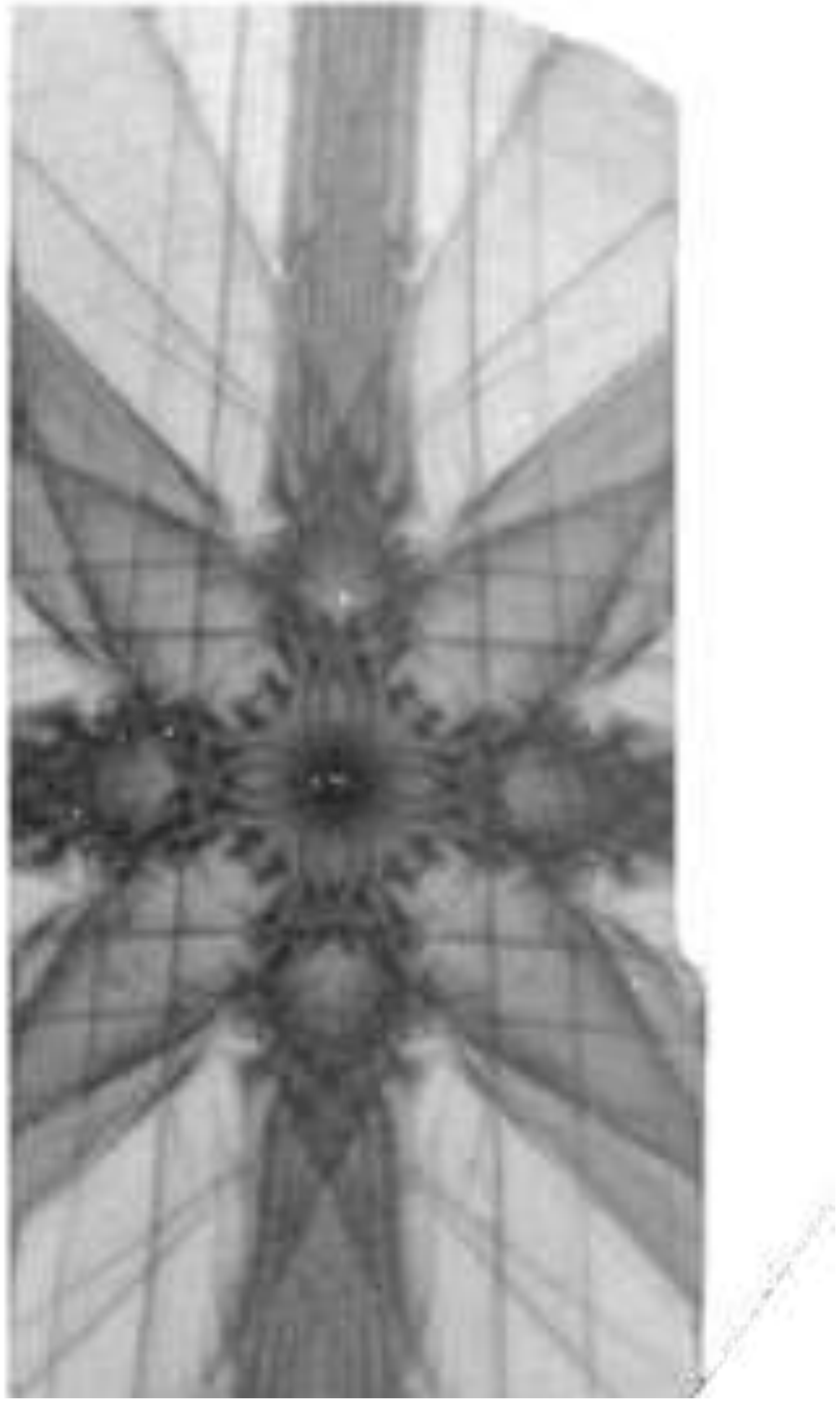
Conclusion

Understanding diffraction contrast in CTEM requires an understanding of how diffraction intensities are generated

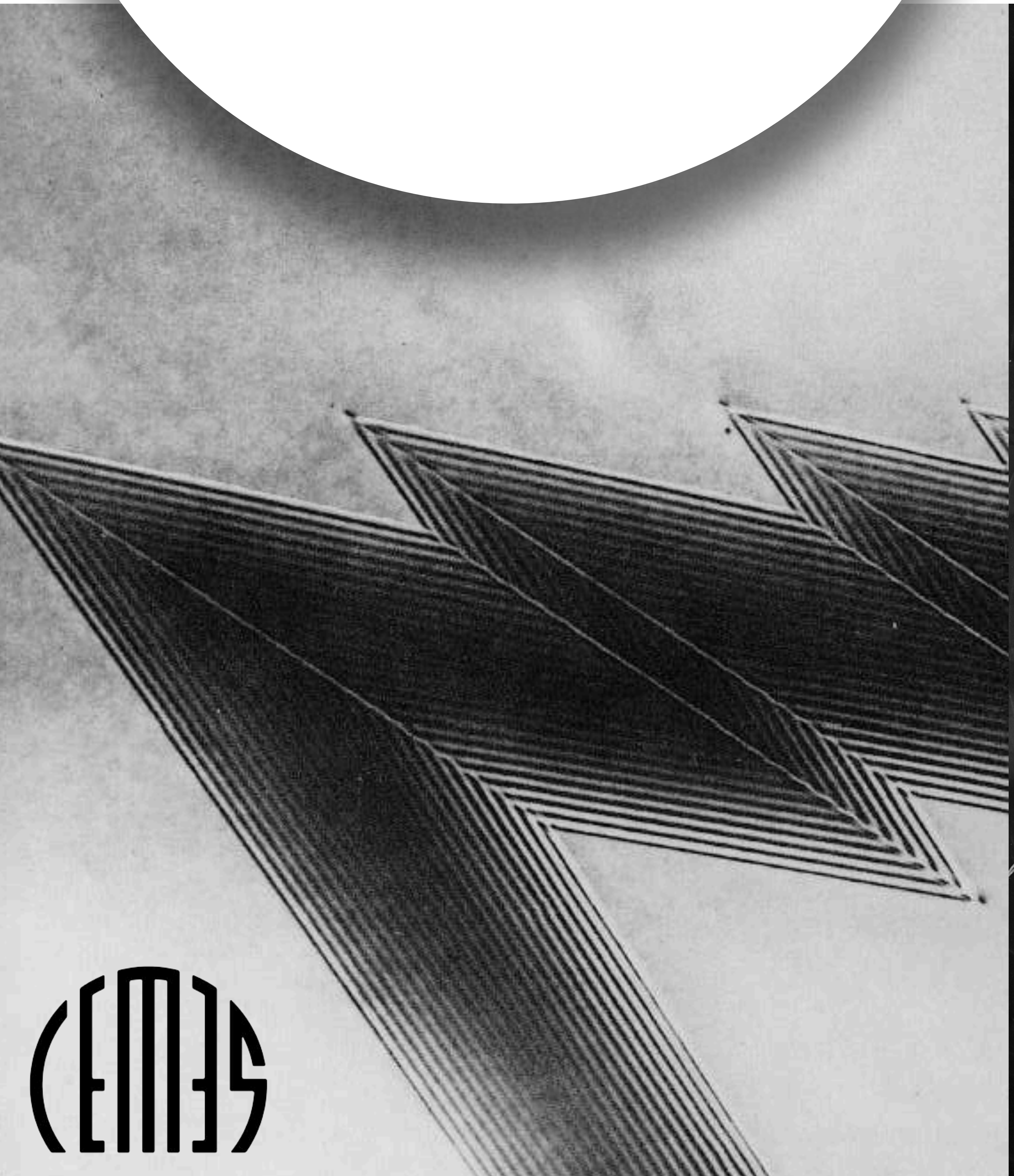
Diffraction

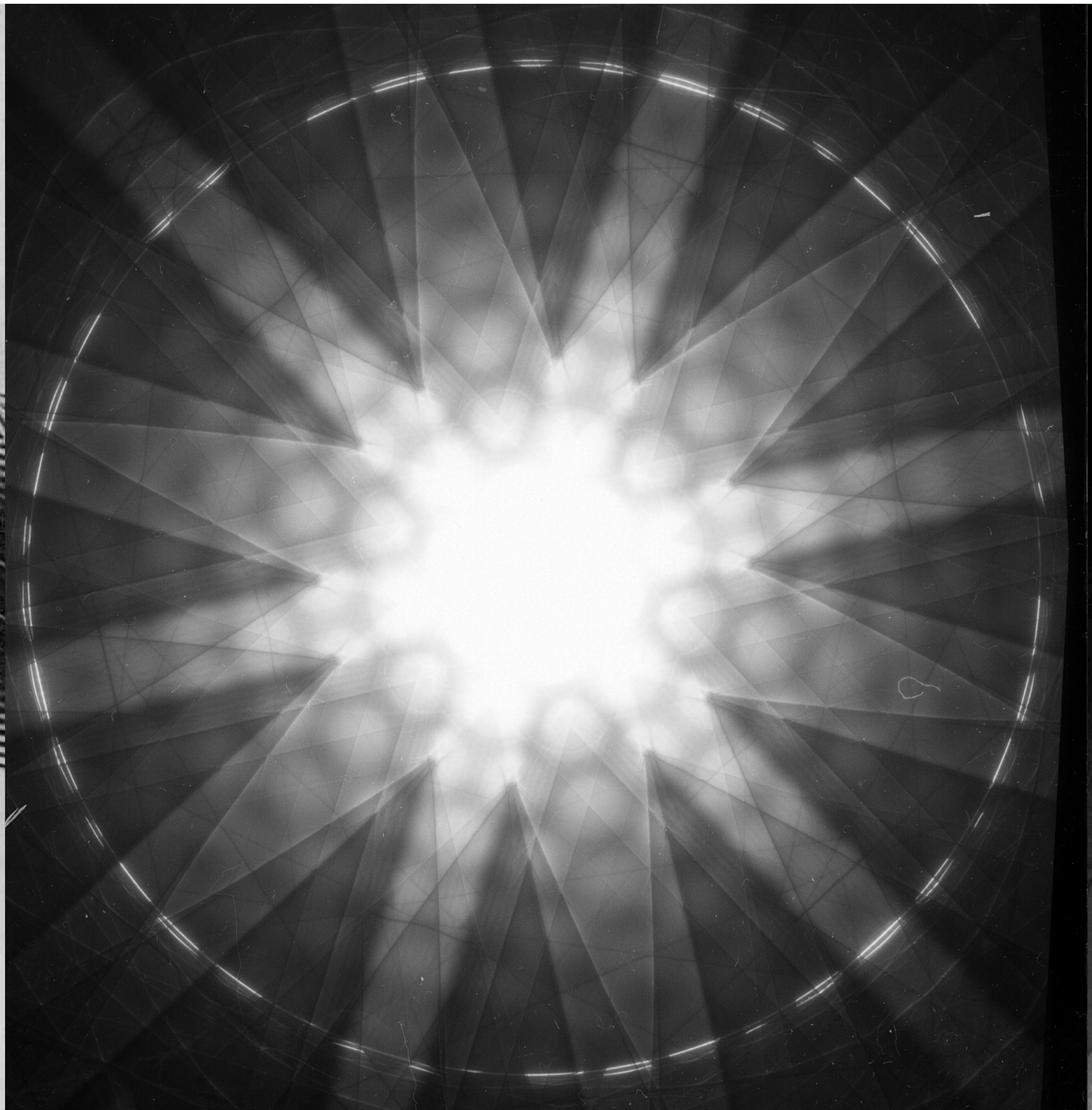


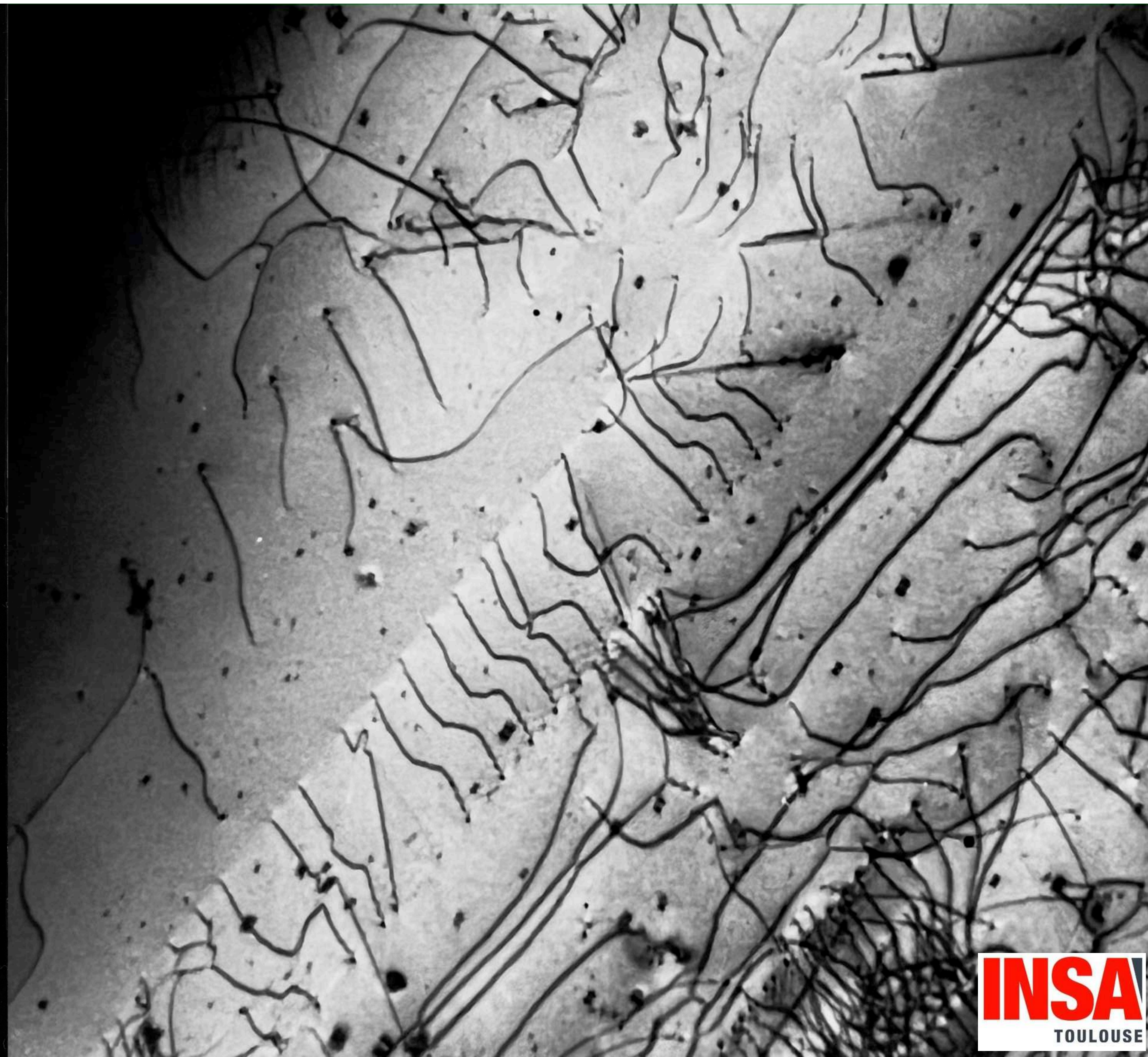
Bright field

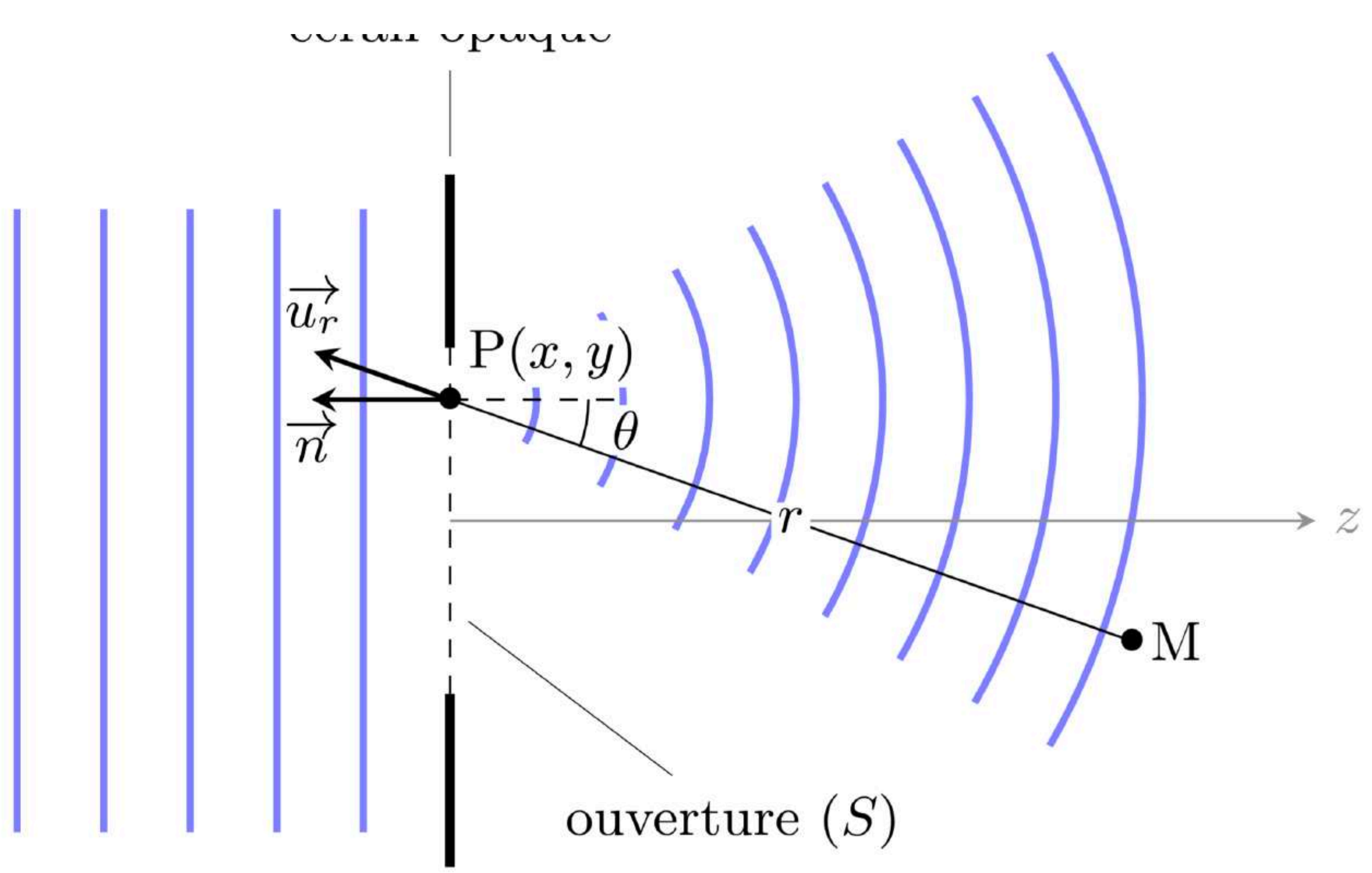


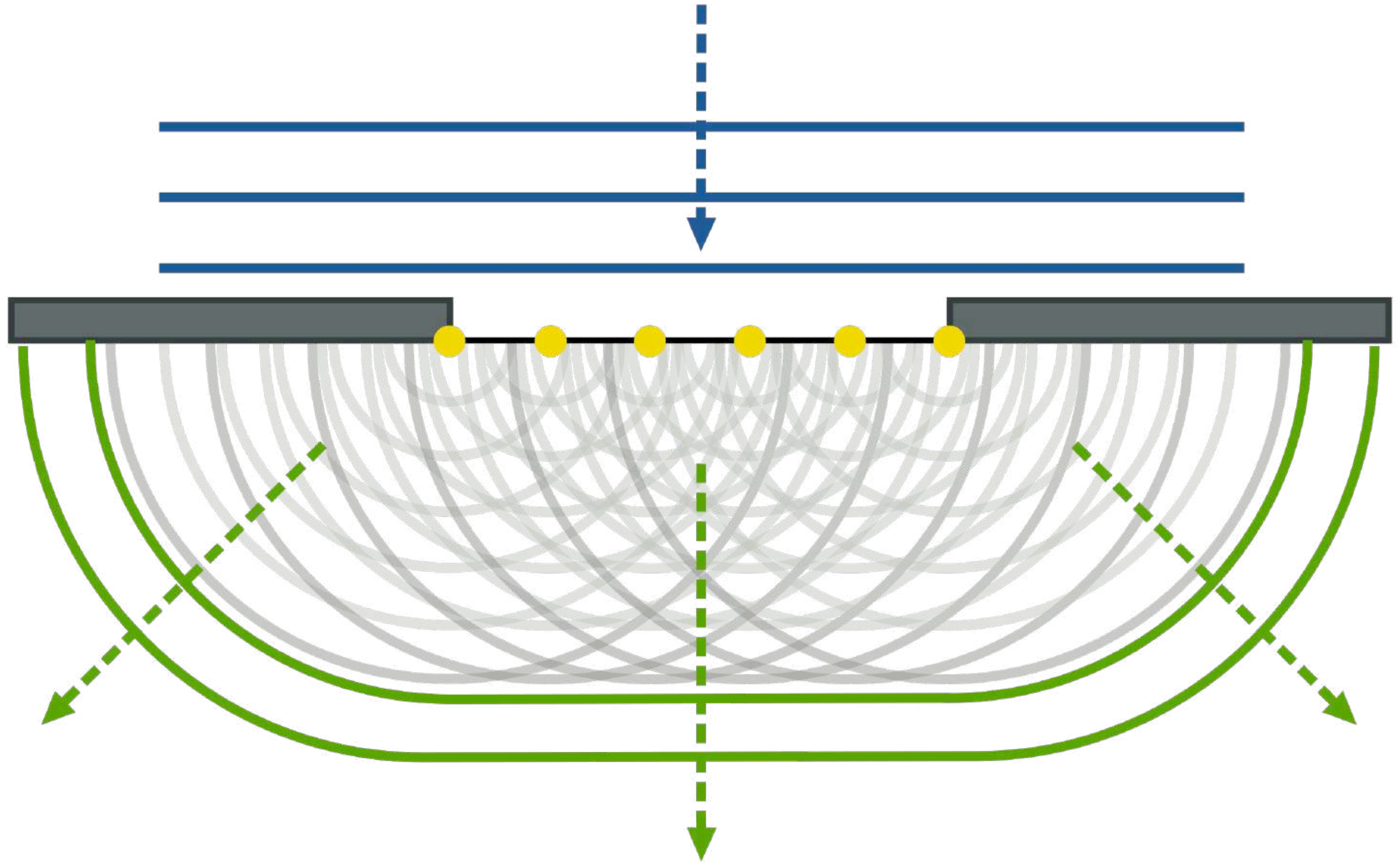
Thank you

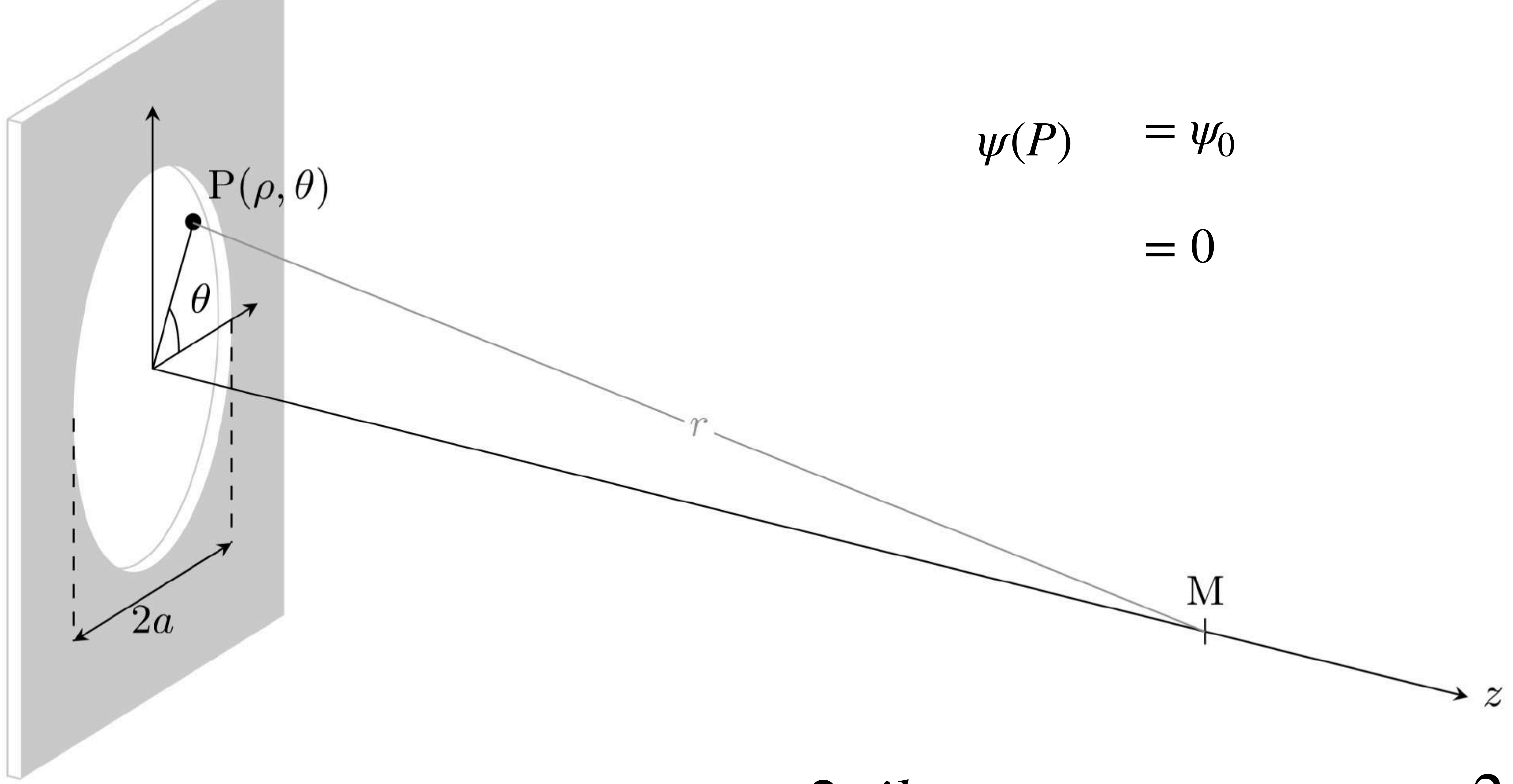







écran opaque
P(x, y)
θ
r
z
M
ouverture (S)





$$\psi(P) = \psi_0$$
$$= 0$$

$$\psi(M) = \iint_S K(\theta)\psi(P)\frac{e^{2\pi ikr}}{r}d\tau = K(\theta)\psi_0\int_{\theta=0}^{2\pi}\int_{\rho=0}^{a}\frac{e^{-ik\sqrt{\rho^2+z^2}}}{\sqrt{\rho^2+z^2}}\rho d\rho d\theta$$

Le calcul de l'intégrale double se découple en un produit de deux intégrales simples (théorème de Fubini)

$$\psi(M) = K(\theta)\psi_0 2\pi\int_{\rho=0}^{a}\frac{e^{-ik\sqrt{\rho^2+z^2}}}{\sqrt{\rho^2+z^2}}\rho d\rho d\theta = K(\theta)\psi_0 2\pi\left[\frac{i}{k}e^{-ik\sqrt{\rho^2+z^2}}\right]_0^a$$