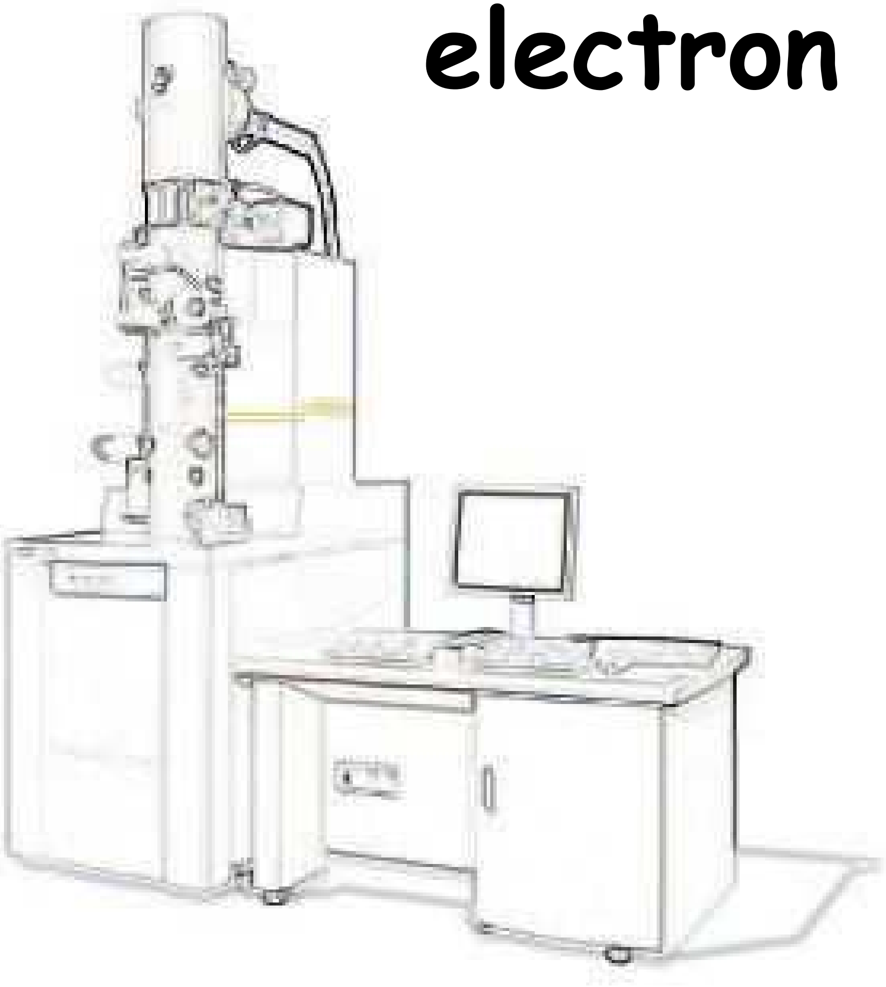
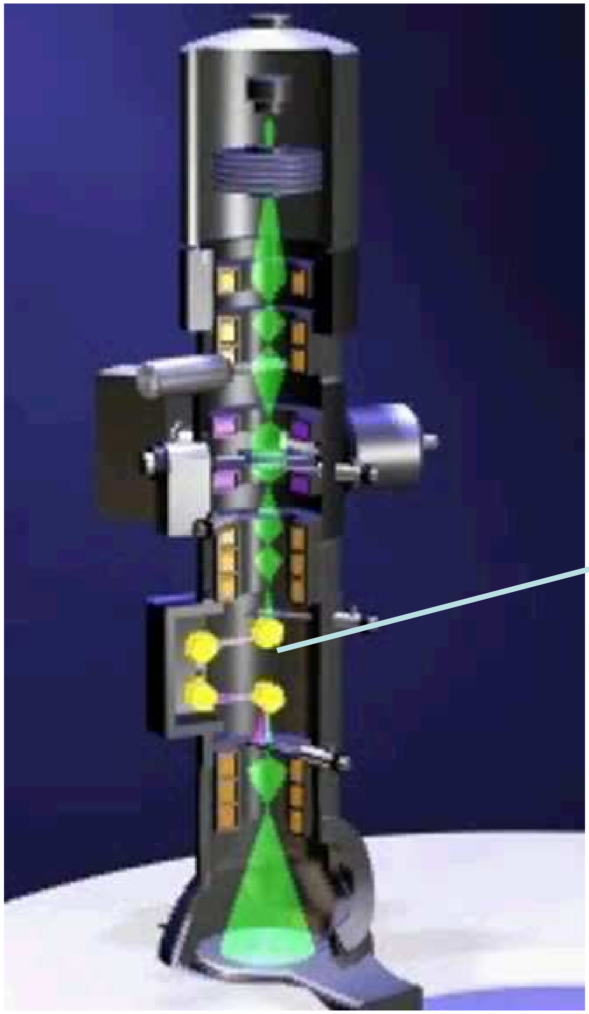
# High resolution transmission electron microscopy

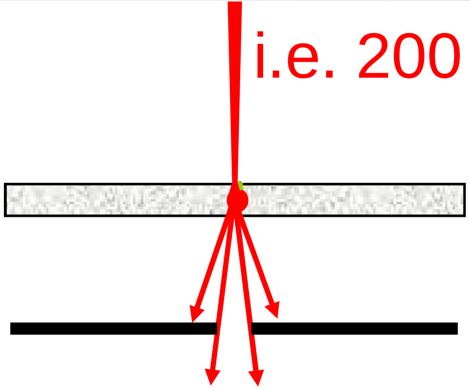


# Overview

- Last time we had a look at inelastic interactions between the electron beam and the sample…

# Electron - Sample Interactions

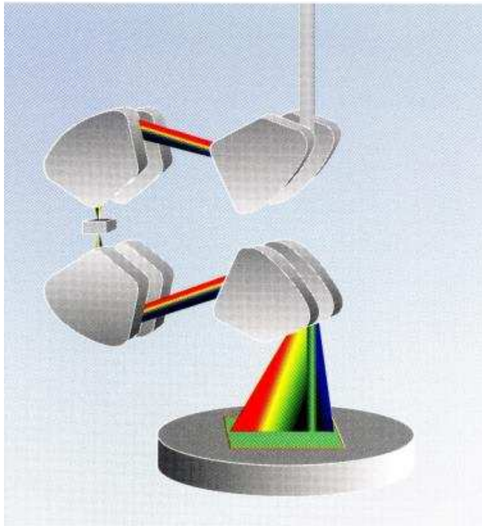


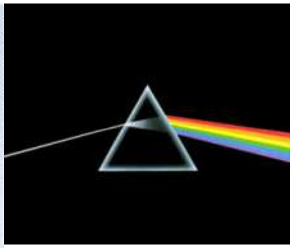


i.e. 200 keV

**Sample**

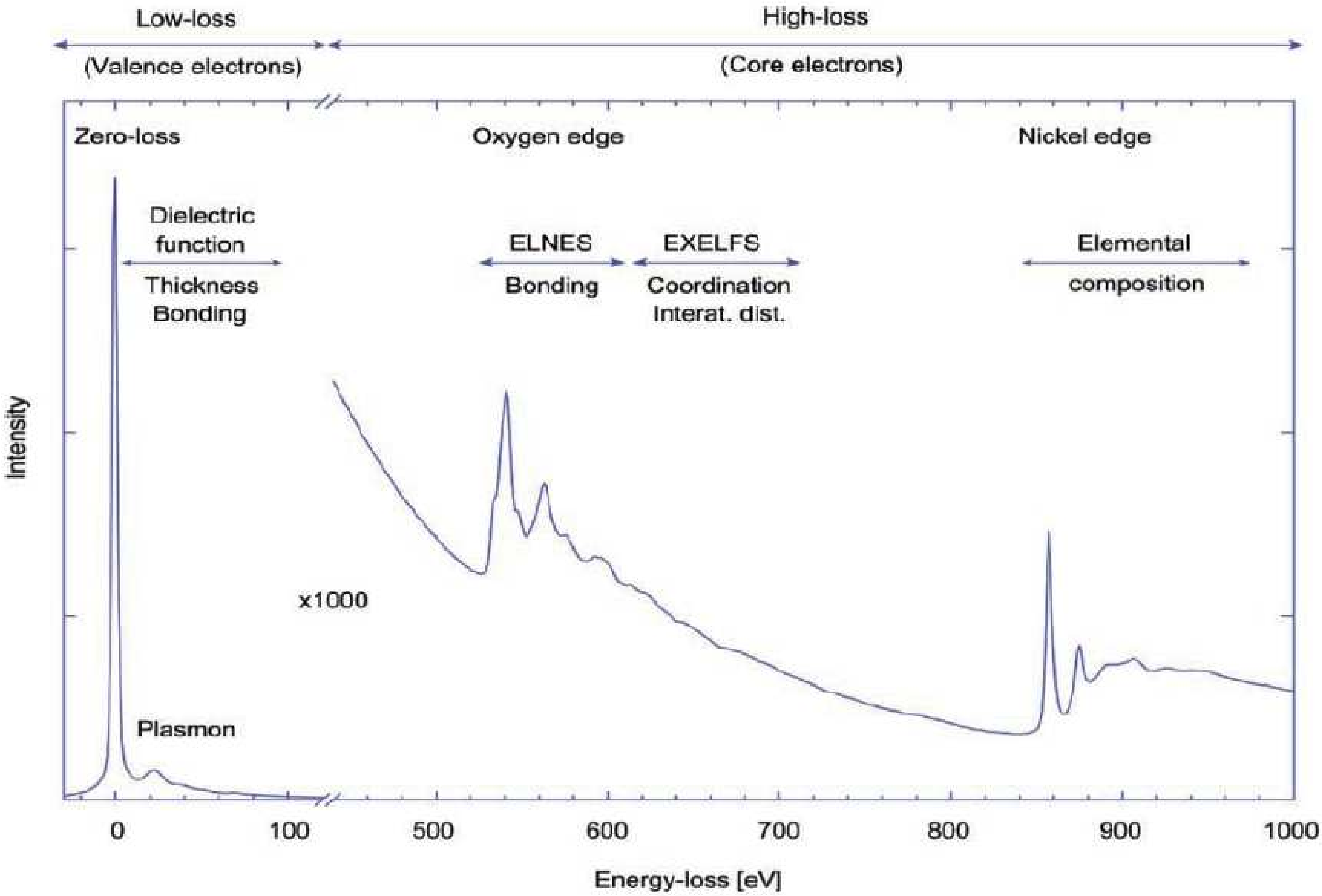
**Angle-limiting aperture**



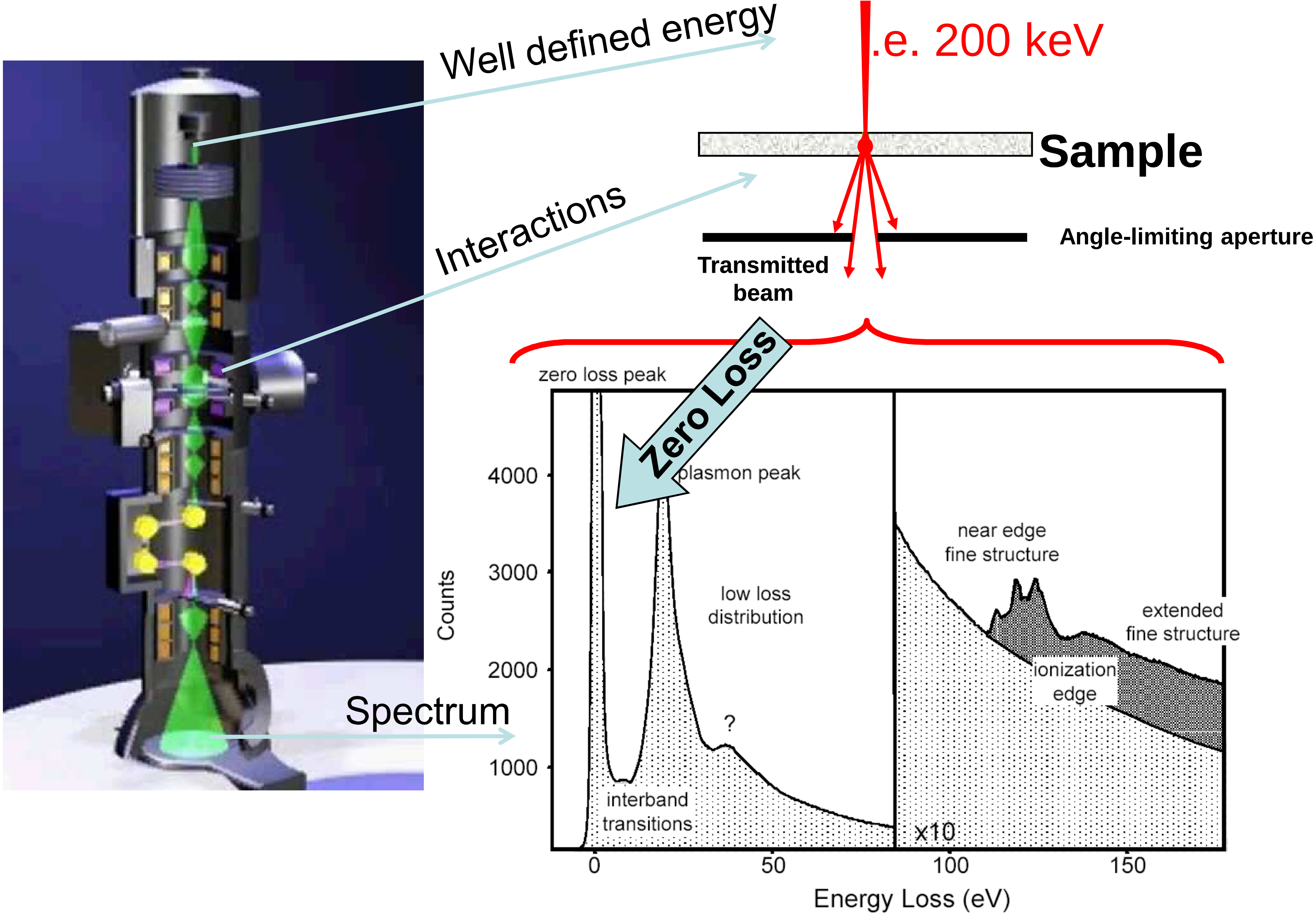


**E**lectron
**E**nergy
**L**oss
**S**pectrum

# Energy Distribution = Energy Loss Spectrum



# Energy Loss Spectrum



Well defined energy

i.e. 200 keV

**Sample**

Interactions

**Angle-limiting aperture**

**Transmitted beam**

**Zero Loss**

Spectrum

# Overview

- Last time we had a look at inelastic interactions between the electron beam and the sample
- This time, we focus more on the image generation and interpretation

# Why electrons?

Smallest visible objects…

-with eye : 0.1 mm = $10^{-4}$ m
(size of one eye «"stick"»)

- with light microscope ~ 300nm
(magnification max ~ 2000x)

**Can we simply magnify the image of an object to observe every detail ?**

Abbe’s equation:

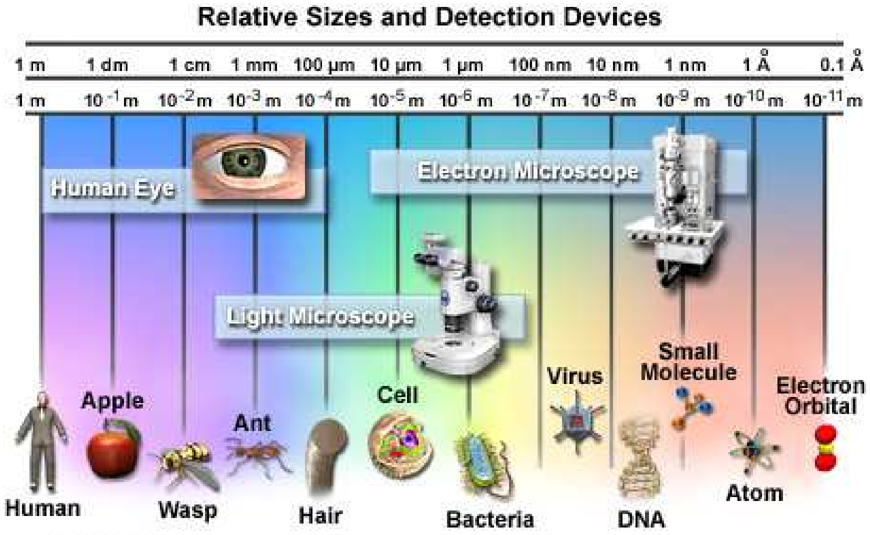
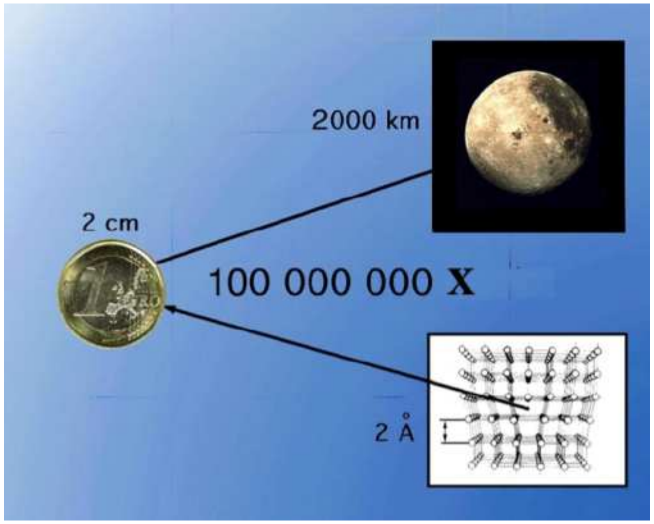
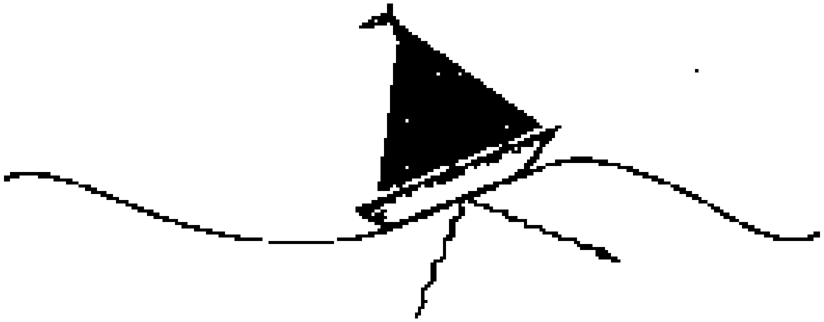


Figure 1



# Why electrons?

The interaction of waves with an obstacle:

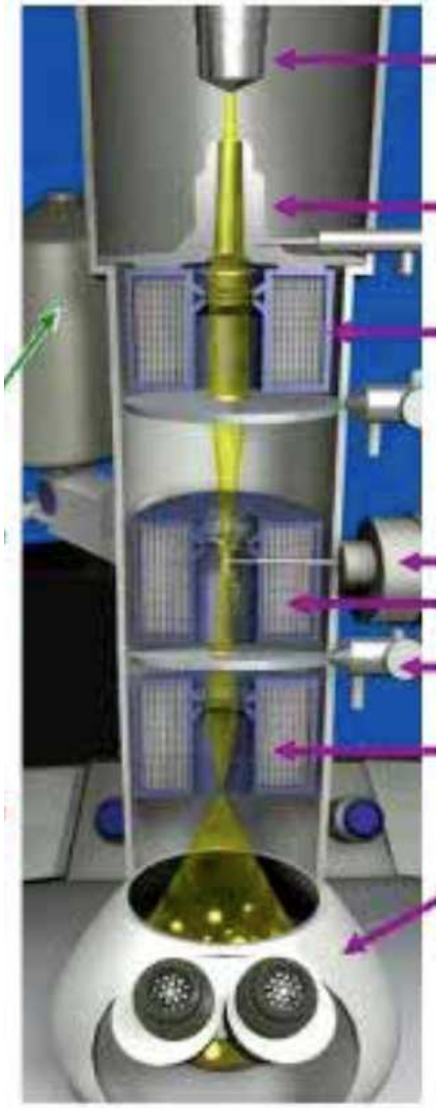


The boat rides the long wavelength ocean wave, but reflects the small wavelength surface ripple. An observer who wishes to detect the presence of the boat can do so only by observing waves which have wavelengths smaller than, or comparable to, the length of the boat. (From Sherwood, p.19)

Waves on water surface

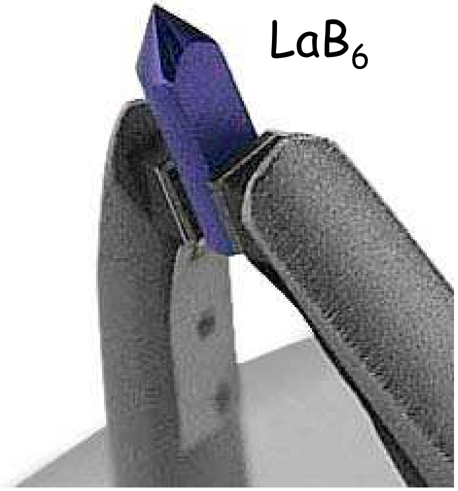
# Ok, so lets use electrons!

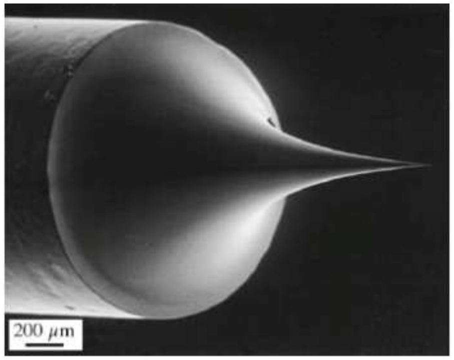
# Basic set-up of the TEM



Electron gun:

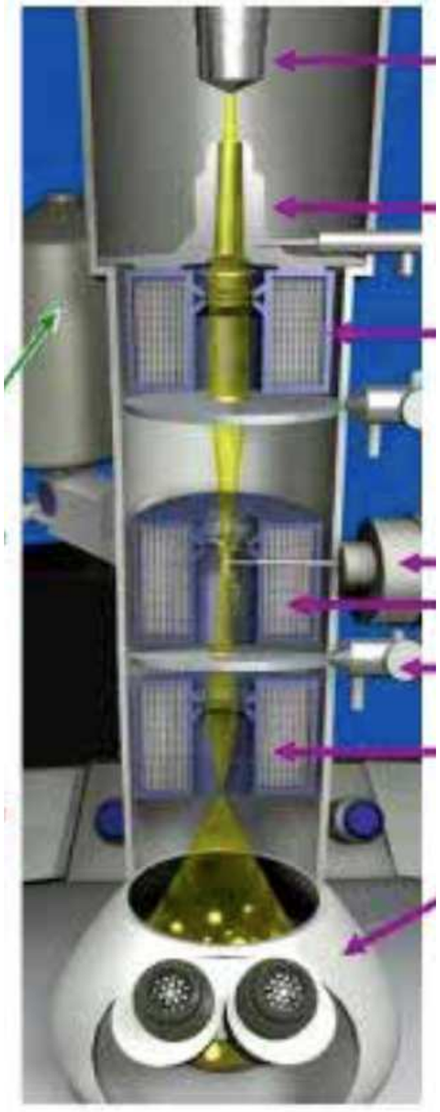
**The electron gun produces a beam of monochromatic (coherent) electrons!!**





a field-emission source: extraordinarily fine W needle

# Basic set-up of the TEM



Electron gun

Acceleration stage:

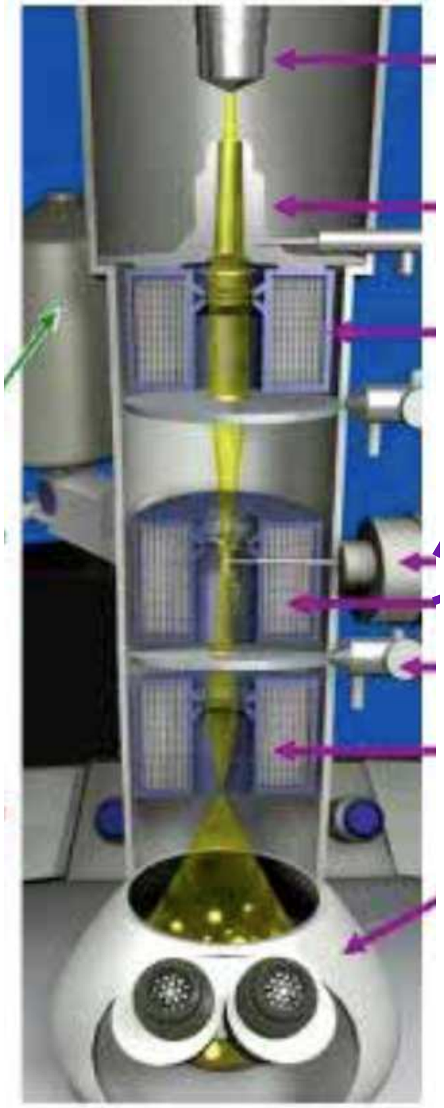
High voltage accelerates the electrons to high kinetic energy.

Wavelength of high-energy electrons:

| $E$ kV | $\gamma$ | $\lambda$ pm | $\frac{v}{c}$ |
|---|---|---|---|
| 50 | 1.098 | 5.362 | 0.412 |
| 100 | 1.119 | 3.706 | 0.548 |
| 200 | 1.391 | 2.511 | 0.695 |
| 500 | 1.978 | 1.423 | 0.862 |
| 1000 | 2.957 | 0.873 | 0.941 |

Abbe's equation!

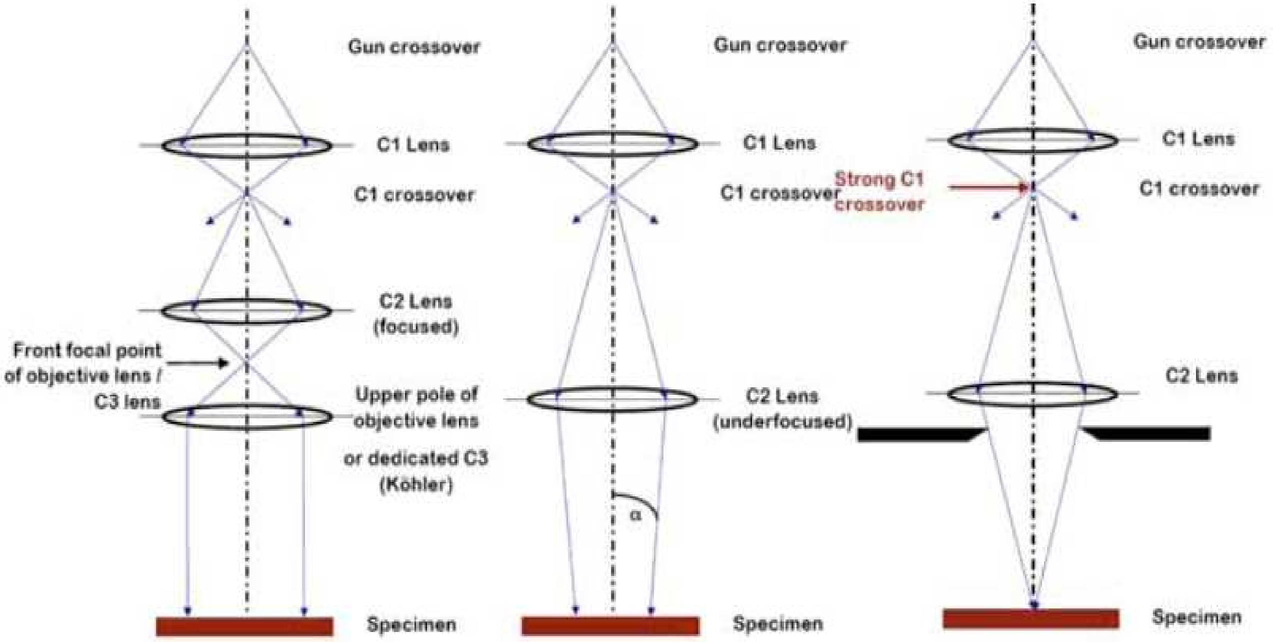
# Basic set-up of the TEM



Electron gun

Acceleration stage

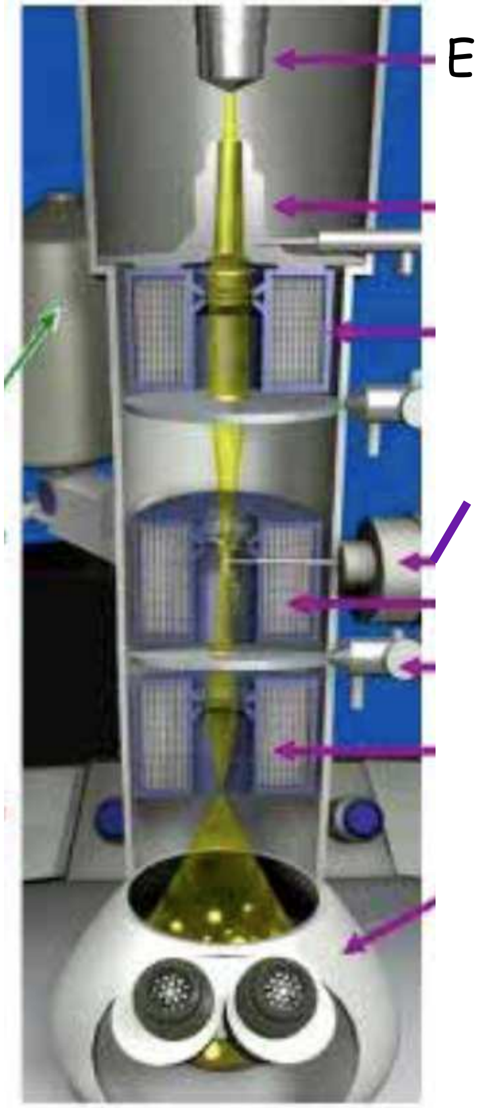
Condenser lens system:



Parallel or converging illumination of the specimen

**M. Willinger, FHI**

# Basic set-up of the TEM



Electron gun

Acceleration stage

Condenser lens system

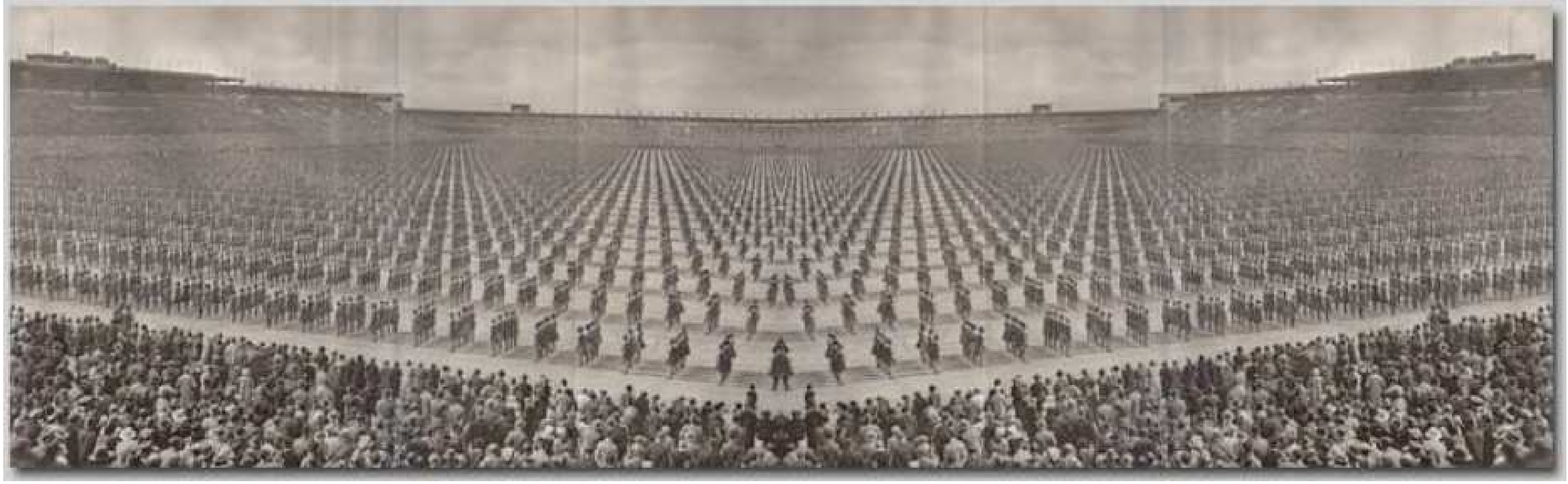
Specimen stage:

Now things get interesting!

M. Willinger, FHI

# Coherence & interference

What is the (crystalline) sample to the electrons?

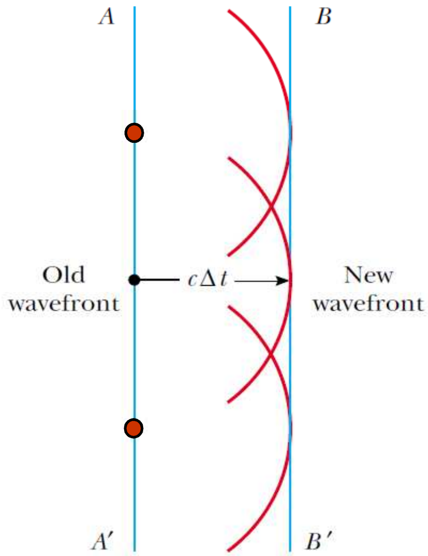


# Coherence & interference

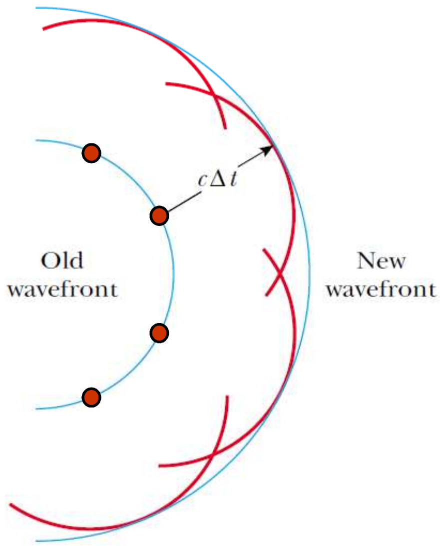
Huygens principle:

The Huygens principle states that every unobstructed point of a wavefront, at a given instant in time, act as a source of spherical secondary waves with same wavelength as that of the primary wave (**wavelets**).The amplitude in any point of the space beyond the obstacle is the superposition of all these wavelets (considering their amplitudes and relative phases).

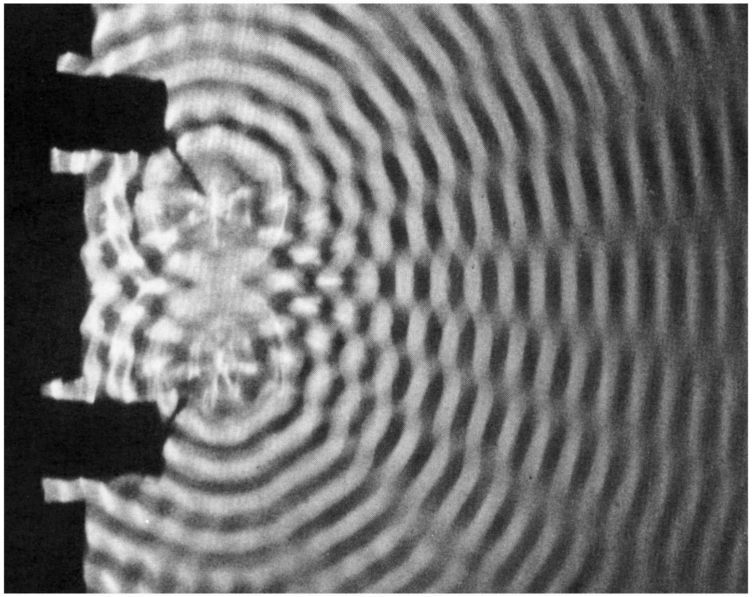
Christiaan
Huygens
1629 – 1695



Propagation of a plane wave

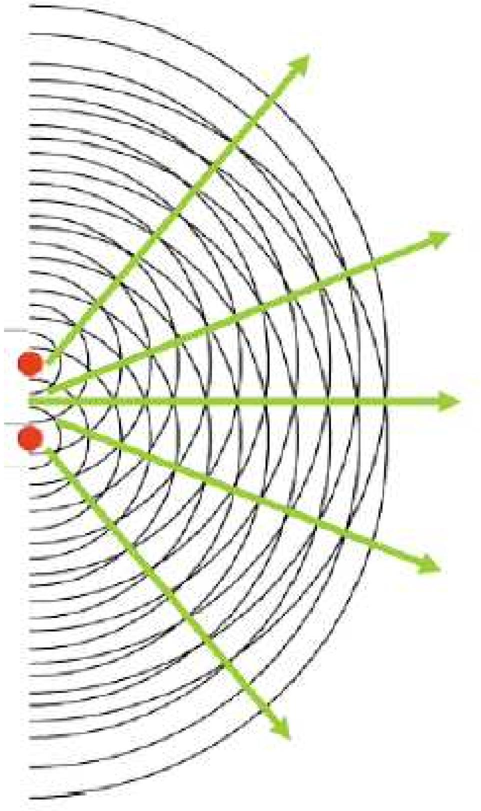


Propagation of a spherical wave

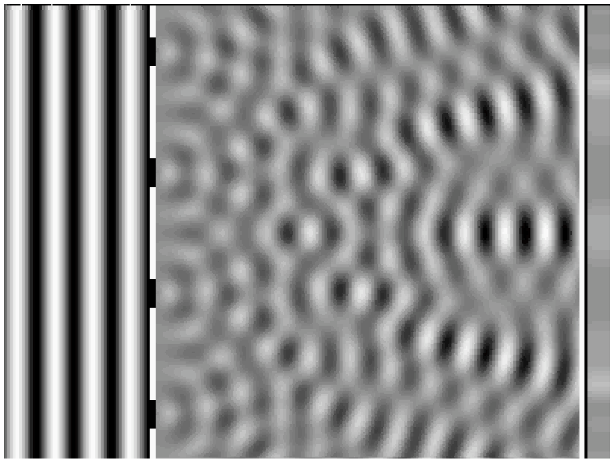


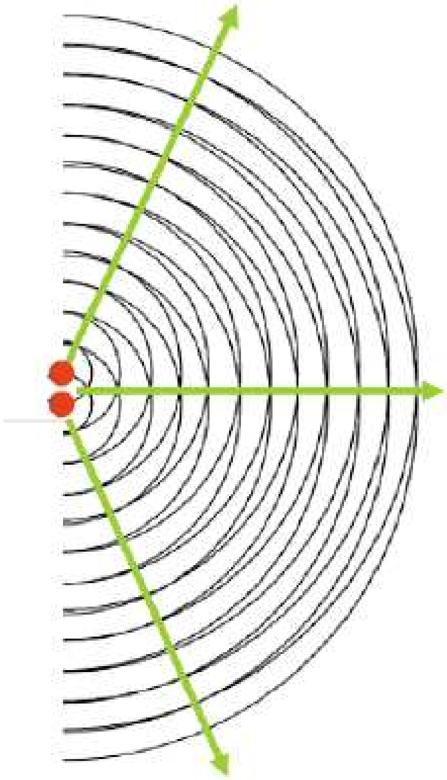
# Coherence & interference

## Scattering by two scattering centers



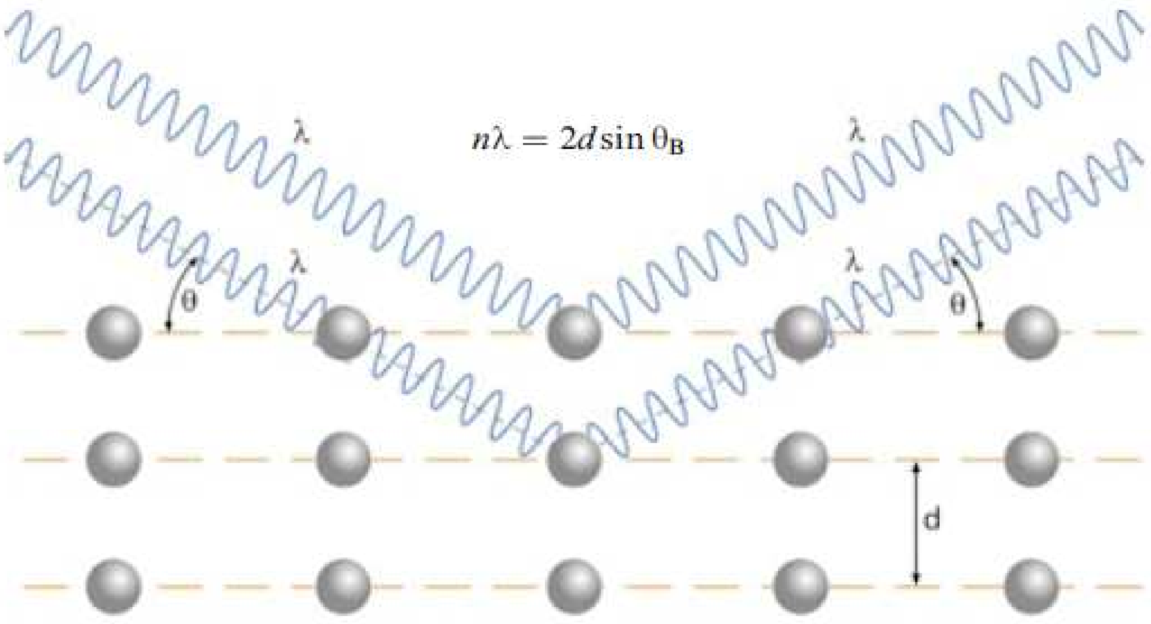
large distance apart

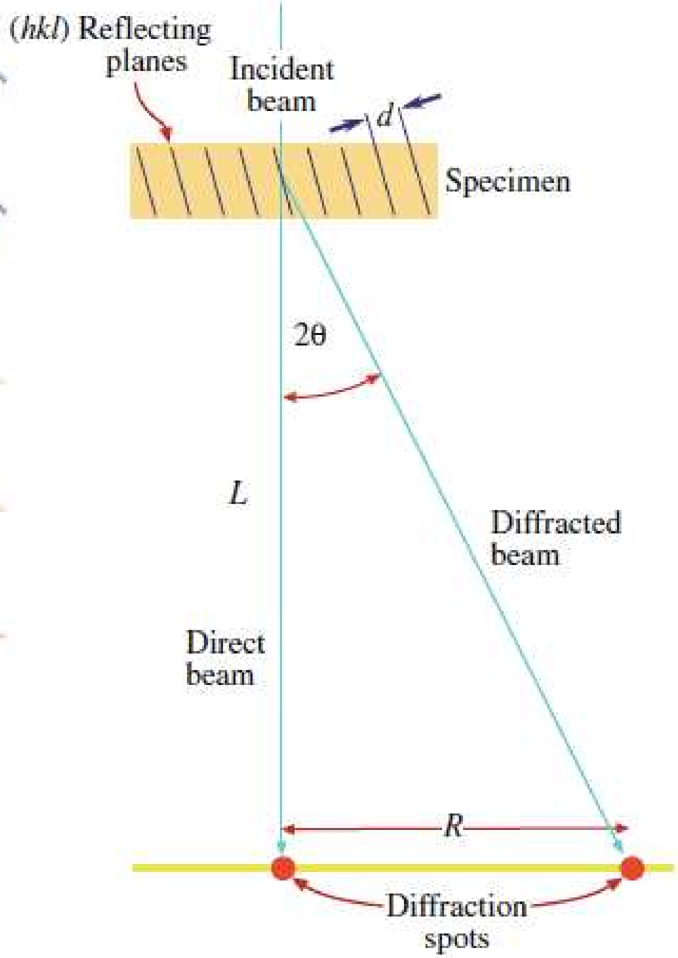




small distance apart

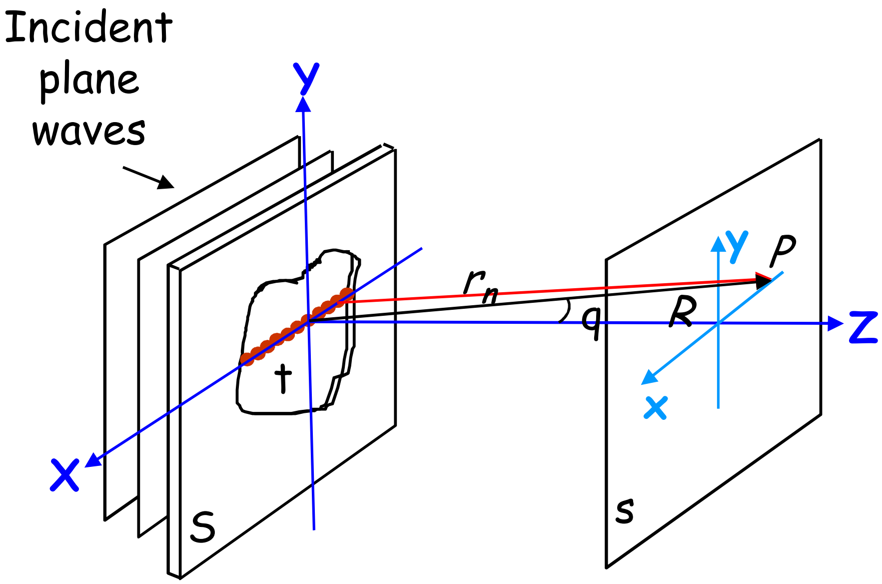
# Coherence & interference

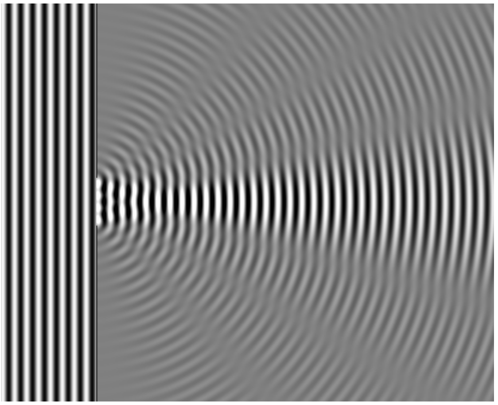


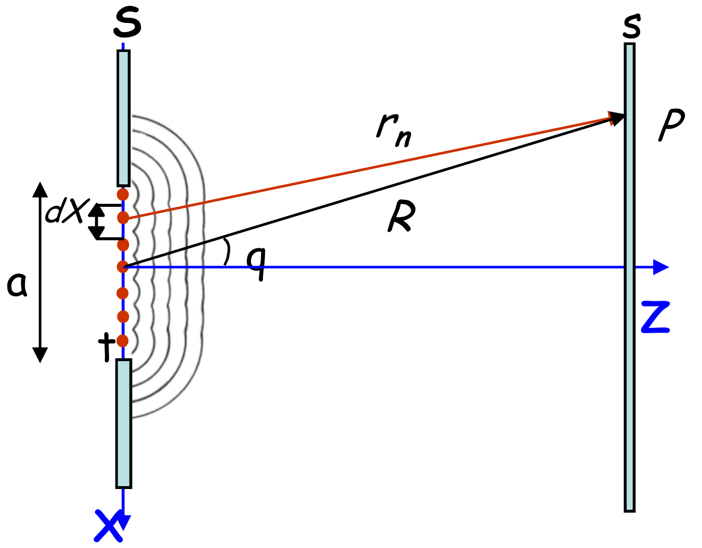


Nothing new… good old Bragg!

# Coherence & interference





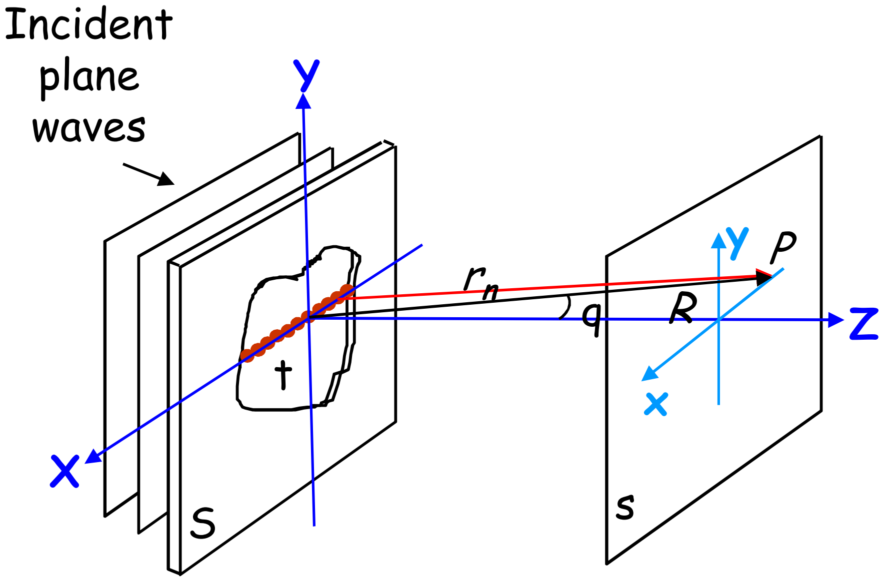


The amplitude at point *P* will be the superposition of all wavelets emitted by the aperture. The contribution of one linear segment *dx* of these secondary sources will be:

$$d\psi_P = \frac{A'_n}{r_n} e^{i(\omega t - 2\pi k r_n)} dX$$

where $A'_n$ is the wavelet amplitude per unit length of the aperture

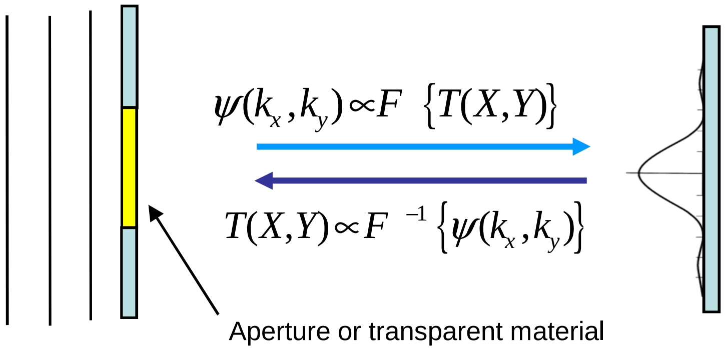
# Coherence & interference



The wave amplitude at any point *P* on the screen is given by:

$$\psi_P = \psi(k_x,k_y) = A_0 \int_{-\infty}^{+\infty}\!\!\int T_{(X,Y)}\, e^{i\,2\pi\left(k_x X + k_y Y\right)}\, dX\, dY$$

$$\psi(k_x,k_y) \propto F\left\{T_{(X,Y)}\right\}$$



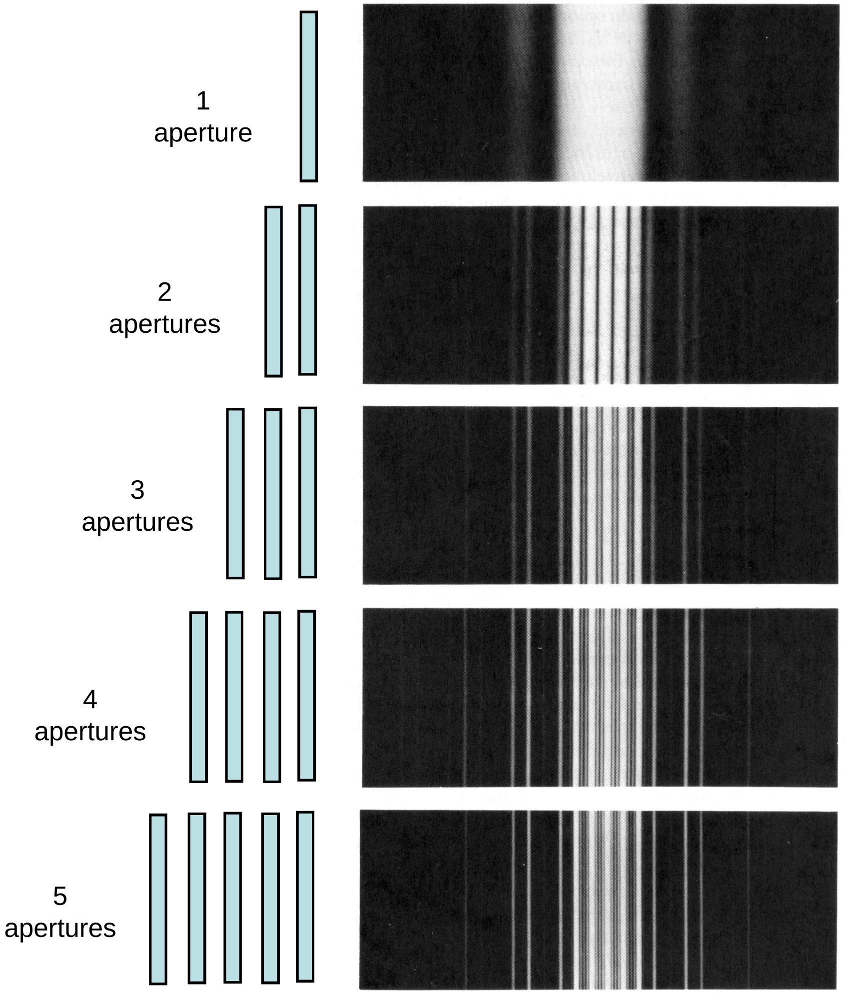
$$\psi(k_x,k_y) \propto F\{T(X,Y)\}$$

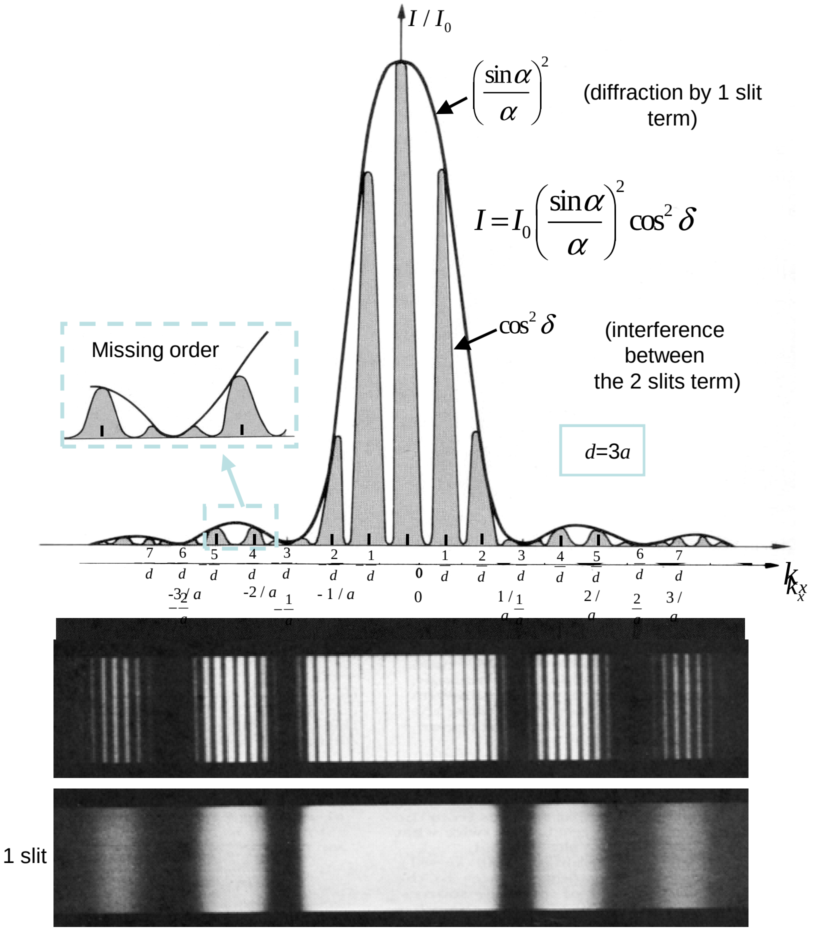
$$T(X,Y) \propto F^{-1}\{\psi(k_x,k_y)\}$$

***The Fraunhofer diffracted field is proportional to the Fourier transform of aperture function***

**M. Willinger, FHI**

# …demonstration FFT transformation





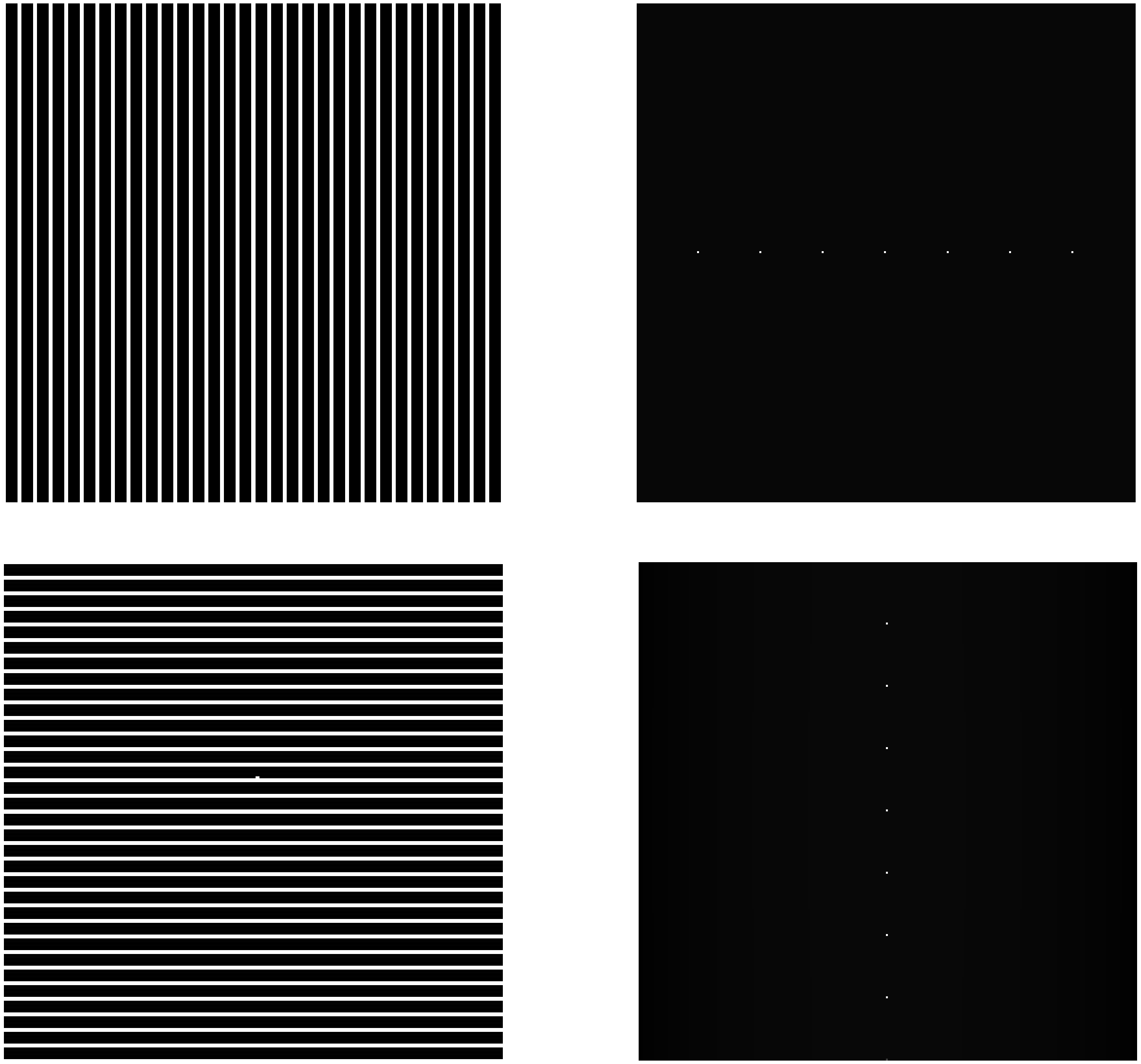
$\left(\frac{\sin\alpha}{\alpha}\right)^2$ (diffraction by 1 slit term)

$$I = I_0\left(\frac{\sin\alpha}{\alpha}\right)^2 \cos^2\delta$$

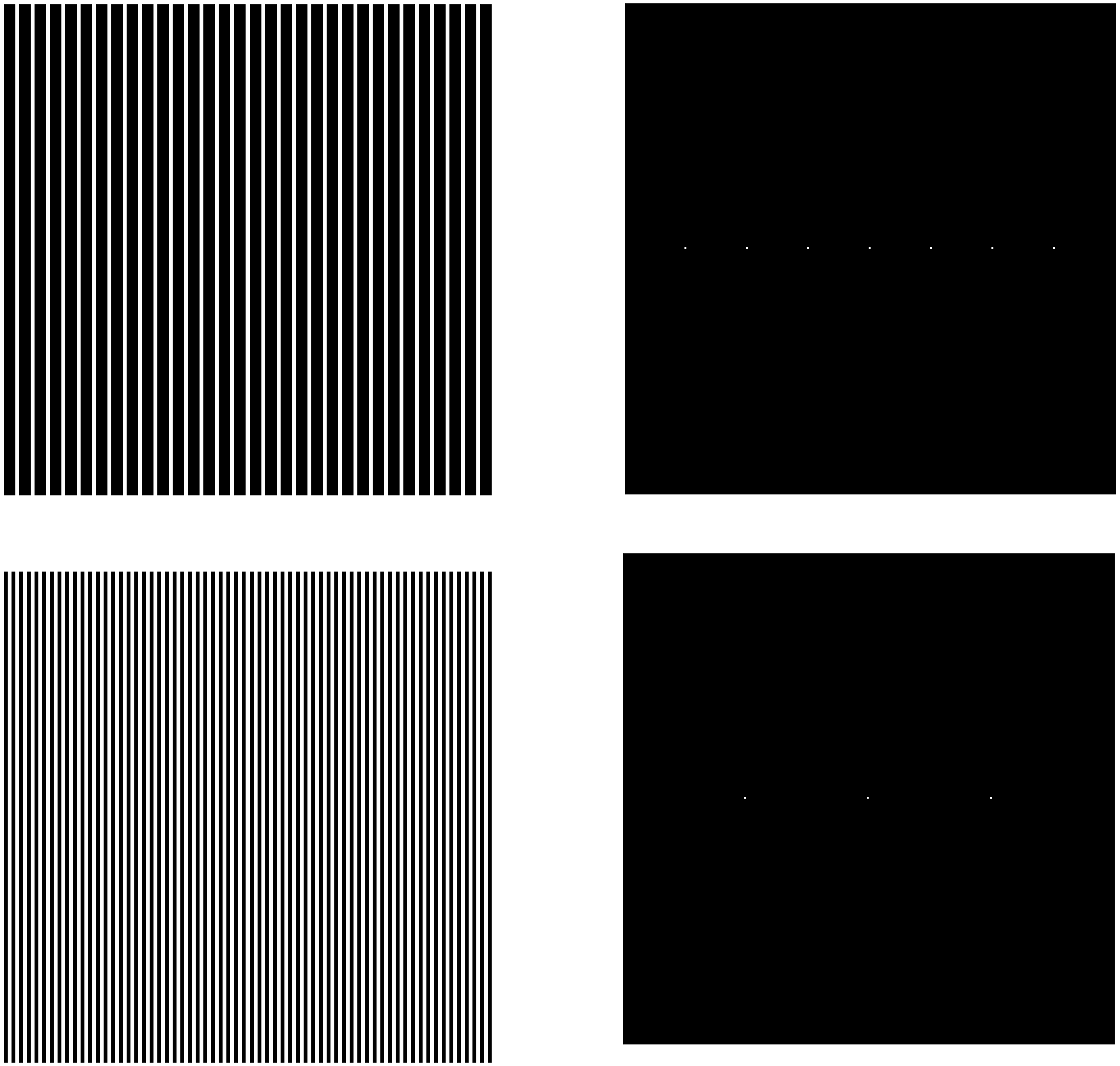
$\cos^2\delta$ (interference between the 2 slits term)

**M. Willinger, FHI**

# FFT of 2-dim grating

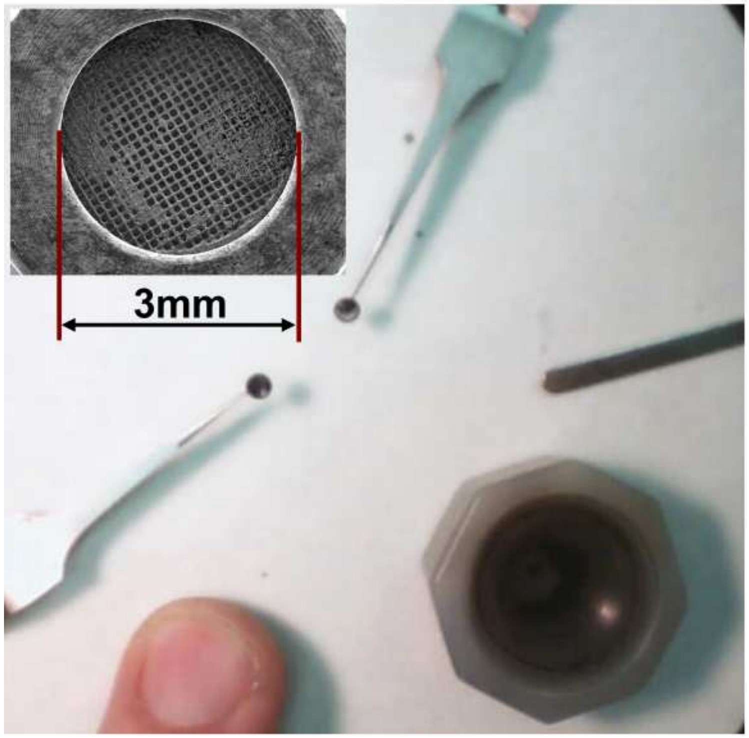


# FFT of 2-dim grating

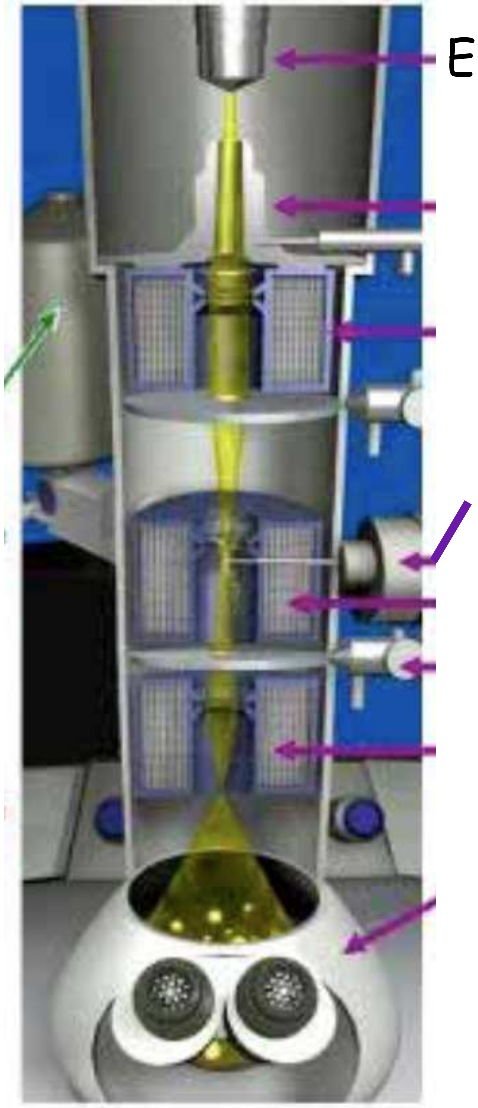


# Examples:

$$n\lambda = 2d\sin\theta_{\mathrm{B}}$$



# Basic set-up of the TEM



Electron gun

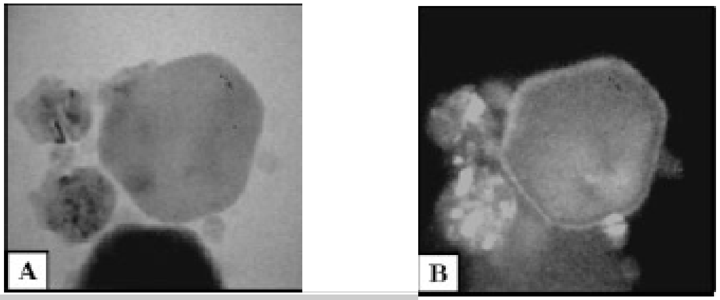
Acceleration stage

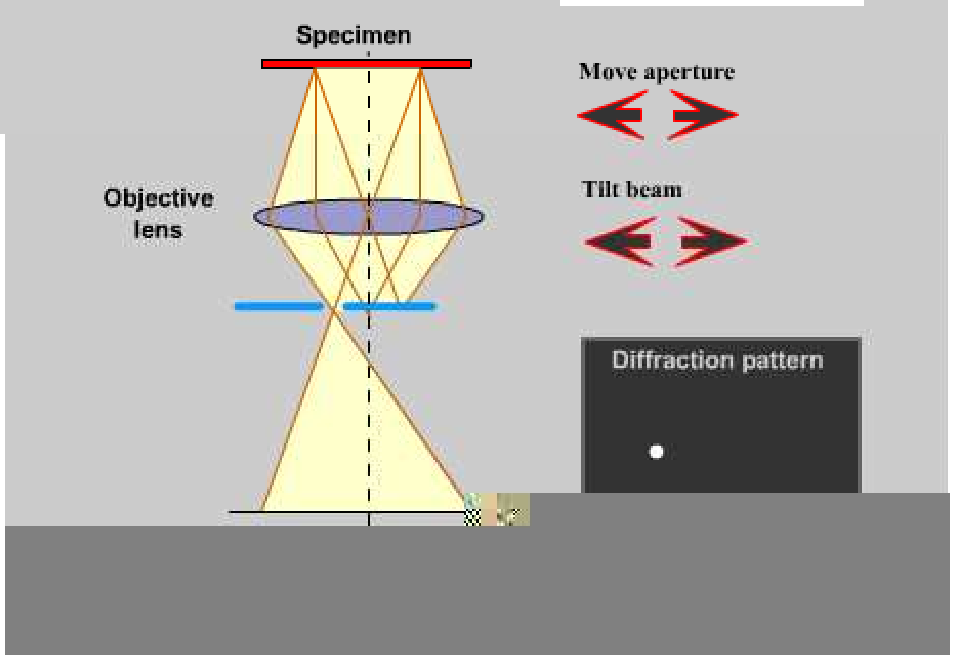
Condenser lens system:

Specimen stage

Objective aperture

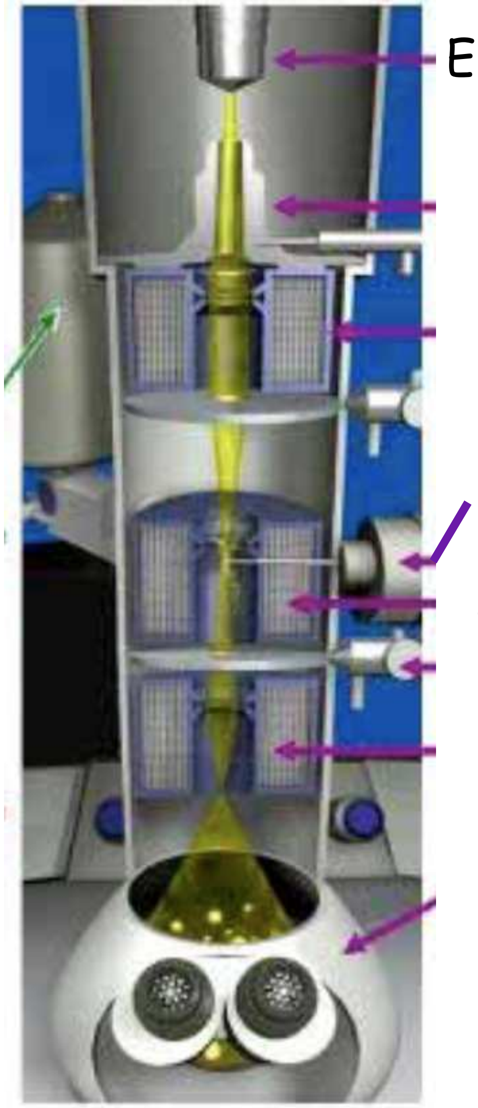
**Bright field (BF)** **Dark field (DF)**





M. Willinger, FHI

# Basic set-up of the TEM



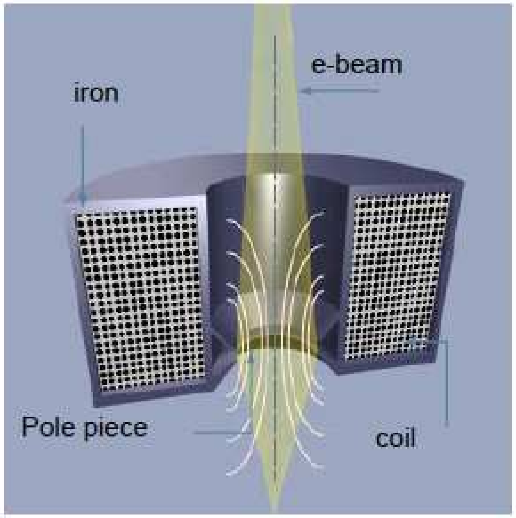
Electron gun

Acceleration stage

Condenser lens system:

Specimen stage

Objective lens



www.x-raymicroanalysis.com

… a few words on this one…

# Lens aberrations

Electrons are focused by simple round magnetic lenses which properties resemble the optical properties of a wine glass….

Unlike in

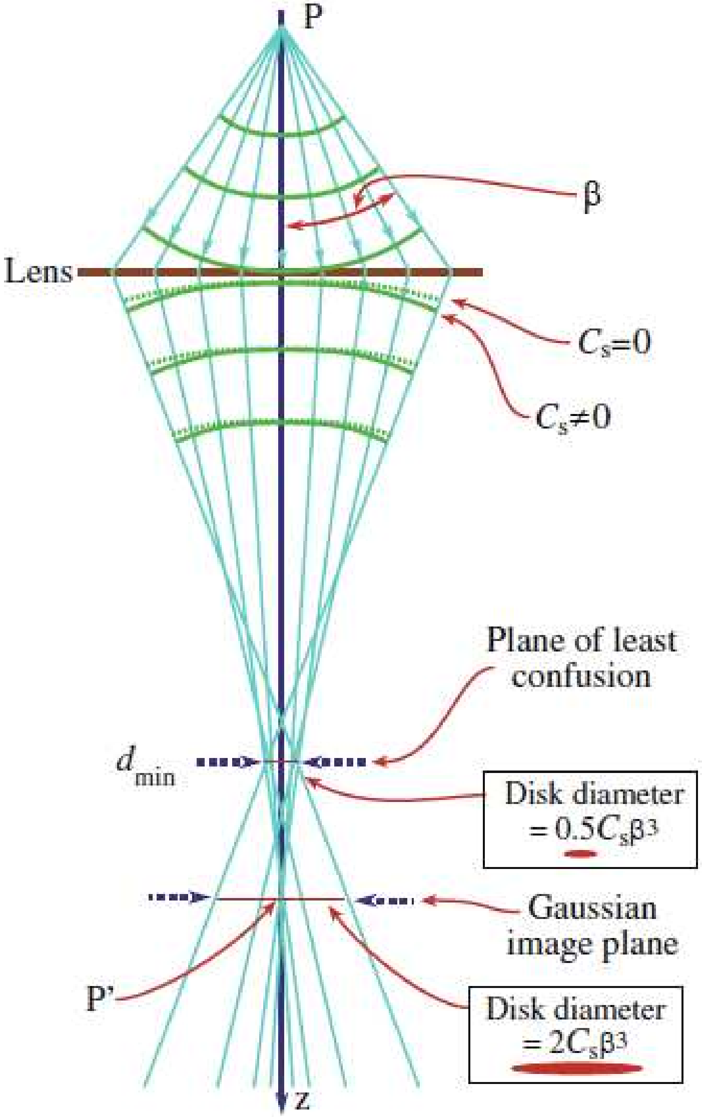
light optics the wavelength (2pm for 300kV) is not the resolution

limiting factor. However lens aberrations and instabilities of the

electronics (lens currents etc.) limit the resolution of even the best and

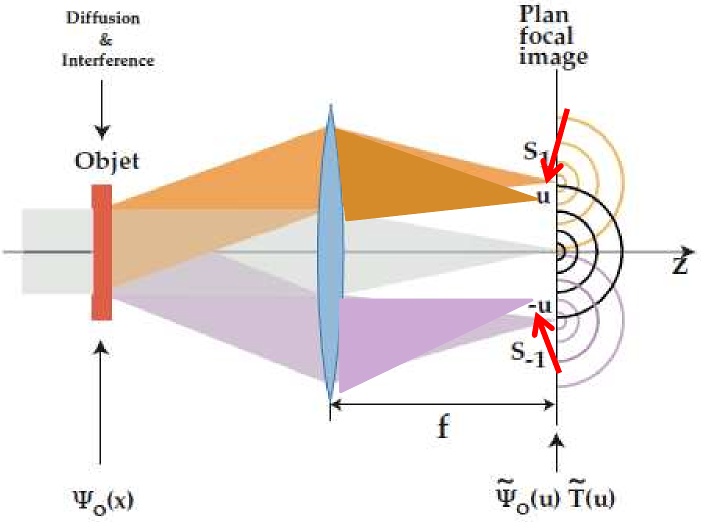
most expensive transmission electron microscopes to about 50pm.

# Lens aberrations

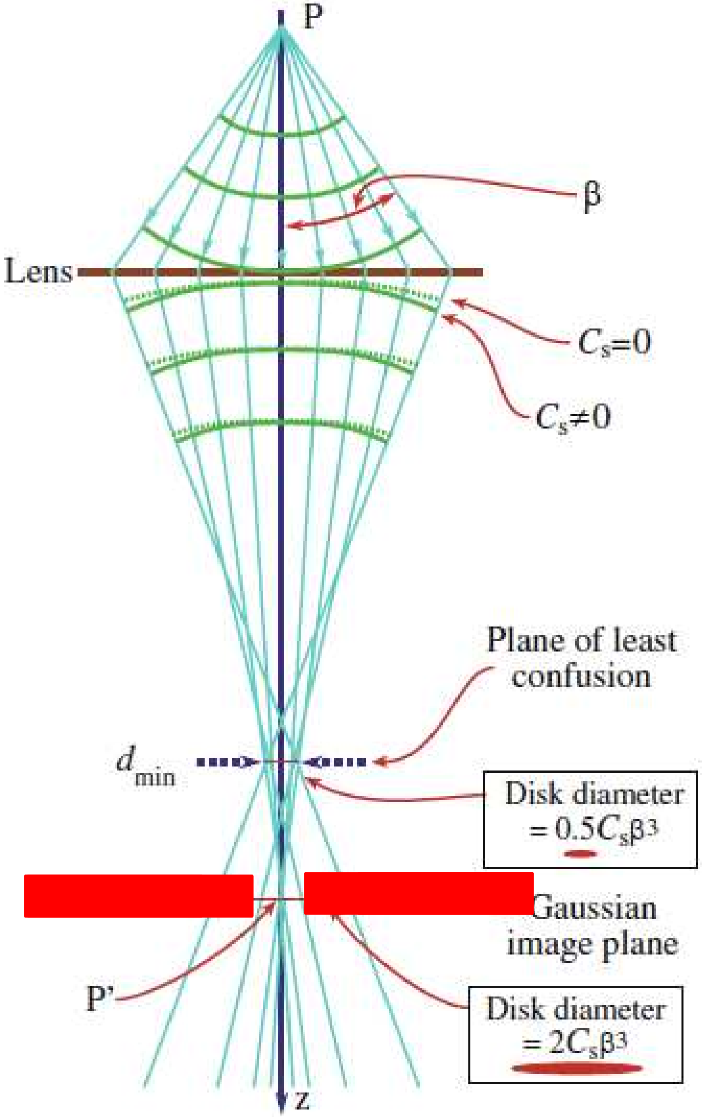


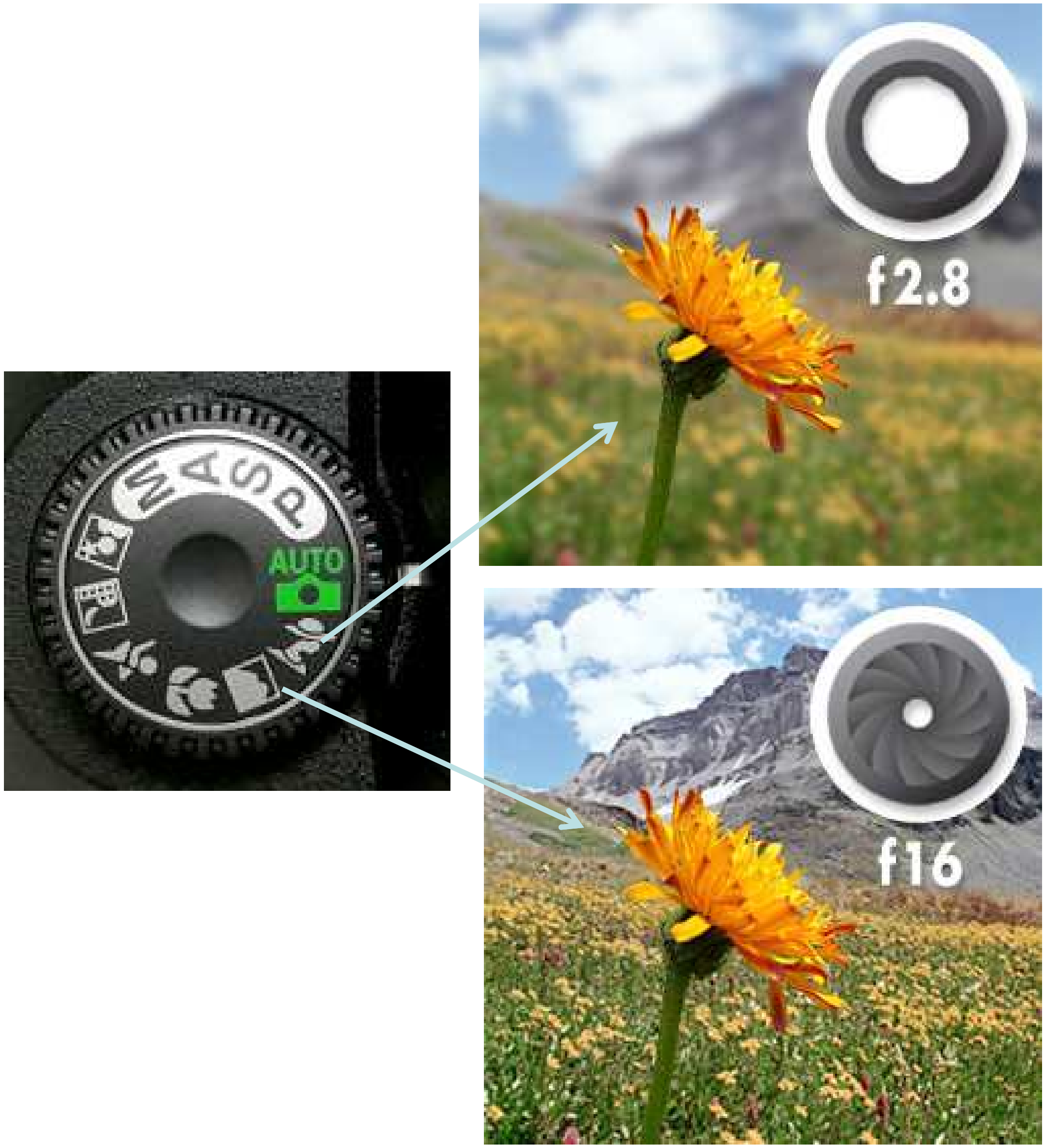
Spherical aberration ($C_S$):

Spherical aberration causes wave fronts to bend more strongly at the outside of the lens than those close to the axis



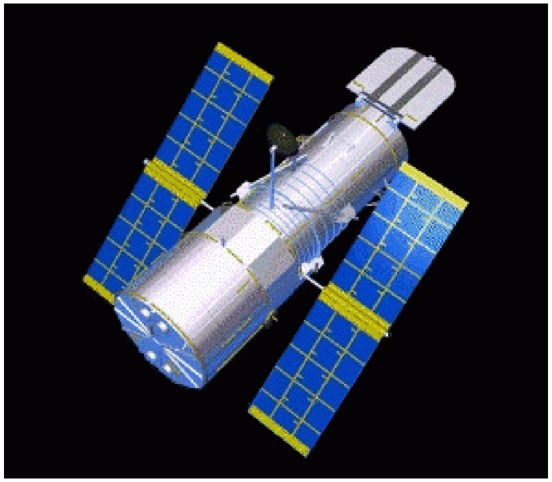
# Lens aberrations





M. Willinger, FHI

# A famous $C_s$-afflicted instrument



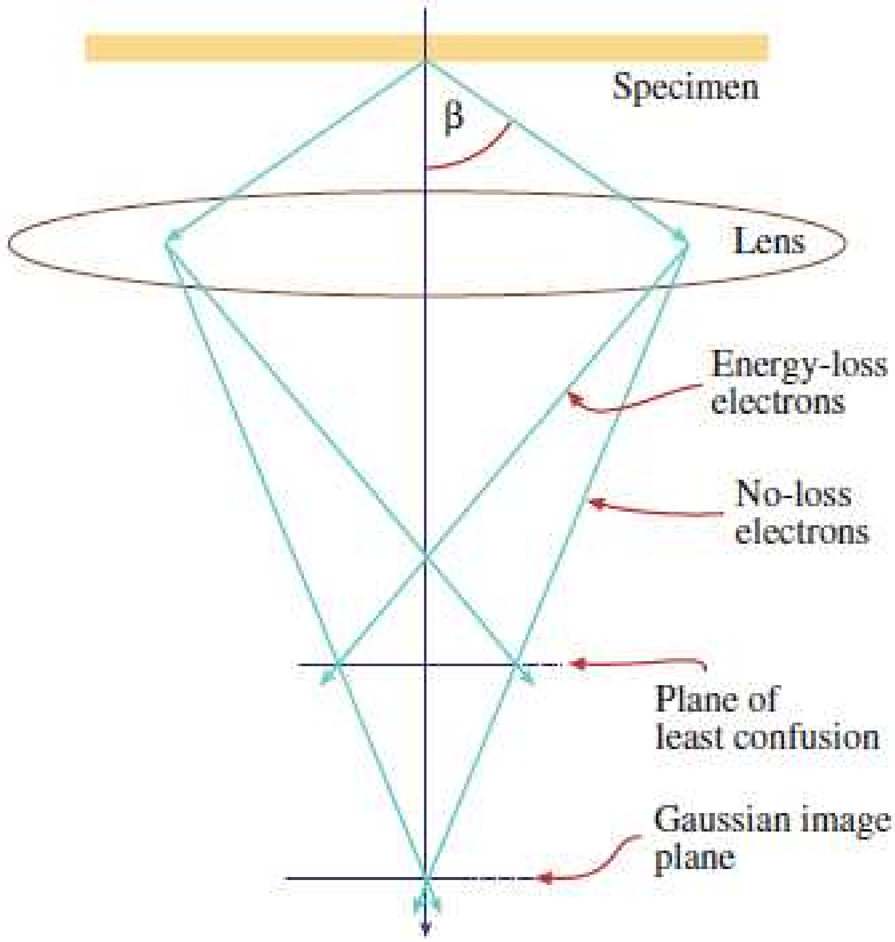
**Hubble telescope:**
the sides of its Ø 2.5 m primary mirror are 2 μm too low (negative $C_s$) - the mirror was ground very precisely to the wrong shape. The error was avoidable.

**Hubble repair:**
a modified camera lens assembly corrected for the too-low phaseshift of marginal rays and resulted in a spectacular improvement of image quality. Primary mirror was not changed.

**Related problem:** imperfect images of ground-based telescopes due to phase shifts caused by atmospheric turbulence.

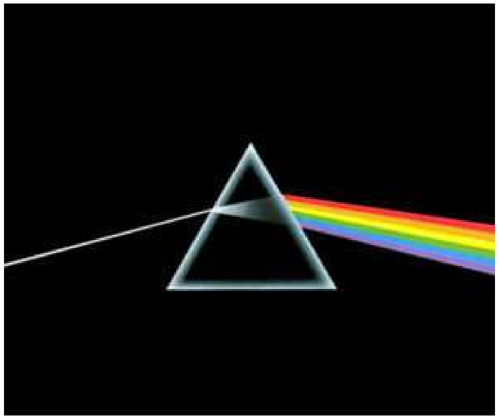
**Solution:** Adaptive optics - the imperfections are quantified in real time and the exact shape of the mirror is adjusted to compensate for them.

# Lens aberrations



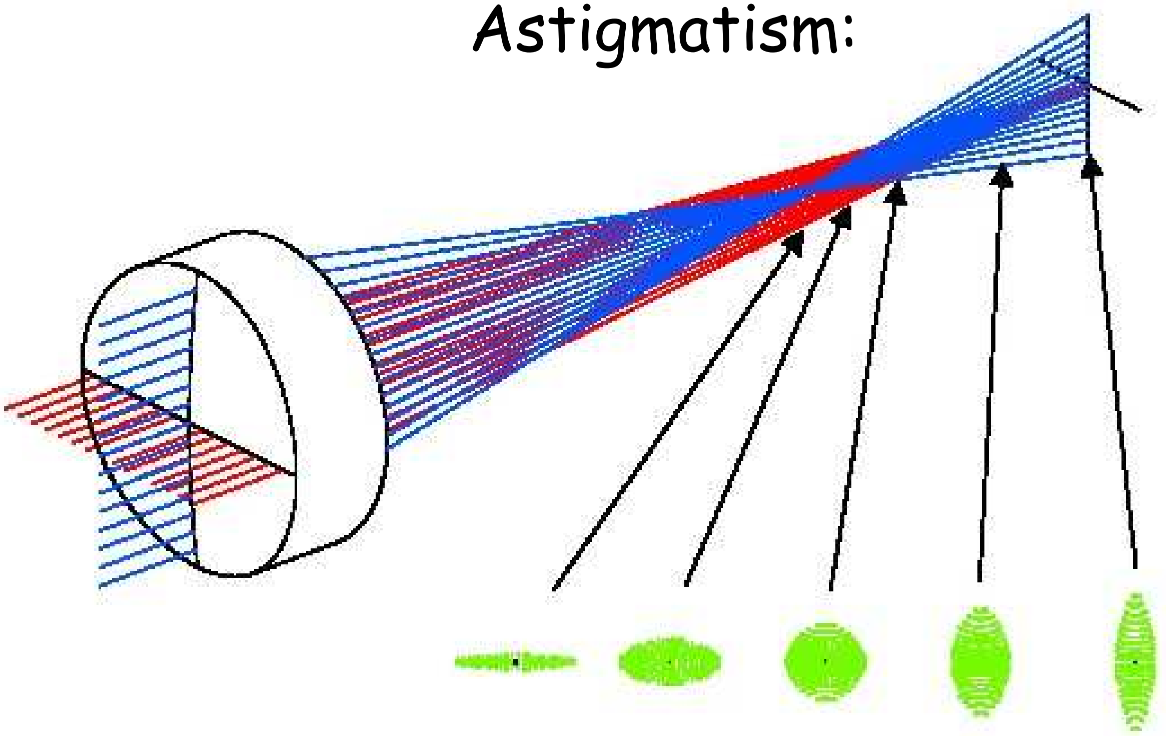
Chromatic aberration:

Chromatic aberration results in electrons with a range of energies being focused in different planes



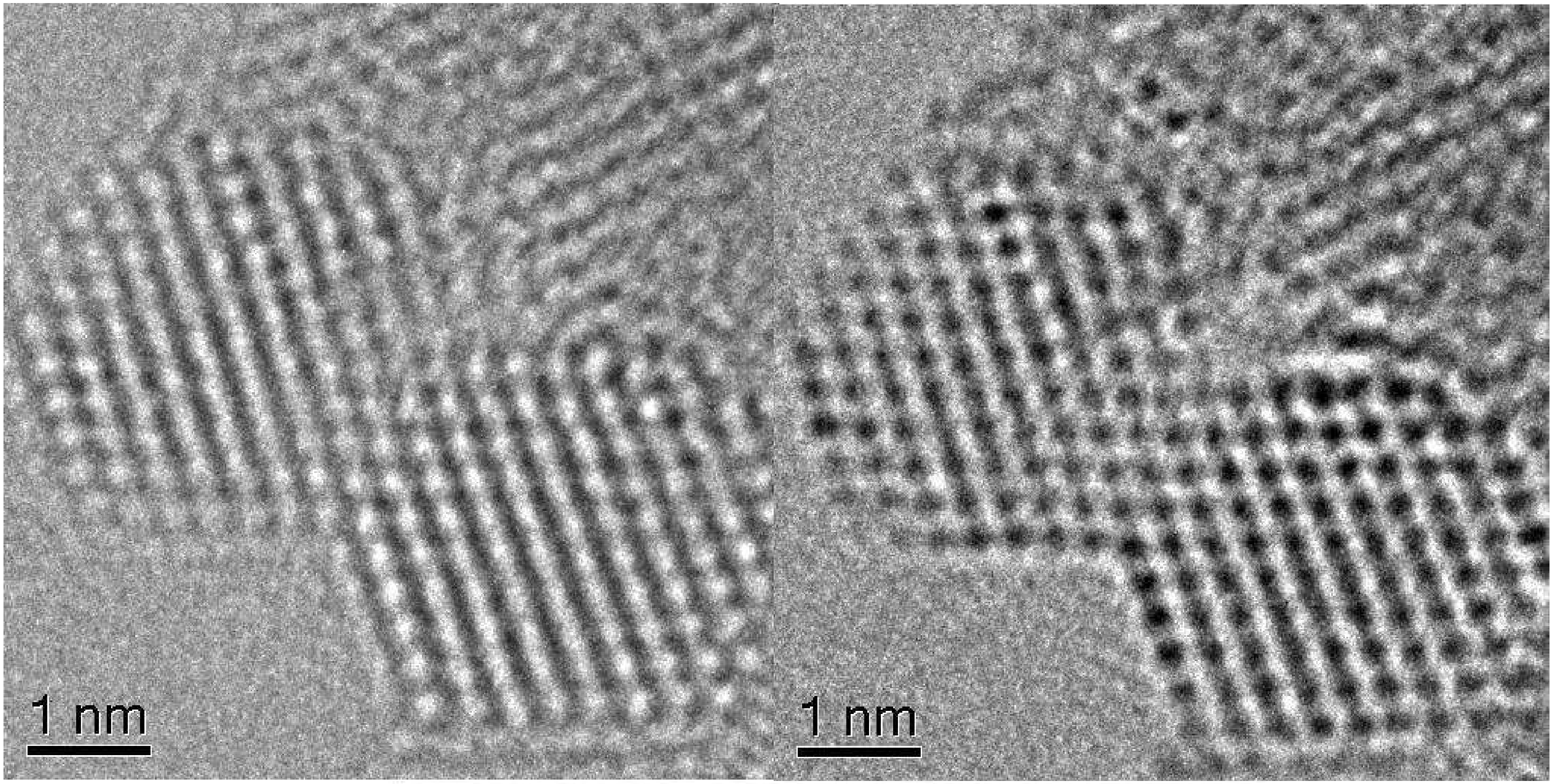
# Lens aberrations

Astigmatism:



Electrons passing at different directions away from the optic axis have different focal lengths.

# Interpretation of TEM images

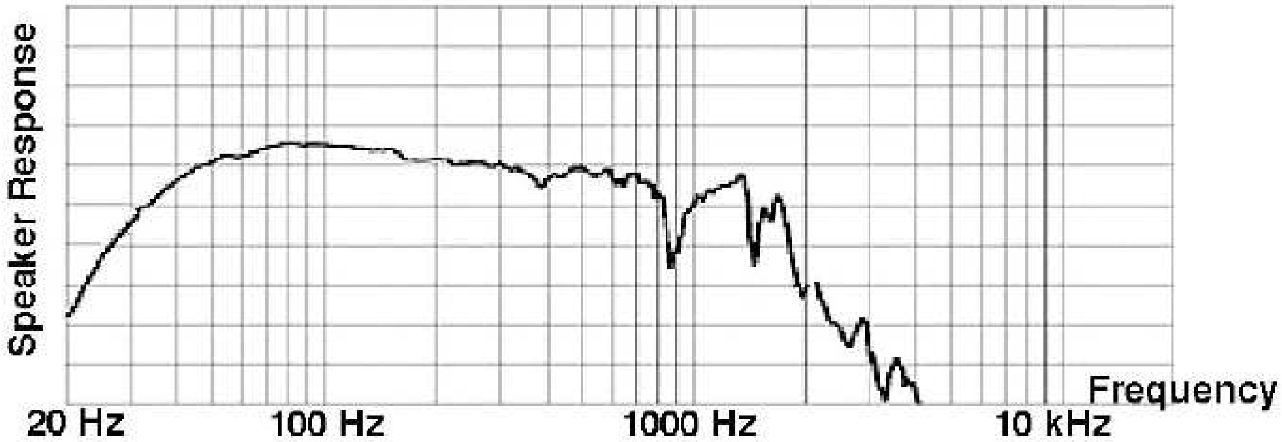


Question : do the atoms appear as white or dark spots ?

how does the microscope transfer the information about the sample down the column?

# Transfer function

Transfer Function



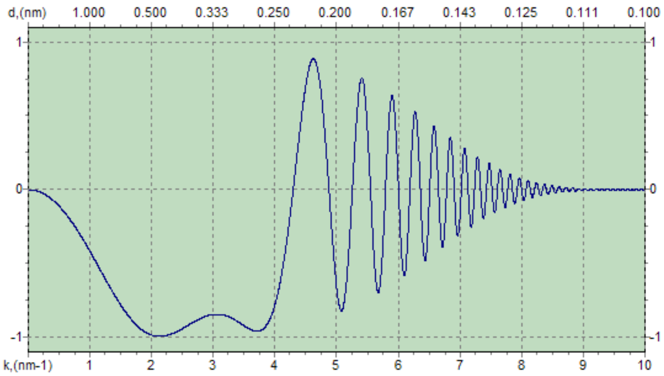
# Transfer function

## Phase contrast transfer function:

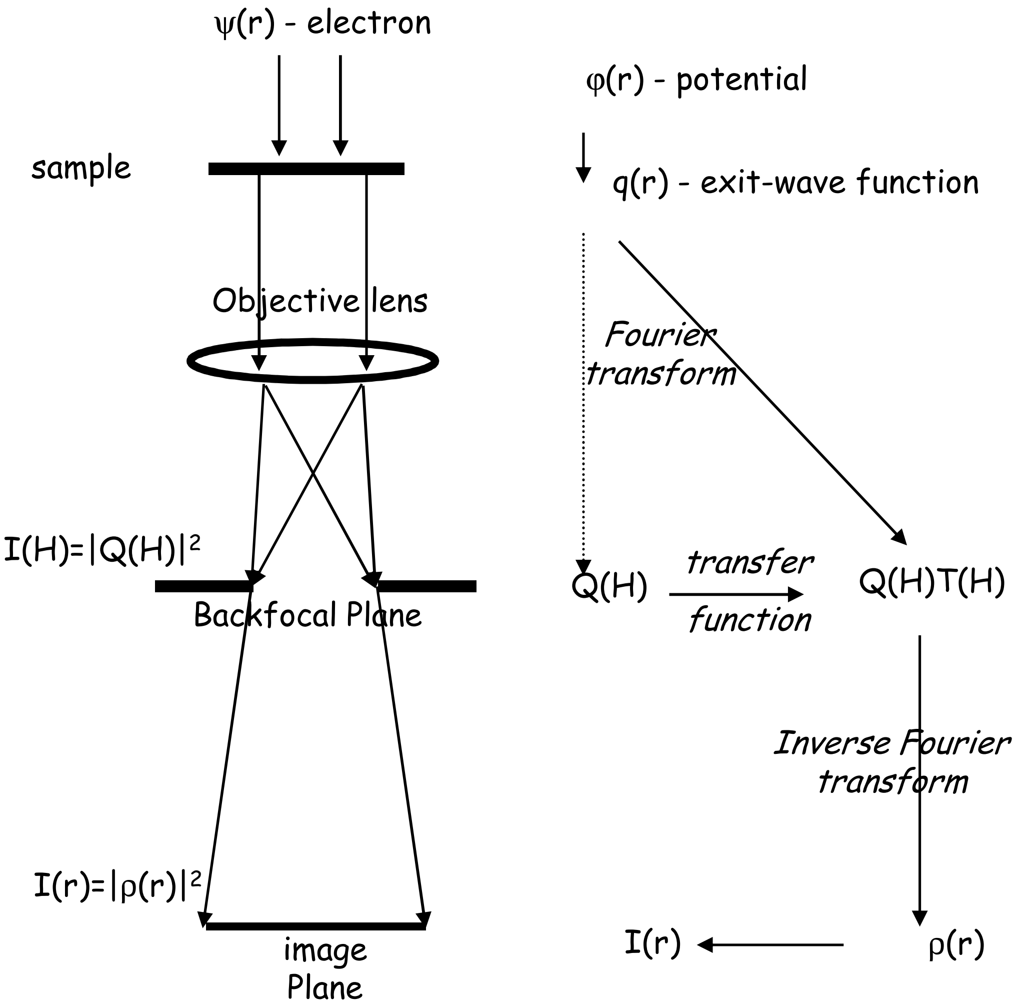
$T(H)=\sin(\pi C_s\lambda^3H^4/2+\pi\Delta f\lambda H^2)$.

Cs: Spherical aberration constant.

$\Delta f$: defocus value.



Phase contrast transfer function calculated at $\Delta f$=-61 nm with Cs=1.0 mm.



$\psi(r)$ - electron

sample

Objective lens

$I(H)=|Q(H)|^2$

Backfocal Plane

$I(r)=|\rho(r)|^2$

image Plane

$\varphi(r)$ - potential

$q(r)$ - exit-wave function

*Fourier transform*

Q(H) *transfer function* Q(H)T(H)

*Inverse Fourier transform*

I(r) ← $\rho(r)$

**M. Willinger, FHI**

# Transfer function

T(H)<0 implies "positive" constrast: atom columns appear dark (in the print, not the negative!).

T(H)>0 implies "negative" contrast: atom columns appear bright.

- T(H) = 0 implies no transfer of the respective spatial frequency at all!

Example: hypothetical crystal with four different sets of planes parallel to the viewing direction

– plane spacing: $d1 > d2 > d3 > d4$

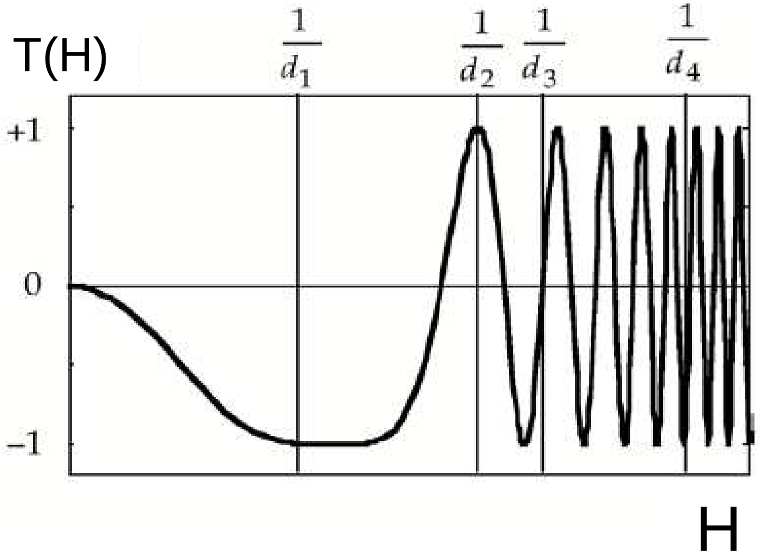
– corresponding spatial frequencies: $1/d1 < 1/d2 < 1/d3 < 1/d4$.

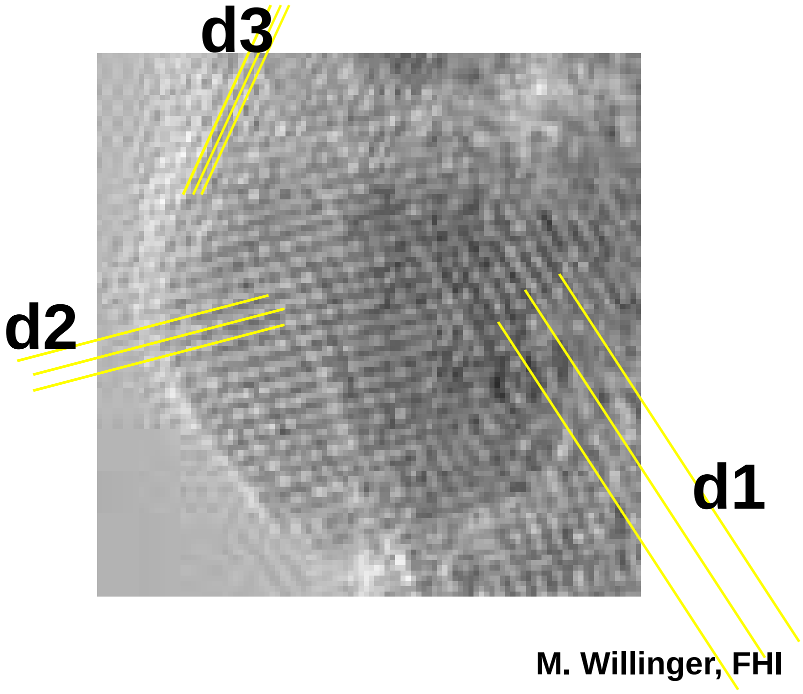
– the planes with spacing $d1$ appear with positive contrast

– the planes with spacing $d2$ appear with negative contrast

– the planes with spacing $d3$ do not appear at all

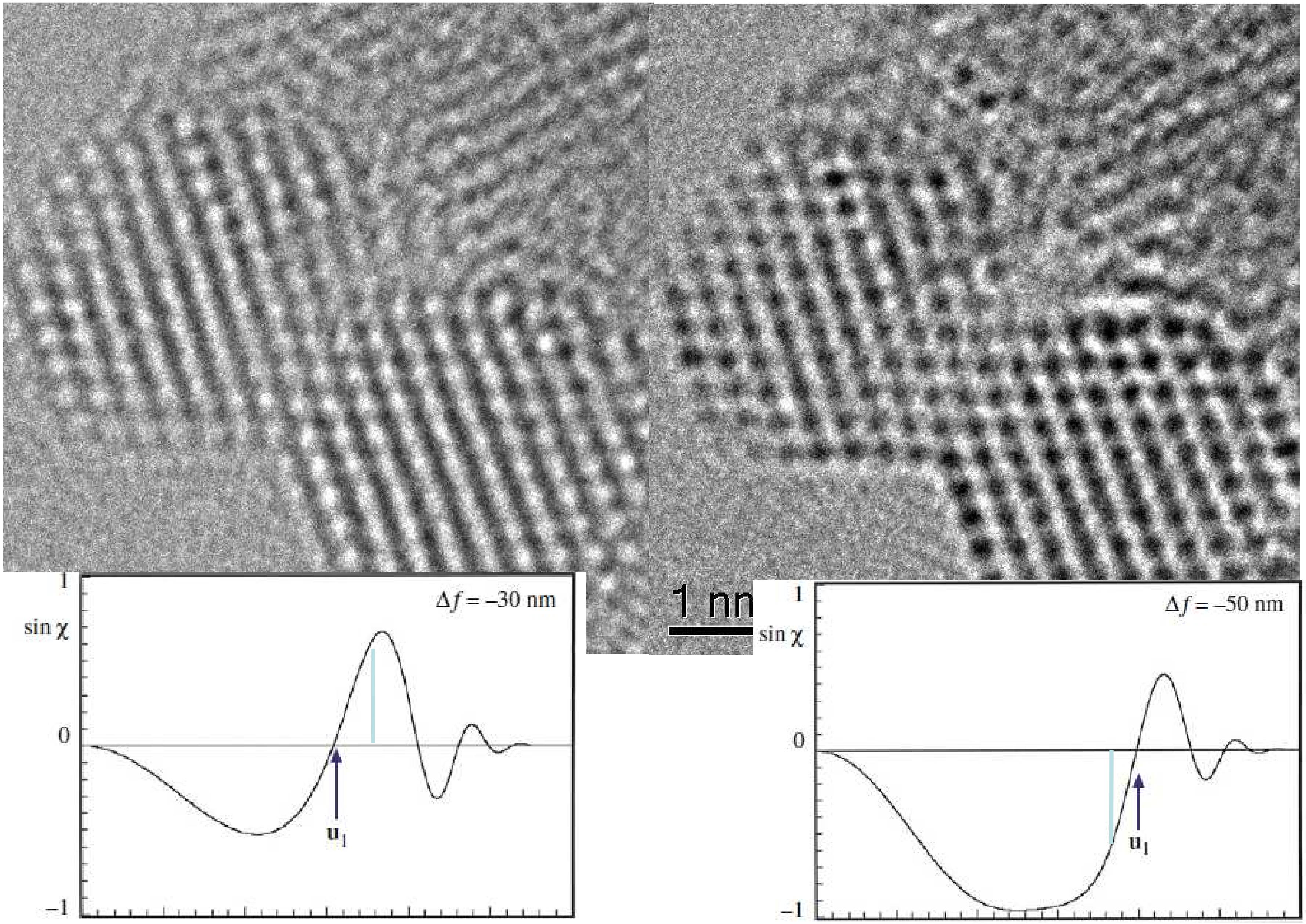
– it is difficult to predict the contrast of the planes with spacing $d4$. We can avoid these problems by introducing an objective aperture.





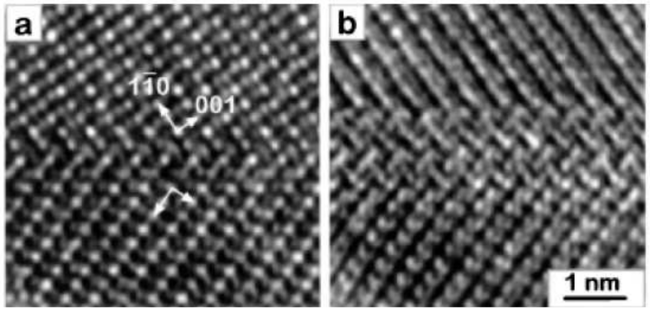
M. Willinger, FHI

# Interpretation of TEM images



# Interpretation of TEM images

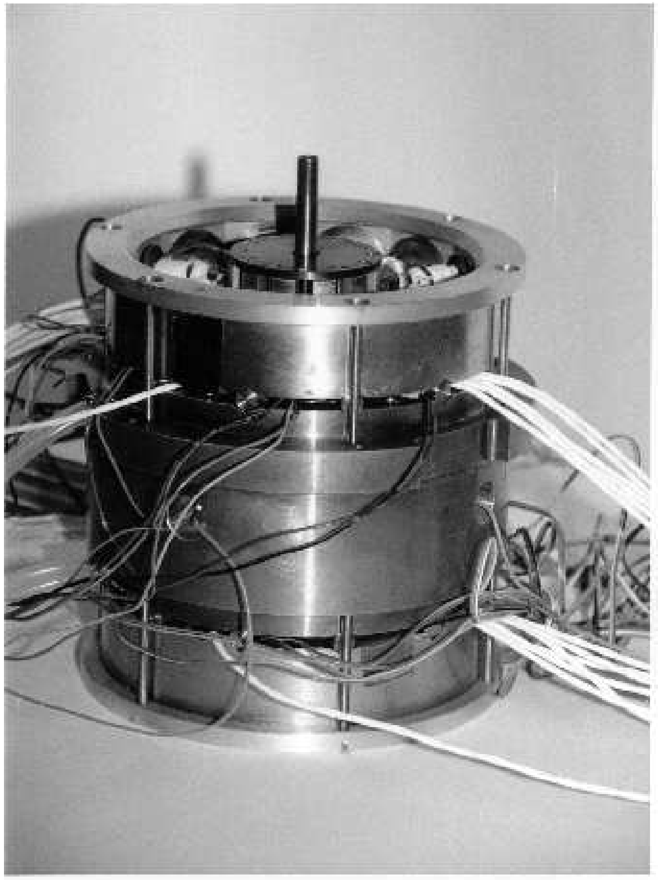
*C. L. Jia and A. Thust*
*PHYSICAL REVIEW LETTERS 82, 25*

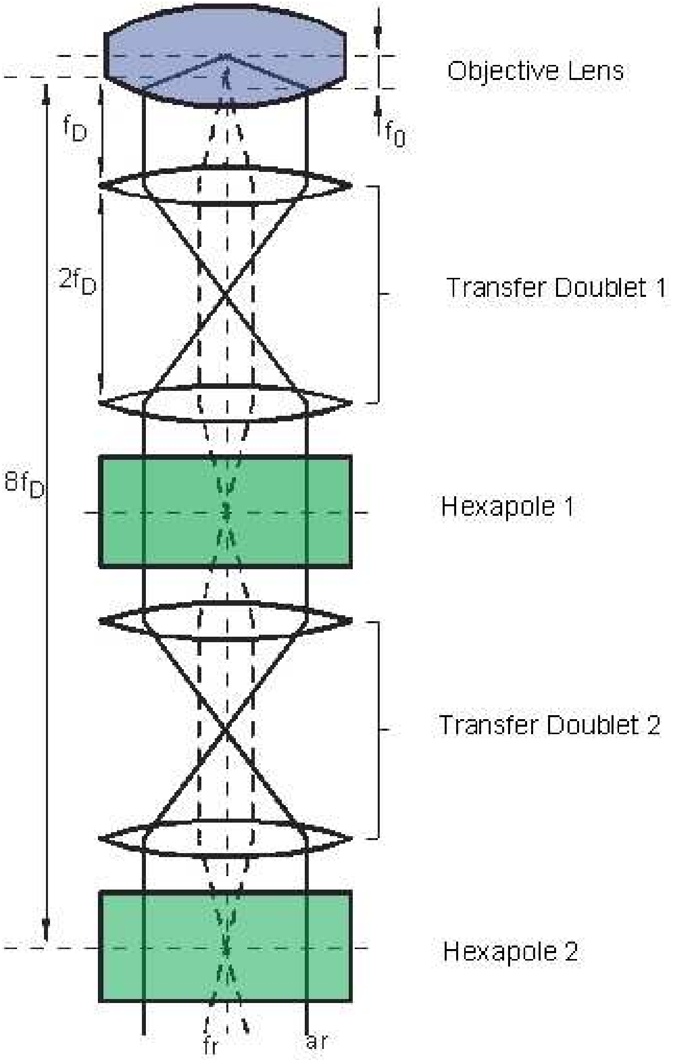


Exit wave function reconstruction

# $C_s$ Corrector: removes Spherical Aberration

$C_S$-Corrector (Rose, Haider and Urban)

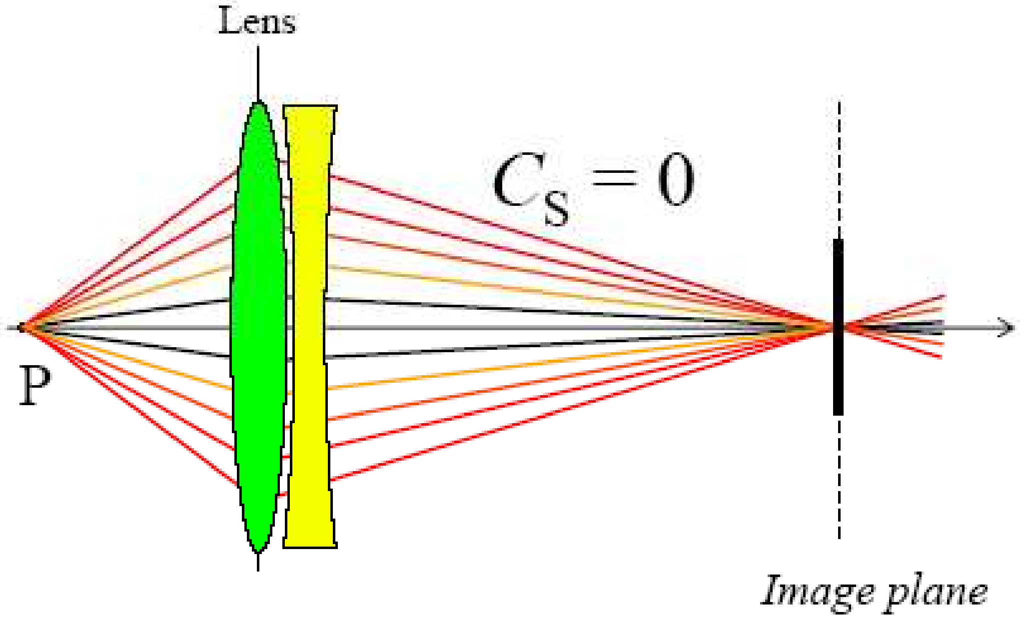




# $C_s$ Corrector: removes Spherical Aberration

## Aberration corrected electron optics

$C_s$ is adjustable!

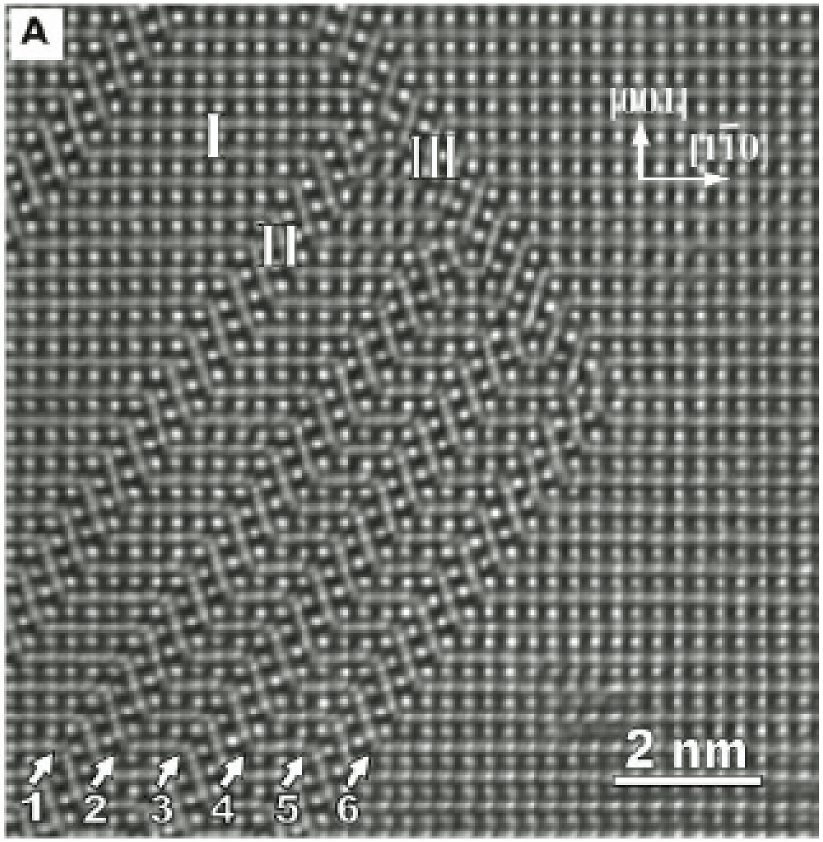


- TU Darmstadt (H. Rose)
- EMBL Heidelberg (M. Haider)
- Forschungszentrum Jülich (K. Urban)

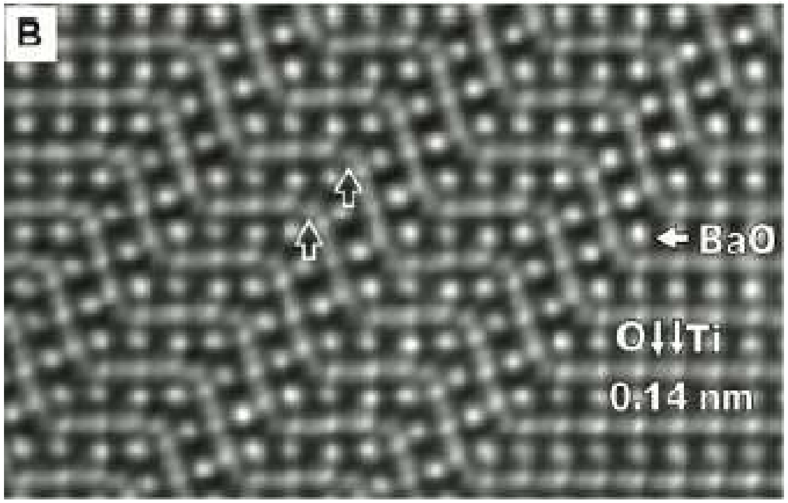
Haider, Rose, Urban et al.
**Nature 392**, 768 (1998)

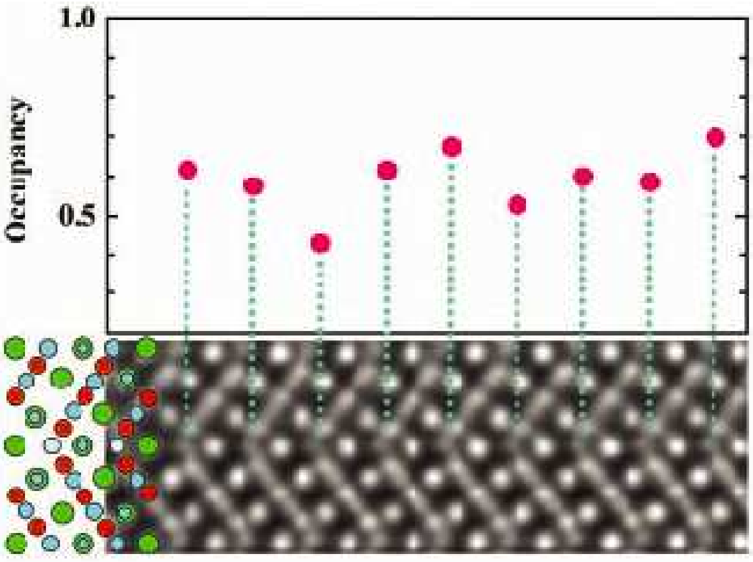
# Aberration - corrected TEM (Example)

## Twin Boundaries in $BaTiO_3$



Jia and Urban, Science 303 (2004)





# Interpretation of TEM images

In the TEM we see 2D projections of 3D specimens, viewed in transmission

Our eyes and brain routinely understand reflected light images but are ill-equipped to interpret TEM images and so we must be cautious

This problem is well illustrated by the picture of the two rhinoceros side by side such that the head of one appears attached to the rear of the other

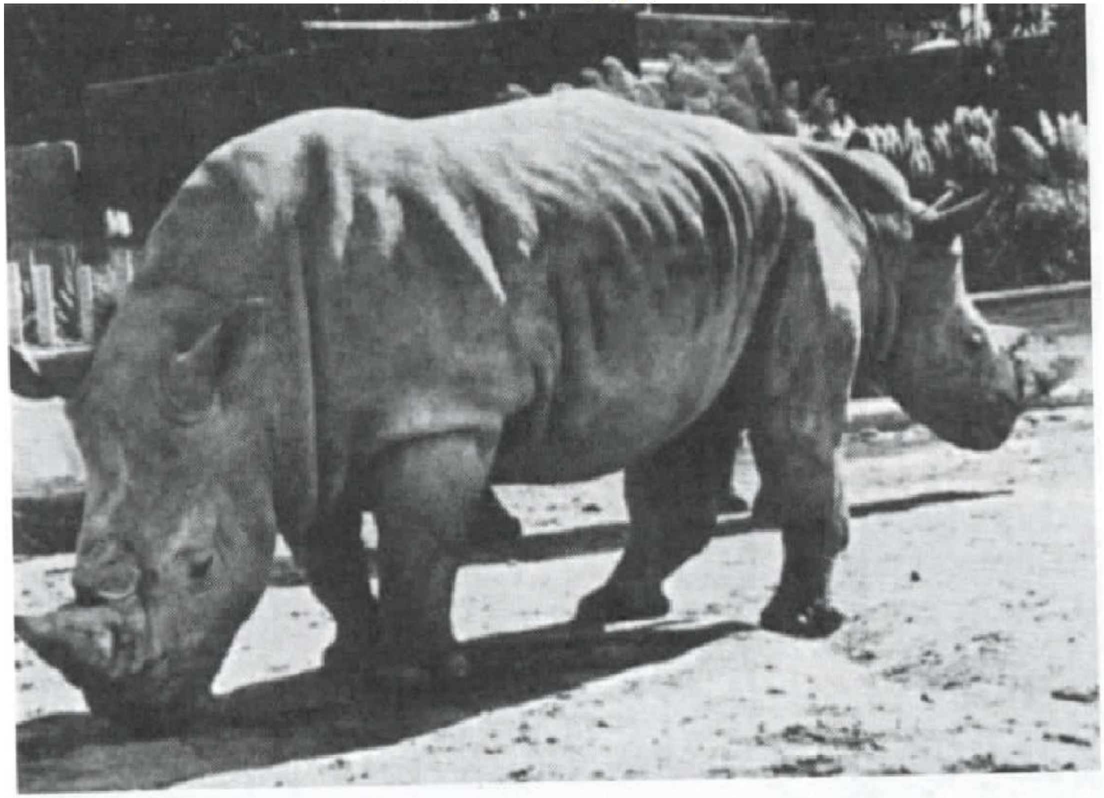
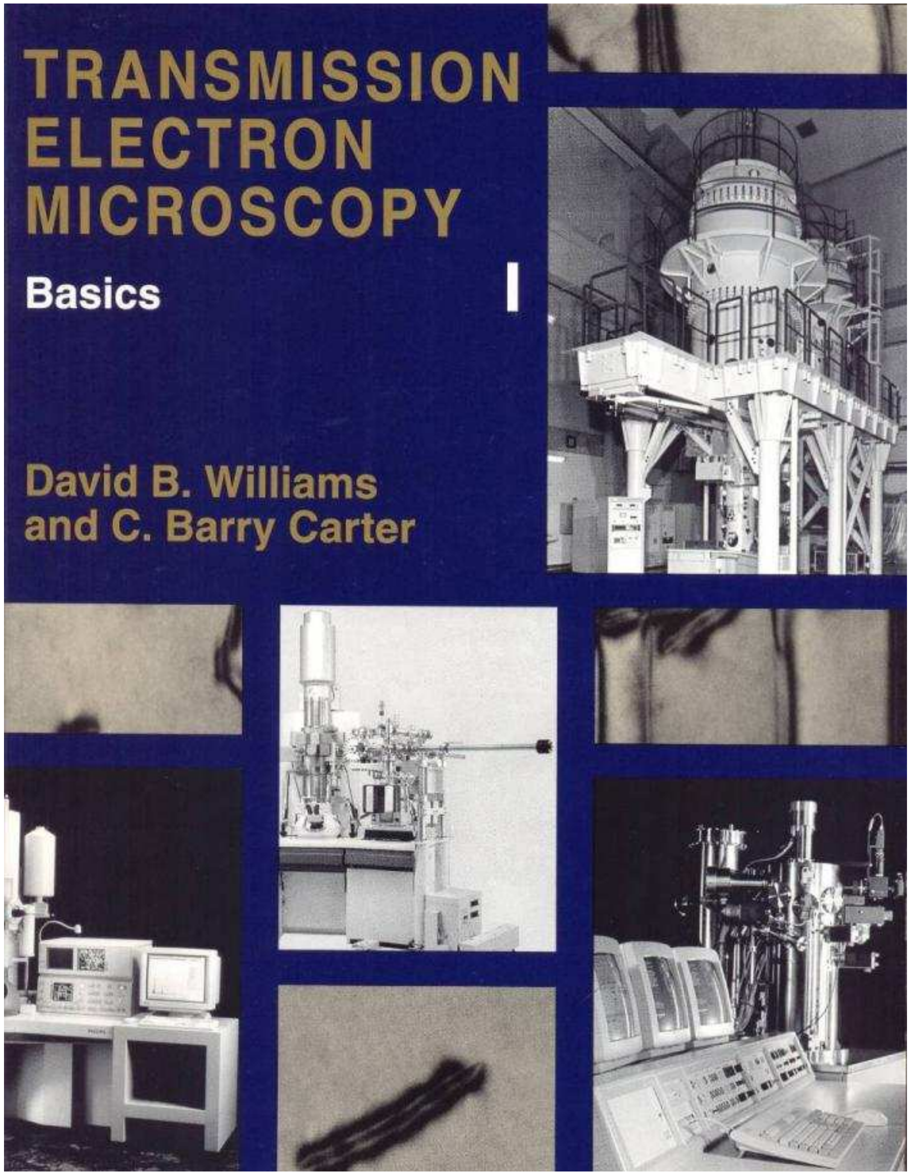


**Figure 1.7.** Photograph of two rhinos taken so that, in projection, they appear as one two-headed beast. Such projection artifacts in reflected-light images are easily discernible to the human eye but similar artifacts in TEM images are easily mistaken for "real" features.

# Literature

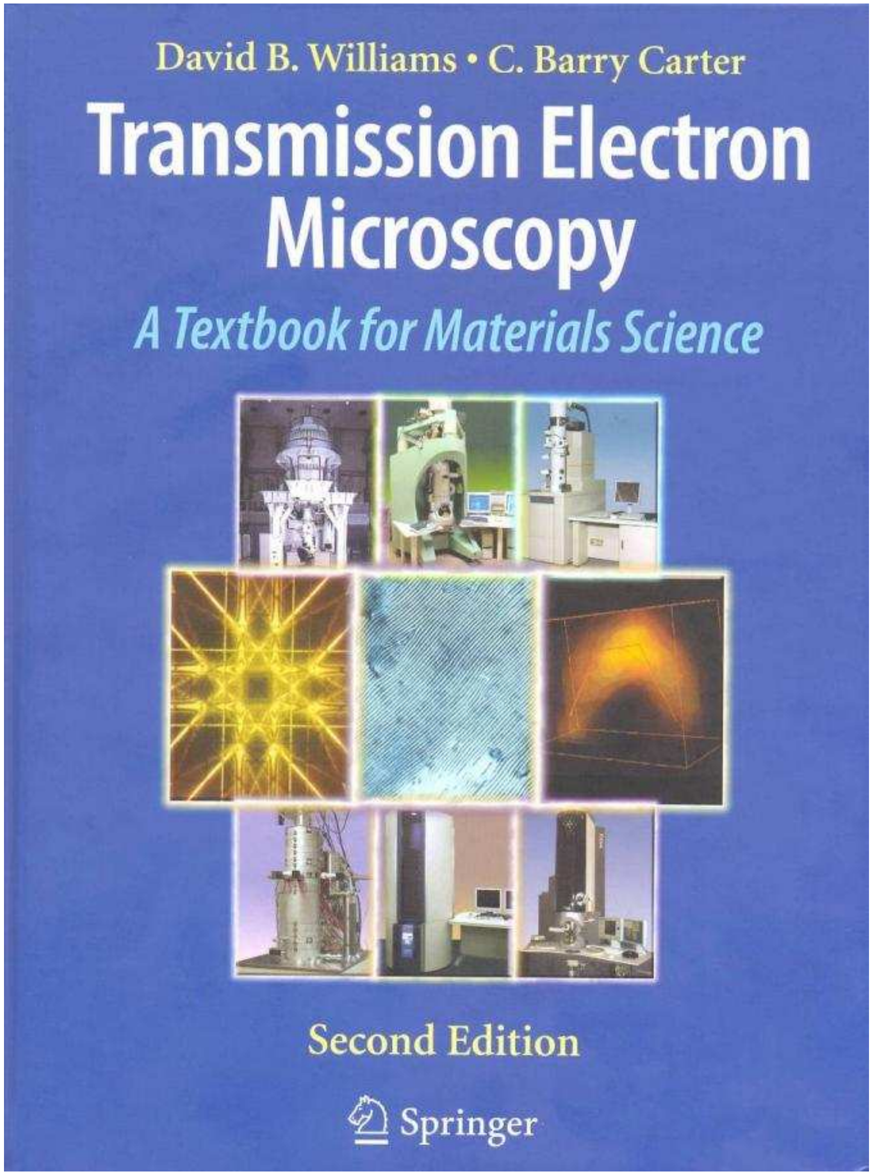


TRANSMISSION
ELECTRON
MICROSCOPY

Basics

I

David B. Williams
and C. Barry Carter



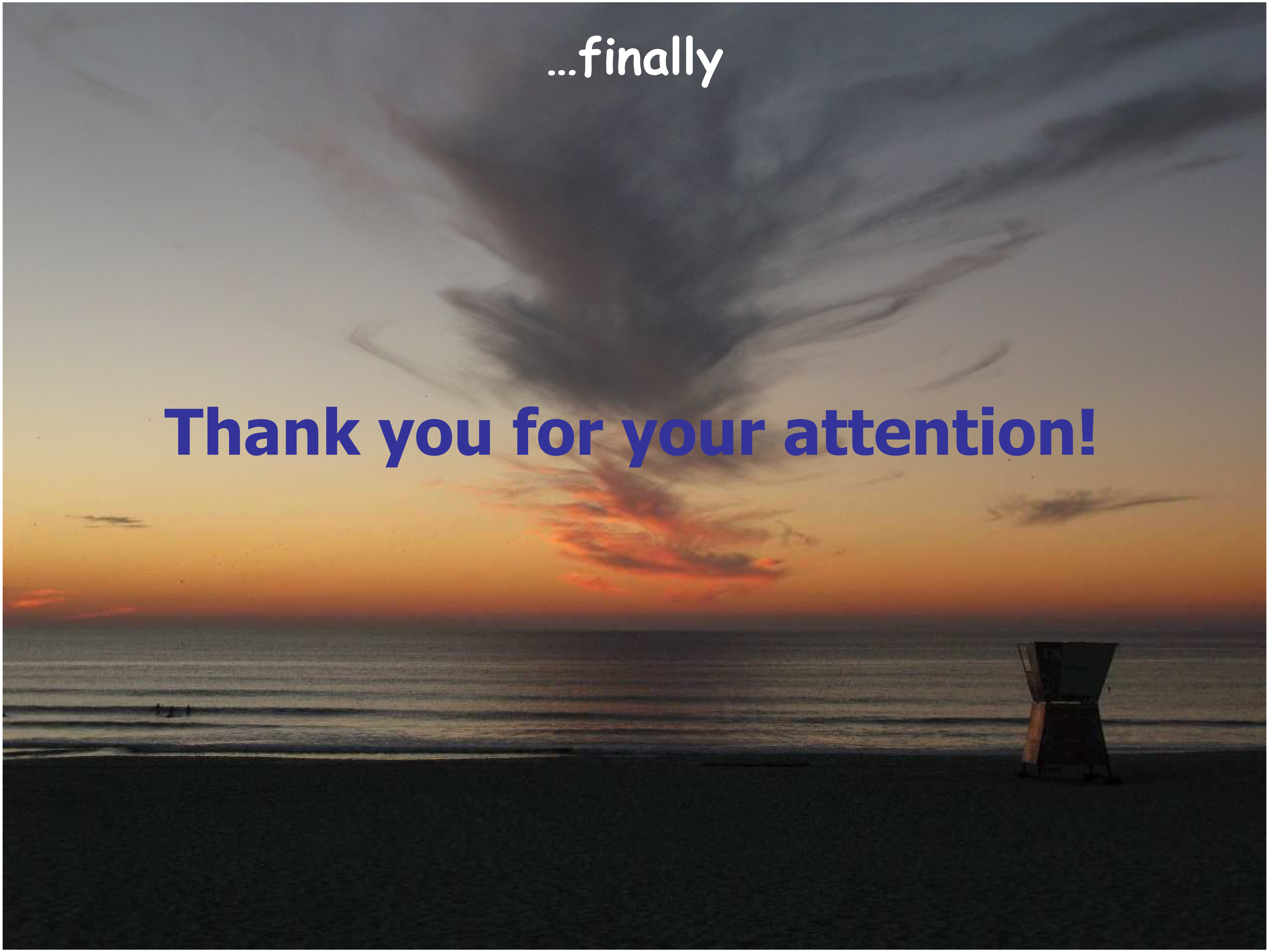
David B. Williams • C. Barry Carter

Transmission Electron
Microscopy

*A Textbook for Materials Science*

Second Edition

Springer



# …finally

**Thank you for your attention!**