FYS 4340/9340
Repetition Class

- TEM Specimen Preparation
- TEM Instrumentation
- TEM Imaging Techniques
- Ray Diagrams

TEM Specimen Preparation

What to consider before preparing a TEM specimen

- Ductile/fragile
- Bulk/surface/powder
- Insulating/conducting
- Heat resistant
- Irradiation resistant
- Single phase/multi phase
- Can mechanical damage be tolerated?
- Can chemical changes be accepted?
- Etc, etc…….

What is the objective of your TEM study?

TEM specimen preparation Philosophy

- Your region of interest in the specimen has to be electron transparent

(Thinning down to thickness of ~100 nm or less)

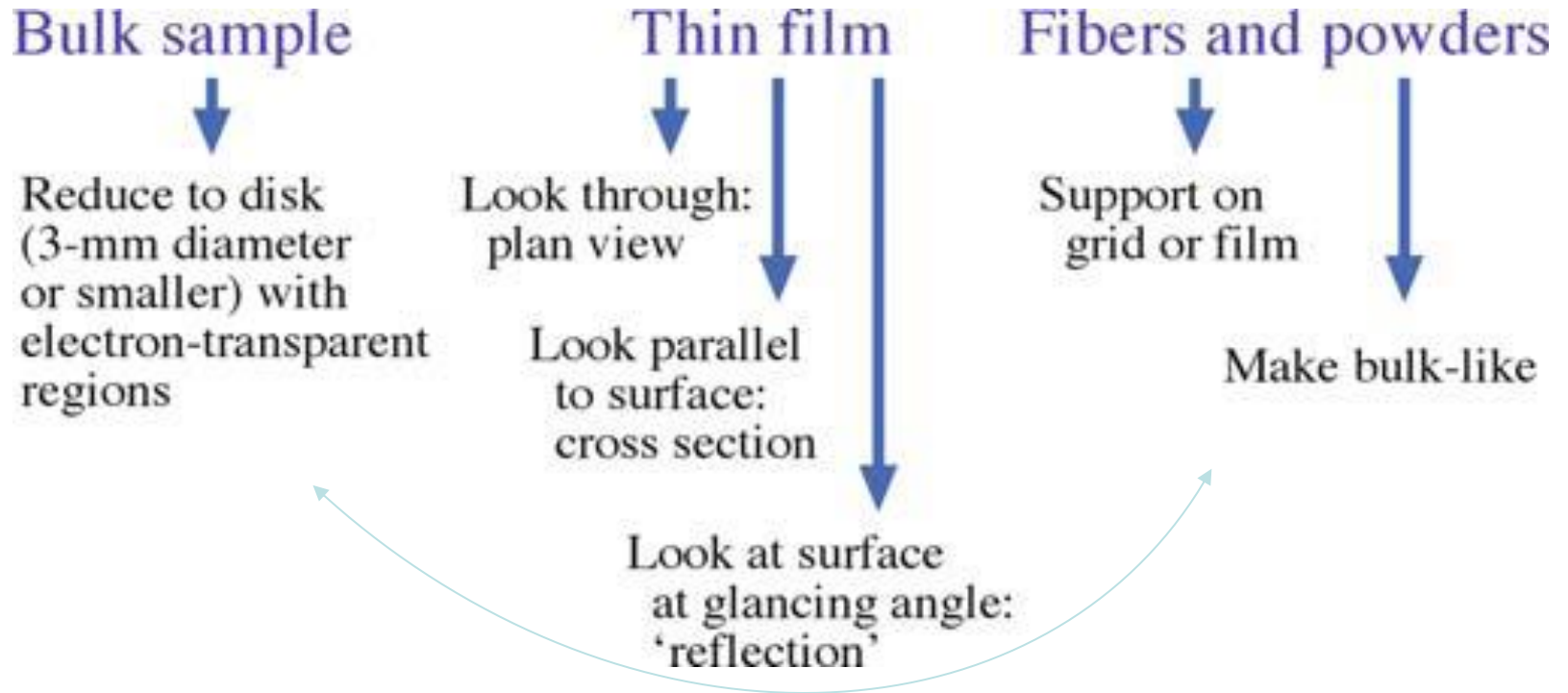- The specimen should fit into the TEM holder

(3mm dia disc)

Courtesy: http://asummerinscience.blogspot.no

Classification of TEM specimens

Self-supporting discs or **specimen supported on a grid or washer**

Preparation philosophy

Bulk sample → Reduce to disk (3-mm diameter or smaller) with electron-transparent regions

Thin film → Look through: plan view

Look parallel to surface: cross section

Look at surface at glancing angle: 'reflection'

Fibers and powders → Support on grid or film

Make bulk-like

Preparation of self-supporting discs

- Cutting
  - Ductile material or not?
- Grinding
  - 100-200 µm thick
  - polish
- Cut the 3mm disc
- Dimple
- Final thinning
  - Ion beam milling
  - Electropolishing
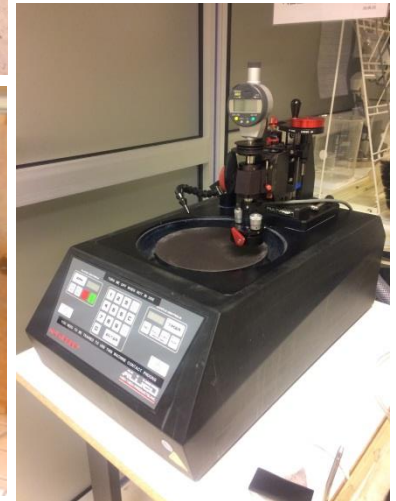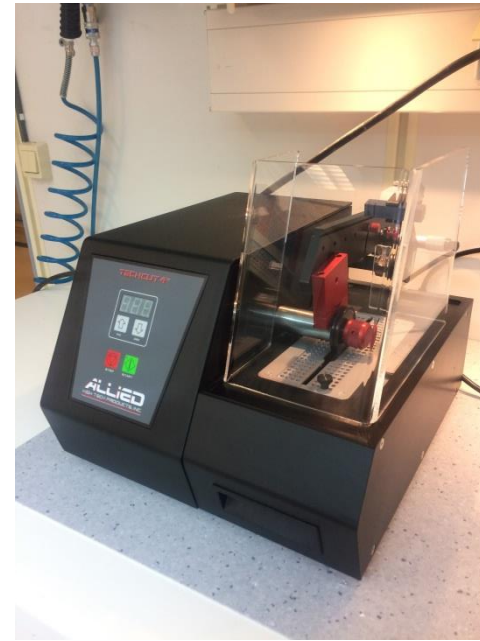
Cutting and cleaving

Cutting with a saw (non-brittle materials):

Brittle materials with well-defined cleavage plane

- Si
- GaAs
- NaCl
- MgO

Razor blade, scratching with Diamond tool or ultramicrotome

Cutting a 3 mm disc

Soft or brittle material?
Mechanical damage OK?

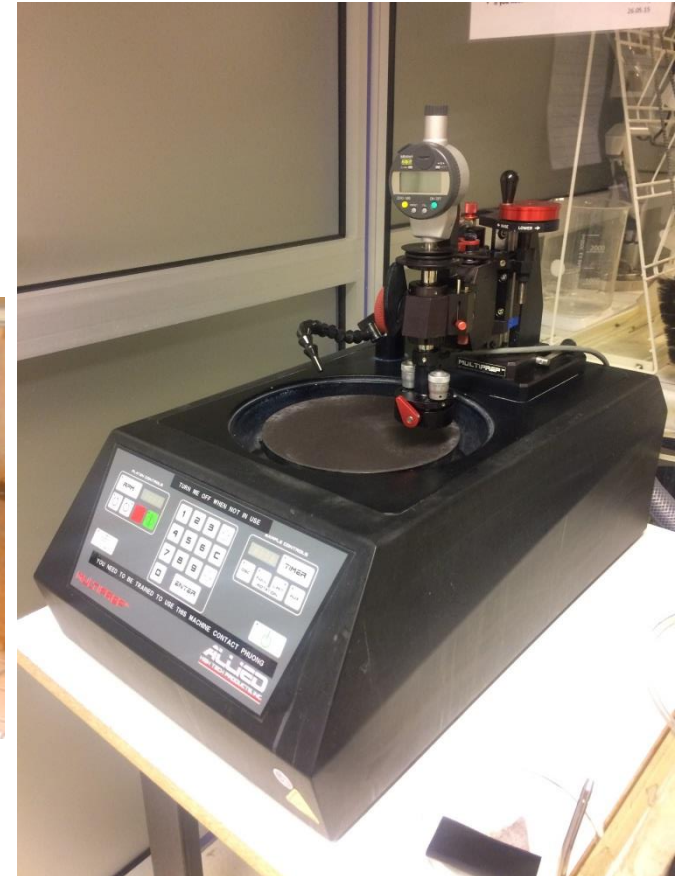**Brittle: Spark erosion, ultrasonic drill, grinding drill**

Preparation of self-supporting discs

- Grinding
  - 100-200 µm thick
  - polish
- Prethinning
  - Dimpling
  - Tripod polishing
  (Wedge polishing)
  Wedge angle ~ 1-2°

**Grinding
(thinning down to ~ 200 µm)**

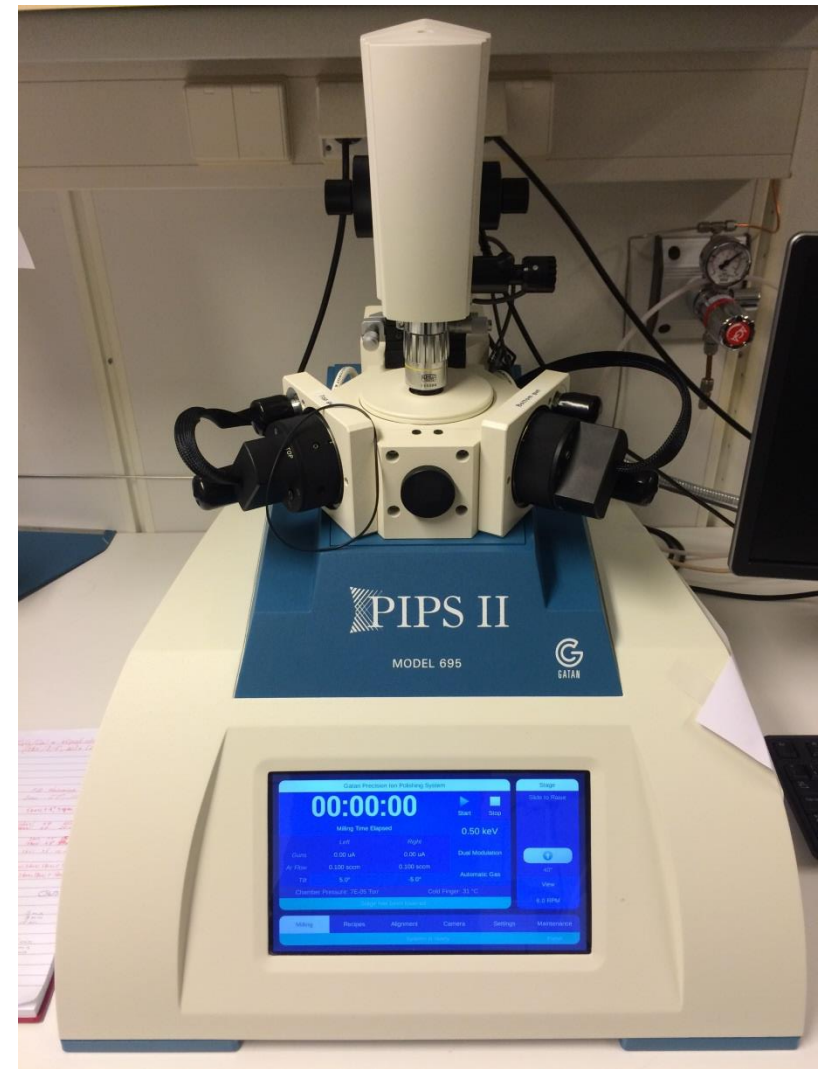**Multi-Prep Precision Polishing
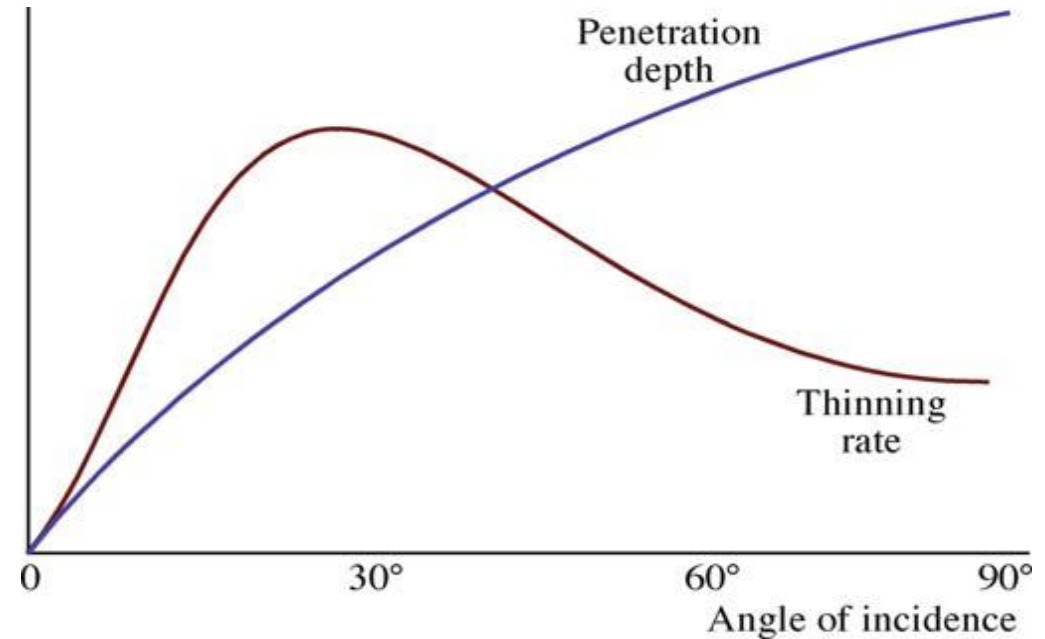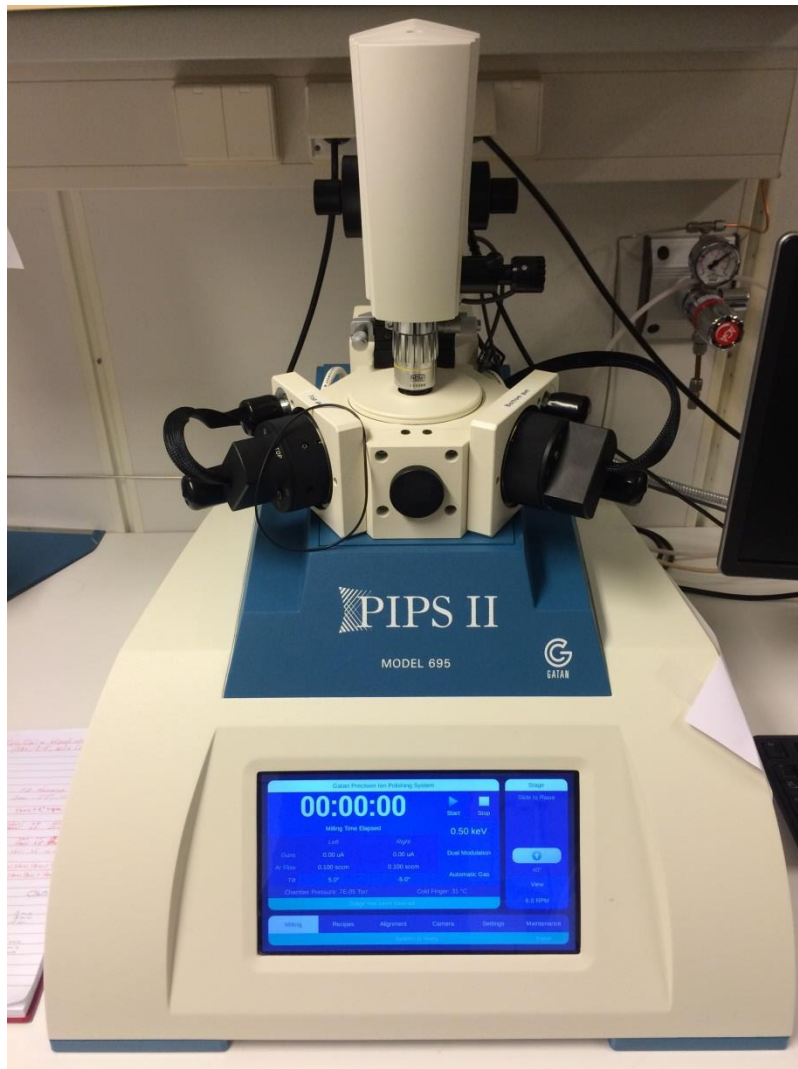(thinning down to ~ 50 – 10 µm)**

Final thinning
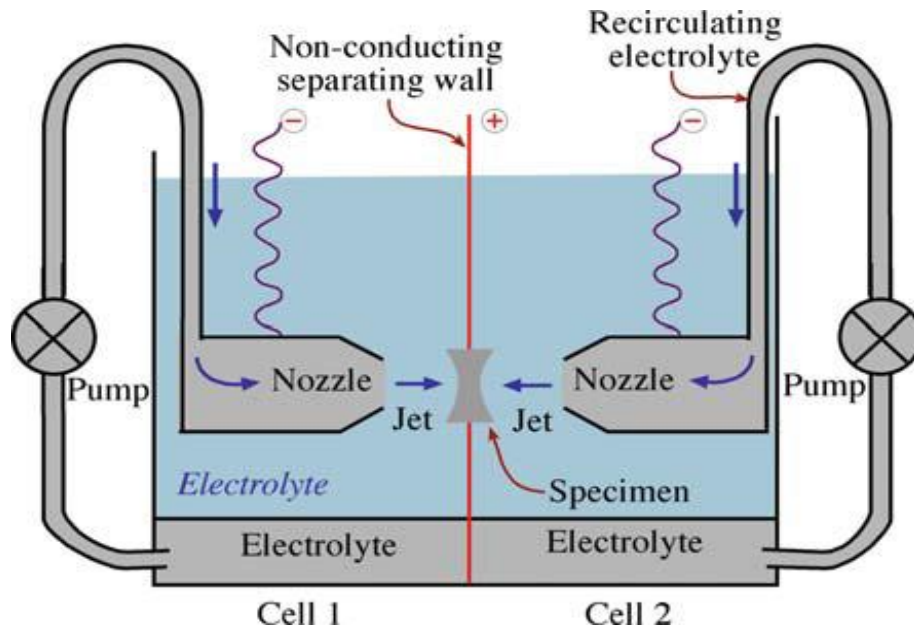
- Ionmilling
- Electropolishing

Ar ion beam thinning

Variation in penetration depth and thinning rate with the angle of incidence.
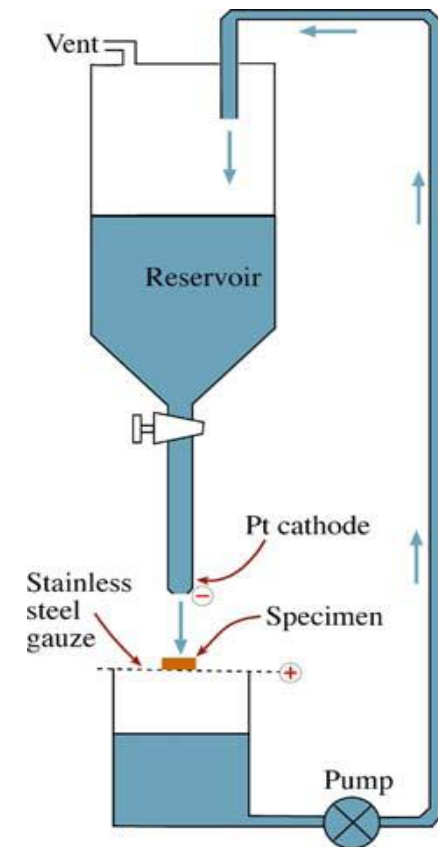
Typical Ar-ion beam milling conditions
Beam Energy: 6 – 0.1 keV
Milling Angle : 8° - 1°
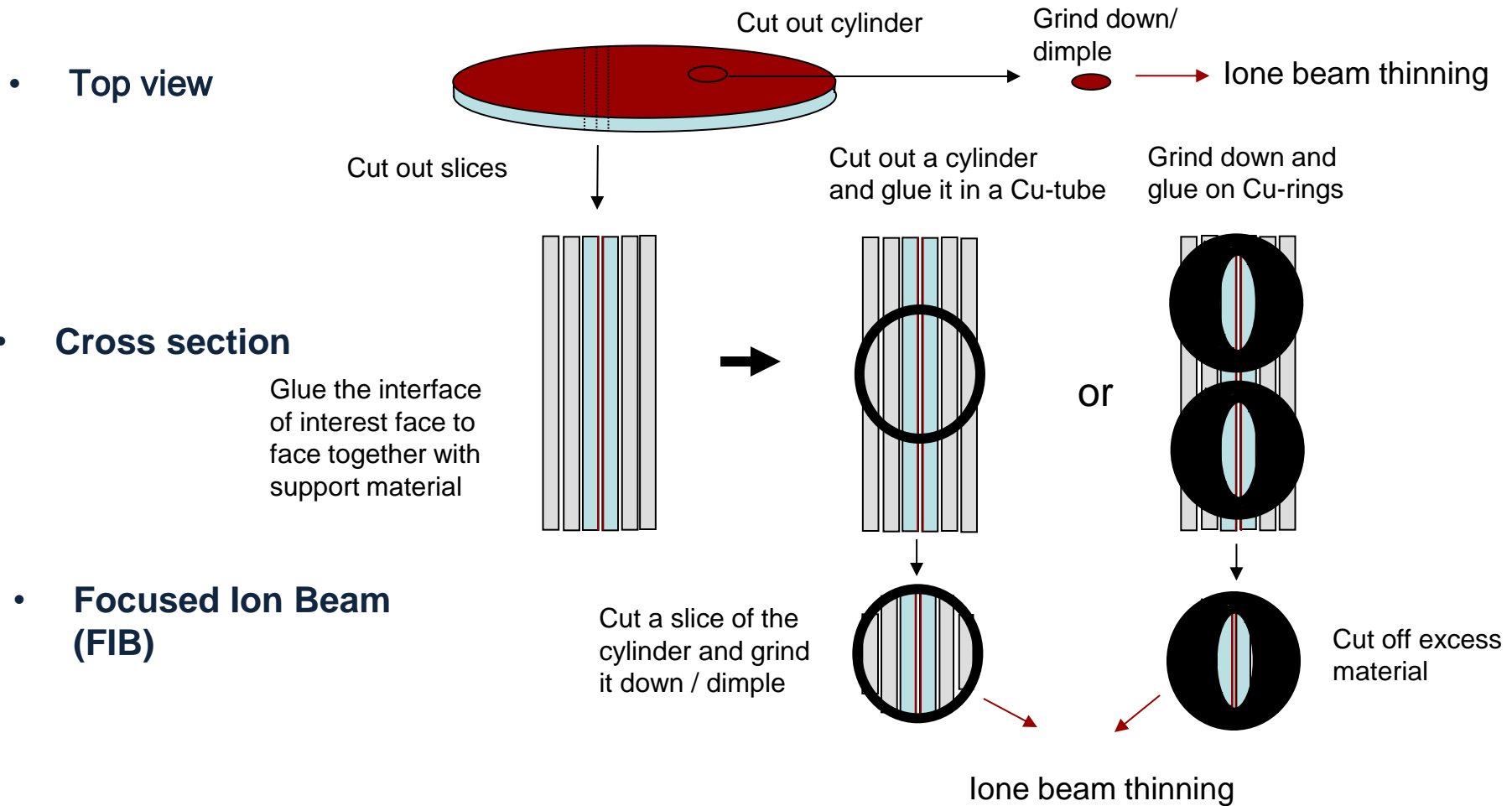
Electrochemical Jet polishing

Twin-jet electropolishing apparatus.

The positively charged specimen is held in a Teflon holder between the jets. A light pipe (not shown) detects perforation and terminates the polishing.

A single jet of gravity fed electrolyte thin a disk supported on a positively charged gauze. The disk has to be rotated periodically.

THIN FILMS TEM specimen preparation

- **Top view**

Cut out cylinder

Grind down/dimple

Ione beam thinning

Cut out slices

Cut out a cylinder and glue it in a Cu-tube

Grind down and glue on Cu-rings

- **Cross section**

Glue the interface of interest face to face together with support material

or

- **Focused Ion Beam (FIB)**

Cut a slice of the cylinder and grind it down / dimple

Cut off excess material

Ione beam thinning

Preparation of **powders, particles and fibers**

Crushing
in mortar and pestle
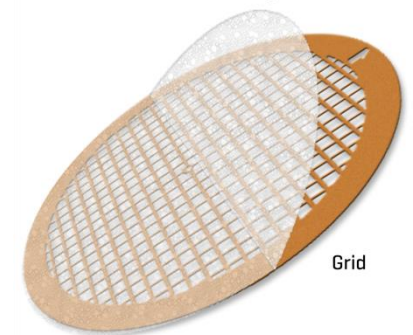in a neutral volatile solvent (eg. Ethanol)

+

Drop-casting on Support film TEM grids

Be Aware that organic solvents should be allowed to dry before inserting the specimen loaded support grids into TEM. To avoid electron beam induced reaction and contamination effects inside TEM.
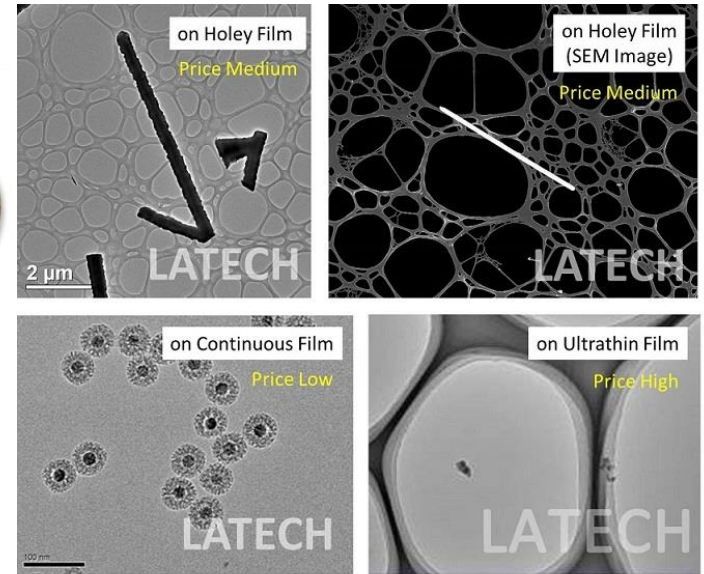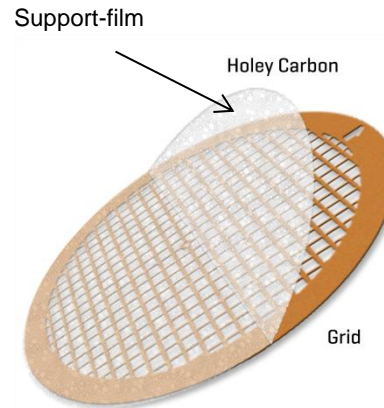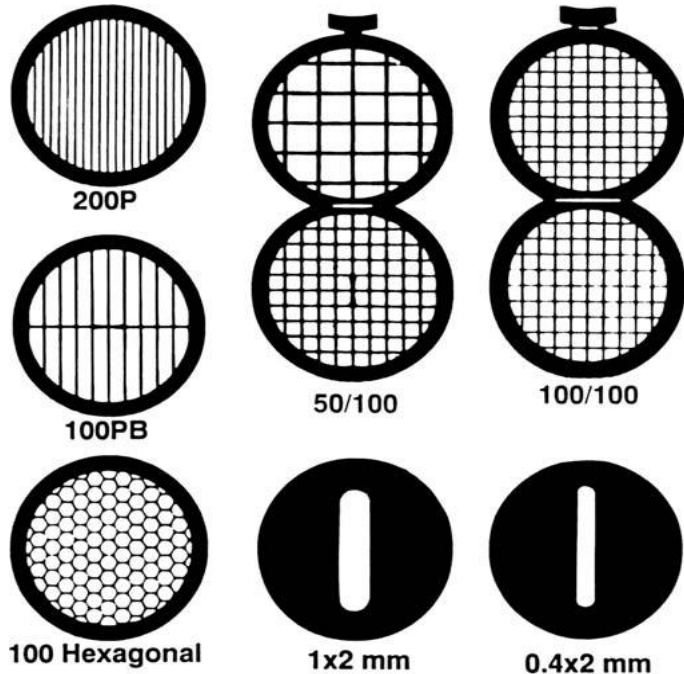
Holey Carbon

Grid

Courtesy: http://emresolutions.com

Supporting Grids and Support film Grids

200P
100PB
100 Hexagonal
50/100
100/100
1x2 mm
0.4x2 mm

Support-film
Holey Carbon
Grid

on Holey Film — Price Medium — 2 µm — LATECH
on Holey Film (SEM Image) — Price Medium — LATECH
on Continuous Film — Price Low — LATECH
on Ultrathin Film — Price High — LATECH

- **Continuous Amorphous carbon film (~ 200 nm – 20 nm thick)**
- **Holey Carbon support film**
- **Lacey Carbon support film**
- **Formvar support film**

Support grids material (Cu, Ni, Mo, Au) may contribute to the EDS signal.

Common size: 3 mm.
Smaller specimen diameters can be used for certain holders.

Courtesy: http://emresolutions.com
http://latech.com.sg

# Preparation of **powders, particles and fibers**

Embedding powders/fibers in conducting epoxy and supported by brass tube ring
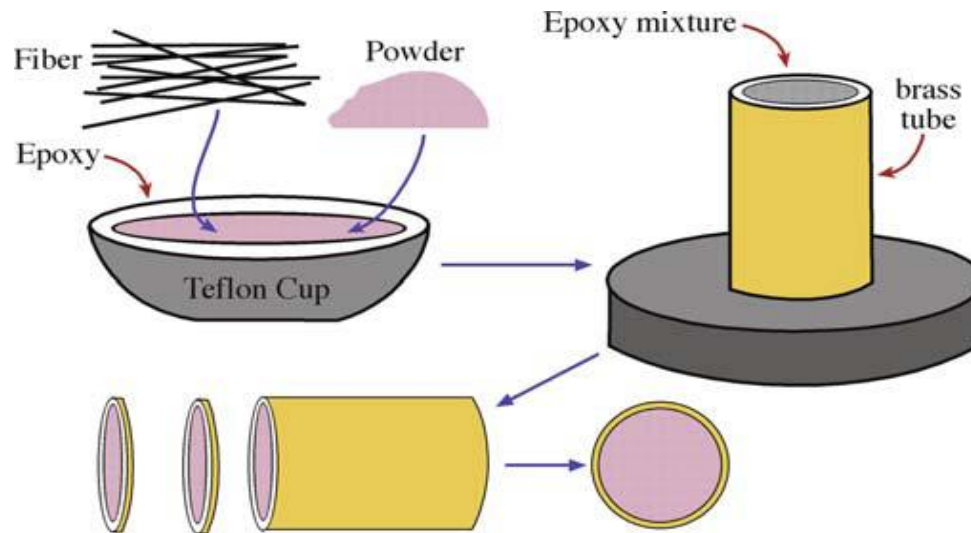
First embedding them in epoxy and forcing the epoxy into a 3-mm (outside) diameter brass tube prior to curing the epoxy. The tube and epoxy are then sectioned into disks with a diamond saw, dimpled, and ion milled to transparency.

FIB – To get very local site specific TEM samples

Marker

Bar

Pad

Trench

Final cut

TEM specimen

FIB

SEM
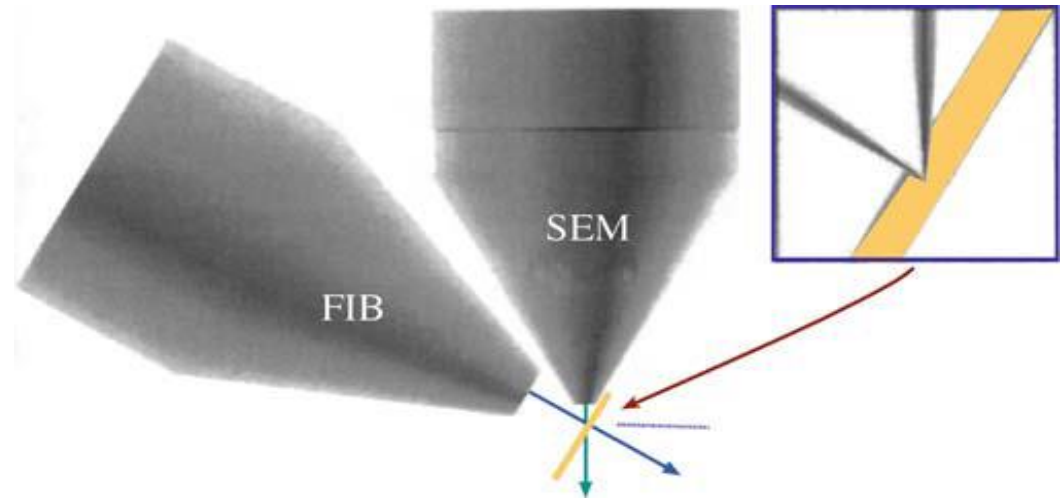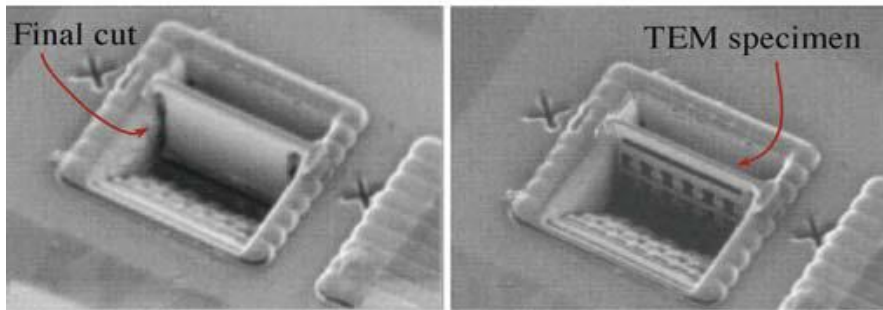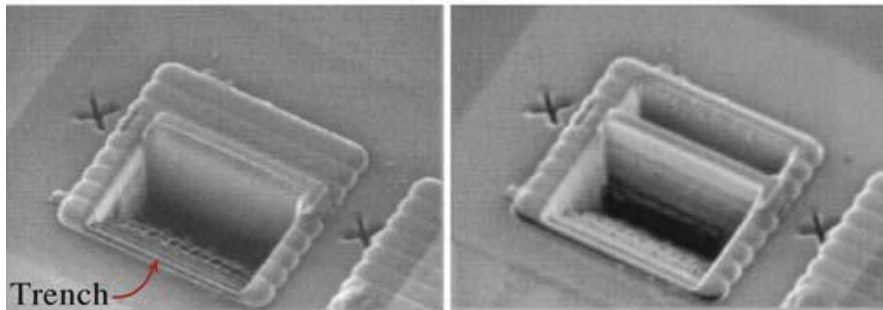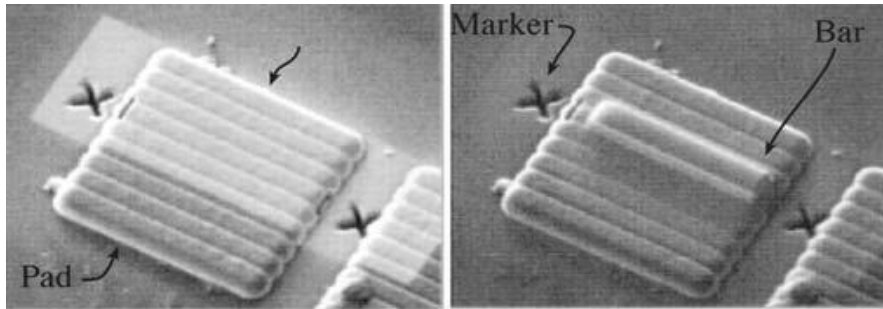
Schematic of a two-beam (electron and ion) FIB instrument.

-The area of interest has been marked.
-A Pt bar is deposited to protect this area from the Ga beam.
-The two trenches are cut.
-The bottom and sides of the slice are (final) cut.
-The TEM specimen is polished in place before extracting it.

Focused Ion Beam (FIB) instrument is usually integrated into a Scanning Electron Microscope (SEM)
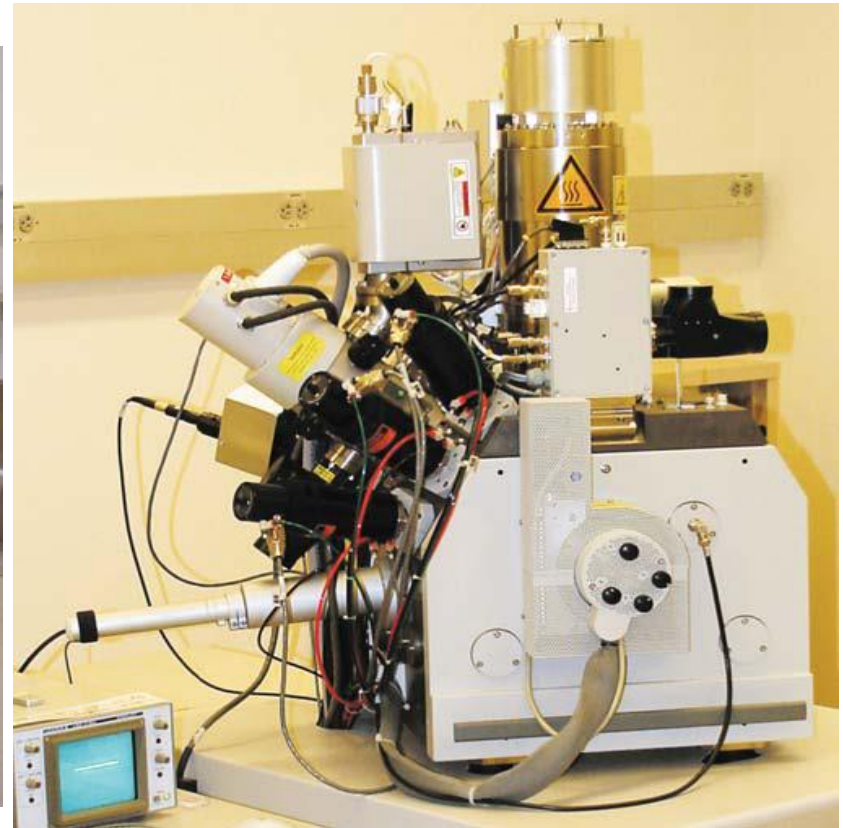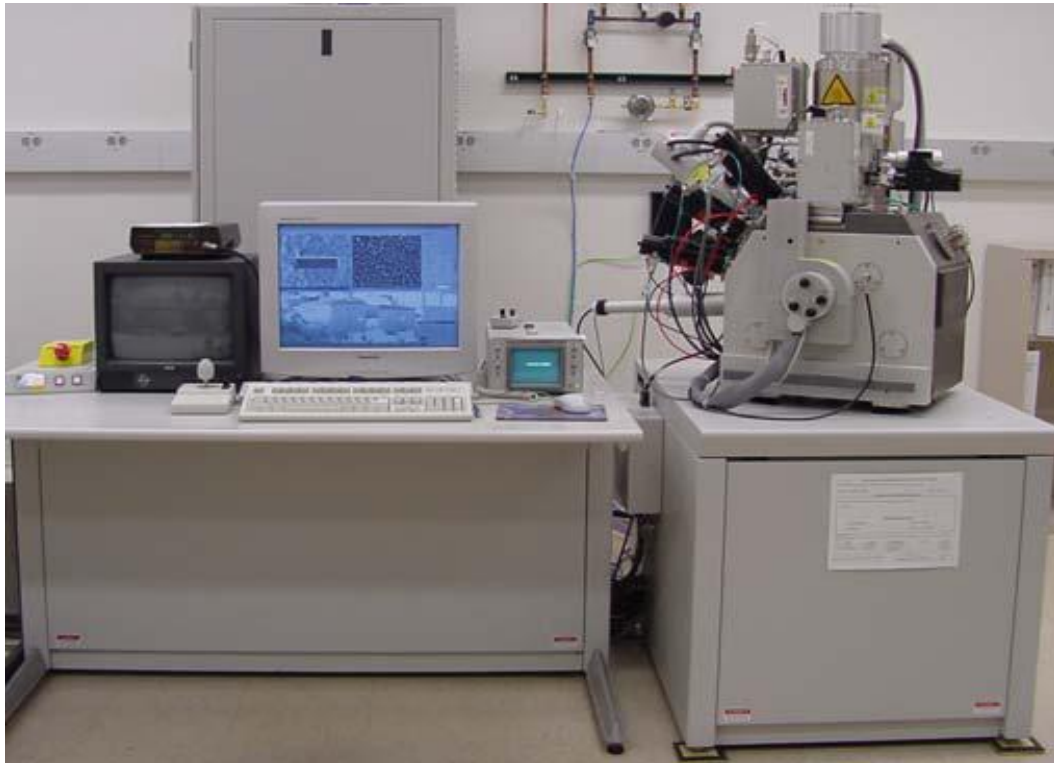
Ultramicrotomy

The sample is first embedded in epoxy or some other medium or the whole sample is clamped and moved across a knife edge.
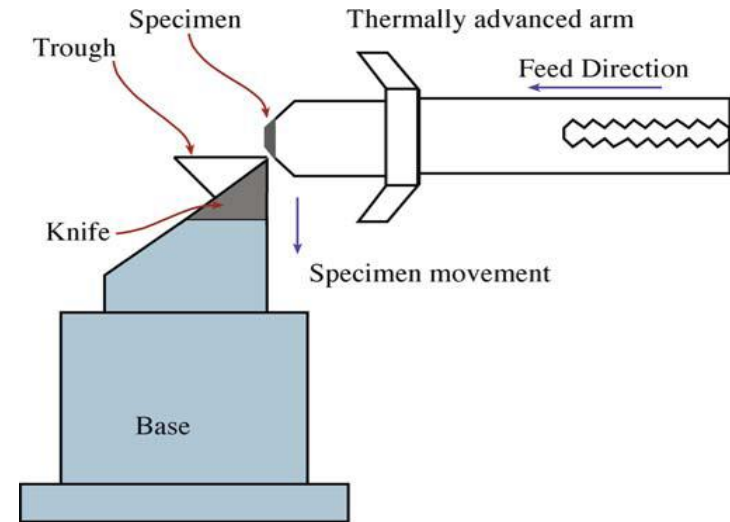
The thin flakes float off onto water or an appropriate inert medium, from where they are collected on grids.

Summary flow chart for specimen preparation

TEM Instrumentation

FEG gun
Extraction Anode
Gun lens
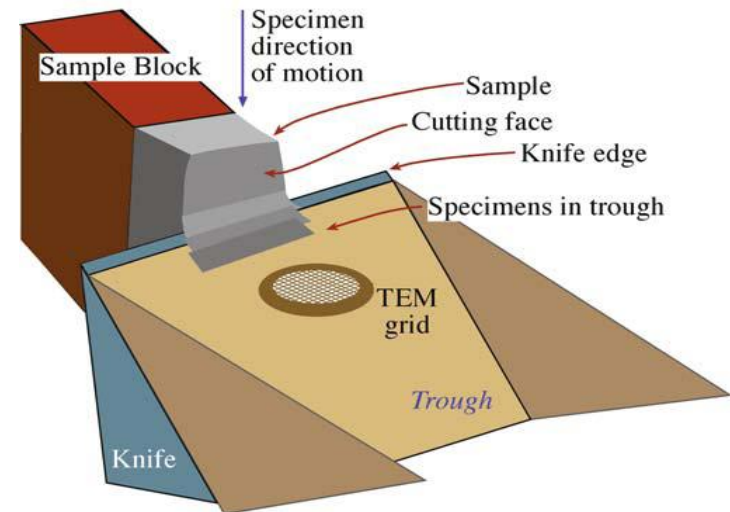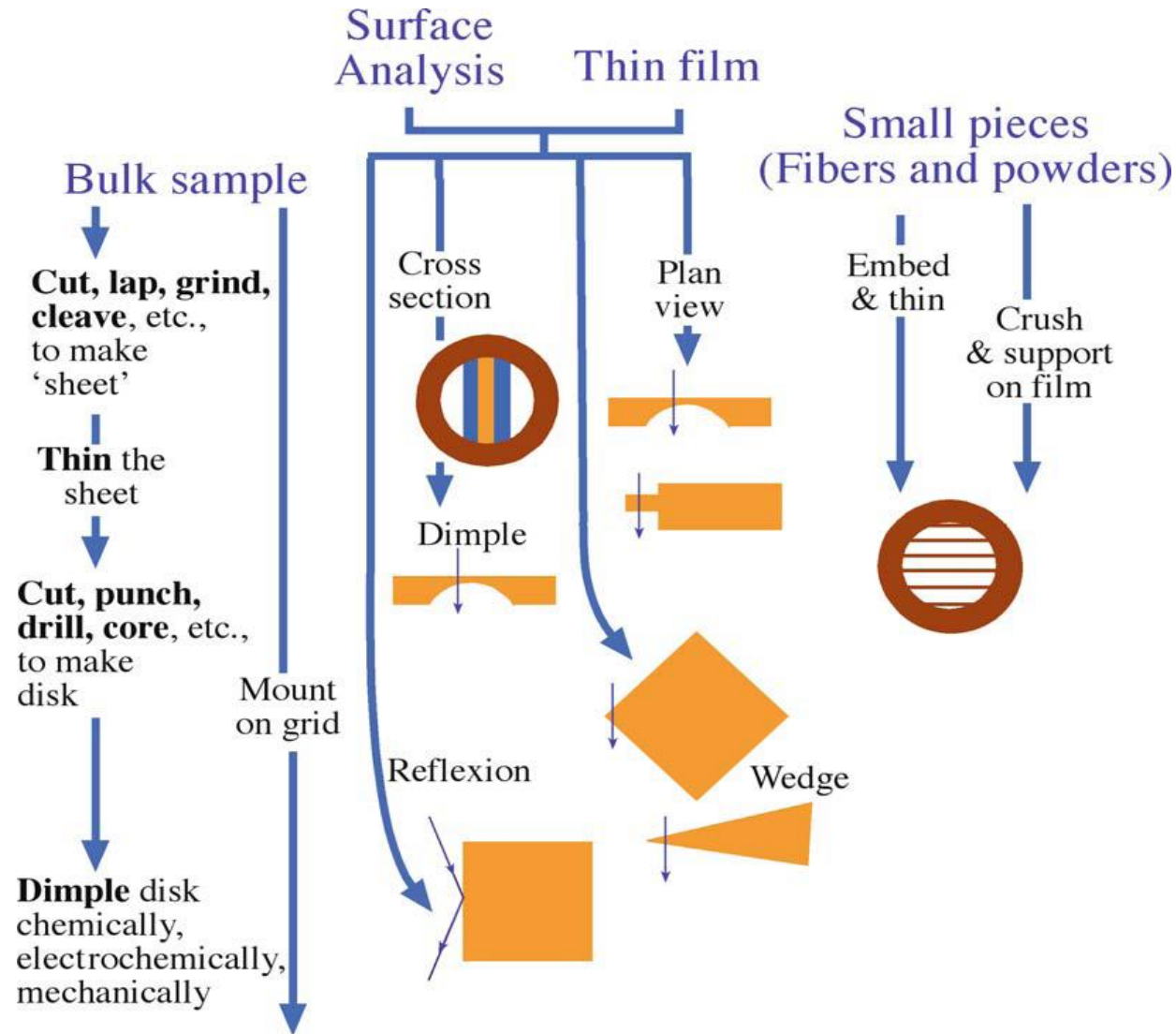Monochromator Aperture
Monochromator
Accelerator
Gun Shift coils
C1 aperture/mono energy slit
C1 lens
C2 lens
C2 aperture
Condenser alignment coils
C3 lens
C3 aperture
Beam shift coils
Mini condenser lens
Objective lens upper
Specimen Stage
Objective lens upper
Image Shift coils
Objective aperture
Cs Corrector
SA Aperture
Diffraction lens
Intermediate lens
Projector 1 lens
Projector 2 lens
HAADF detector
Viewing Chamber
Phosphorous Screen
BF/CCD detectors
EELS prism   GIF CCD detector

Courtesy: David Rassouw, CCEM, Canada

Electron gun

Illumination system

Specimen stage

Imaging system

Projection and Detection system

GIF CCD detector

Courtesy: David Rassouw

The diagram labels (top to bottom):

- FEG gun
- Extraction Anode
- Gun lens
- Monochromator Aperture
- Monochromator
- Accelerator
- Gun Shift coils
- C1 aperture/mono energy slit
- C1 lens
- C2 lens
- C2 aperture
- Condenser alignment coils
- C3 lens
- C3 aperture
- Beam shift coils
- Mini condenser lens
- Objective lens upper
- Specimen Stage
- Objective lens upper
- Image Shift coils
- Objective aperture
- Cs Corrector
- SA Aperture
- Diffraction lens
- Intermediate lens
- Projector 1 lens
- Projector 2 lens
- HAADF detector
- Viewing Chamber
- Phosphorous Screen
- BF/CCD detectors
- EELS prism
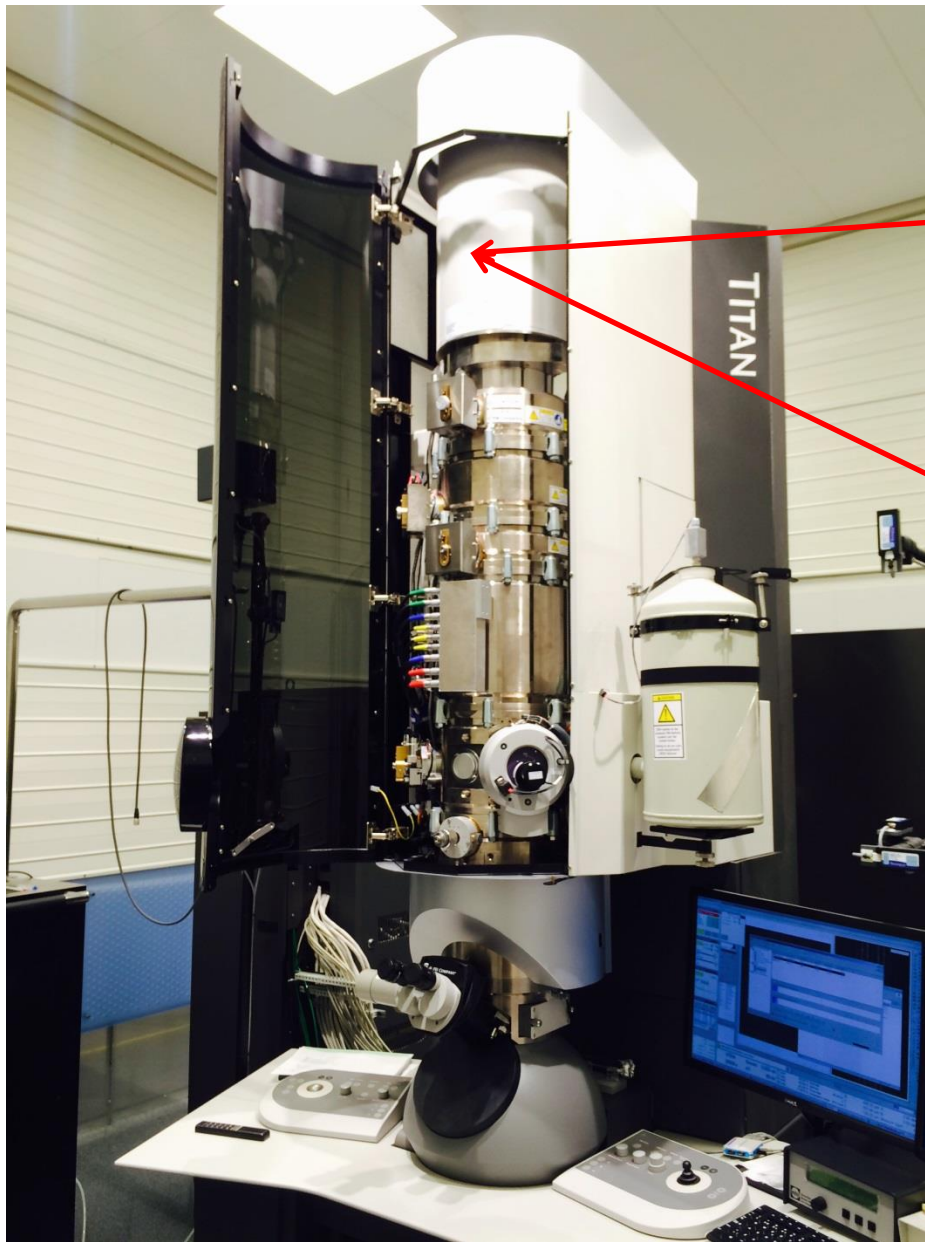- GIF CCD detector

- **Electron Gun**

- **Electron Lens**

- **Apertures**

- **Stigmators, scan coils and beam deflecting coils**

- **Specimen Stage/Holders**

- **Lq. N$_2$ Coldtrap**

- **Image Viewing/Recording system**
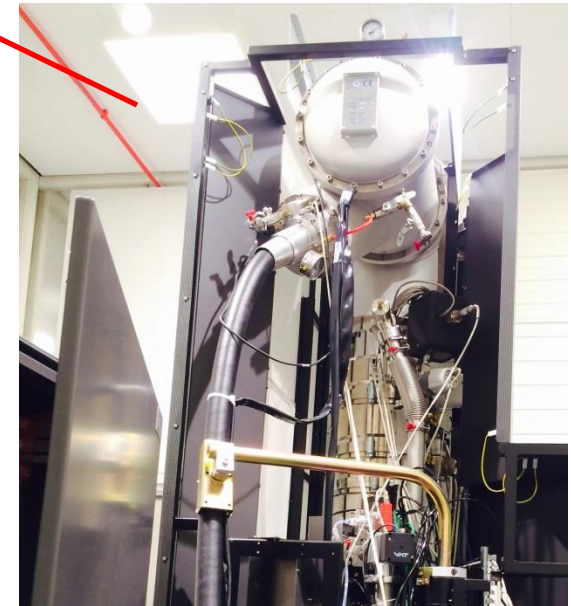
- **Spectrometers**

FEG Electron gun source

The electron source

- Two types of emission sources
  - **Thermionic emission**
    - W or LaB6
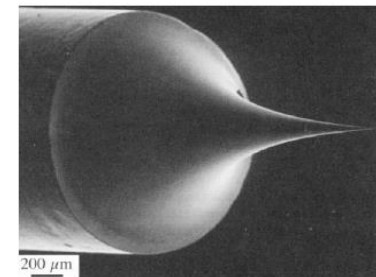
  - **Field emission**
    - **Cold FEG**   W
    - **Schottky FEG**   ZnO/W

University of Oslo
Centre for Materials Science and Nanotechnology (SMN)
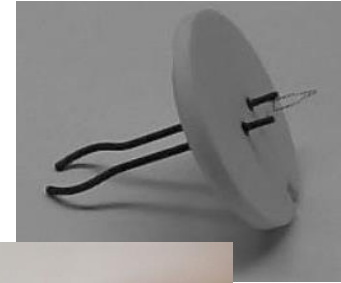
The electron gun

FEG

Emission of electrons induced by electrostatic field

Field emission tip →

First anode →

Second anode →

V_1 V_0
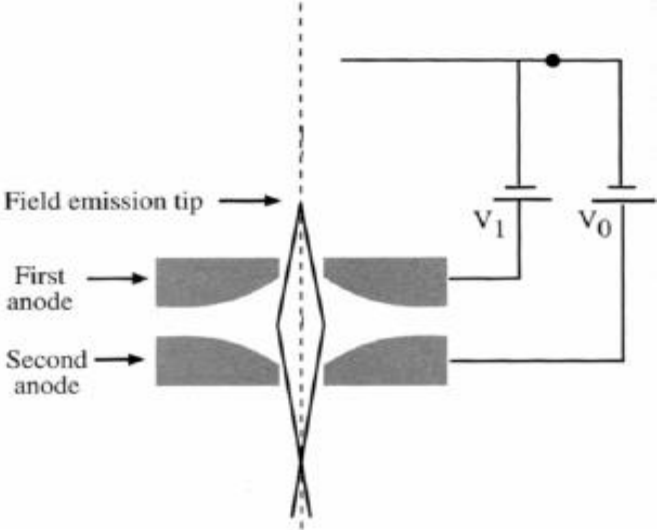
Thermionic gun

Emission of electrons induced by thermal heat

Wehnelt cylinder

Cathode

-200 kV

Bias -200 V

Equipotential lines

Anode

Ground potential

d_{cr} **Cross over**

α_{cr}
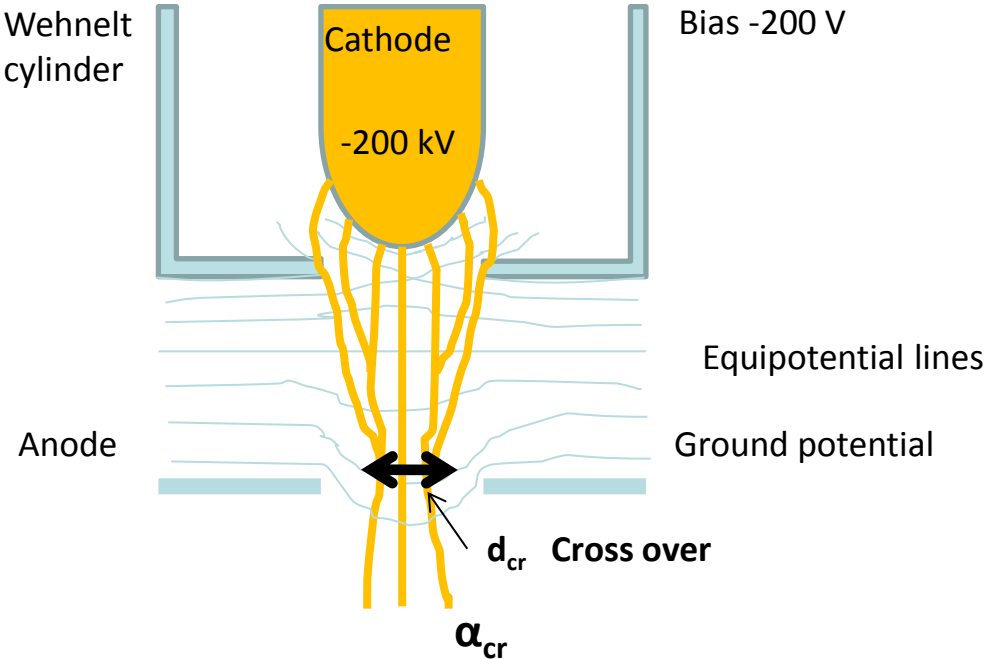
The electron gun

- The performance of the gun is characterised by:

    - Beam diameter, d_{cr}
    - Divergence angle, α_{cr}
    - Beam current, I_{cr}
    - Beam brightness, β_{cr}

    at the cross over

d $\longrightarrow$ $\longleftarrow$ Cross over

α
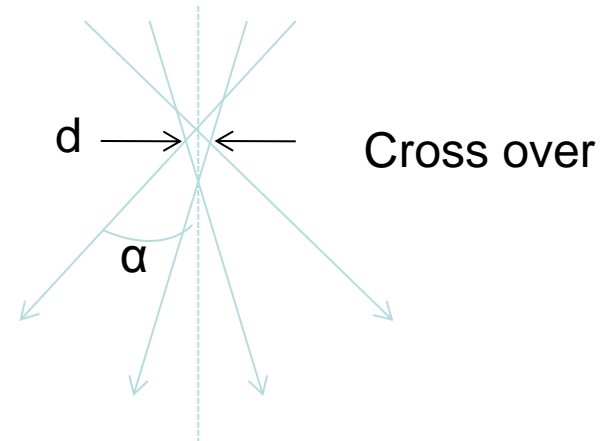
Image of source

Brightness

- Brightness (β) is the current density per unit solid angle of the source

- $\beta = i_{cr}/(\pi d_{cr} \alpha_{cr})^2$

Beam diameter, d_{cr}
Divergence angle, α_{cr}
Beam current, I_{cr}
Beam brightness, β_{cr}
at the cross over

UiO : University of Oslo
Centre for Materials Science and Nanotechnology (SMN)

Characteristics of principal electron sources at 200 kV

	W Thermionic	LaB6 Thermionic	FEG Schottky (ZrO/W)	FEG cold (W)
Current density J_c (A/m^2)	$2\text{-}3*10^4$	$25*10^4$	$1*10^7$	
Electron source size (µm)	50	10	0.1-1	0.010-0.100
Emission current (µA)	100	20	100	20~100
Brightness B (A/m^{2}sr)	$5*10^9$	$5*10^{10}$	$5*10^{12}$	$5*10^{12}$
Energy spread ΔE (eV)	2.3	1.5	0.6~0.8	0.3~0.7
Vacuum pressure (Pa)*	10^{-3}	10^{-5}	10^{-7}	10^{-8}
Vacuum temperature (K)	2800	1800	1800	300

* Might be one order lower

Lower the Gun Energy Spread than better for the energy and spatial resolution as it lowers chromatic aberration

Advantages and disadvantages of the different electron sources

W Advantages:	LaB$_6$ advantages:	FEG advantages:
Rugged and easy to handle	High brightness	Extremely high brightness
Requires only moderat vacuum	High total beam current	Long life time, more than 1000 h.
Good long time stability	Long life time (500-1000h)	
High total beam current		

W disadvantages:	LaB$_6$ disadvantages:	FEG disadvantages:
Low brightness	Fragile and delicate to handle	Very fragile
Limited life time (100 h)	Requires better vacuum	Current instabilities
	Long time instabilities	Ultra high vacuum to remain stable

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Centre for Materials Science and Nanotechnology (SMN)

Electron lenses

Any axially symmetrical electric or magnetic field have the properties
of an ideal lens for paraxial rays of charged particles.

- Electrostatic
  - Require high voltage- insulation problems   **F= -eE**
 - Not used as imaging lenses, but are used in modern monochromators

- ElectroMagnetic
  - Can be made more accurately   **F= -e(v x B)**
  - Shorter focal length

General features of magnetic lenses

- Focus near-axis electron rays with the same accuracy as a glass lens focusses near axis light rays

- Same aberrations as glass lenses

- Converging lenses

- The bore of the pole pieces in an objective lens is about 4 mm or less

- A single magnetic lens rotates the image relative to the object

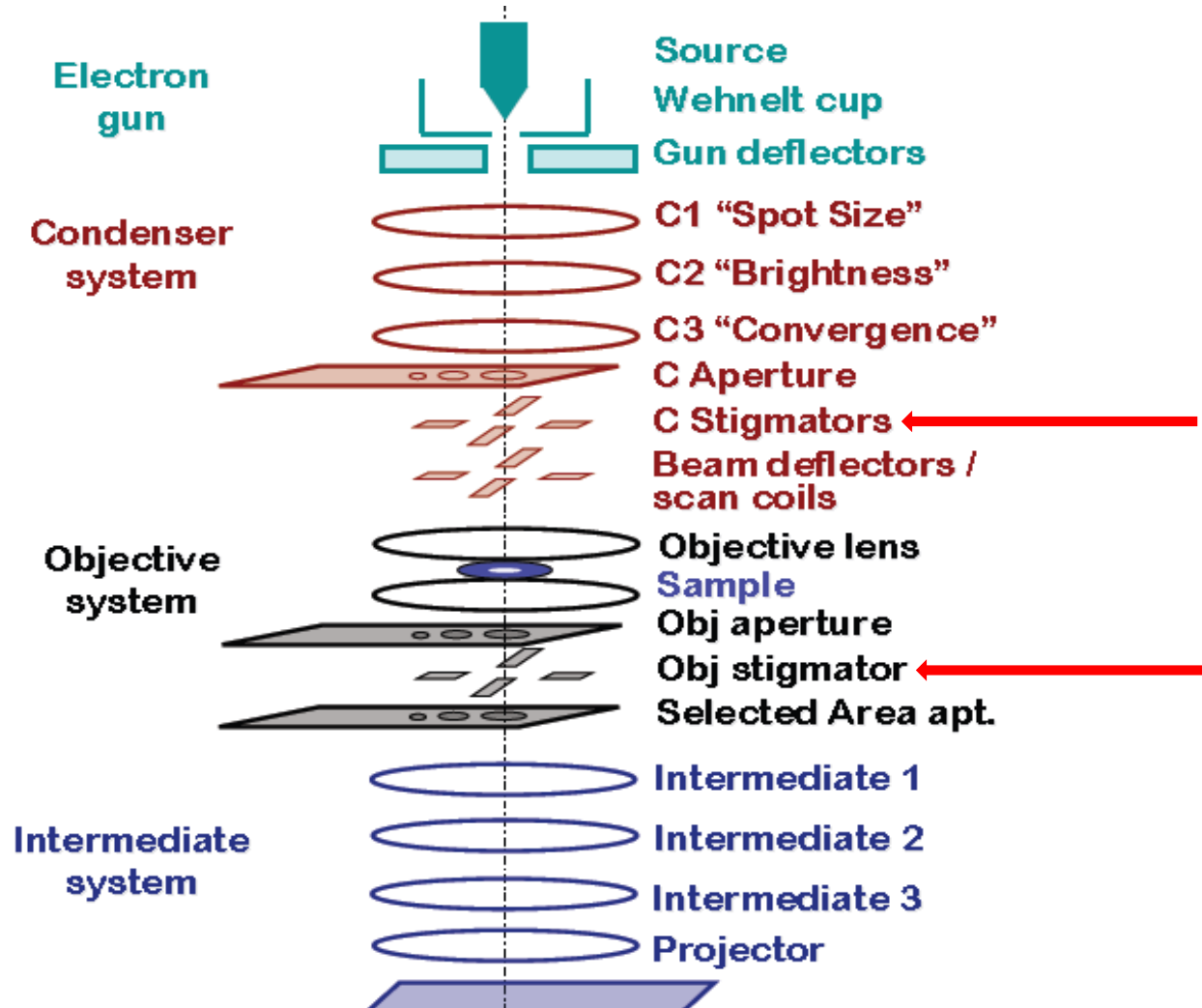- Focal length can be varied by changing the field between the pole pieces. (Changing magnification)
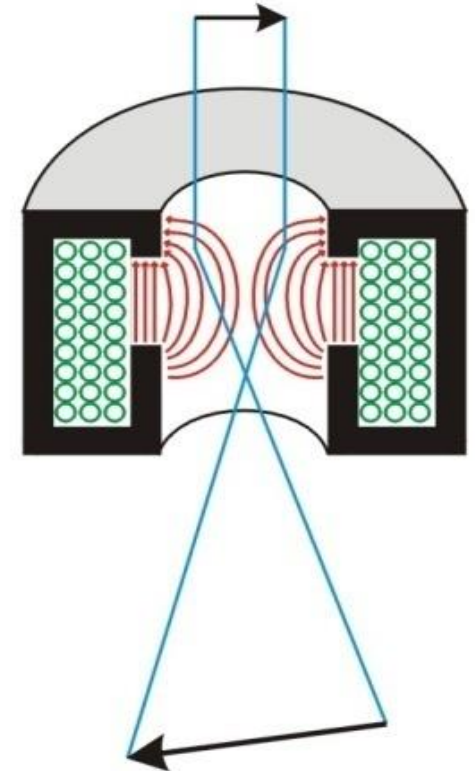
http://www.matter.org.uk/tem/lenses/electromagnetic_lenses.htm

The Objective lens
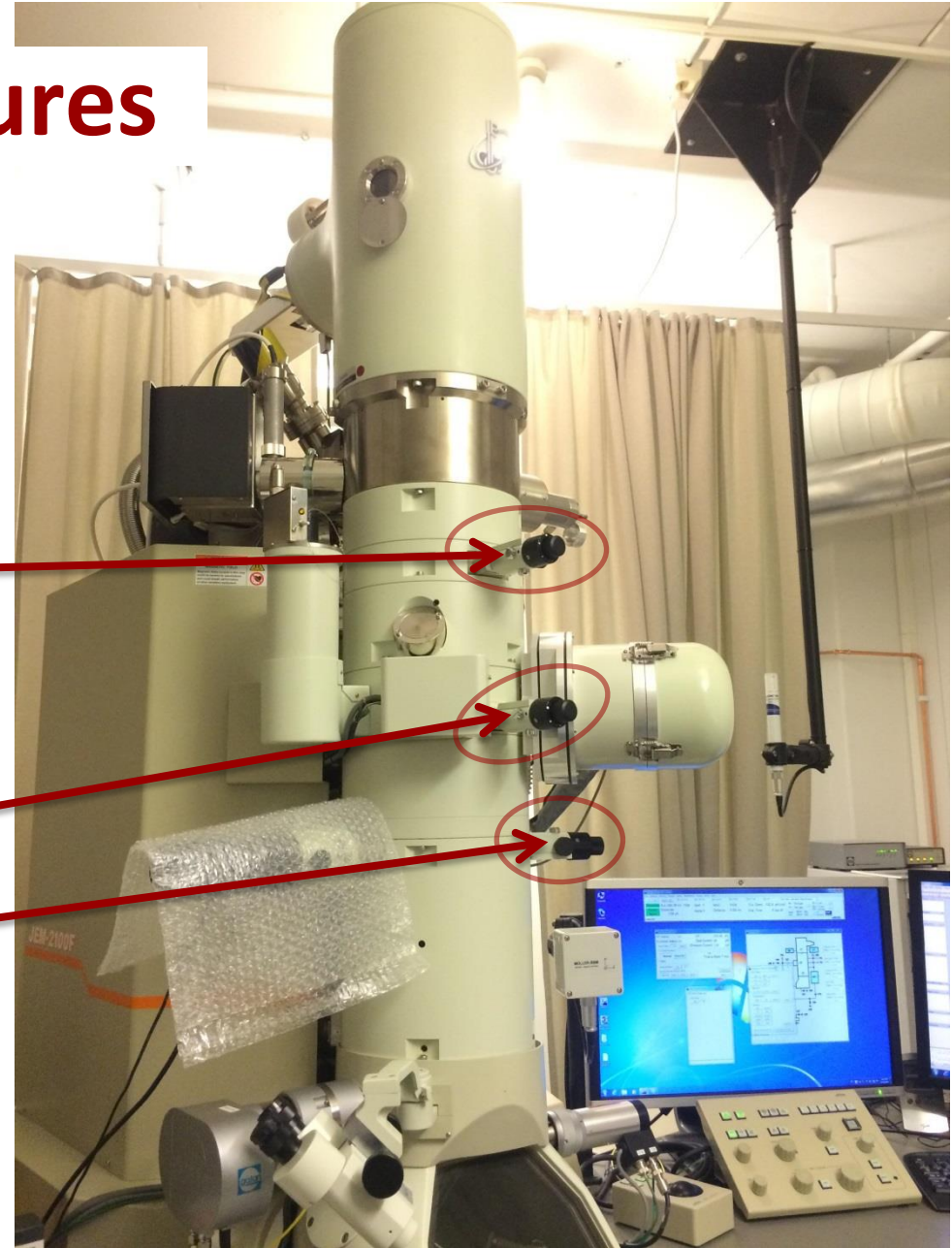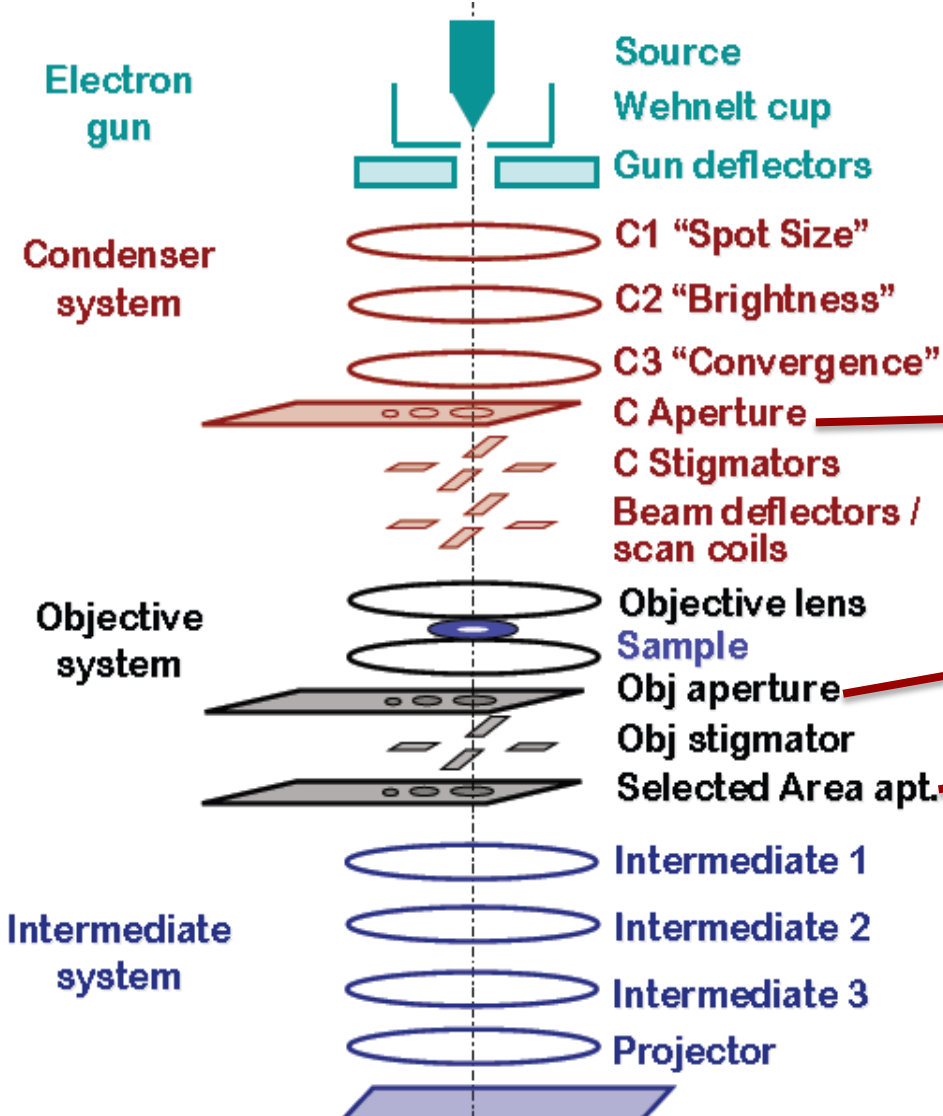
- Often a double or twin lens

- The most important lens
  - Determines the reolving power of the TEM
    - All the aberations of the objective lens are magnified by the intermediate and projector lens.

- The most important aberrations
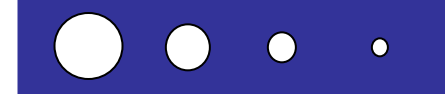 - Asigmatism
  - Spherical aberration
  - Chromatic aberration

Stigmators – to correct astigmatism

Electron gun
- Source
- Wehnelt cup
- Gun deflectors

Condenser system
- C1 "Spot Size"
- C2 "Brightness"
- C3 "Convergence"
- C Aperture
- C Stigmators ←
- Beam deflectors / scan coils

Objective system
- Objective lens
- Sample
- Obj aperture
- Obj stigmator ←
- Selected Area apt.

Intermediate system
- Intermediate 1
- Intermediate 2
- Intermediate 3
- Projector

Apertures

Electron gun
- Source
- Wehnelt cup
- Gun deflectors

Condenser system
- C1 "Spot Size"
- C2 "Brightness"
- C3 "Convergence"
- C Aperture
- C Stigmators
- Beam deflectors / scan coils

Objective system
- Objective lens
- Sample
- Obj aperture
- Obj stigmator
- Selected Area apt.

Intermediate system
- Intermediate 1
- Intermediate 2
- Intermediate 3
- Projector

Use of apertures

Condenser aperture:

Limit the beam divergence (reducing the diameter of the discs in the convergent electron diffraction pattern).
Limit the number of electrons hitting the sample (reducing the intensity),
.

Objective aperture:

Control the contrast in the image. Allow certain reflections to contribute to the image. Bright field imaging (central beam, 000), Dark field imaging (one reflection, **g**), High resolution Images (several reflections from a zone axis).

Selected area aperture:

Select diffraction patterns from small (> 1µm) areas of the specimen.
Allows only electrons going through an area on the sample that is limited by the SAD aperture to contribute to the diffraction pattern (SAD pattern).

Objective aperture: Contrast enhancement
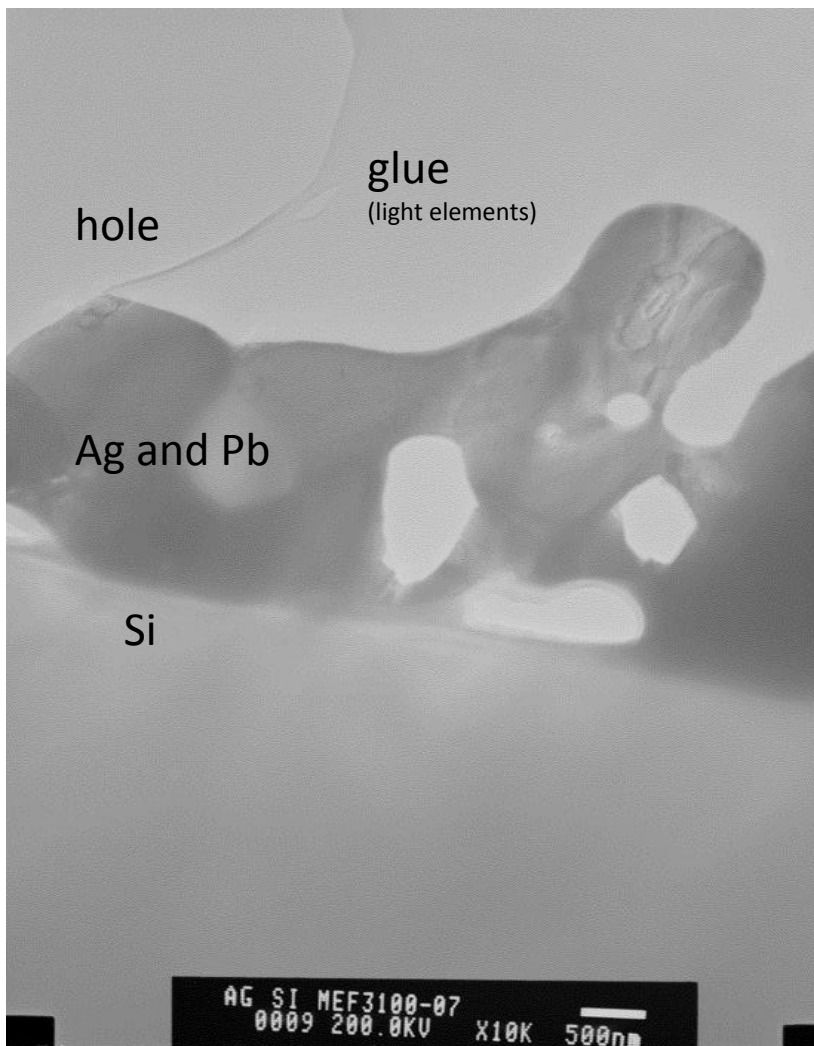
All electrons contributes to the image.
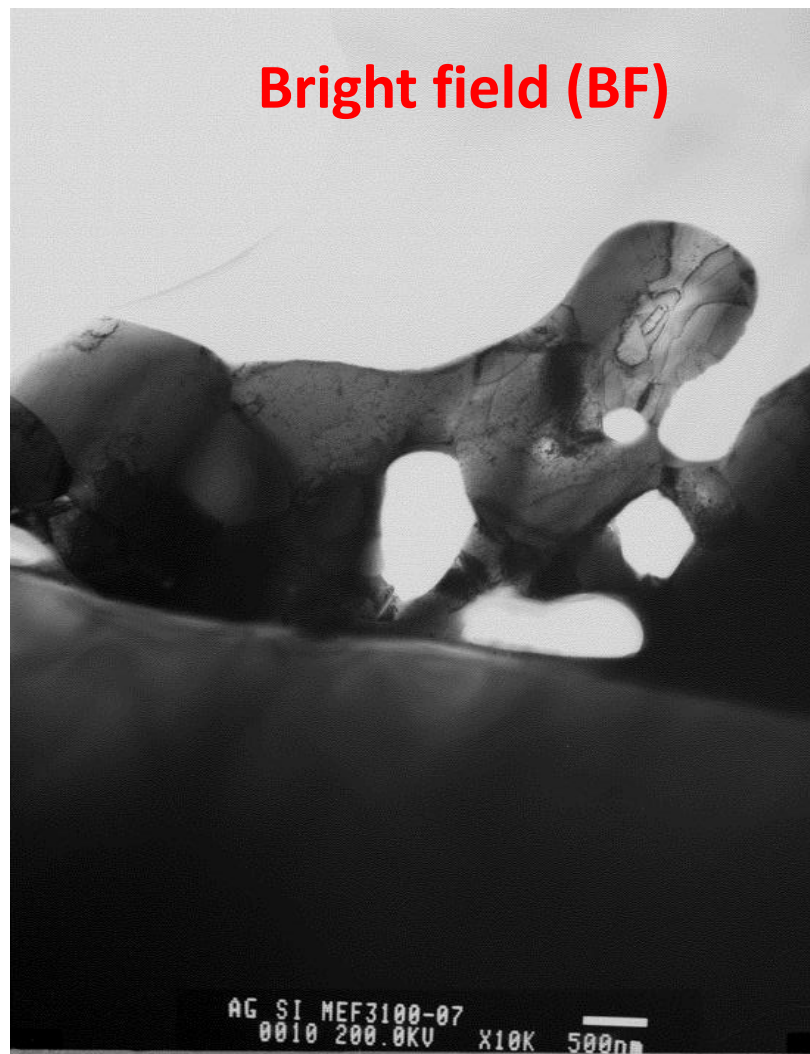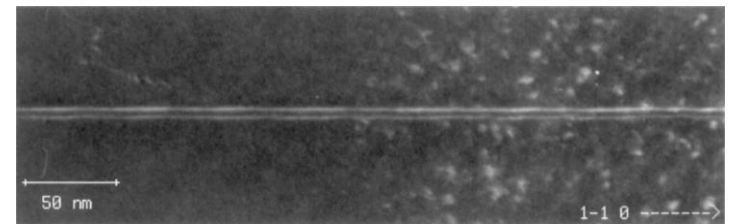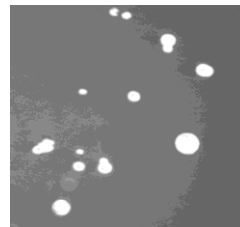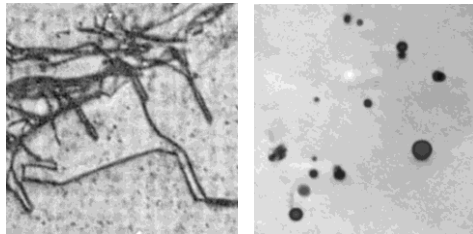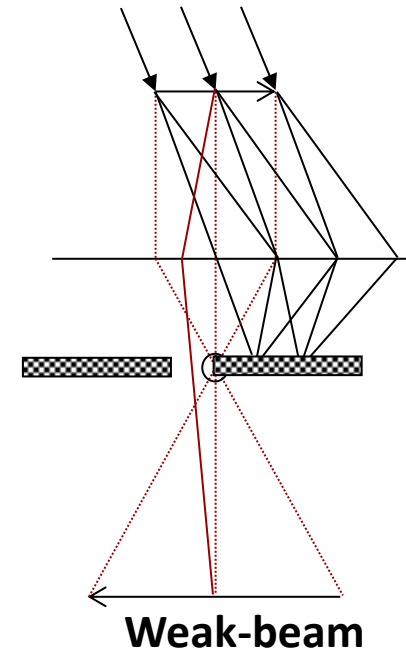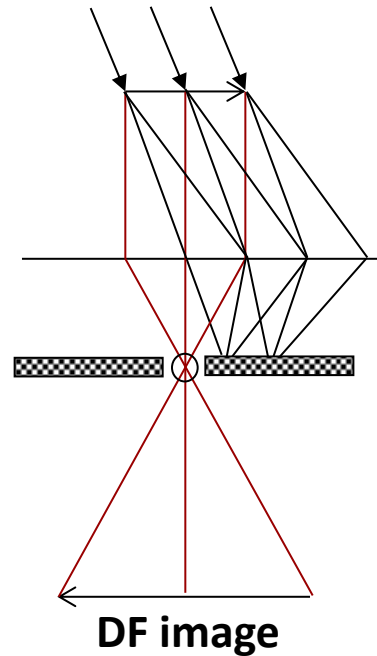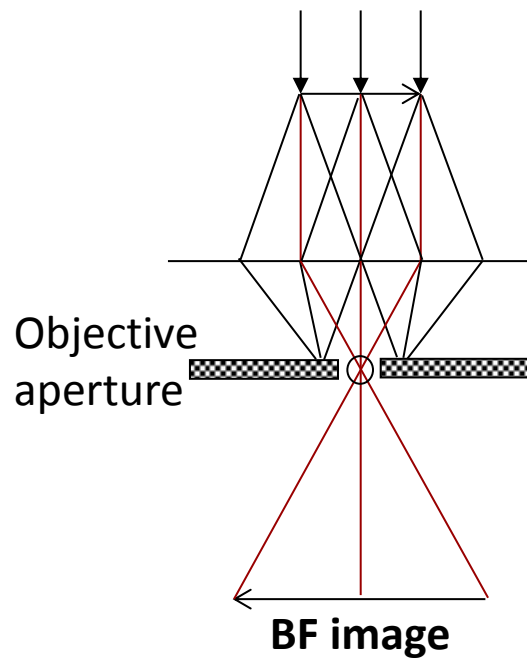
Only central beam contributes to the image.

Small objective aperture

## Bright field (BF), dark field (DF) and weak-beam (WB)

(Diffraction contrast)

Objective aperture

BF image   **DF image**   **Weak-beam**

Dissociation of pure screw dislocation In Ni$_3$Al, Meng and Preston, J. Mater. Scicence, 35, p. 821-828, 2000.
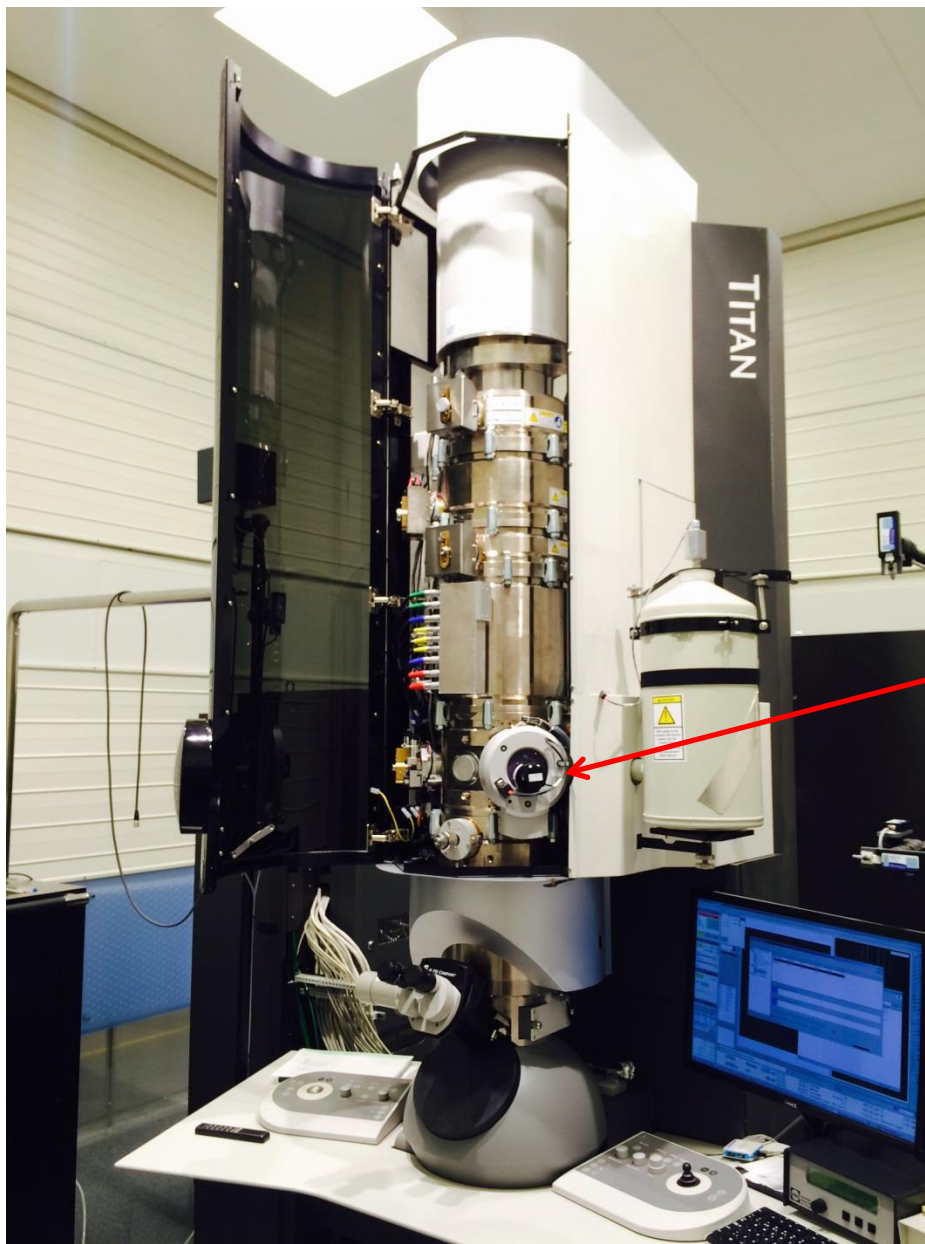
Selected Area Diffraction Aperture

Selected area diffraction

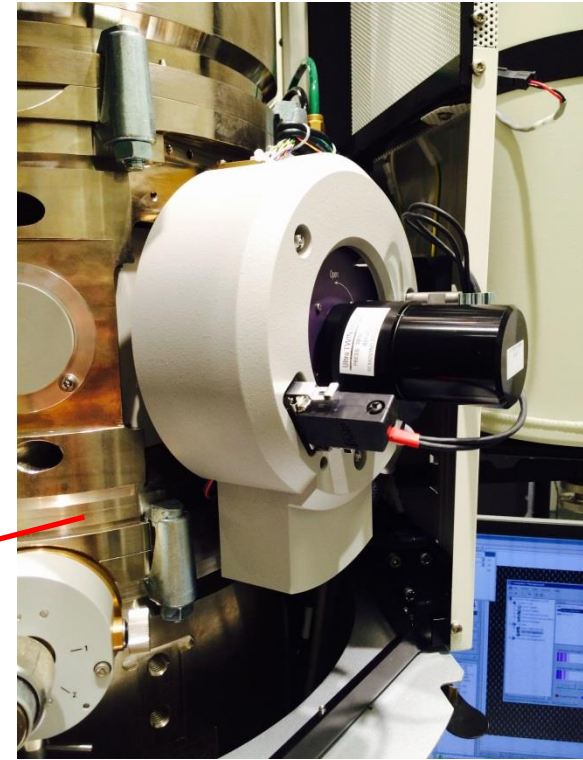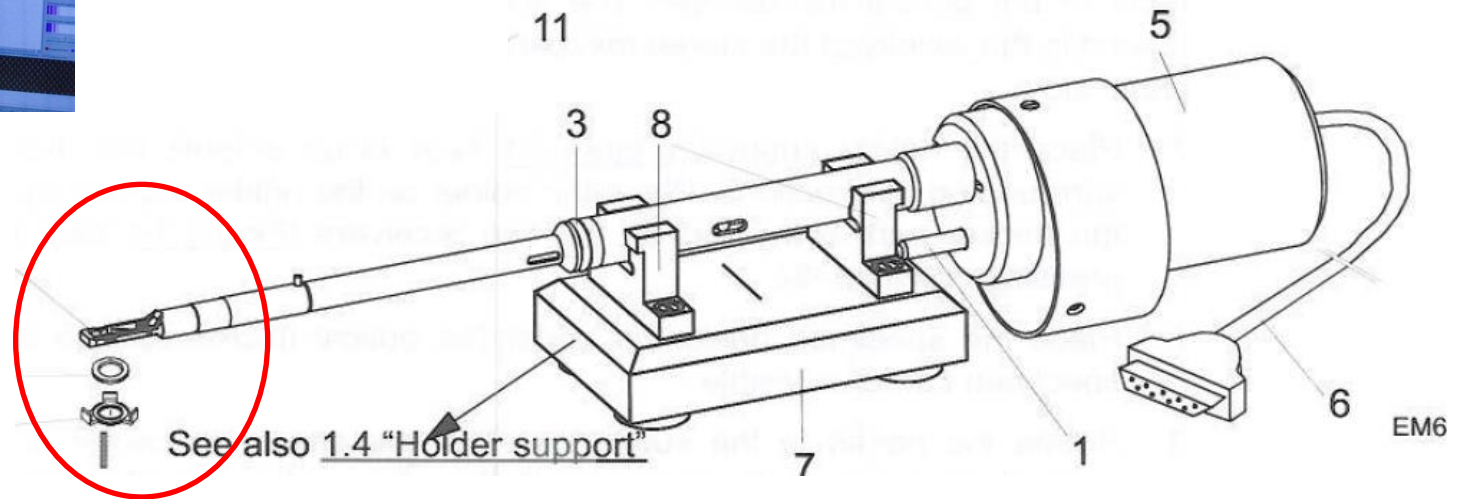Parallel incoming electron beam

Specimen with two crystals (red and blue)

Objective lense

Pattern on the screen
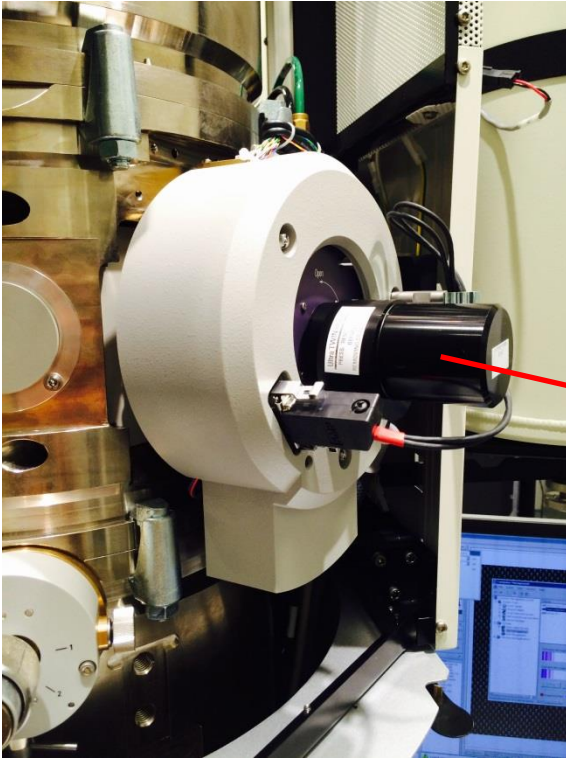
Diffraction pattern

Image plane
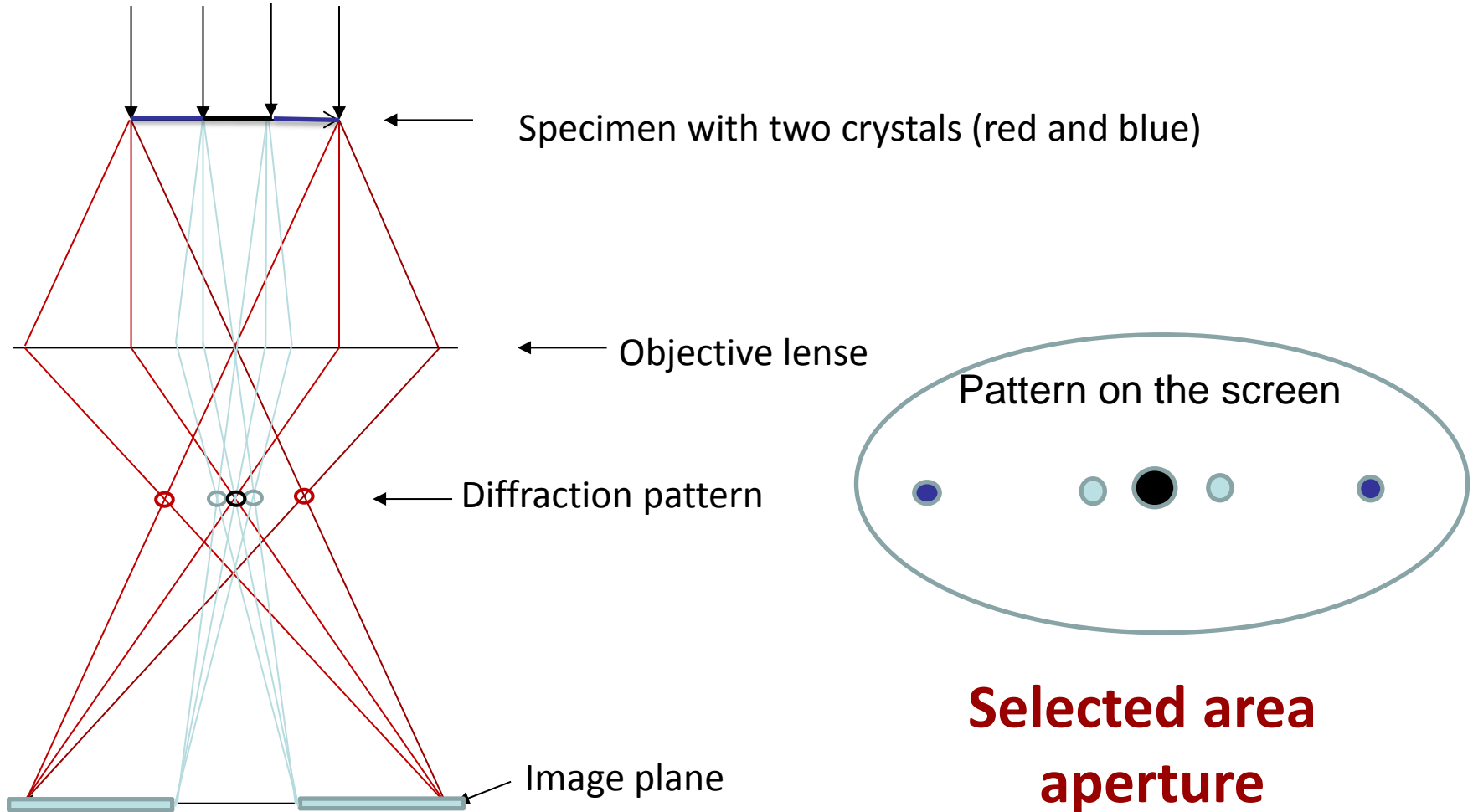
Selected area aperture

TEM Specimen Holder
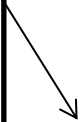
Specimen holders and goniometers

- **Specimen holders**

  - Single tilt holders
  - Double tilt holders
  - High tilt holders $\longrightarrow$ **3D imaging (TOMOGRAPHY)**
  - Rotation holders

  - Heating holders
  - Cooling holders
  - Strain holders
  - Electrical Biasing Holders
  - Environmental cells

- **Goniometers:**

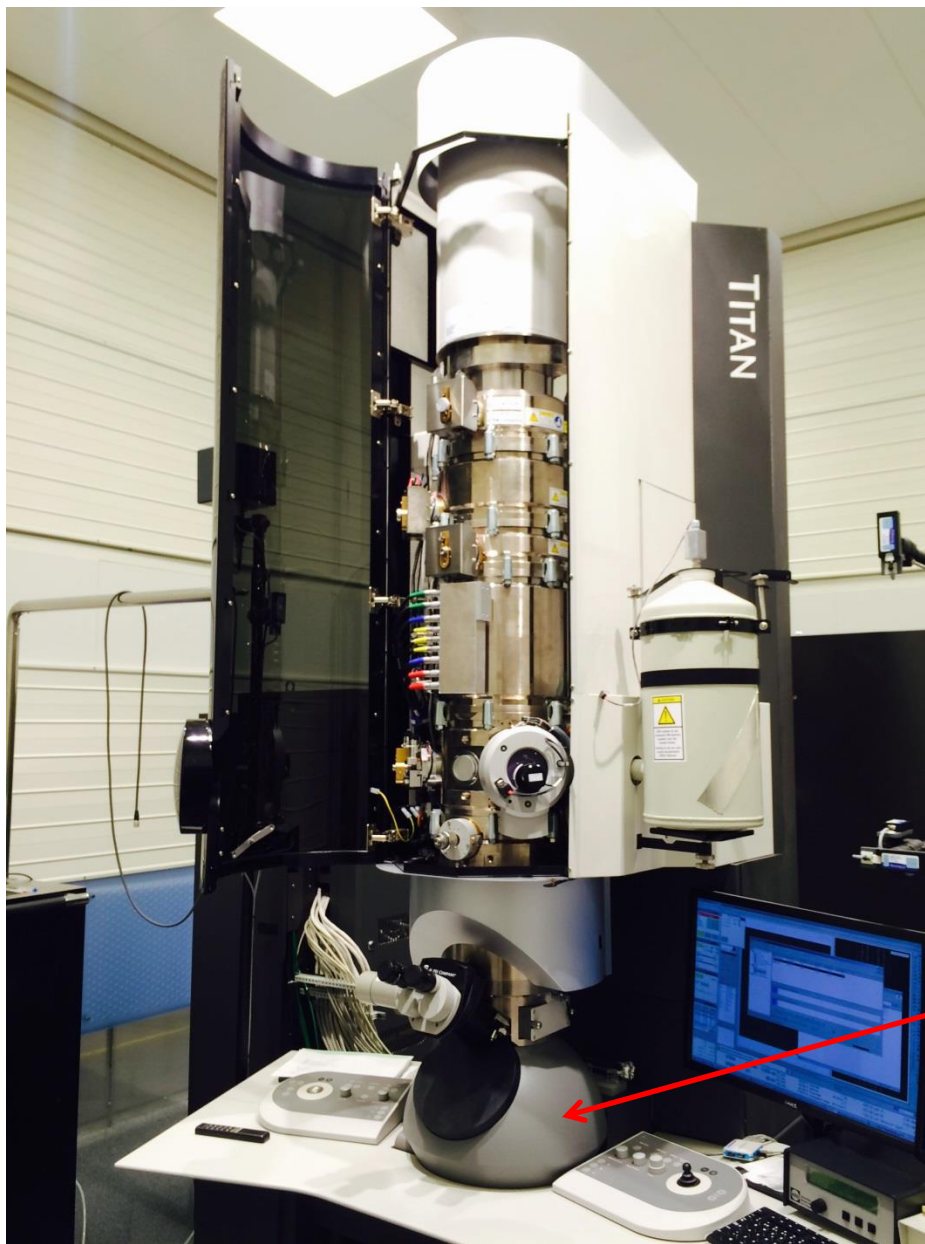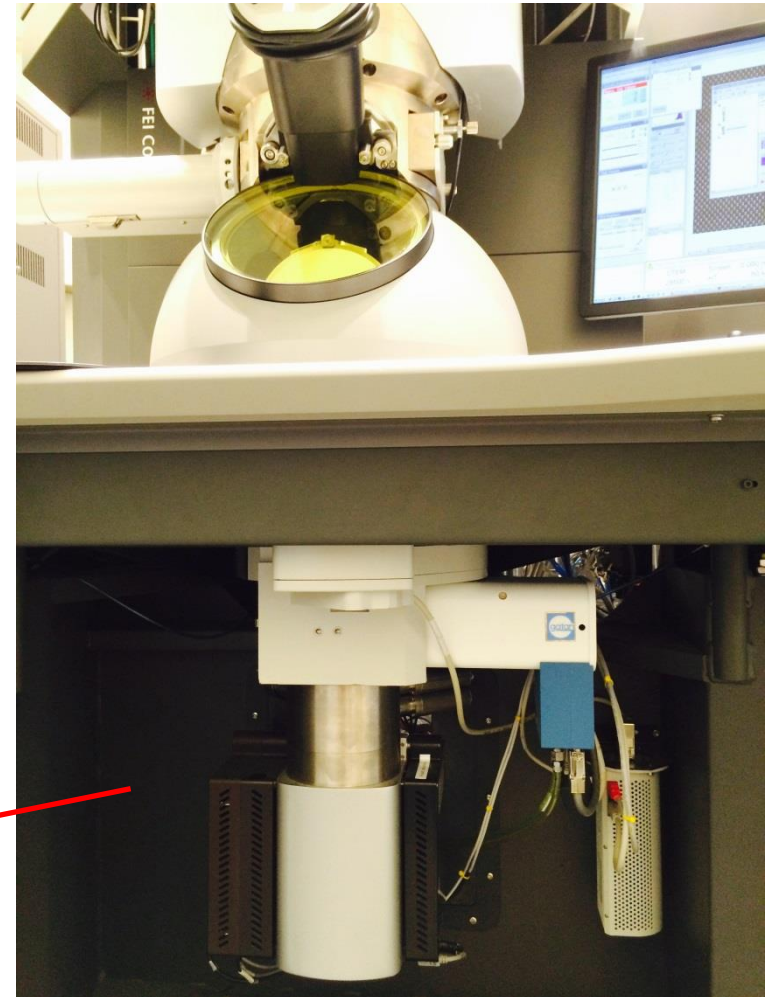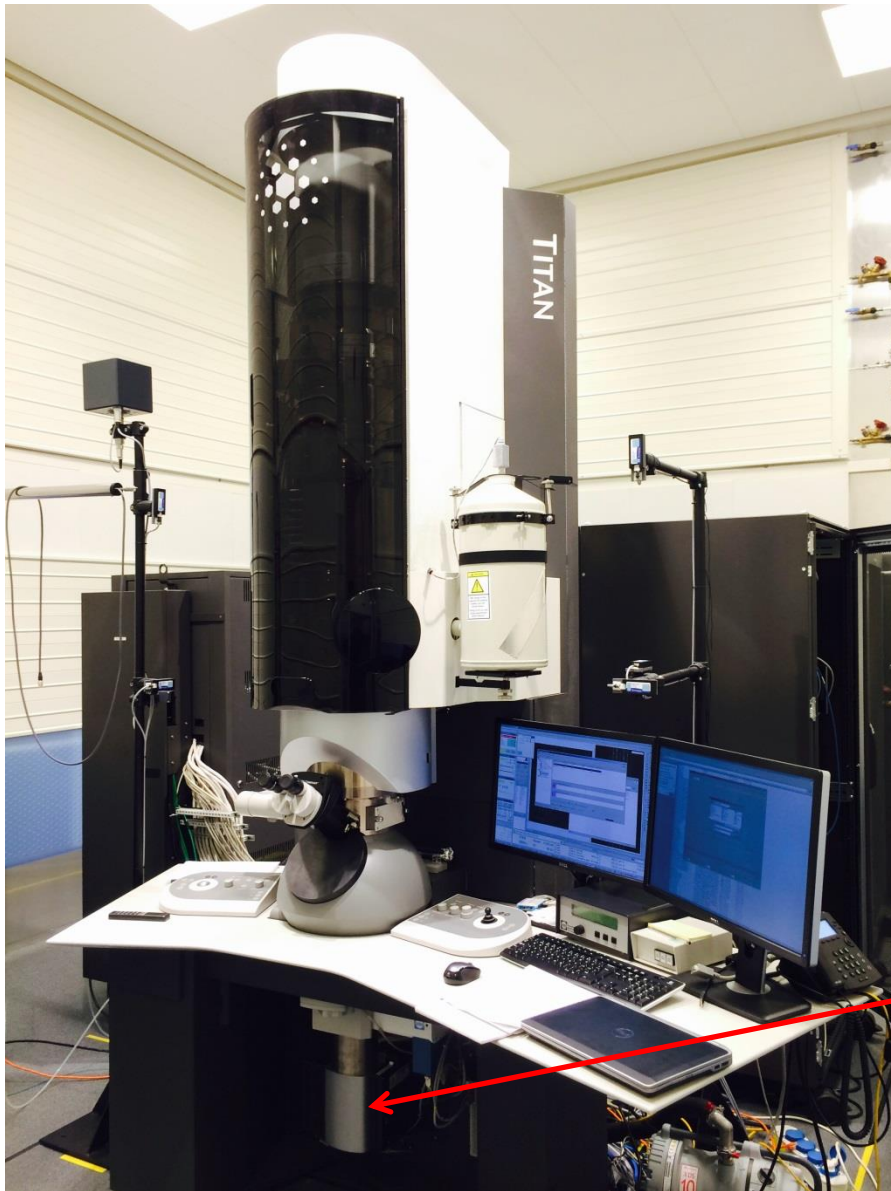 **- Side-entry stage**
  - Most common type
  - Eucentric

  - Top-entry stage
  - Less obj. lens aberrations
  - Not eucentric
  - Smaller tilting angles
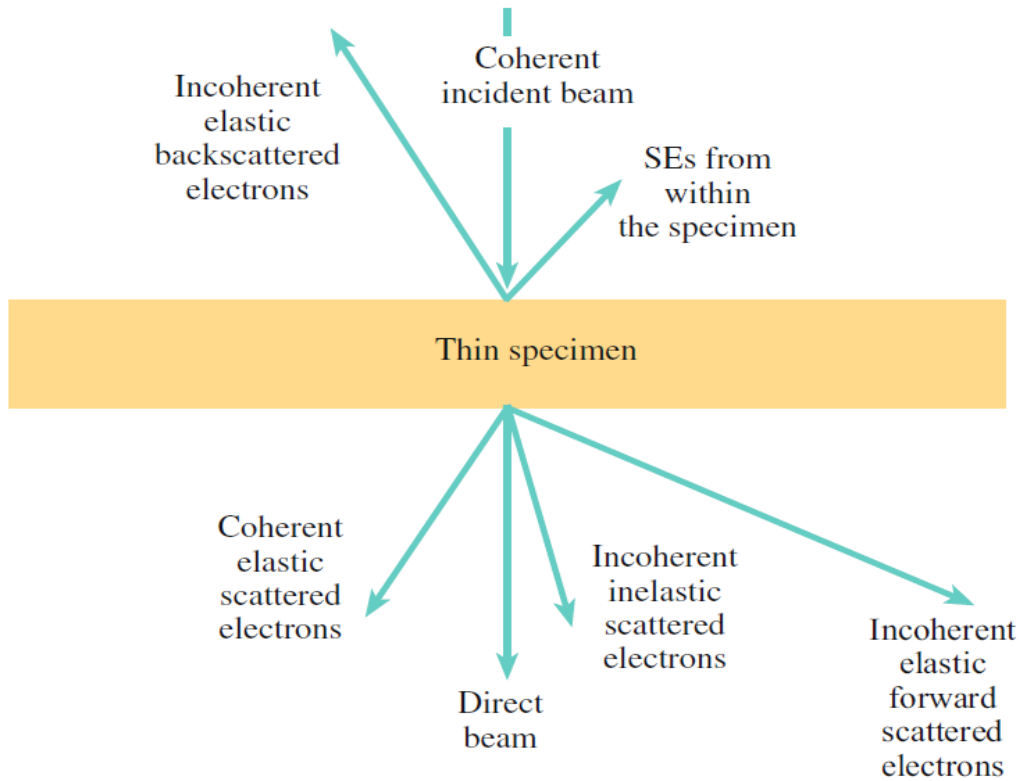
  Allows to perform Insitu-S/TEM experiments

TEM Viewing Chamber – Phosphorous Screen

TEM Imaging

University of Oslo
Centre for Materials Science and Nanotechnology (SMN)

Coherent incident beam

Incoherent elastic backscattered electrons

SEs from within the specimen

Thin specimen

Coherent elastic scattered electrons

Direct beam

Incoherent inelastic scattered electrons

Incoherent elastic forward scattered electrons

- Elastic scattering is usually coherent, if the specimen is thin and crystalline (think in terms of waves).
- Elastic scattering usually occurs at relatively low angles (1–10°), i.e., it is strongly peaked in the forward direction (waves).
- At higher angles (>~10°) elastic scattering becomes more incoherent (now think of particles).
- Inelastic scattering is almost always incoherent and is very low angle (<1°) scattering (think particles).
- As the specimen gets thicker, fewer electrons are forward scattered and more are backscattered. Incoherent, backscattered electrons are the only remnants of the incident beam that emerge from bulk, non-transparent specimens (think particles).

TEM imaging and Diffraction   **– Elastic scattering, Coherent - (1-10°)** and
                                   Forward scattered e⁻ -(0°)

STEM Z-contrast Imaging   **– Elastic scattering, Incoherent - (> ~10°)**

EELS *(Spectroscopy Technique*   **– Inelastic scattering, Incoherent - (< ~1°)**

EDS *(Spectroscopy Technique)*   **– X-rays**

Amplitude contrast and Phase-contrast images

The electron wave can change both its amplitude and phase as it traverses the specimen
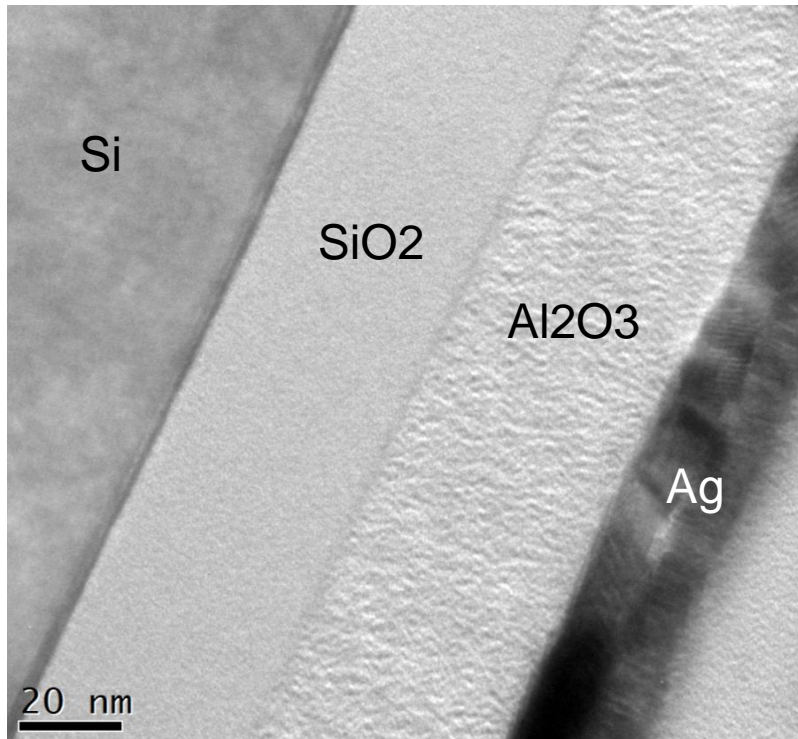
This Gives rise to contrast in TEM images

We select imaging conditions so that one of them dominates.

Contrast mechanisms

The image contrast originates from:

Amplitude contrast

- Mass - The only mechanism that generates contrast for amorphous materials: Polymers and biological materials

- Diffraction - Only exists with crystalline materials: metals and ceramics

Phase (produces images with atomic resolution)
Only useful for THIN crystalline materials (diffraction with NO change in wave amplitude): Thin metals and ceramics

TEM techniques

Main Constrast phenomena in TEM

Imaging

Conventional TEM
Bright/Dark-Field TEM
High Resolution TEM (HRTEM)
Scanning TEM (STEM)
Energy Filtered TEM (EFTEM)

- Mass thickness Contrast
- Diffraction contrast
- Phase Contrast
- Z-contrast

Diffraction

Selected Area Electron Diffraction
Convergent Beam Electron Diffraction

Phase identification, defects, orientation relationship between different phases, nature of crystal structure (amorphous, polycrystalline, single crystal)

Spectroscopy
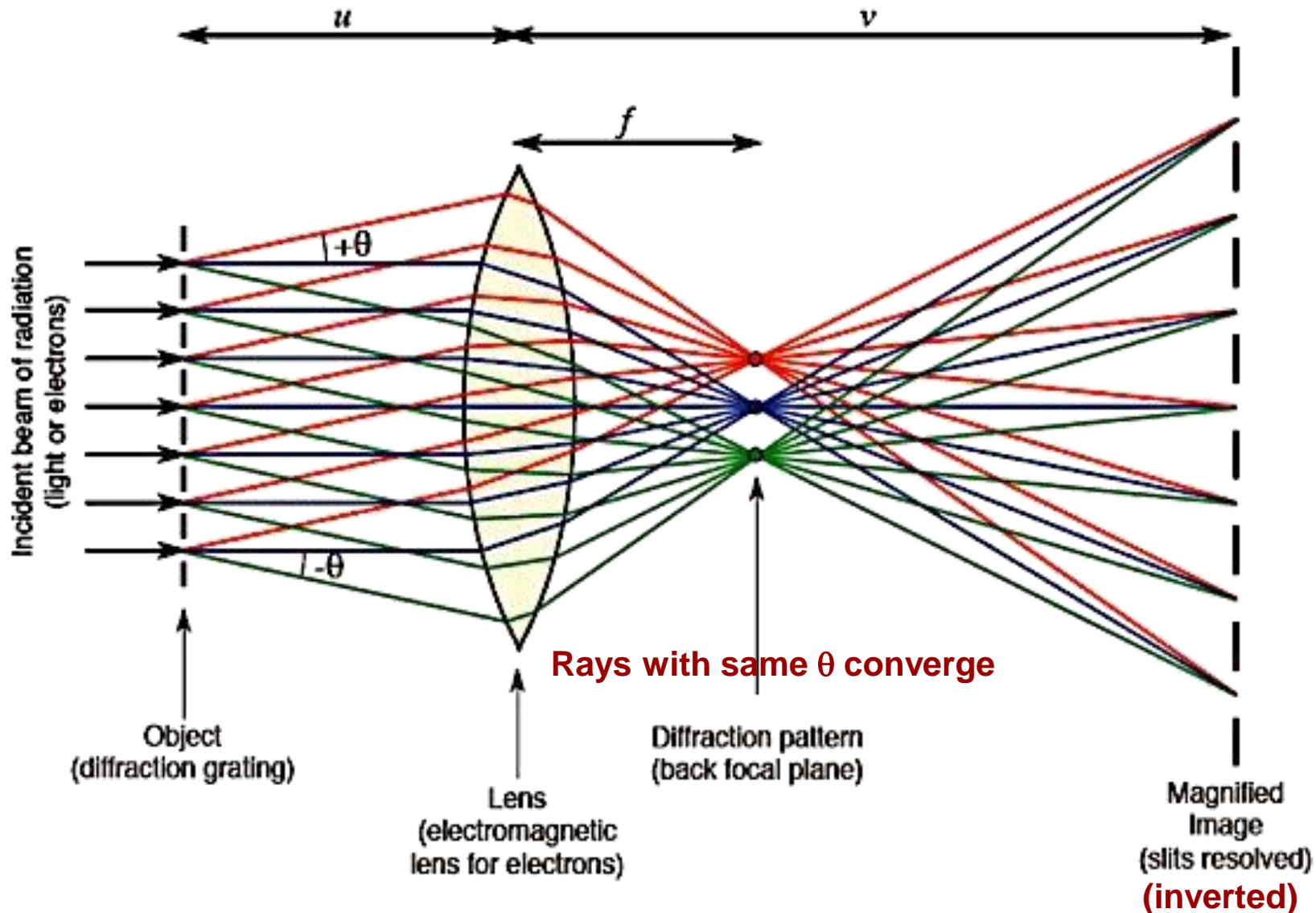
Electron Dispersive X-ray Spectroscopy (EDS)
Electron Energy Loss Spectroscopy (EELS)

Chemical composition, electronic states, nature of chemical bonding (EDS and EELS).
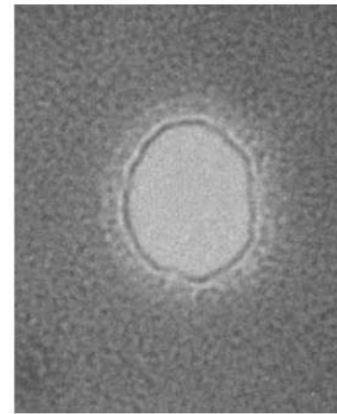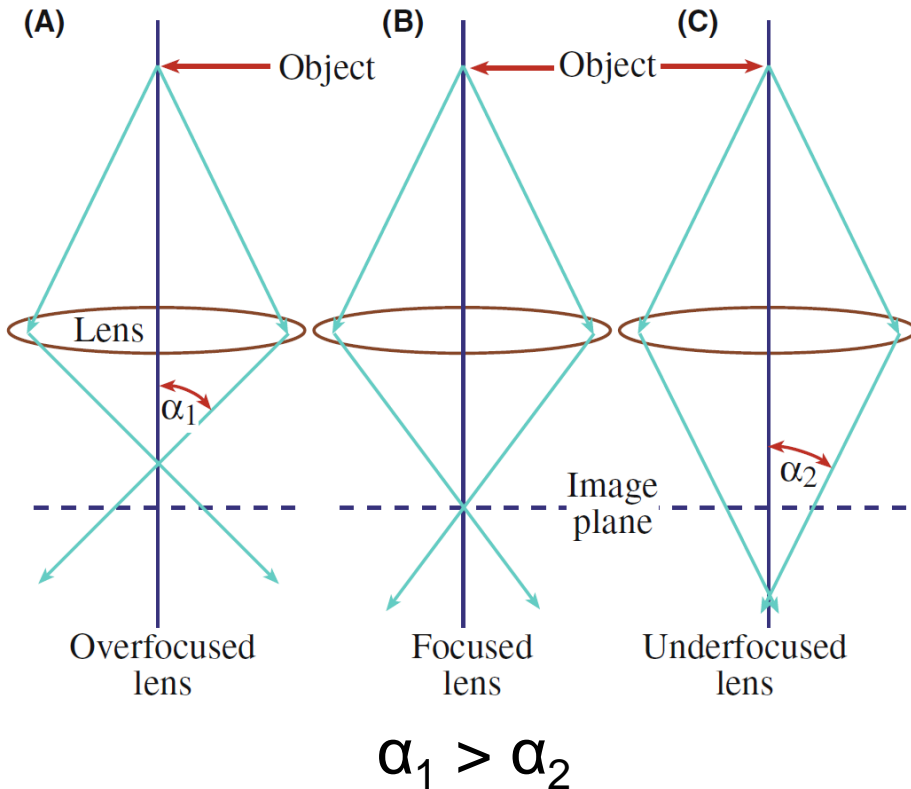Spatial and energy resolution down to the atomic level and ~0.1 eV.

Abbe's principle of imaging

Unlike with visible light, due to the small λ, electrons can be coherently scattered by crystalline samples so the diffraction pattern at the back focal plane of the object corresponds to the sample reciprocal lattice.
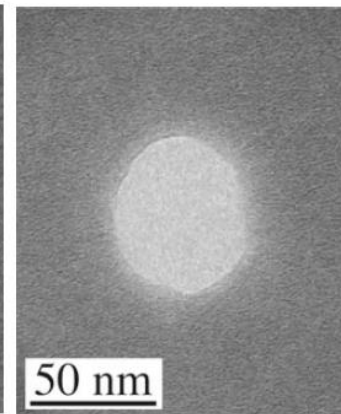
Incident beam of radiation (light or electrons)

$+\theta$

$-\theta$

Rays with same θ converge

Object (diffraction grating)

Lens (electromagnetic lens for electrons)

Diffraction pattern (back focal plane)

Magnified Image (slits resolved) **(inverted)**

UiO : **University of Oslo**
Centre for Materials Science and Nanotechnology (SMN)

WHEN YOU ARE IN FOCUS IN TEM THE CONTRAST IS MINIMUM IN IMAGE *(AT THE THINNEST PART OF THE SAMPLE)*

(A) Object — Lens — α_1 — Overfocused lens

(B) Object — Image plane — Focused lens

(C) Object — α_2 — Underfocused lens

$$\alpha_1 > \alpha_2$$

50 nm
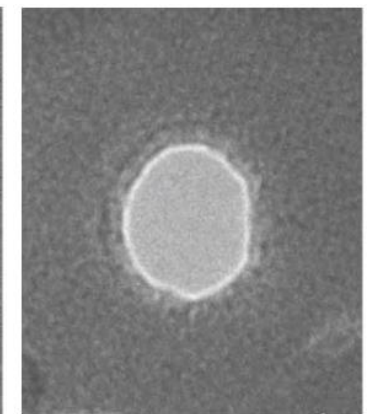
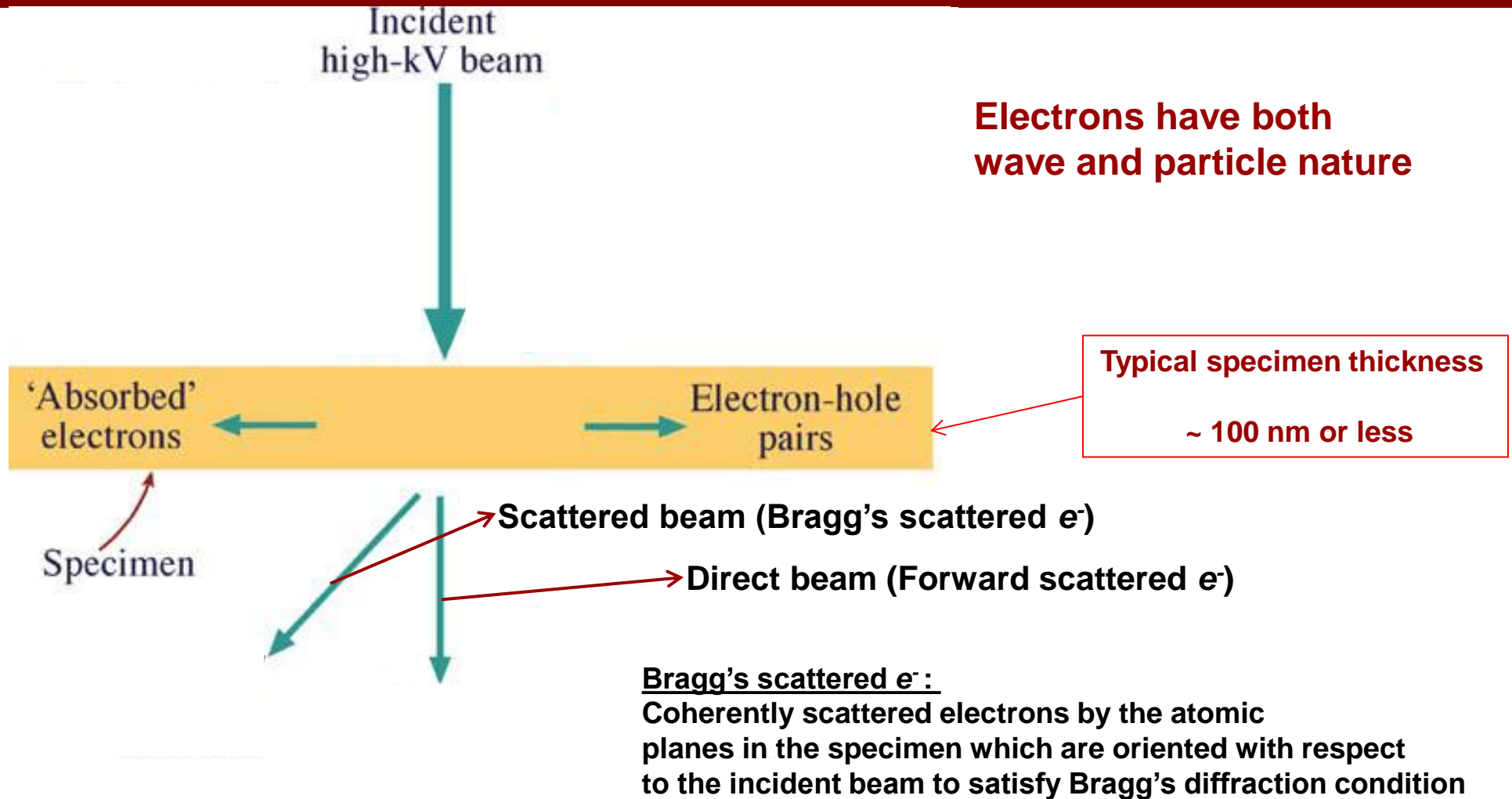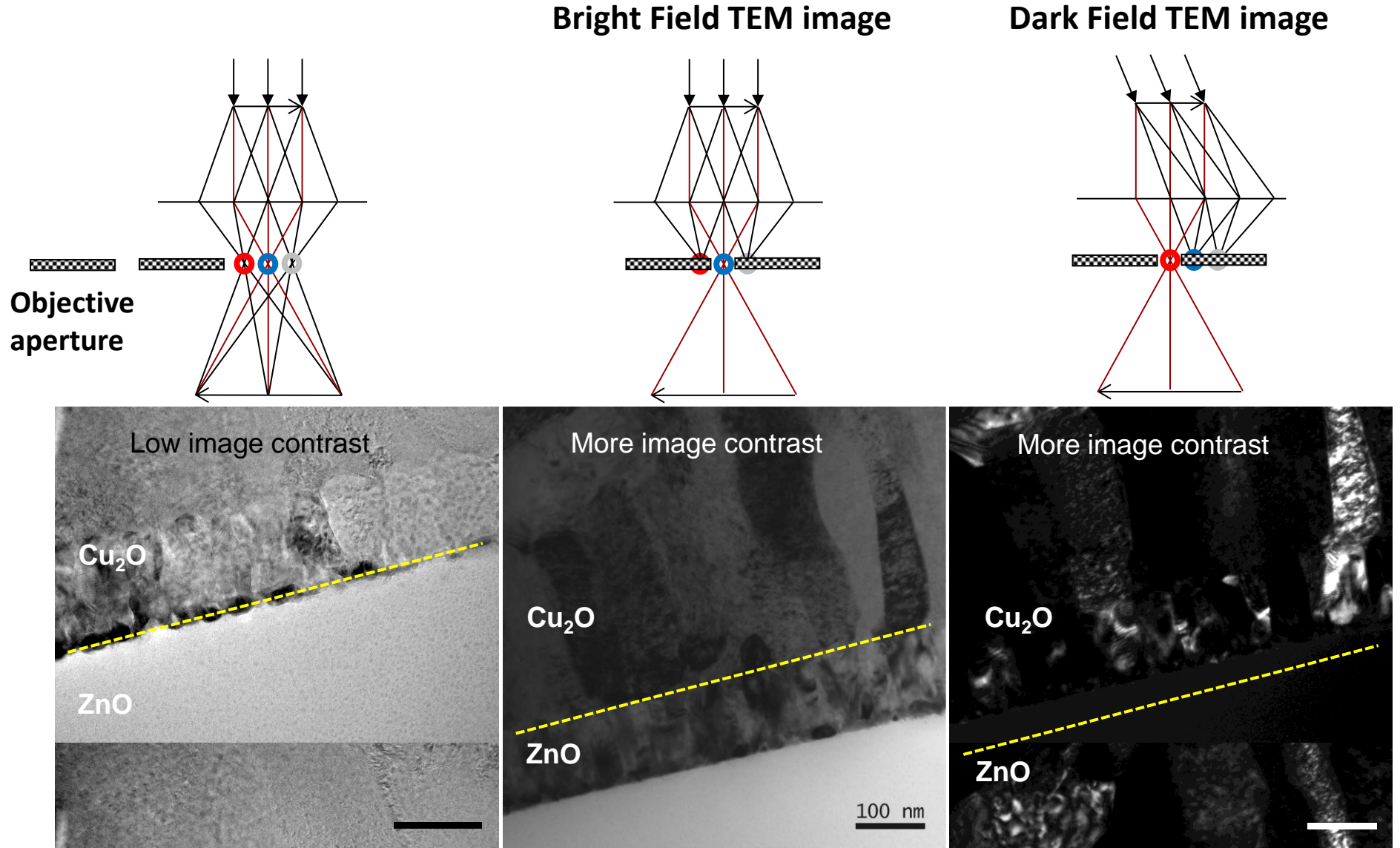| OVER FOCUS	FOCUS	UNDER FOCUS
DARK FRINGE	**NO** FRINGE	**BRIGHT** FRINGE

FRINGES OCCURS AT EDGE DUE TO FRESNEL DIFFRACTION

Courtesy: D.B. Williams & C.B. Carter, Transmission electron microscopy

Interaction of Electrons with the specimen in TEM

Incident high-kV beam

'Absorbed' electrons

Specimen

Electron-hole pairs

Electrons have both wave and particle nature

Typical specimen thickness

~ 100 nm or less

Scattered beam (Bragg's scattered e⁻)

Direct beam (Forward scattered e⁻)

Bragg's scattered e⁻ :
Coherently scattered electrons by the atomic planes in the specimen which are oriented with respect to the incident beam to satisfy Bragg's diffraction condition

Courtesy: D.B. Williams & C.B. Carter, Transmission electron microscopy

Bright Field TEM image

Dark Field TEM image

Objective aperture

Low image contrast

Cu_2O

ZnO

More image contrast

Cu_2O

ZnO

100 nm

More image contrast

Cu_2O

ZnO
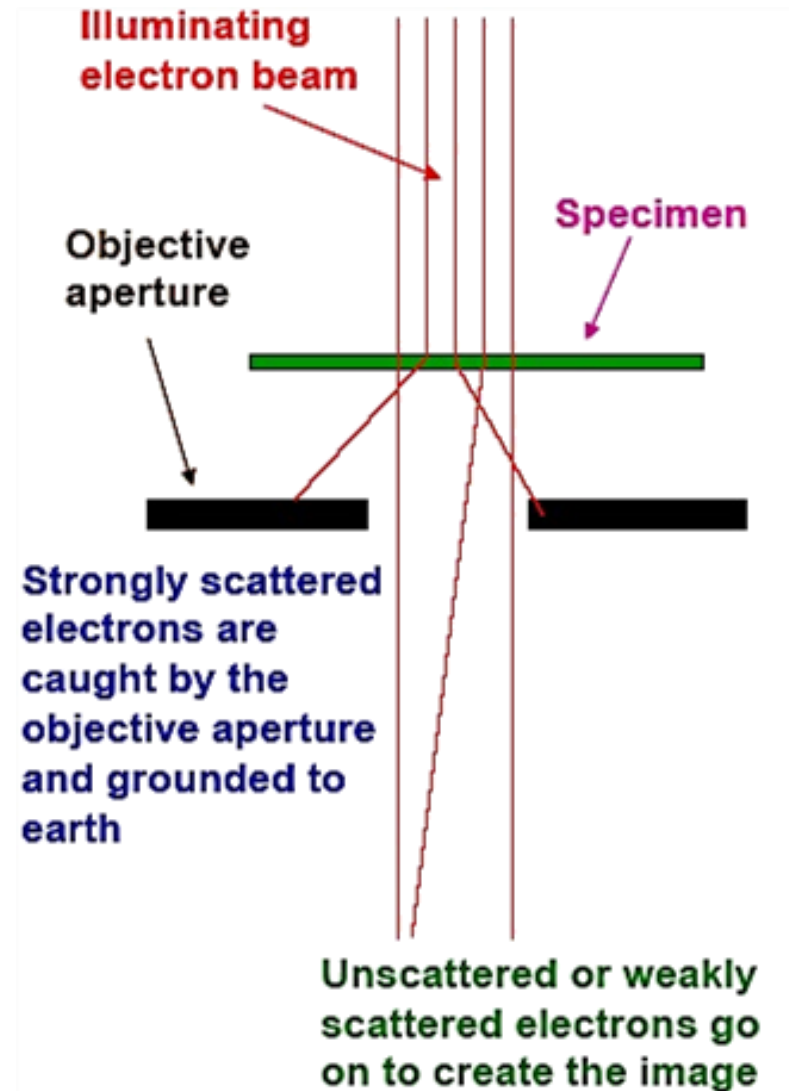
Mass contrast

- Mass contrast: Variation in mass, thickness or both

- Bright Field (BF): The basic way of forming mass-contrast images
- No coherent scattering

Mechanism of mass-thickness contrast in a BF image. Thicker or higher-Z areas of the specimen (darker) will scatter more electrons off axis than thinner, lower mass (lighter) areas. Thus fewer electrons from the darker region fall on the equivalent area of the image plane (and subsequently the screen), which therefore appears darker in BF images.
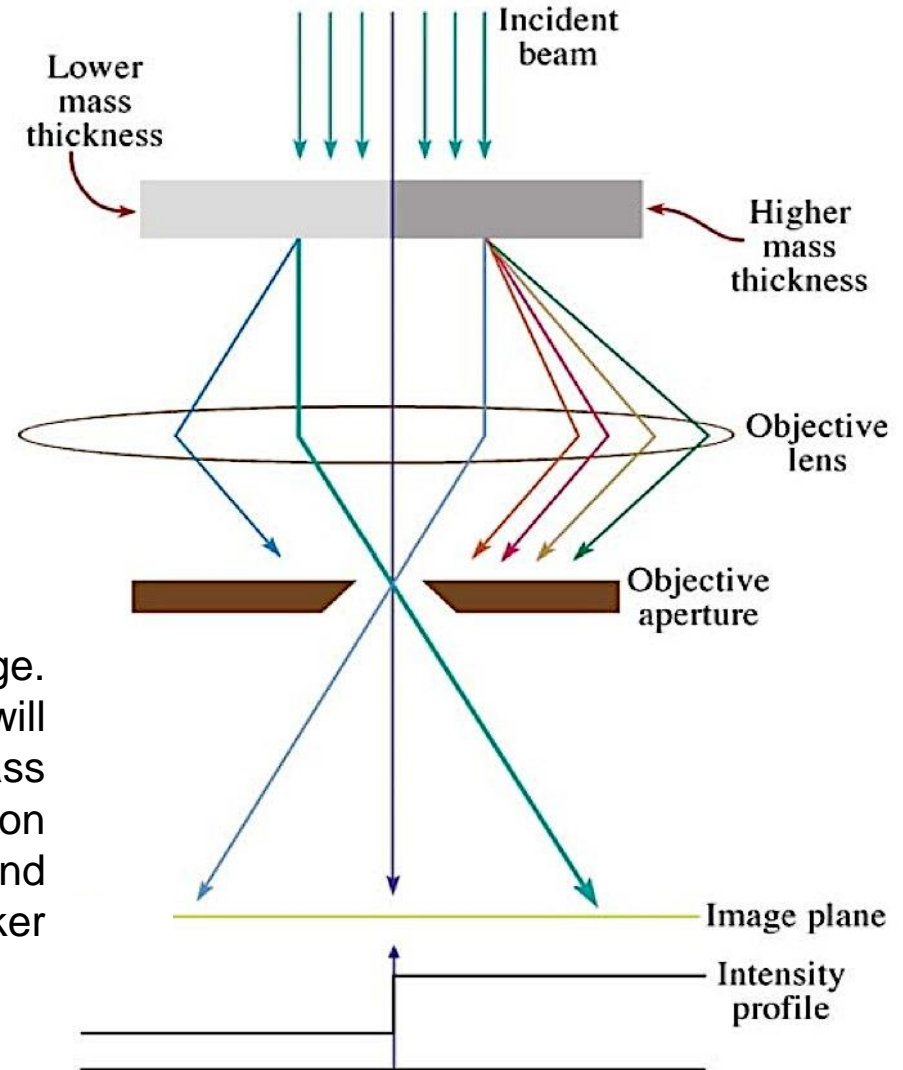
Mass contrast

• Heavy atoms scatter more intensely and at higher angles than light ones.

• Strongly scattered electrons are prevented from forming part of the final image by the objective aperture.

• Regions in the specimen rich in heavy atoms are dark in the image.

• The smaller the aperture size, the higher the contrast.

• Fewer electrons are scattered at high electron accelerating voltages, since they have less time to interact with atomic nuclei in the specimen: **High voltage TEM result in lower contrast and also damage polymeric and biological samples**
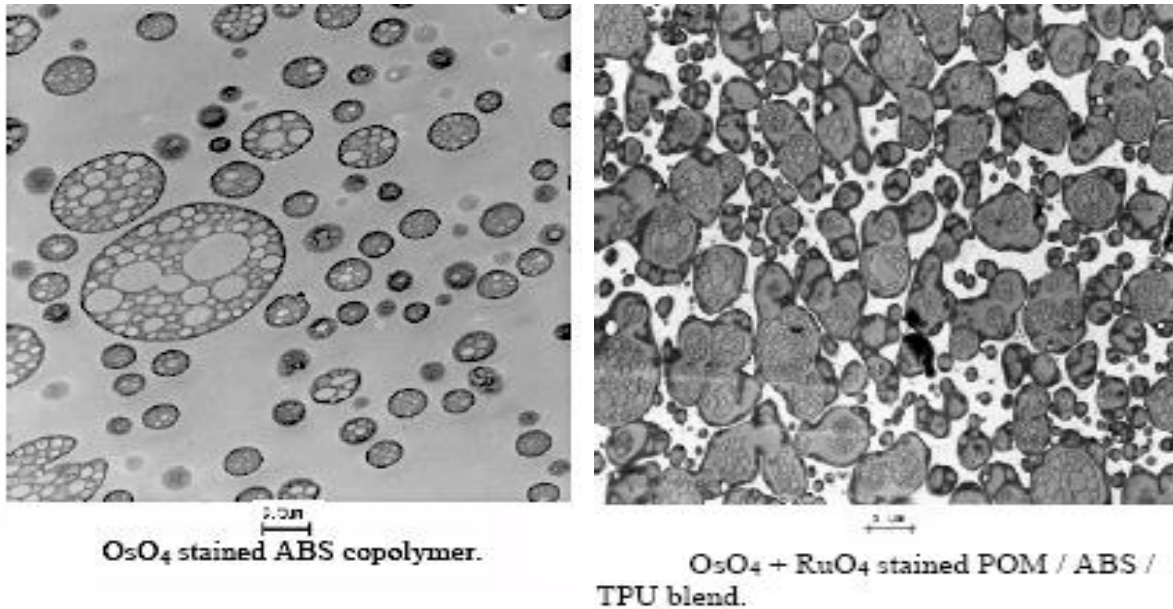
Illuminating electron beam

Specimen

Objective aperture

Strongly scattered electrons are caught by the objective aperture and grounded to earth

Unscattered or weakly scattered electrons go on to create the image

Mass contrast

Bright field images
(J.S.J. Vastenhout, Microsc Microanal 8 Suppl. 2, 2002)

OsO$_4$ stained ABS copolymer.

OsO$_4$ + RuO$_4$ stained POM / ABS / TPU blend.

Stained with OsO$_4$ and RuO$_4$ vapors
Os and Ru are heavy metals…

In the case of polymeric and biological samples, i.e., with low atomic number and similar electron densities, staining helps to increase the imaging contrast and mitigates the radiation damage.

The staining agents work by selective absorption in one of the phases and tend to stain unsaturated C-C bonds. Since they contain heavy elements with a high scattering power, the stained regions appear dark in bright field.

Diffraction Contrast

- **Thickness Fringes**

Some of the Microstructural defects that can be observed

- **Stacking faults**
- **Dislocations**
- **Strain fields due to Dislocations**
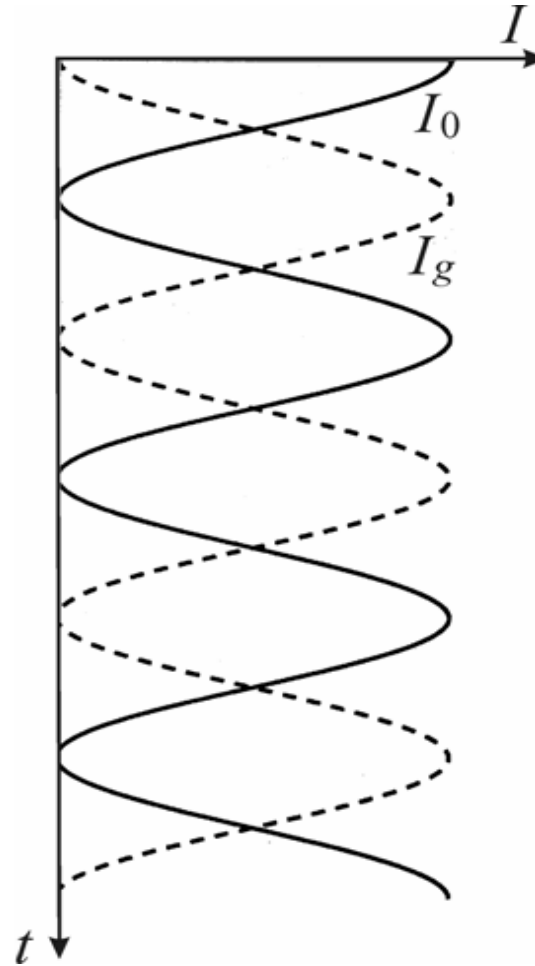
Two-beam conditions
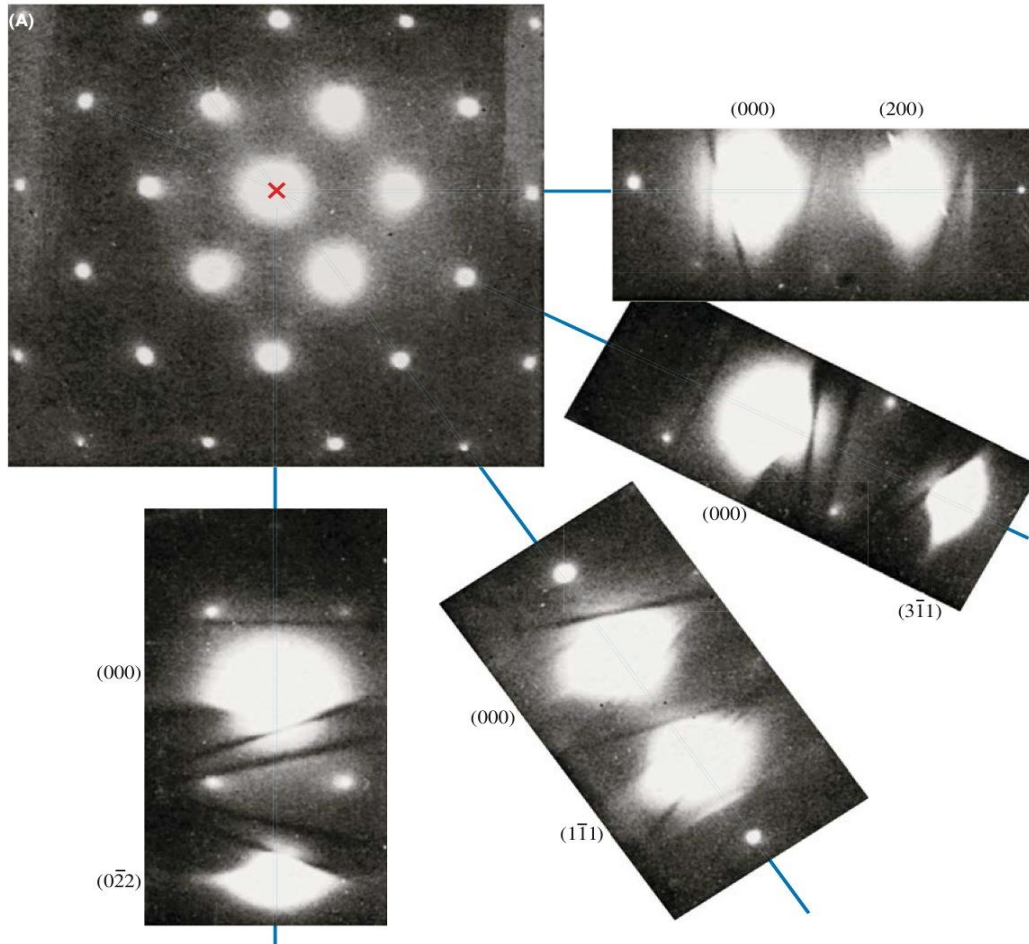
The [011] zone-axis diffraction pattern has many planes diffracting with equal strength. In the smaller patterns the specimen is tilted so there are only two strong beams, the direct 000 on-axis beam and a different one of the hkl off-axis diffracted beams.

dynamical scattering for 2-beam conditions

$$\frac{d\Psi_0}{dz} = \frac{i\pi}{\xi_0}\Psi_0 + \frac{i\pi}{\xi_g}\Psi_g \exp\left(2\pi i s_g z\right)$$

$$\frac{d\Psi_g}{dz} = \frac{i\pi}{\xi_0}\Psi_g + \frac{i\pi}{\xi_g}\Psi_0 \exp\left(-2\pi i s_g z\right)$$

$$I_g = \Psi_g \Psi_g^* = \frac{\pi^2}{\xi_g^2}\frac{\sin^2 \pi t s_g}{(\pi s_g)^2}$$

Coupling: interchange of intensity between the two beams as a function of thickness t for a perfect crystal

Originates thickness fringes, in BF or DF images of a crystal of varying t

dynamical scattering for 2-beam conditions
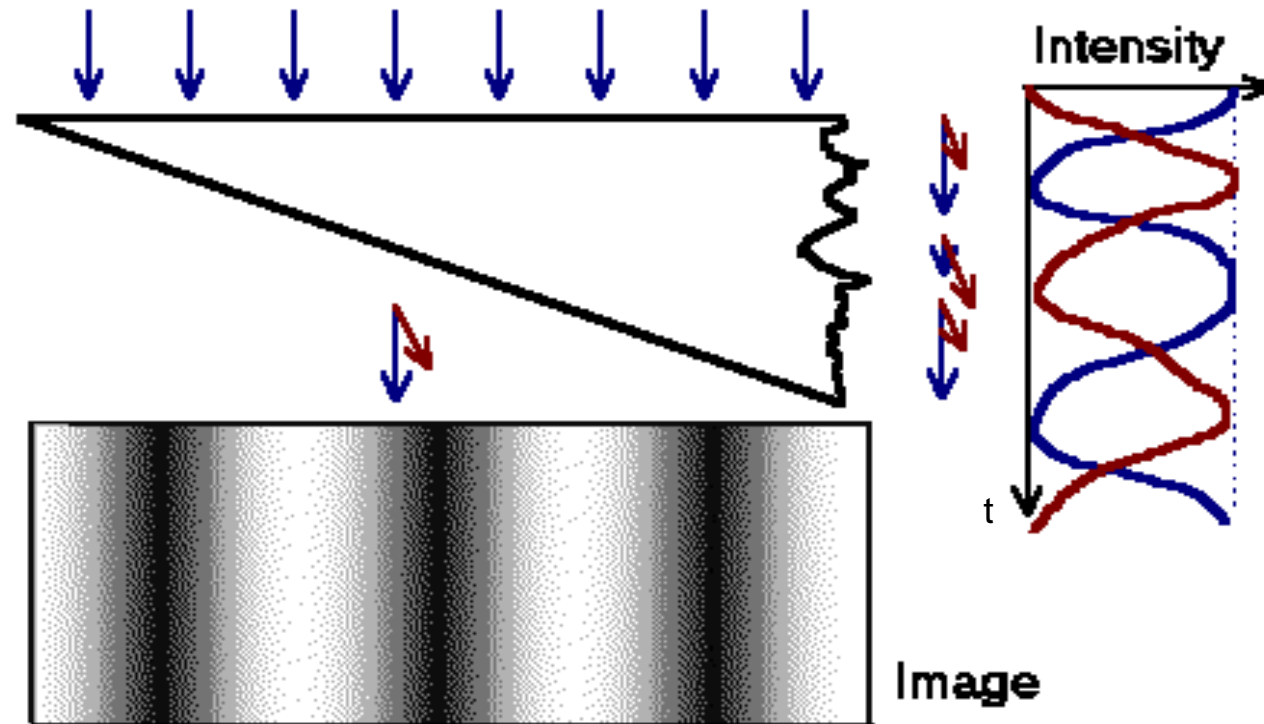
The images of wedged samples present series of so-called thickness fringes in **BF** or **DF** images (only one of the beams is selected).

dynamical scattering for 2-beam conditions

The image intensity varies sinusoidally depending on the thickness and on the beam used for imaging.
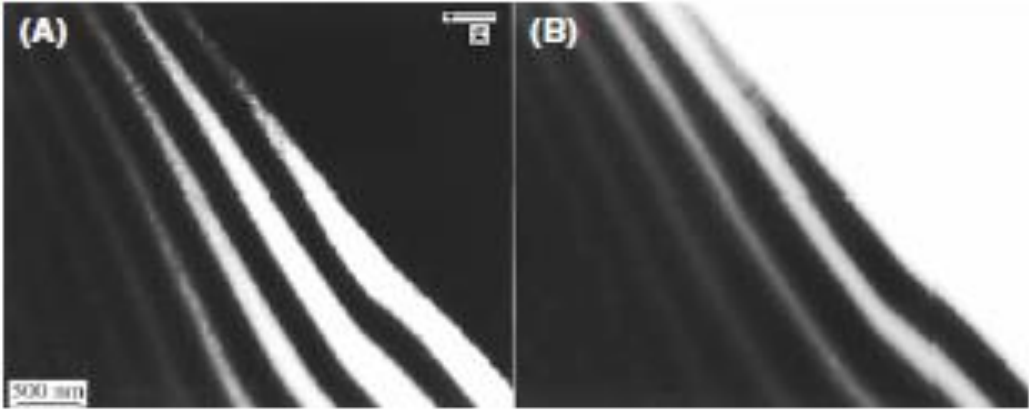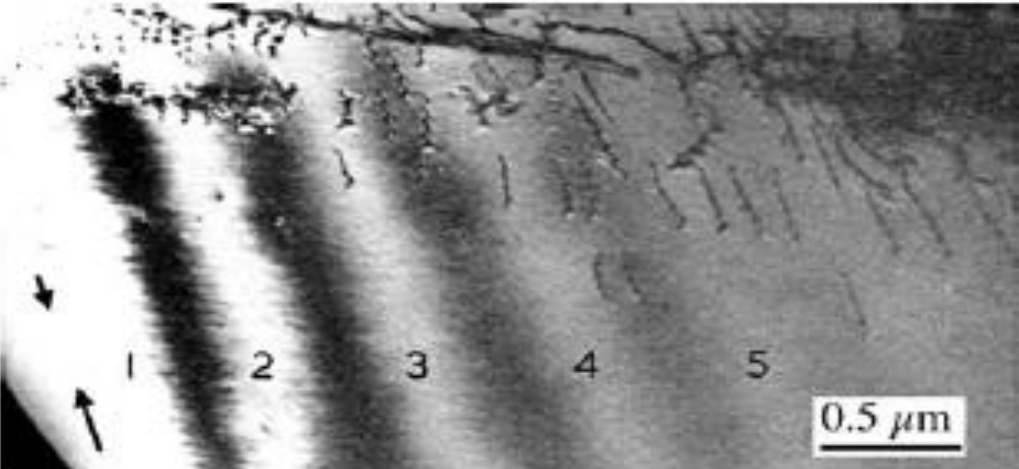
FIGURE 24.10. The contrast of thickness fringes in a two-beam BF image decreases when the effect of anomalous absorption is included. Note that the defects are still visible when the fringes have disappeared at a thickness of −5 ξ_g.

FIGURE 24.1. (A) BF and (B) DF images from the same region of a wedge-shaped specimen of Si at 300 kV tilted so that g(220) is strong. The periodicity and contrast of the fringes are similar and complementary in

Reduced contrast as thickness increases due to absorption

2-beam condition
A: image obtained with transmitted beam (Bright field)
B: image obtained with diffracted beam (Dark field)
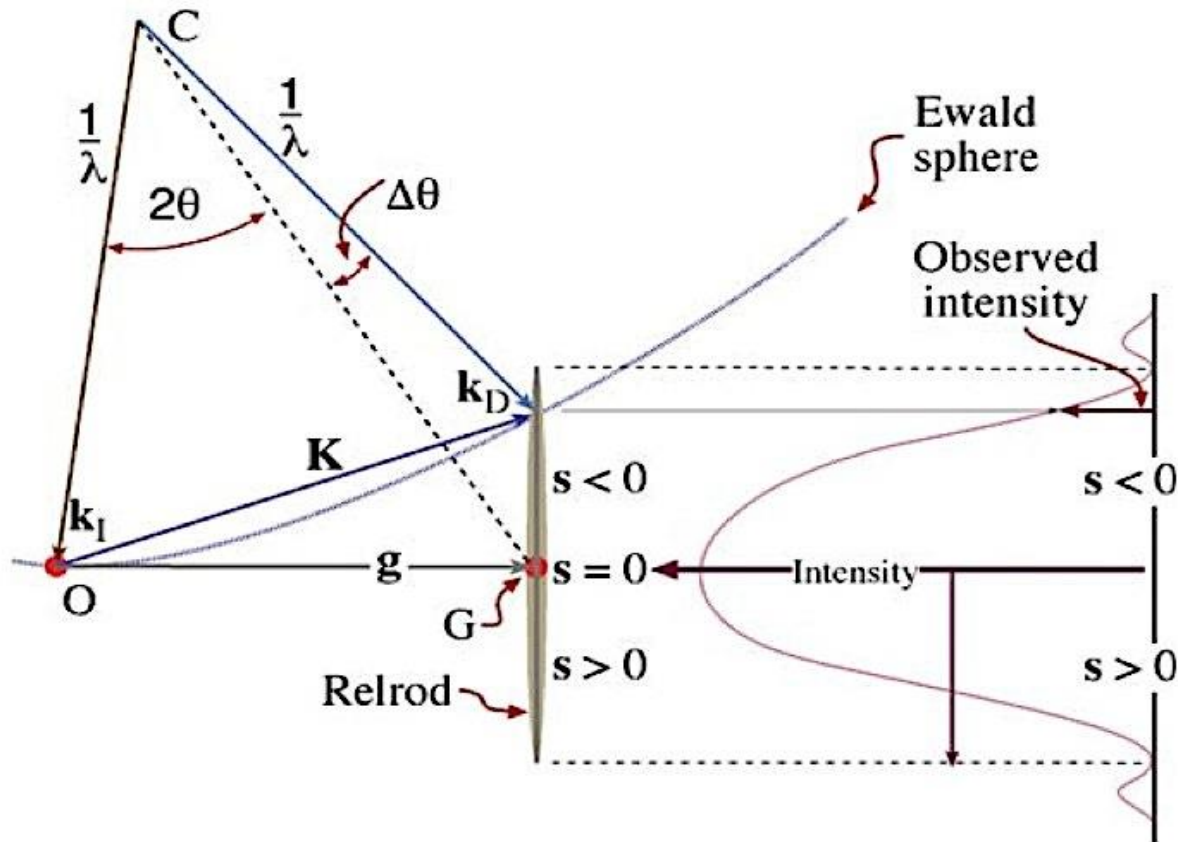
Diffraction contrast

As **s** increases the defect images become narrower but the contrast is reduced:
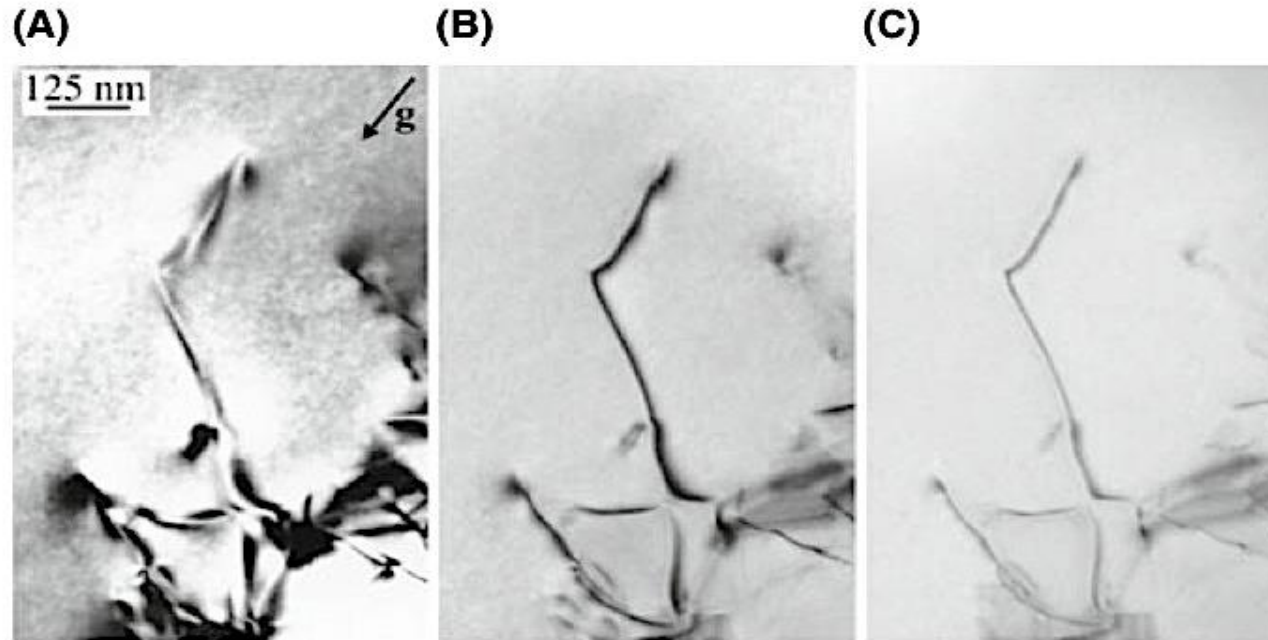
Variation in the diffraction contrast when s is varied from (A) zero to (B) small and positive and (C) larger and positive.

Bright field two-beam images of defects should be obtained with s small and positive.

the excitation error or deviation parameter

Other notation
(Williams and Carter):
$K=k_D-k_I=g+s$

The relrod at g_{hkl} when the beam is $\Delta\theta$ away from the exact Bragg condition. The Ewald sphere intercepts the relrod at a negative value of **s** which defines the vector **K = g + s**. The intensity of the diffracted beam as a function of where the Ewald sphere cuts the relrod is shown on the right of the diagram. In this case the intensity has fallen to almost zero.
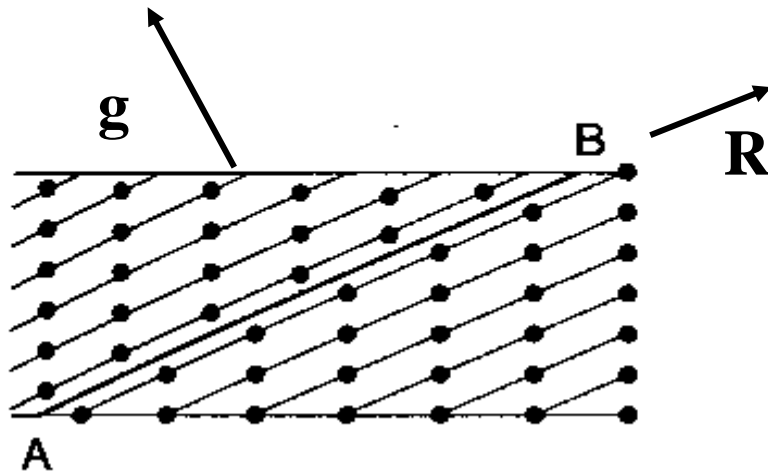
Kikuchi lines

Useful to determine s…
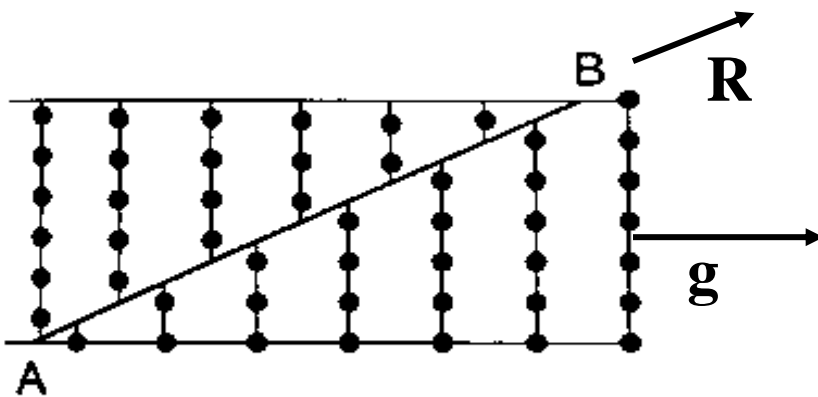

$s > 0$

$s = 0$
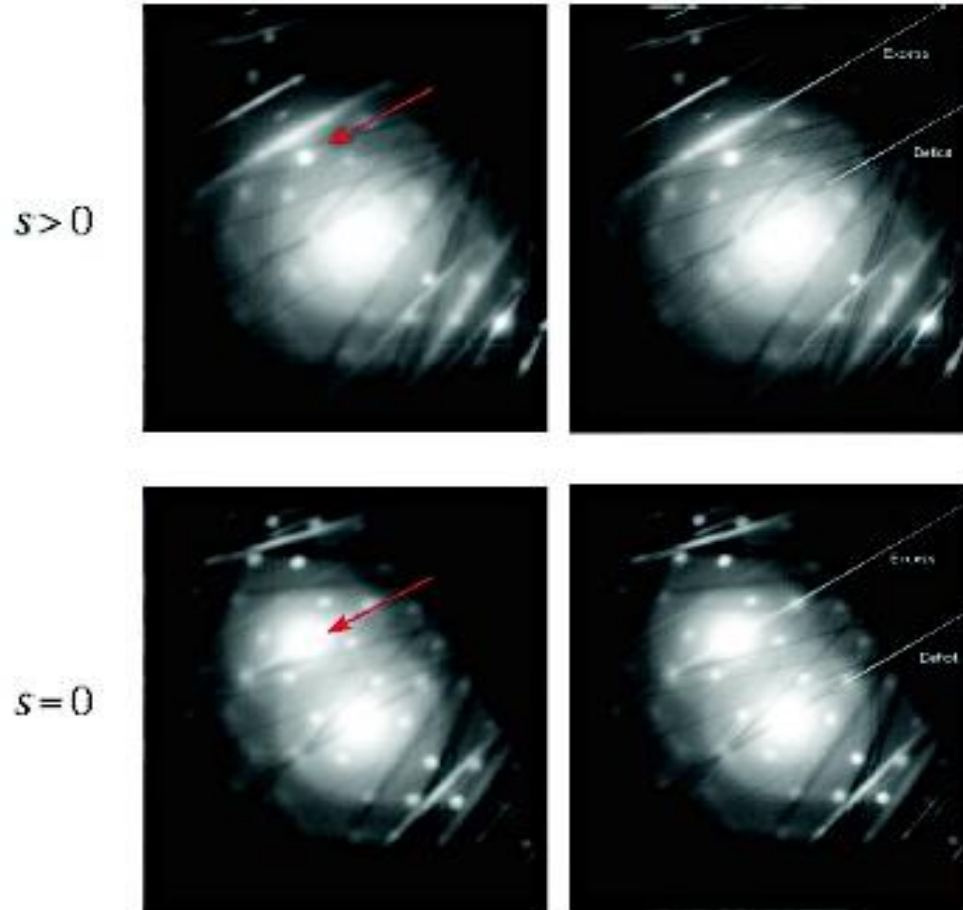
Excess Kikuchi line on G spot
Deficient line in transmitted spot

Planar defects under two-beam conditions

The upper crystal is considered fixed while the lower one is translated by a vector $\mathbf{R}(r)$ and/or rotated through some angle θ about any axis, v.

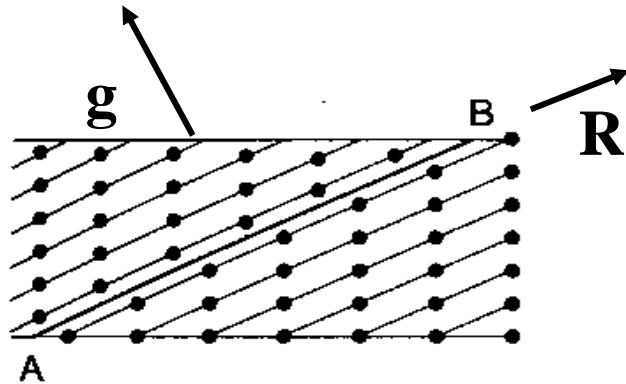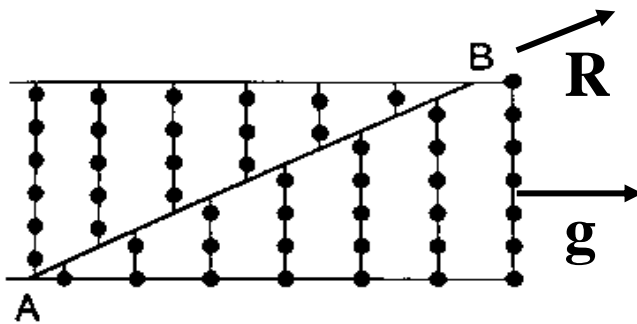In (a) the stacking fault does not disrupt the periodicity of the planes (solid lines).

In (b) the stacking fault disrupts the periodicity of the planes (solid lines).

Planar defects under two-beam conditions

(a)

Invisible **g.R** = 0 or even integer

(b)

Visible **g.R** ≠ 0

(max contrast for 1 or odd integer)

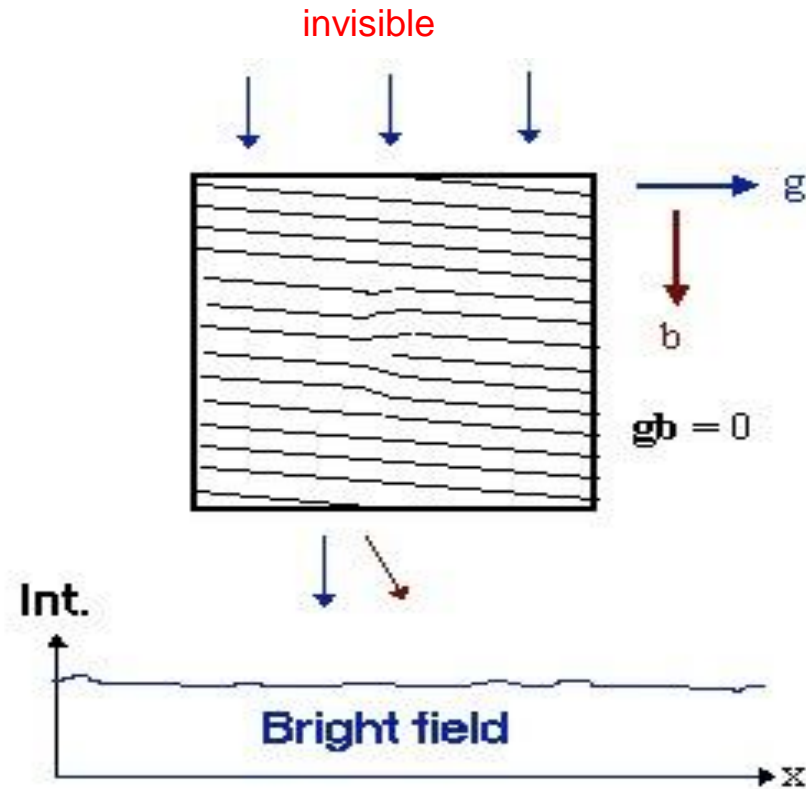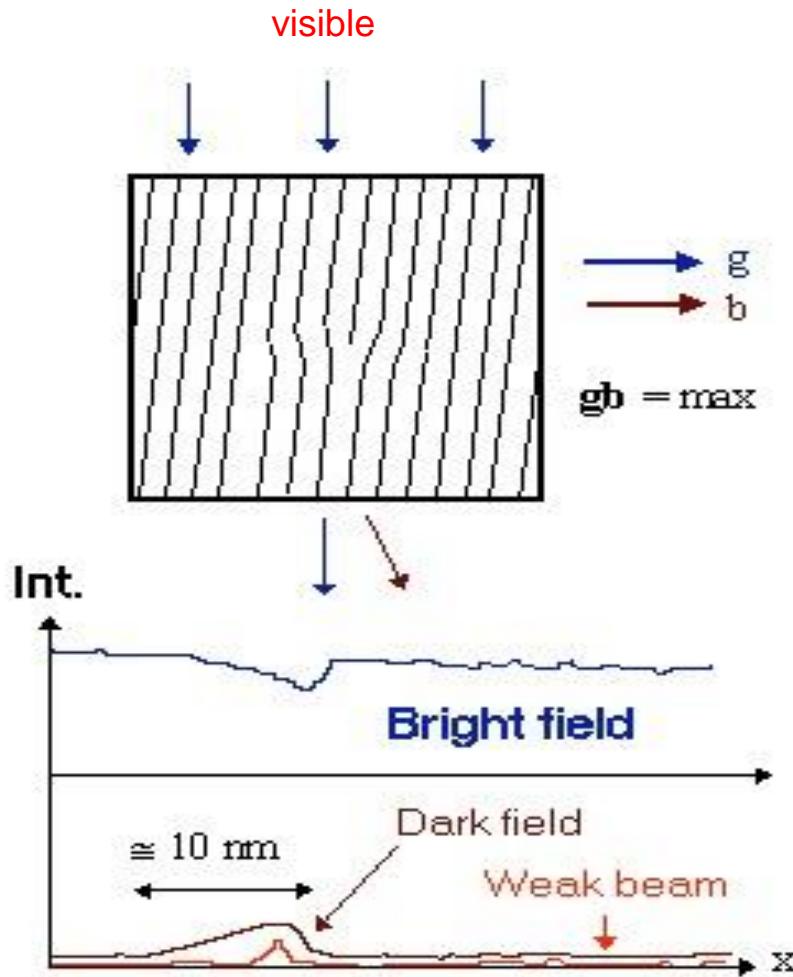**from two invisibility conditions: $g_1 \times g_2$: direction of R!**

Imaging strain fields
(typically dislocations)
(quantitative information from 2-beam conditions)
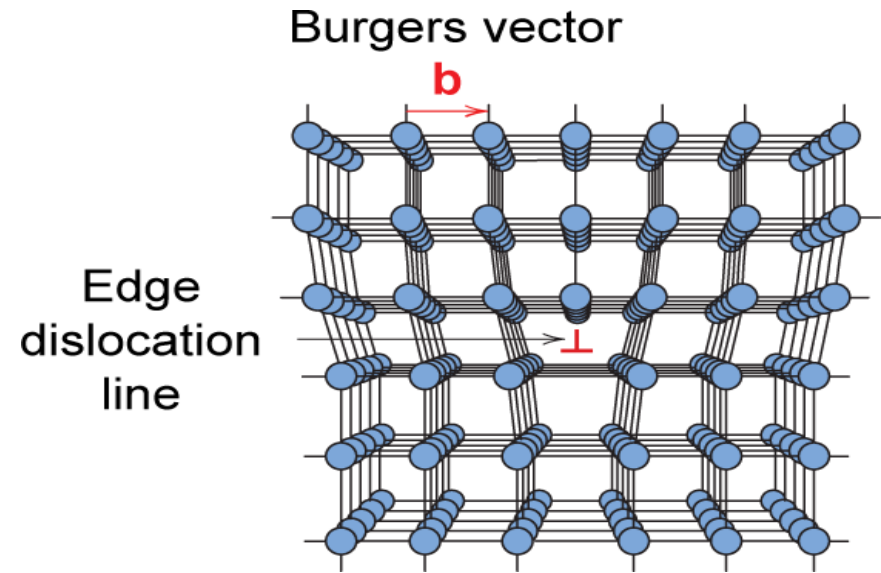
Imaging strain fields, In summary:

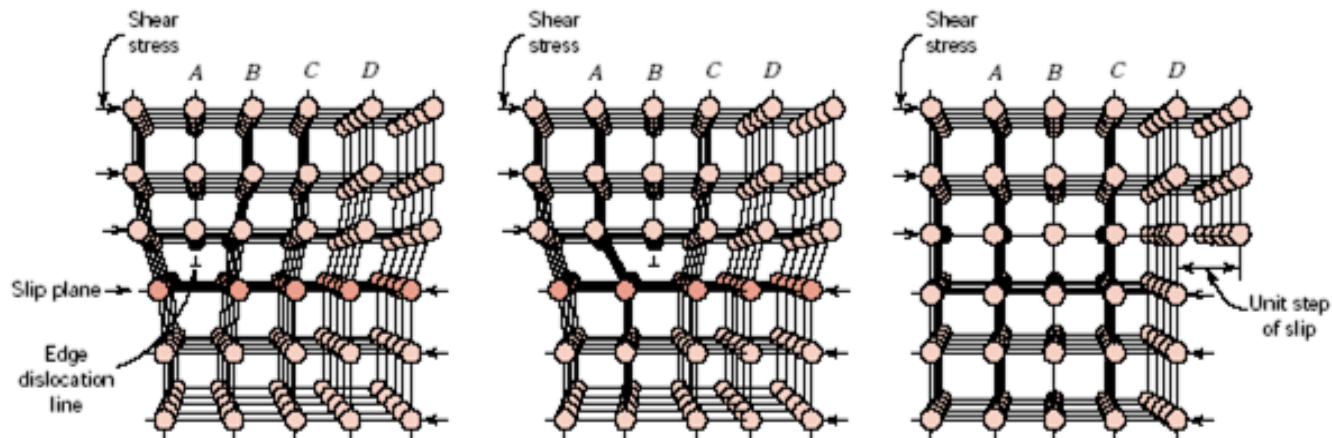**b** is referred as Burgers Vector of Dislocation

Dislocations

Edge dislocation:

– extra half-plane of atoms inserted in a crystal structure
– **b** ⊥ to dislocation line

Dislocation movement: slip

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dislocations

Burgers circuit

Definition of the Burgers vector, **b**, relative to an edge dislocation.

(a) In the perfect crystal, an m × n atomic step loop closes at the starting point.

(b) In the region of a dislocation, the same loop does not close, and the closure vector **(b)** represents the magnitude of the structural defect.
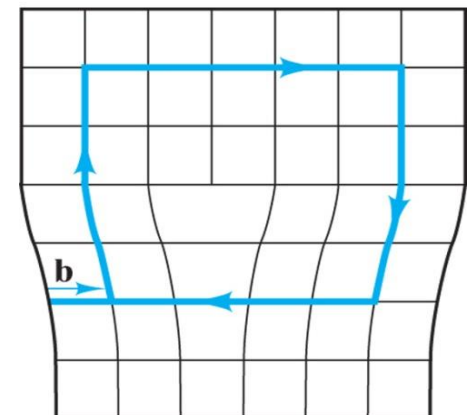
In an edge dislocation the Burgers vector is perpendicular to the dislocation line.

The Burgers vector is an invariant property of a dislocation (the line may be very entangled but **b** is always the same along the dislocation)

The Burgers vector represents the step formed by the dislocation when it slips to the surface.
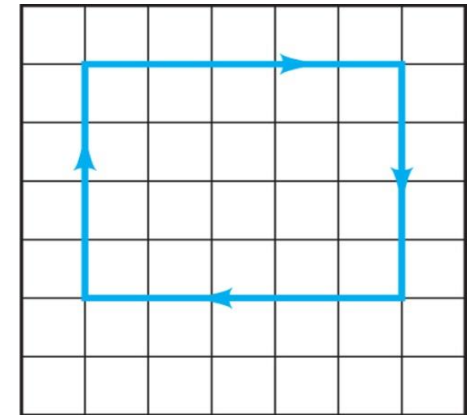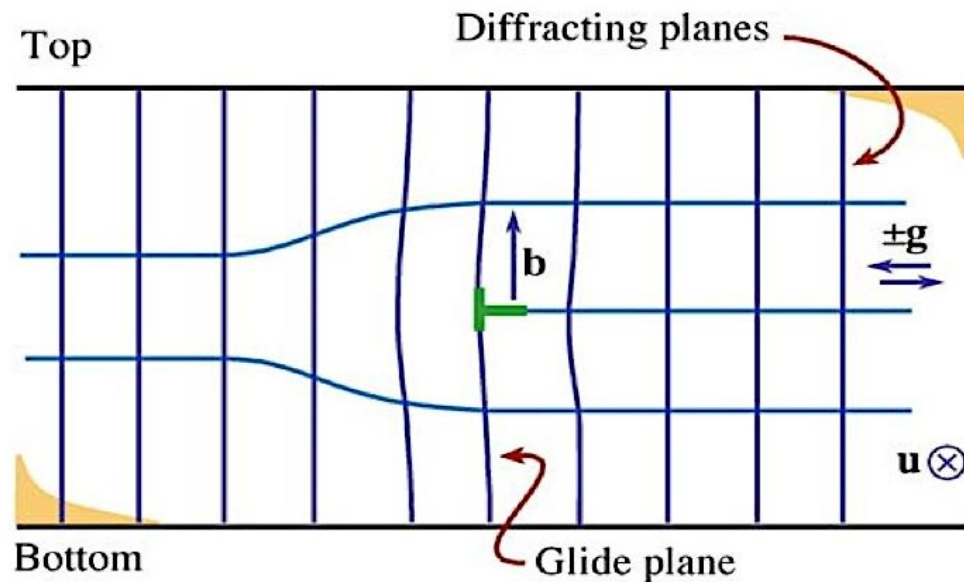

(a)

(b)

Imaging strain fields

Determination of **b** from the visibility conditions of the strain field associated with dislocations

Due to some stress relaxation complete invisibility is never achieved for edge dislocations, unlike screw dislocations

Invisibility criterion: **g.b** = 0

from two invisibility conditions: **g**$_1$ x **g**$_2$: **b** direction

Imaging strain fields

The **g.b** rule

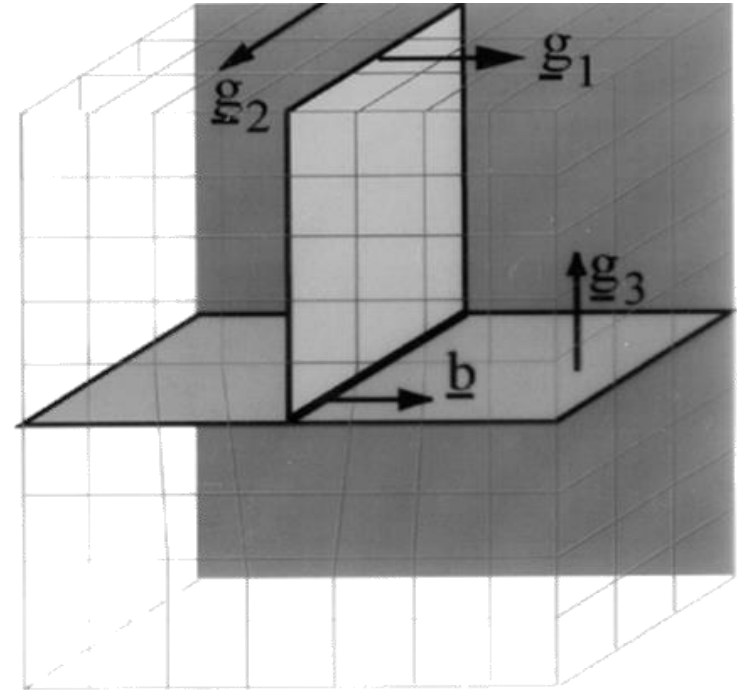Only the planes belonging to $\mathbf{g}_1$ are affected by the presence of the dislocation.

Applying **g.b**:

$$\mathbf{g}_1.\mathbf{b} \neq 0$$
$$\mathbf{g}_2.\mathbf{b} = 0$$
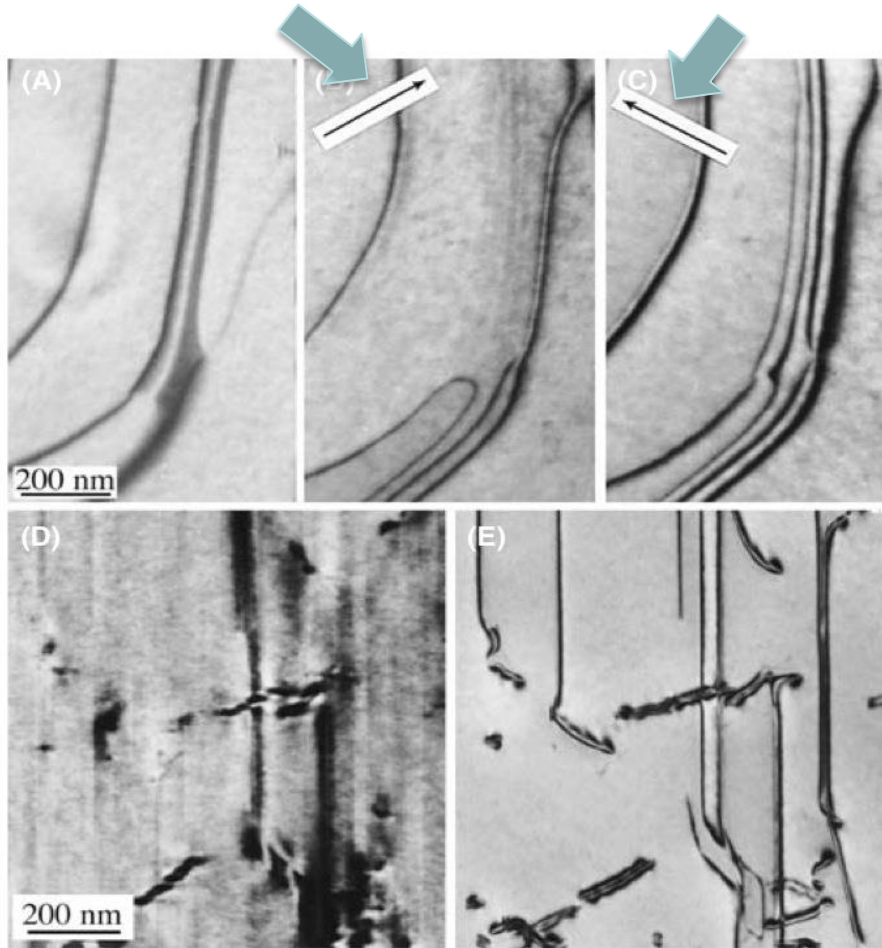$$\mathbf{g}_3.\mathbf{b} = 0$$

$$\mathbf{g}_2 \quad \mathbf{g}_3 = 0$$

\\ Ä

Invisibility criterion: **g.b** = 0

from two invisibility conditions: $\mathbf{g}_1 \times \mathbf{g}_2$: **b** direction

Imaging strain fields

Direction of **g** in the diffraction pattern gives an indication of the direction of displacement of the dislocation contrast

(A–C) Three strong-beam BF images from the same area using (A) {11-1 } and (B, C) {220} reflections to image dislocations which lie nearly parallel to the (111) foil surface in a Cu alloy which has a low stacking-fault energy.

(D, E) Dislocations in Ni_3Al in a (001) foil imaged in two orthogonal {220} reflections. Most of the dislocations are out of contrast in (D).

Williams and Carter book

Imaging strain fields
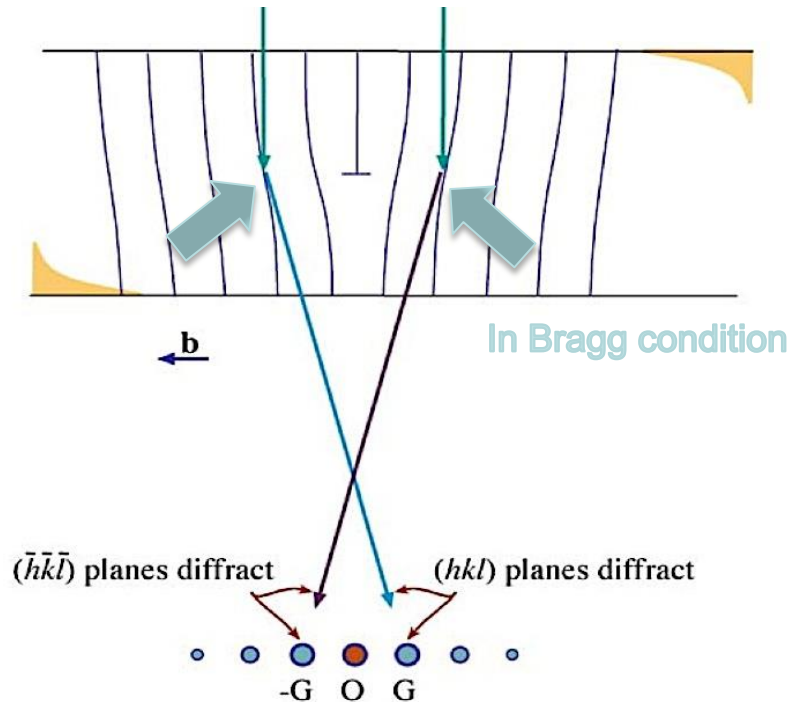
The specimen is tilted slightly away from the Bragg condition ($\mathbf{s} \neq 0$). The distorted planes close to the edge dislocation are bent back into the Bragg-diffracting condition ($\mathbf{s} = 0$), diffracting into G and –G as shown.

Imaging strain fields

In Bragg condition

Intensity

BF for G excited

DF using G

Projected position of dislocation
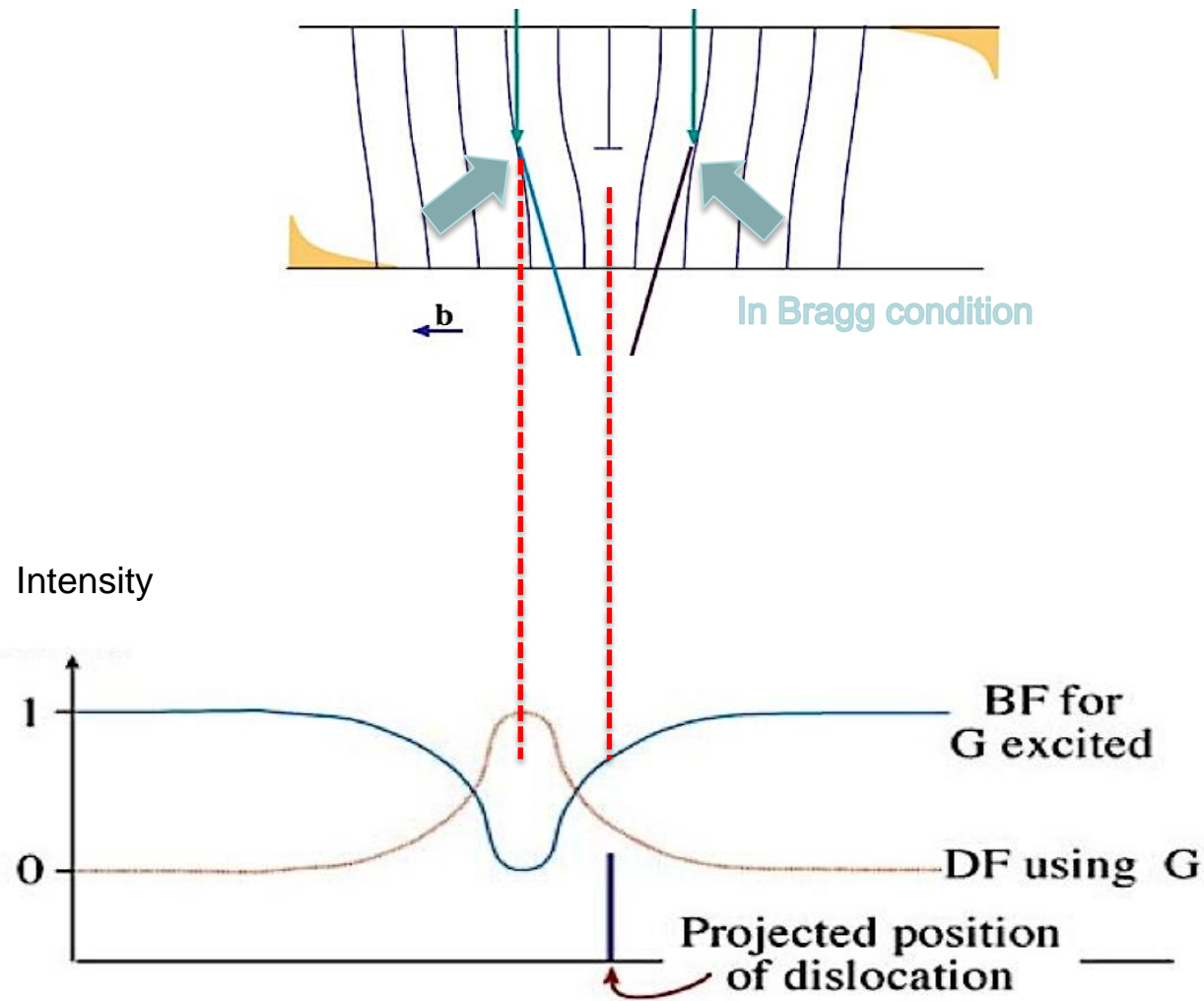
Schematic profiles across the dislocation image showing that the defect contrast is displaced from the projected position of the defect. (As usual for an edge dislocation, **u** points into the paper).

Imaging strain fields

Weak beam = kinematical approximation

Before we saw for 2-beam condition:

$$I_g = \left(\frac{\pi t}{\xi_g}\right)^2 \frac{\sin^2(\pi t s_{eff})}{(\pi t s_{eff})^2} \qquad \text{where:} \qquad s_{eff} = \sqrt{s^2 + \frac{1}{\xi_g^2}}$$

For **g**

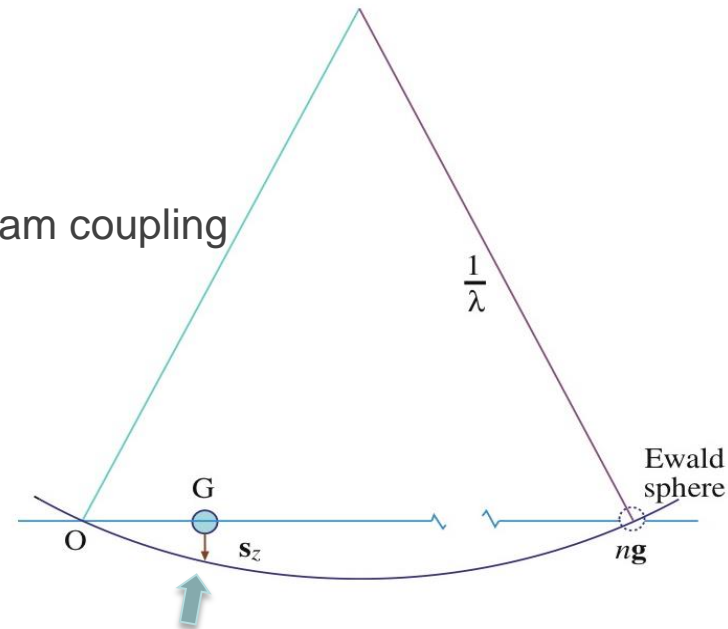Weak-beam imaging: make s large (~0.2 nm^{-1})

Now I_g is effectively independent of ξ_g - i.e. beam coupling "kinematical" conditions!

=> dark-field image intensity easier to interpret

$\frac{1}{\lambda}$

Ewald sphere

O   G s_z $n\mathbf{g}$

Imaging strain fields

In general we need to tilt both the specimen and the beam to achieve weak beam conditions
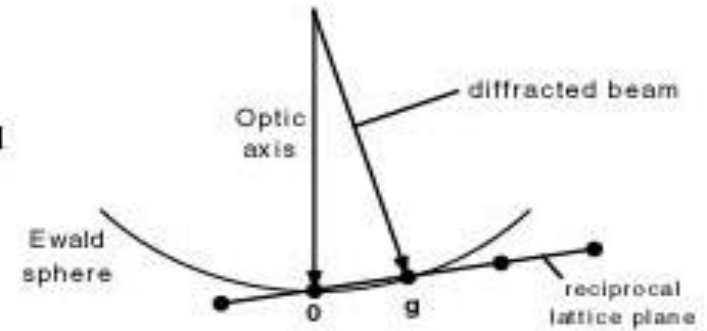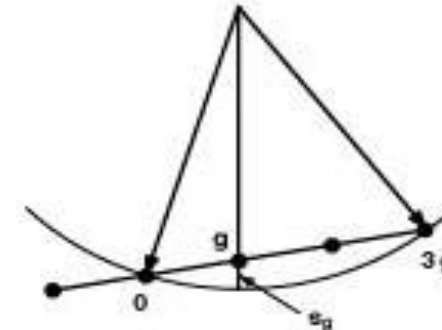
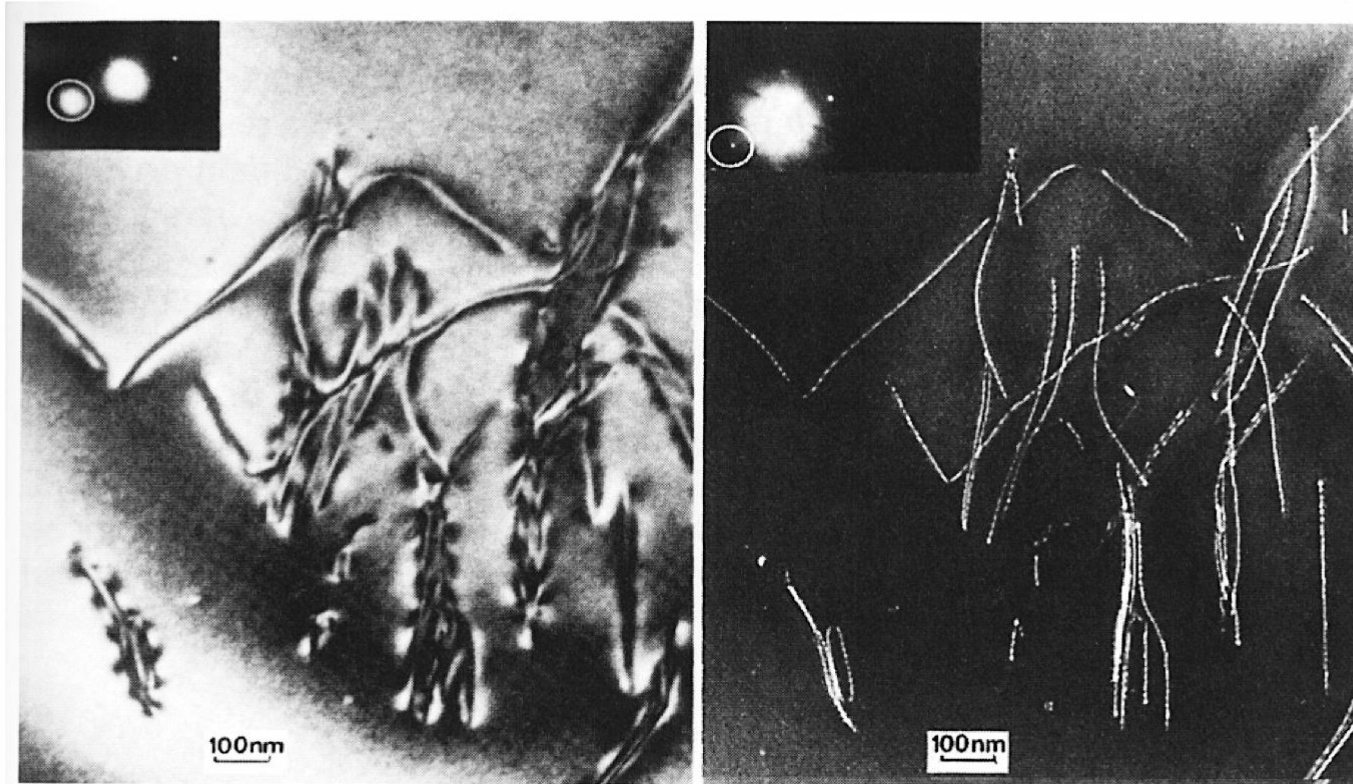# Imaging strain fields

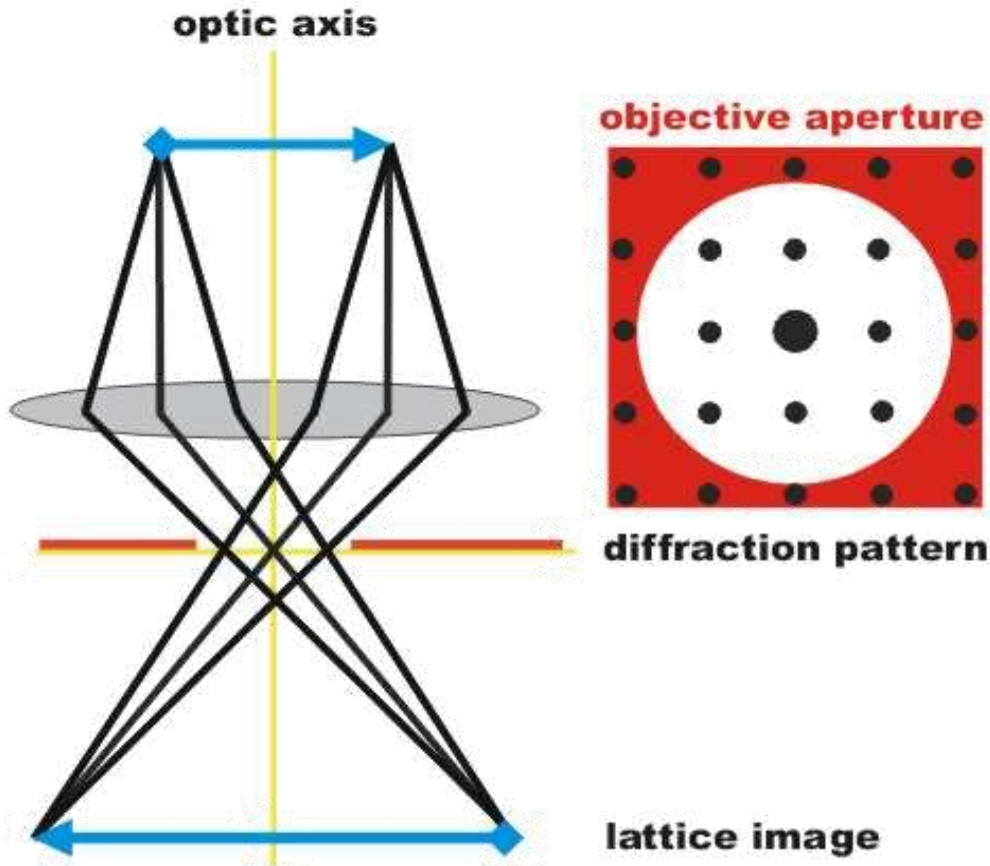Two beam BF   WBDF

Fig. 7.41. Dislocations in Si. Left: BF image in two-beam condition with strong $(2\bar{2}0)$ diffraction. Right: g-$3g$ WBDF image with weak $(2\bar{2}0)$ diffraction. Compare the intensities of the active diffractions (circled in inserts). After [7.9].

Weak beam: finer details easier to interpret!

Phase contrast

optic axis

objective aperture

diffraction pattern

lattice image

Contrast in TEM images can arise due to the differences in the phase of the electron waves scattered through a **thin specimen**.

Many beams are allowed to pass through the objective aperture (as opposed to bright and dark field where only one beam pases at the time).

To obtain lattice images, a large objective aperture has to be selected that allows many beams to pass including the direct beam.
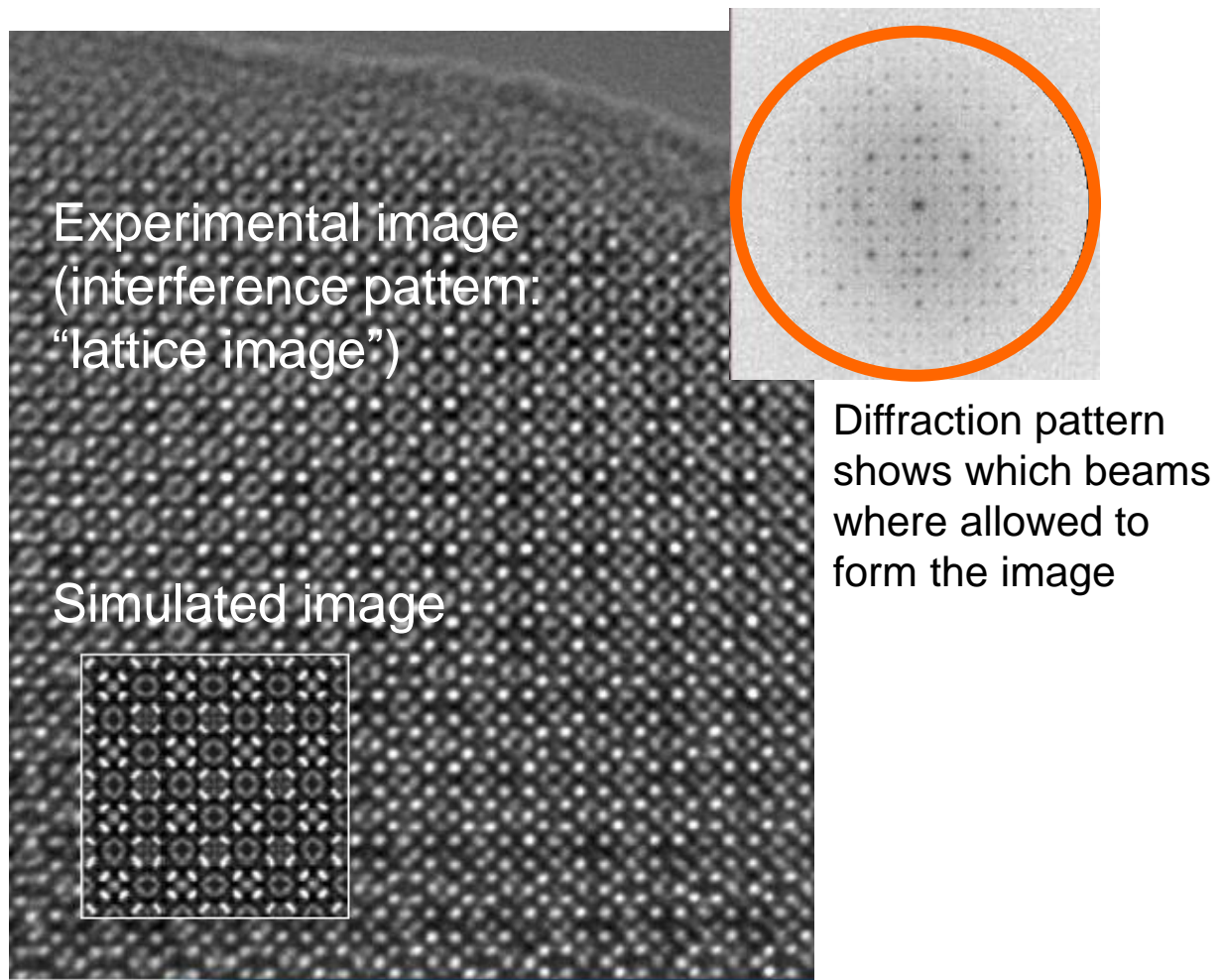
The image is formed by the interference of the diffracted beams with the direct beam (phase contrast). If the point resolution of the microscope is sufficiently high and a suitable crystalline sample is oriented along a low-index zone axis, then high-resolution TEM (HRTEM) images are obtained.

In many cases, the atomic structure of a specimen can directly be investigated by HRTEM

Phase contrast

An atomic resolution image is formed by the "phase contrast" technique, which exploits the differences in phase among the various electron beams scattered by the **THIN** sample in order to produce contrast. A large objective lens aperture allows the transmitted beam and at least four diffracted beams to form an image.

Experimental image (interference pattern: "lattice image")

Simulated image

Diffraction pattern shows which beams where allowed to form the image

Courtesy : ETH Zurich

Phase contrast

- However, the location of a fringe does not necessarily correspond to the location of a lattice plane.

- So lattice fringes are not direct images of the structure, but just give information on lattice spacing and orientation.

- Image simulation is therefore required.

Phase contrast

Some of the Microstructural defects that can be imaged

- **Stacking Faults**
- **Twinning**
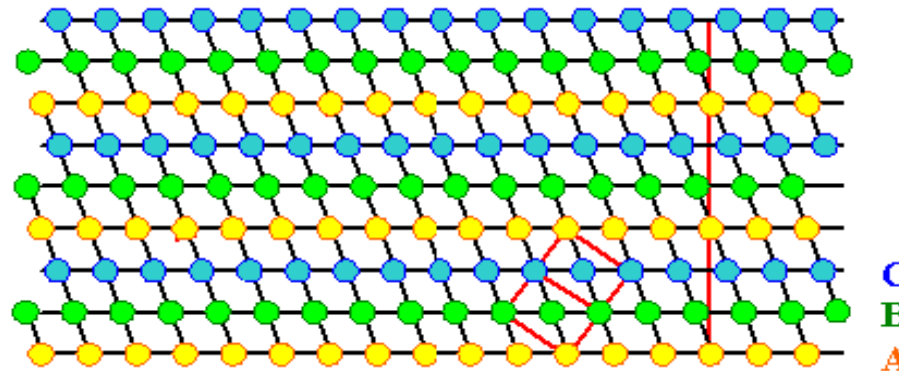- **Interface**
- **Dislocations**
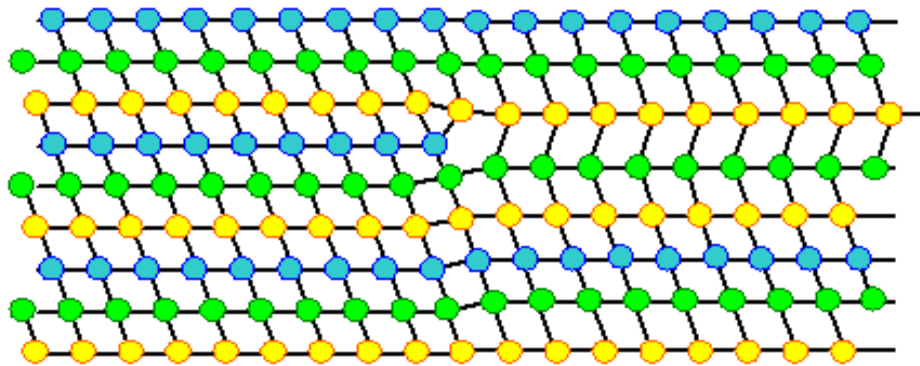
stacking faults

Stacking faults

For FCC metals an error in ABCABC packing sequence

- – Ex: ABCABABC: the local arrangement is hcp
- – Stacking faults by themselves are simple two-dimensional defects. They carry a certain stacking fault energy g~100 mJ/m^2
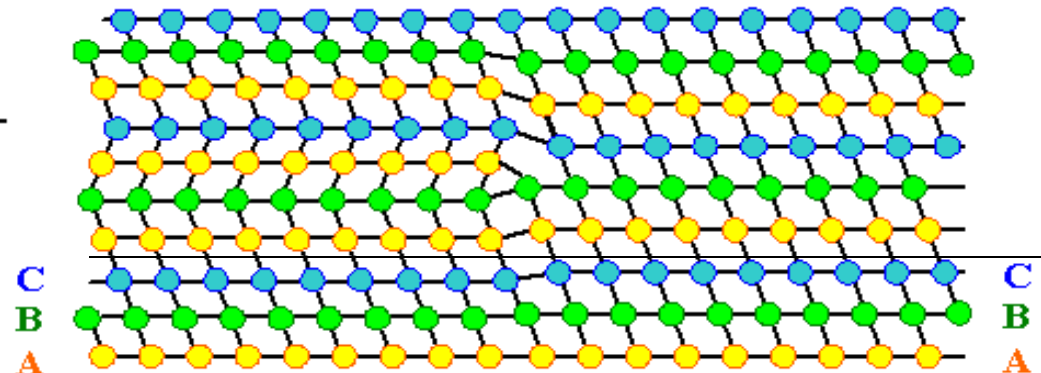
Perfect sequence
<110> projection of
fcc lattice

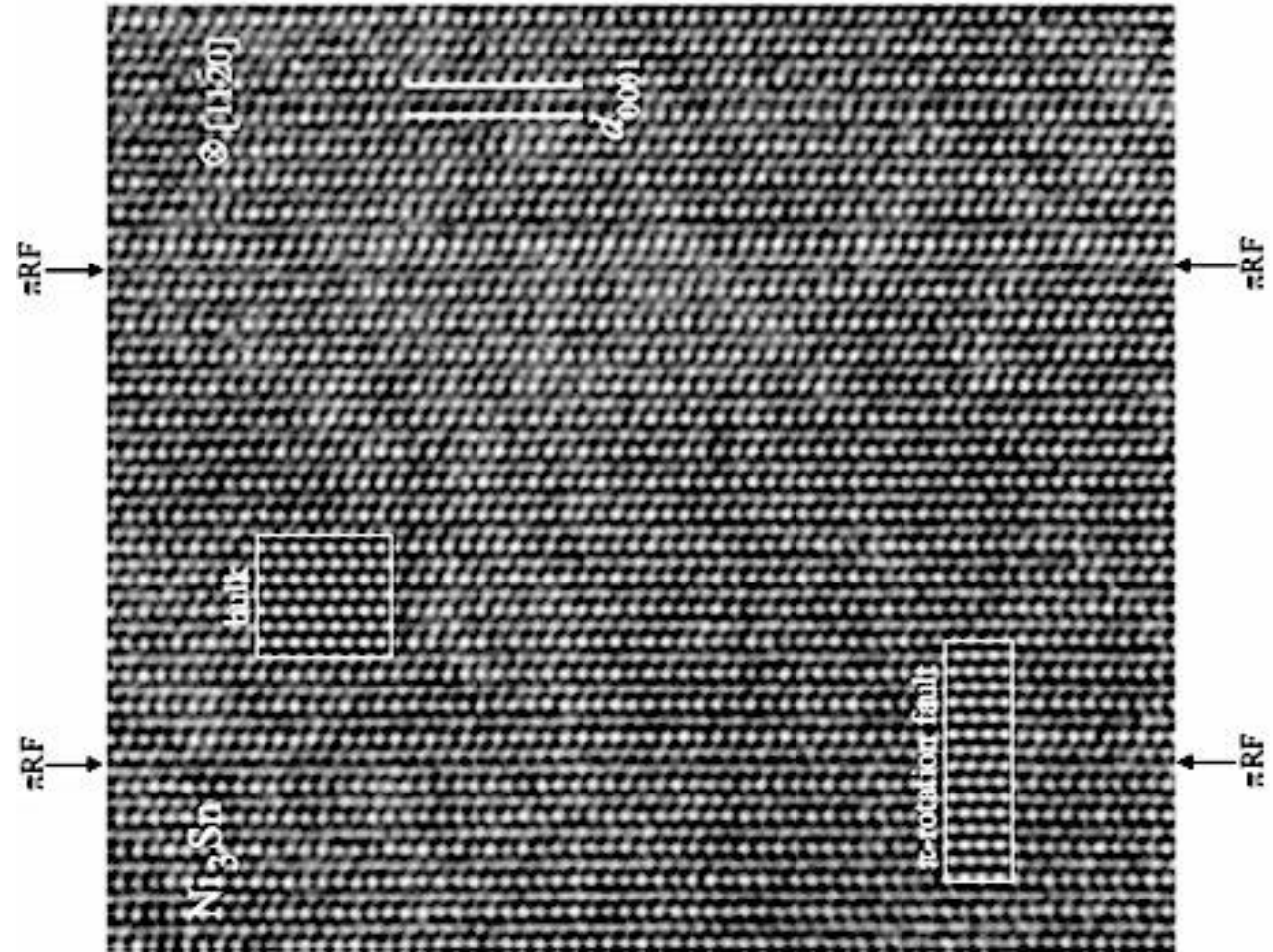
collapse of vacancies disk

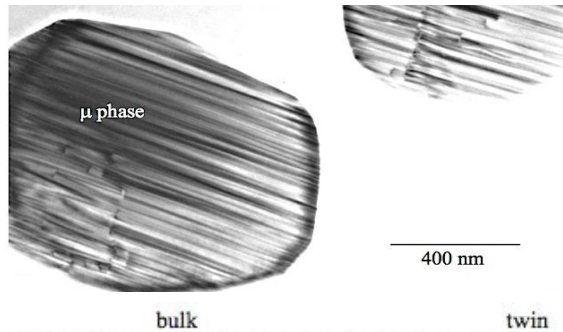condensation of interstitials disk

Phase contrast

Example of easily interpretable information: Stacking faults viewed edge on

Stacking faults are relative displacements
of blocks in relation to the perfect crystal

Phase contrast

Example of easily interpretable information: Polysynthetic twins viewed edge on

400 nm

Co_7W_6

$R\bar{3}m$

bulk   twin

(a)

(b)

(c)

$[1\bar{1}0]_o$
$[001]$

W
Co

mirror planes

twin simulation

bulk simulation

$[1\bar{1}0]_o$
$[\bar{1}10]_r$

$[001]$ mirror plane $d_{(003)} \approx 0.85$ nm
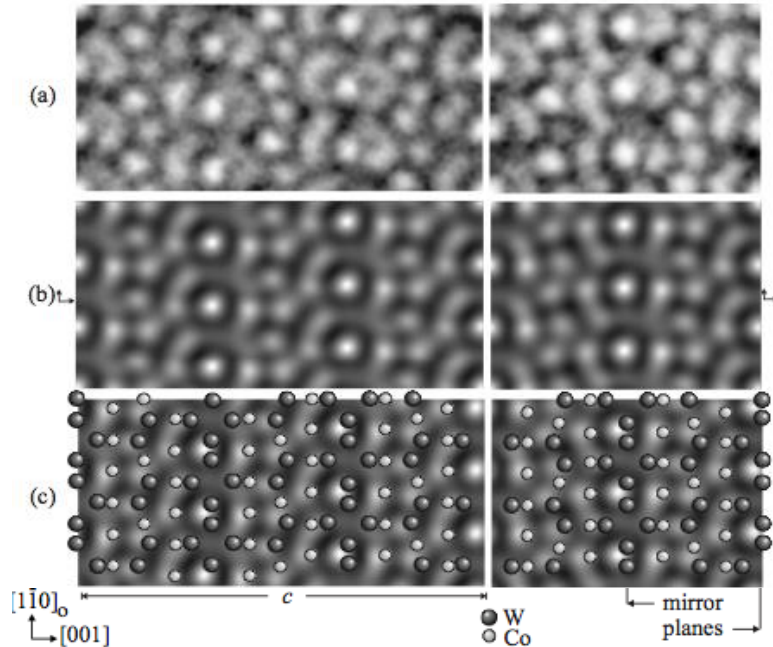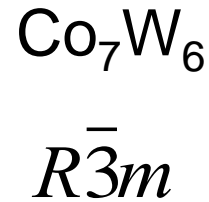
Compare the relative position of the atoms and intensity maxima!
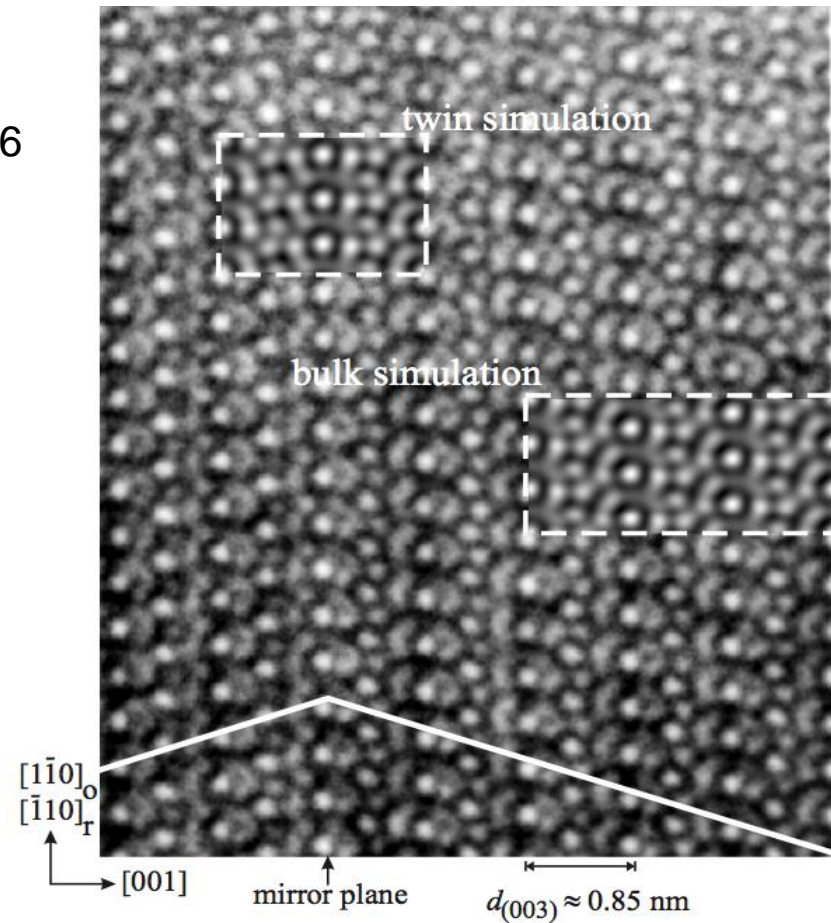
Phase contrast

Example of easily interpretable information:
Faceting at atomic level at a Ge grain boundary
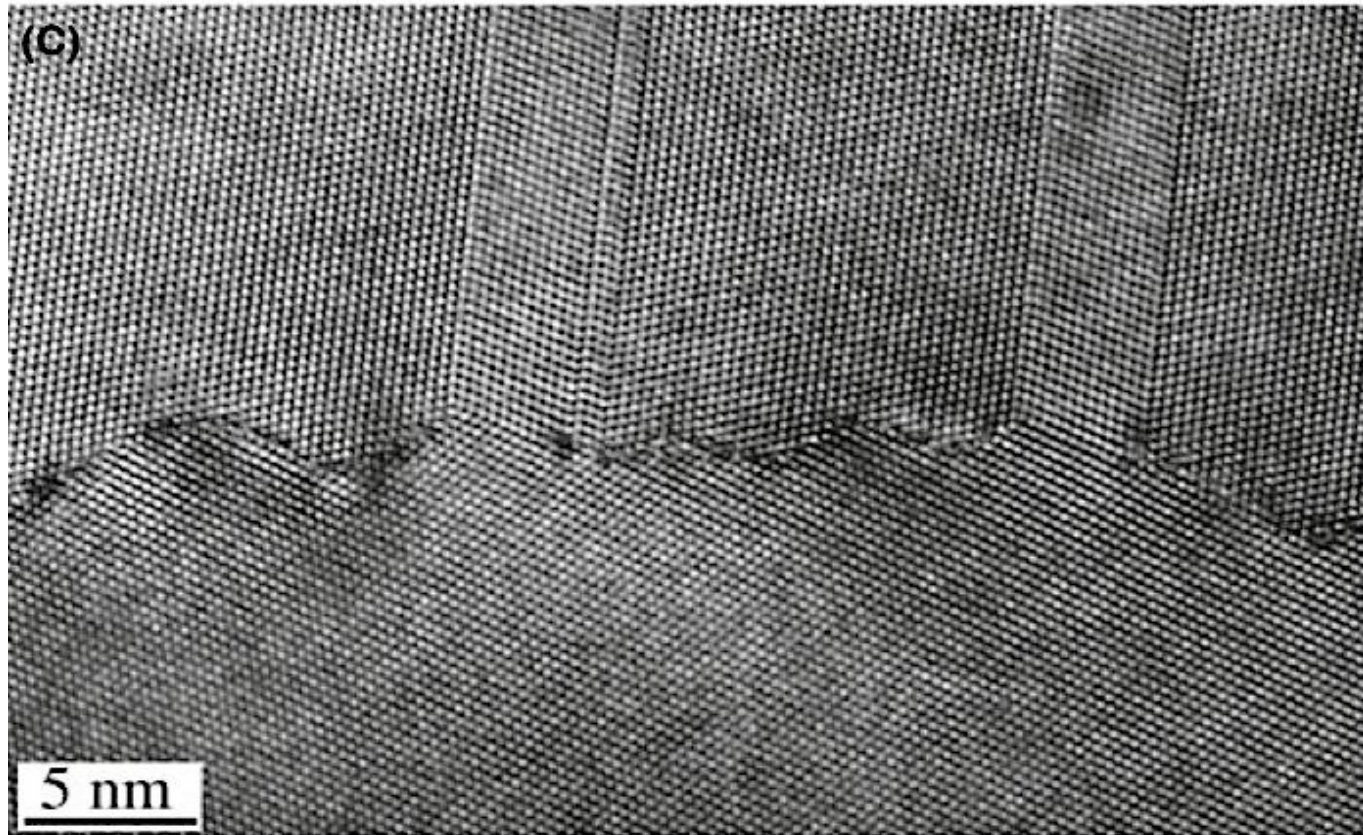
Williams and Carter book

Phase contrast

Example of easily interpretable information:
misfit dislocations viewed end on at a heterojunction between InAsSb and InAs
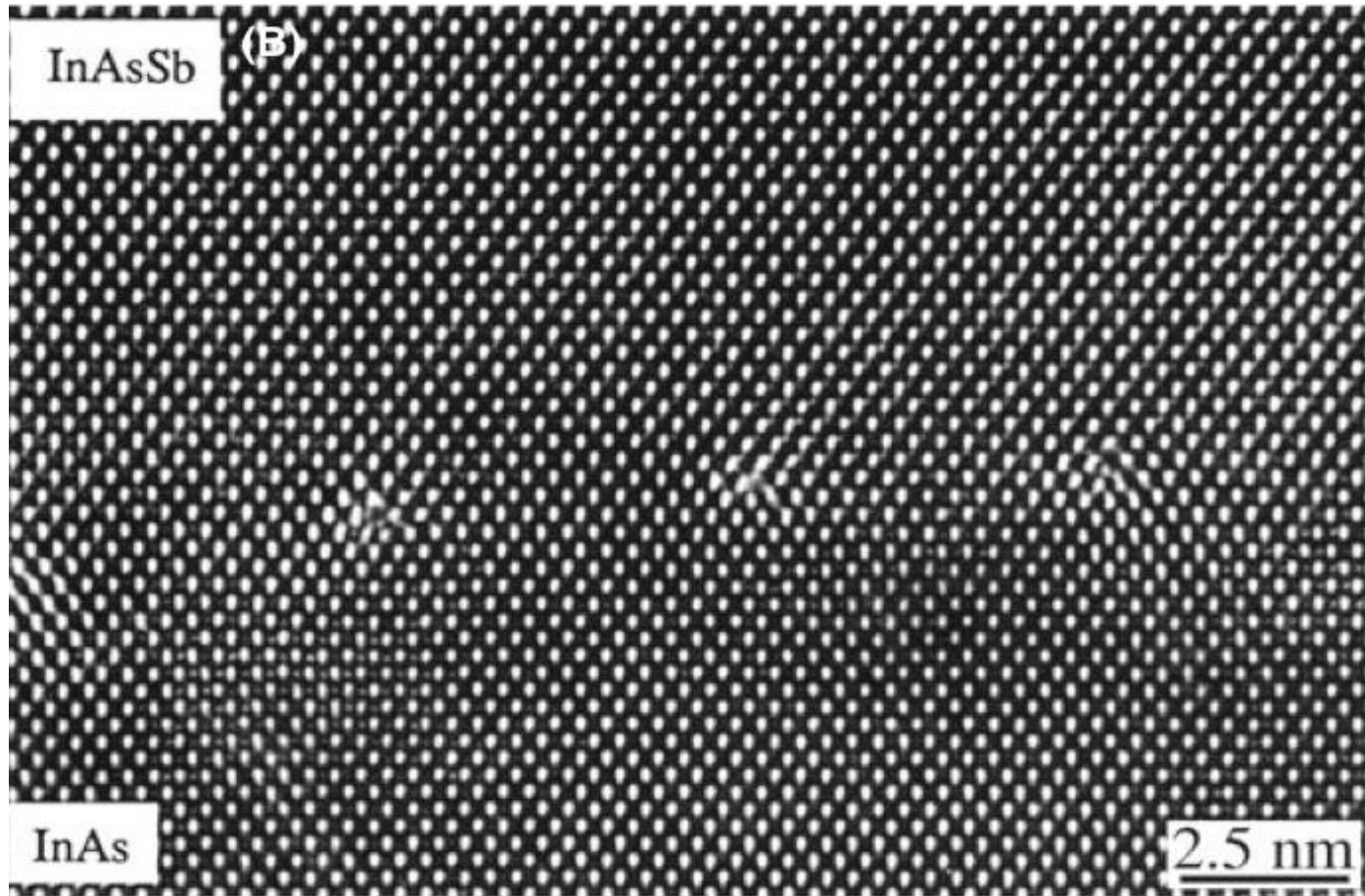
Williams and Carter book

Phase contrast

Example of easily interpretable information:
misfit dislocations viewed end on at a heterojunction between InAsSb and InAs
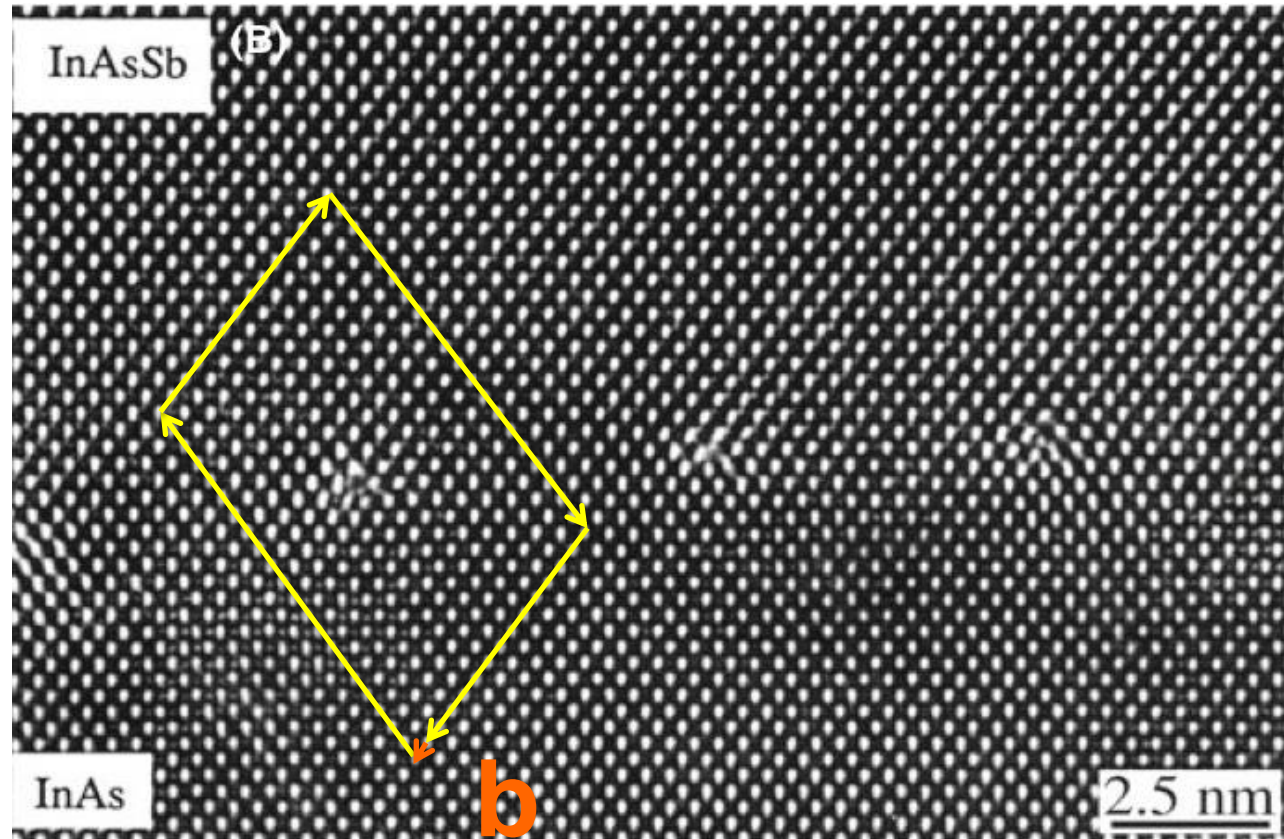
Direct use of the Burgers circuit:

Burgers vector of the dislocation

InAsSb

(E)

InAs

b

2.5 nm

Williams and Carter book

Resolution in HRTEM

Resolution of an Imaging system

Two independent origins

(A)Diffraction limit –

(Inherent nature of bending of light/electron waves when passes through an aperture/lens of finite size)

(B) Aberrations in the image forming lens –

(Inherent nature of the lens used in the imaging system)

EFFECT of the both (A) and (B) combined?

Point in object → Disc/spread out point in the image

(A) Diffraction limit

Resolution of an optical system

Rayleigh criterion

- The resolving power of an optical system is limited by the diffraction occurring at the optical path every time there is an aperture/diaphragm/lens.
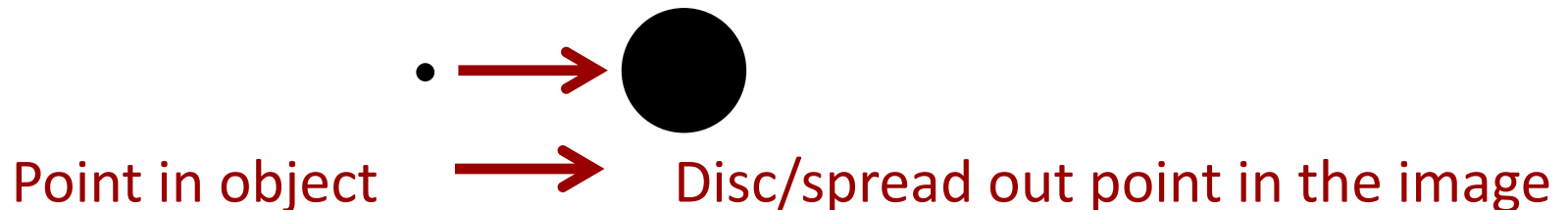- The aperture causes interference of the radiation (the path difference between the green waves results in destructive interference while the path difference between the red waves results in constructive interference).
- An object such as point will be imaged as a disk surrounded by rings.
- The image of a point source is called the **Point Spread Function**

Resolution Limit Imposed by Wave Nature of Light
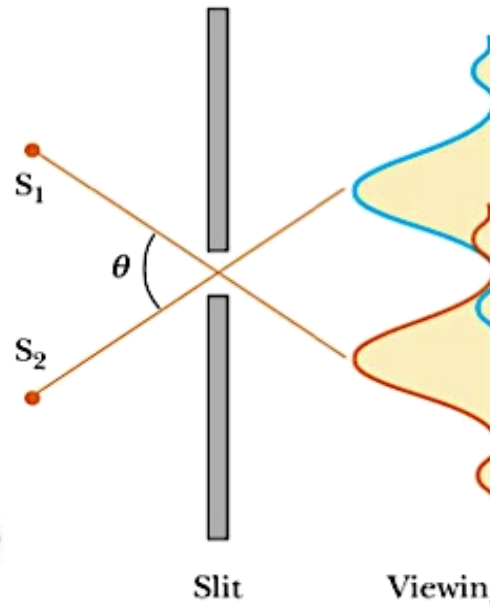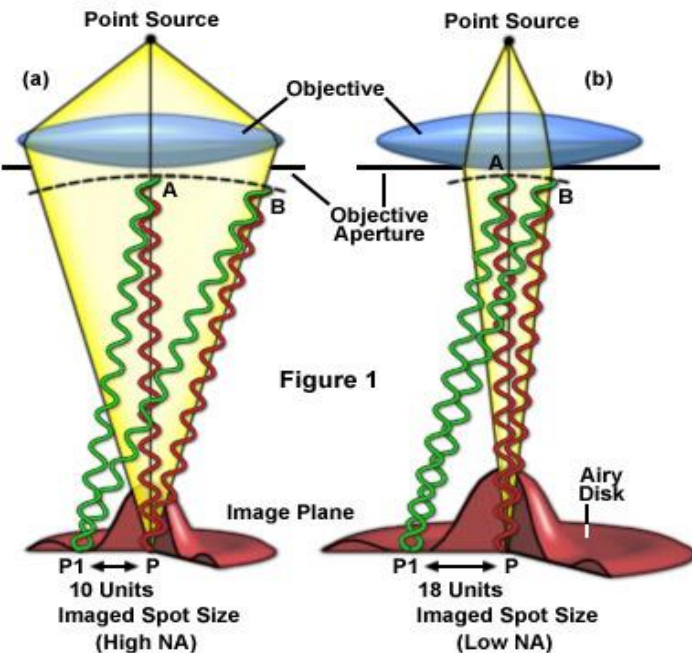
Point spread function (real space)
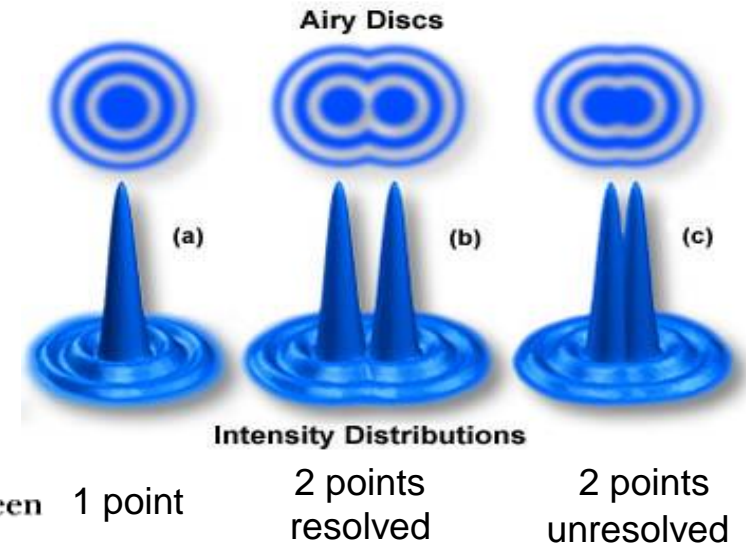
Resolution of an optical system

Diffraction at an aperture or lens - Rayleigh criterion

The Rayleigh criterion for the resolution of an optical system states that two points will be resolvable if the maximum of the intensity of the Airy ring from one of them coincides with the first minimum intensity of the Airy ring of the other. This implies that the resolution, d_0 (strictly speaking, the resolving power) is given by:

$$d_o = 0.61 \cdot \frac{\lambda}{\eta \cdot Sin(\alpha)}$$

where λ is the wavelength, η the refractive index and α is the semi-angle at the specimen.

$\eta \cdot Sin(\alpha)$ = NA (Numerical Aperture).

This expression can be derived using a reasoning similar to what was described for diffraction gratings (path differences...).

When d_0 is small the resolution is high!
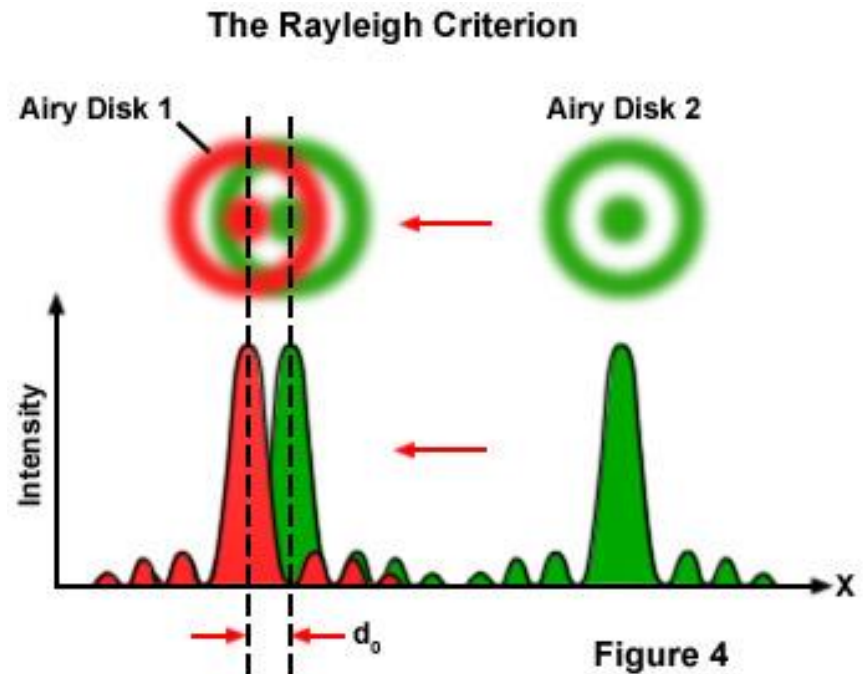
The Rayleigh Criterion

Airy Disk 1   Airy Disk 2

Intensity

X

d_o

Figure 4

Resolution of an Imaging system

Two independent origins

(A) Diffraction limit –

(Inherent nature of bending of light/electron waves when passes through an aperture/lens of finite size)

(B) Aberrations in the image forming lens –

(Inherent nature of the lens used in the imaging system)

EFFECT of the above?

Point in object ⟶ Disc/spread out point in the image
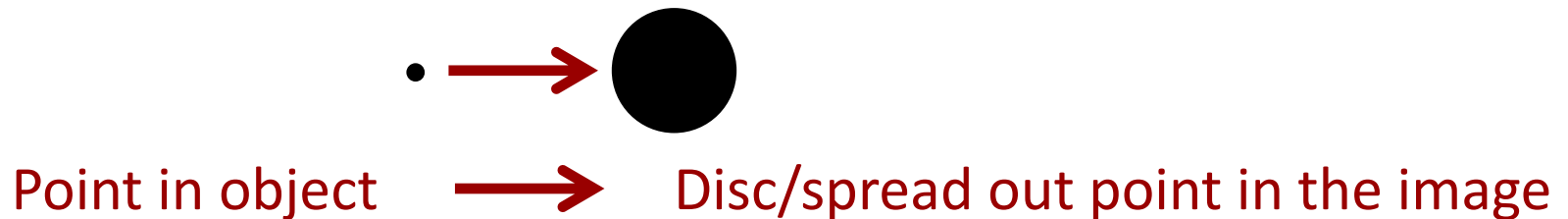
(B) Aberrations in the electro magnetic lens

Aberrations in TEM lens

Electron gun

Energy Spread

Objective lens, imaging process

2-fold, 3-fold Astigmatism
Spherical Aberration (C_S)
Chromatic Aberration (C_C)
Coma

Defocus Spread
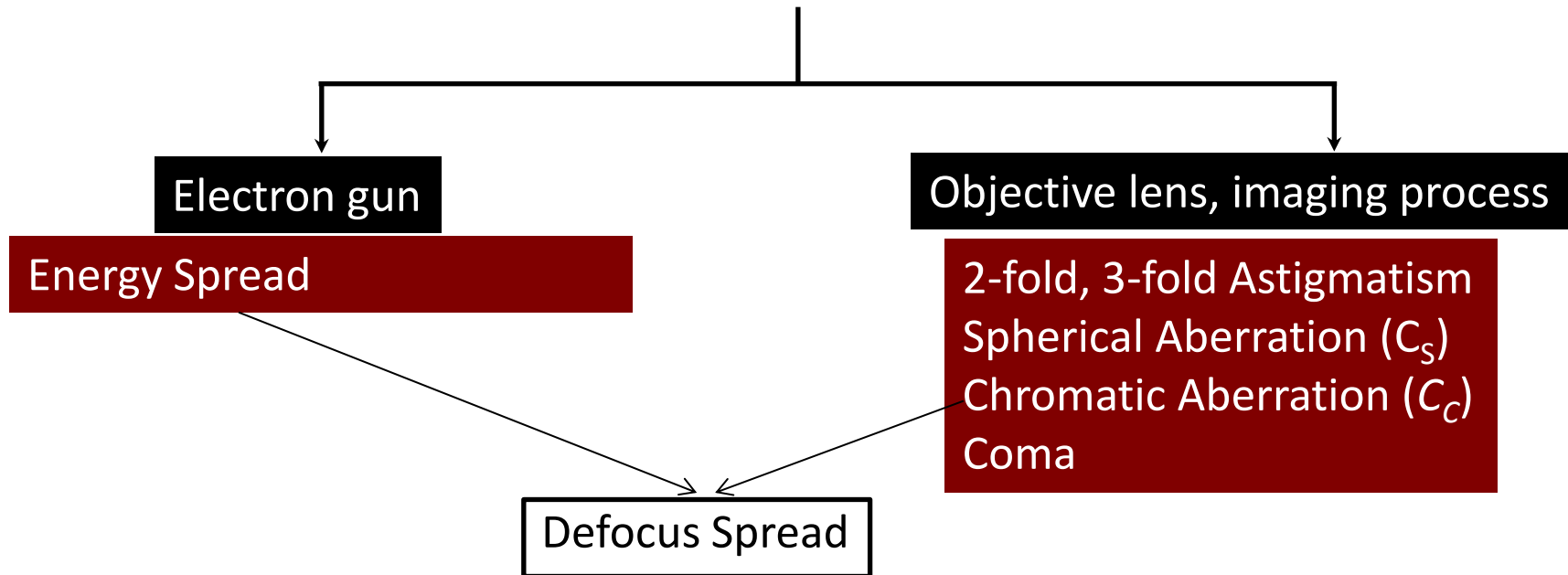
In reality, there are atleast about 10 different kinds of lens aberrations in TEM lenses that impose limitation of final resolution!!!

Spherical aberration coefficient (C_S) Chromatic aberration coefficient (C_c)
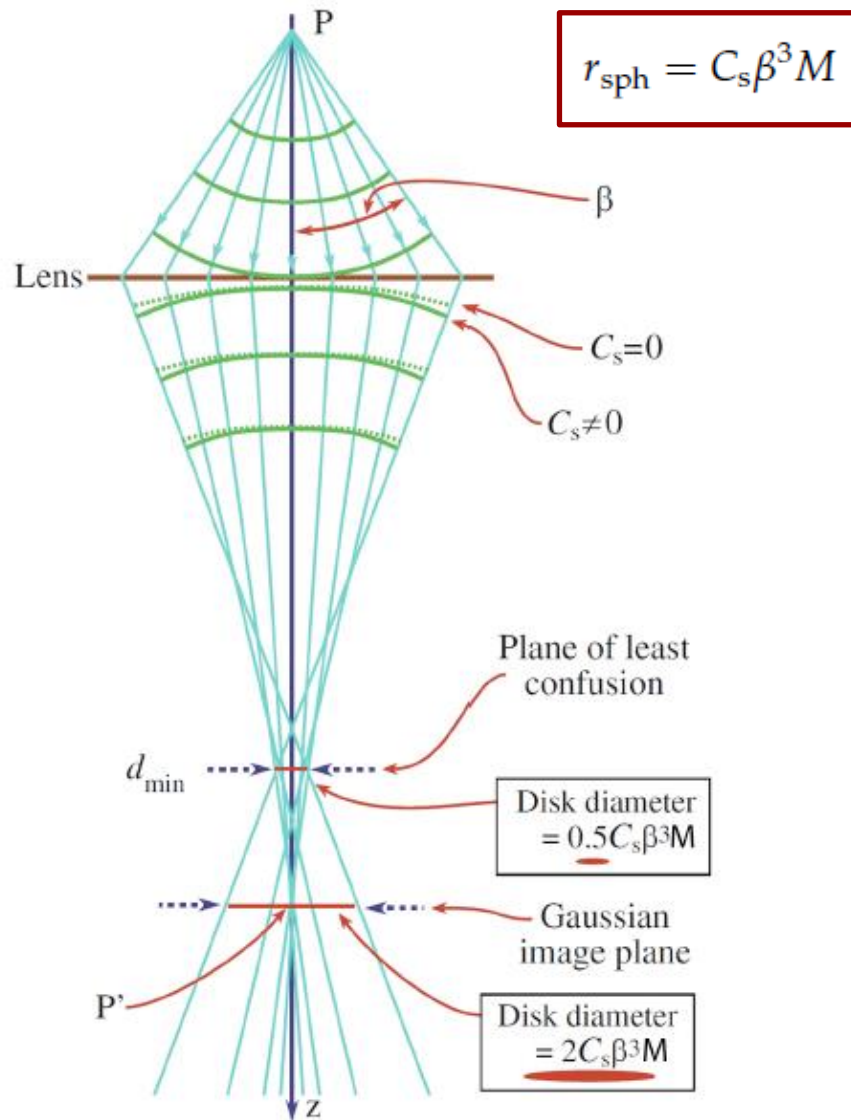
$$r_{sph} = C_s \beta^3 M$$

$$r_{chr} = C_c \cdot \left[\left(\frac{\Delta E}{E_0}\right)^2 + 2\left(\frac{\Delta I}{I}\right)^2 \right]^{-\frac{1}{2}} \beta M$$

P

β

Lens

$C_s = 0$

$C_s \neq 0$

Plane of least confusion

d_{min}

Disk diameter $= 0.5 C_s \beta^3 M$

Gaussian image plane

P'

Disk diameter $= 2 C_s \beta^3 M$

z

Specimen

β

Lens

Energy-loss electrons

No-loss electrons

Plane of least confusion

Gaussian image plane
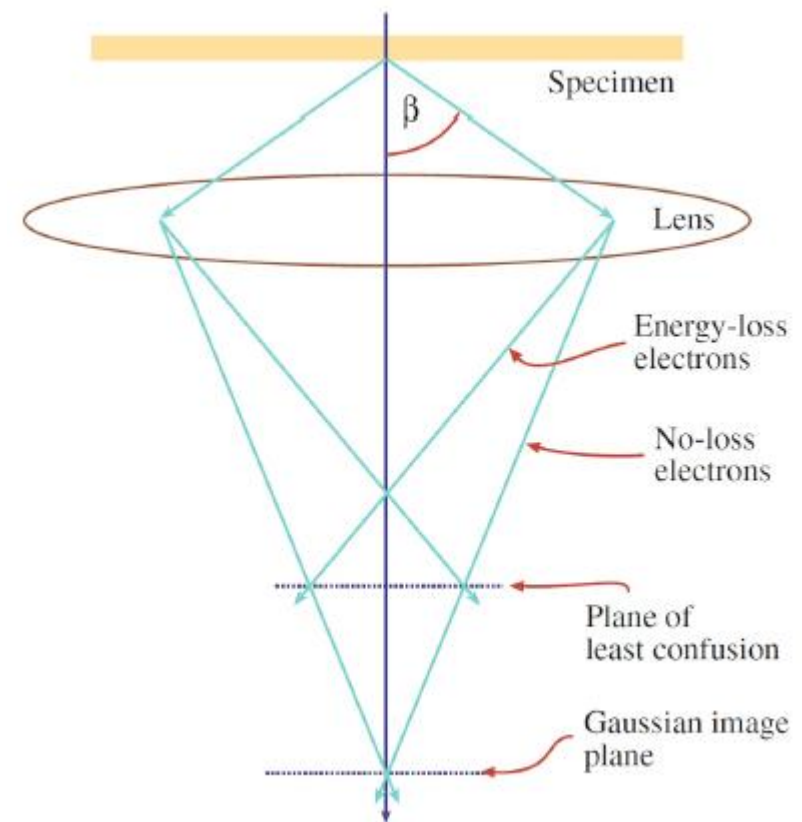
TEM Lens Aberrations

Schematic of spherical aberration correction

(a) ... specimen / lens / image / P / R

(b) ... P / Cs Correction lens system (diverging lens)

Probe-forming Cs corrector (CEOS GmbH)

Image-forming Cs corrector (CEOS GmbH)

25 cm

25 cm

Control system
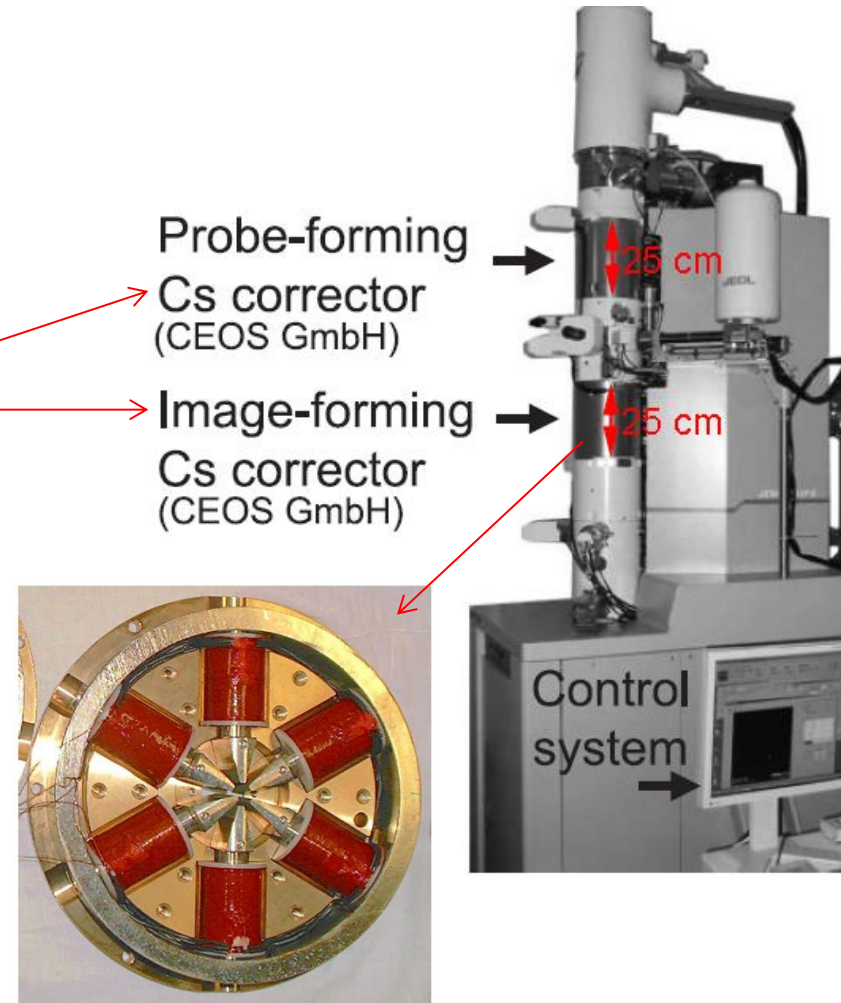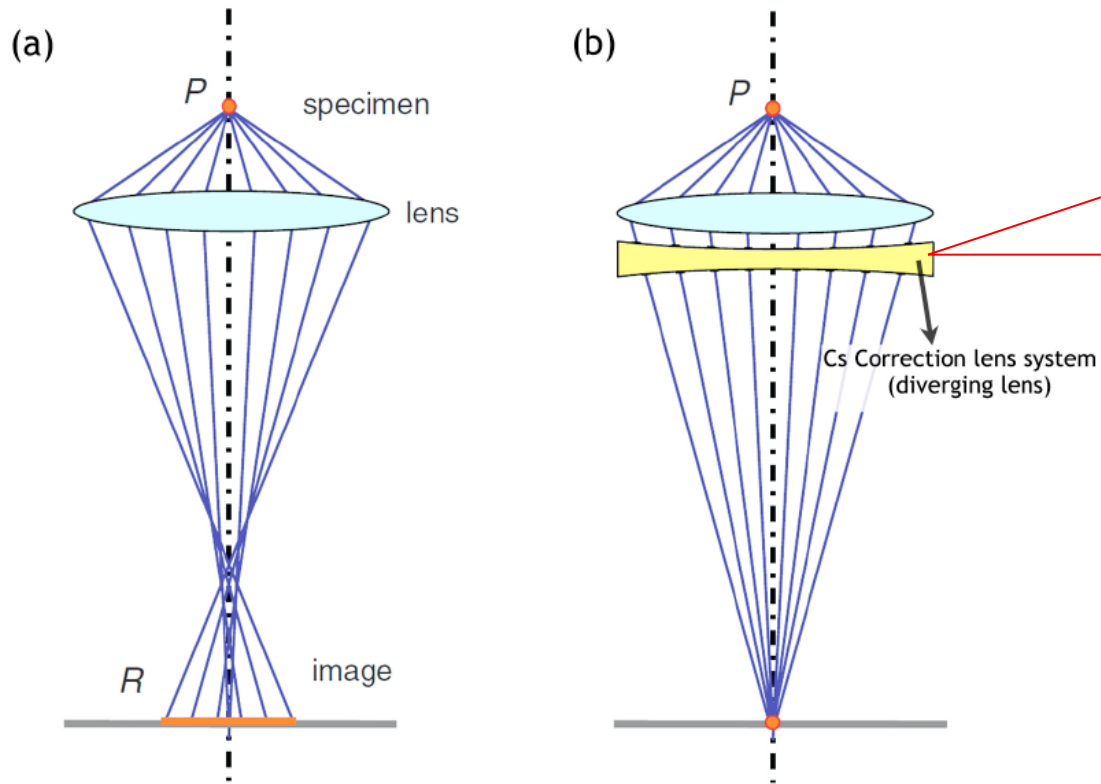
Courtesy: Knut W. Urban, Science 321, 506, 2008; CEOS gmbh, Germany; www.globalsino.com

Resolution of an Imaging system

Two independent origins

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EFFECT of the both (A) and (B) combined?

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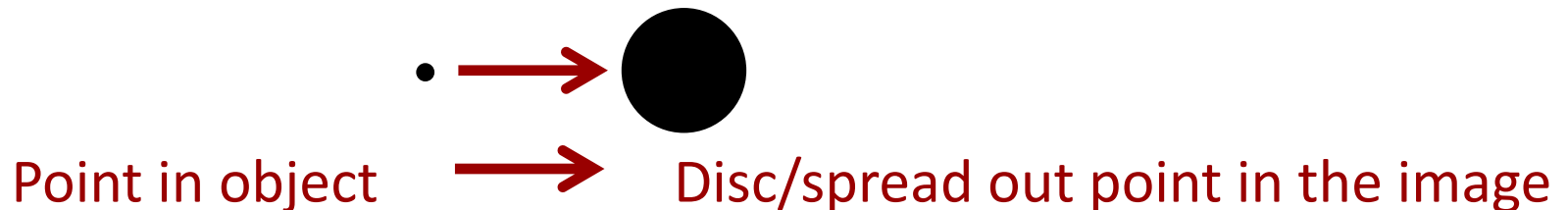
How can we now describe the effect of point spread function of an imaging system mathematically???

FOURIER TRANSFORMATIONS (FT)

FT of PSF in light Microscope **= OTF (Optical Transfer Function)**

FT of obj. lens image formation in HRTEM **= CTF (Contrast Transfer Function)**

New concept:
Contrast Transfer Function (CTF)

Optical Transfer Function (OTF)
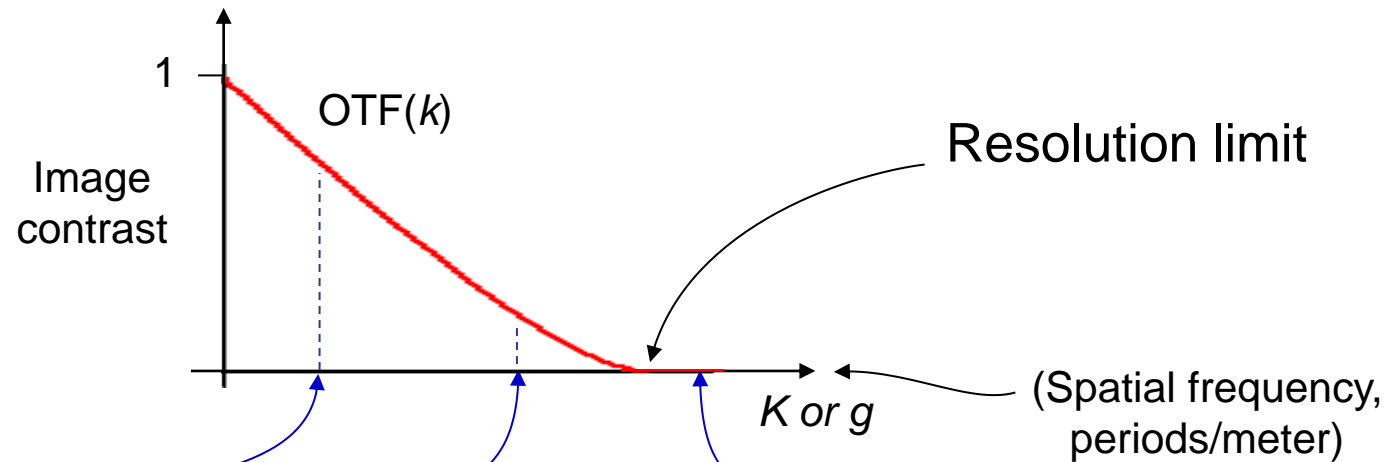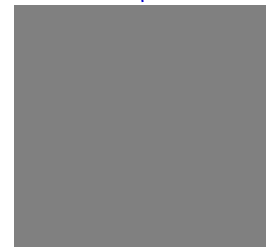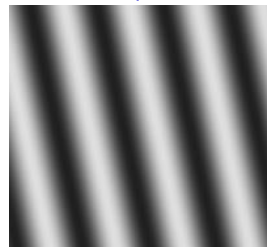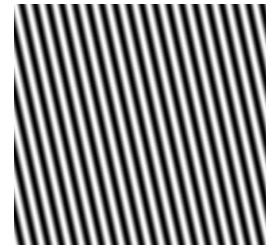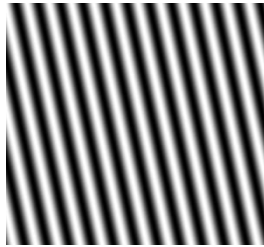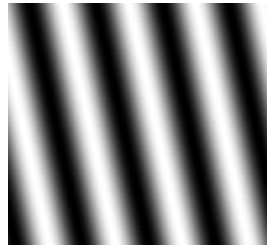
Kurt Thorn, University of California, San Francisco

Definitions of Resolution

As the OTF cutoff frequency

OTF(k)
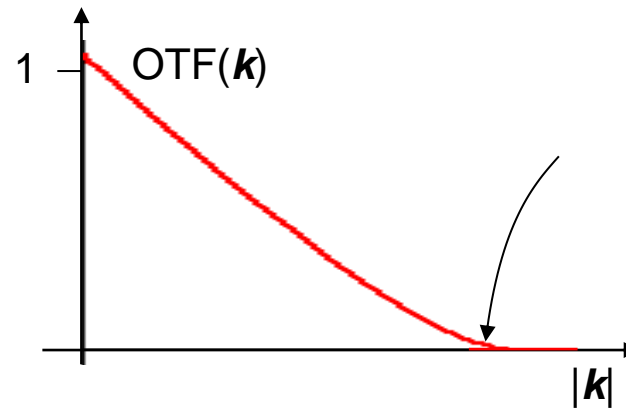
$1/k_{max}$
$= 0.5\ \lambda\ /NA$

As the Full Width at Half Max (FWHM) of the PSF

FWHM
$\approx 0.353\ \lambda\ /NA$
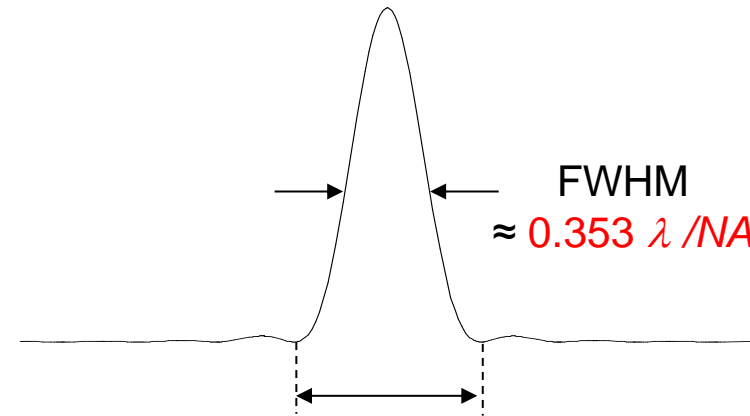
As the diameter of the Airy disk (first dark ring of the PSF) = "Rayleigh criterion"

Airy disk diameter
$\approx 0.61\ \lambda\ /NA$

Kurt Thorn, University of California, San Francisco

The Transfer Function Lives in Frequency Space

Object   Observed image

OTF($\boldsymbol{k}$)

$|\boldsymbol{k}|$

$\boldsymbol{k_y}$

$\boldsymbol{k_x}$

Observable Region
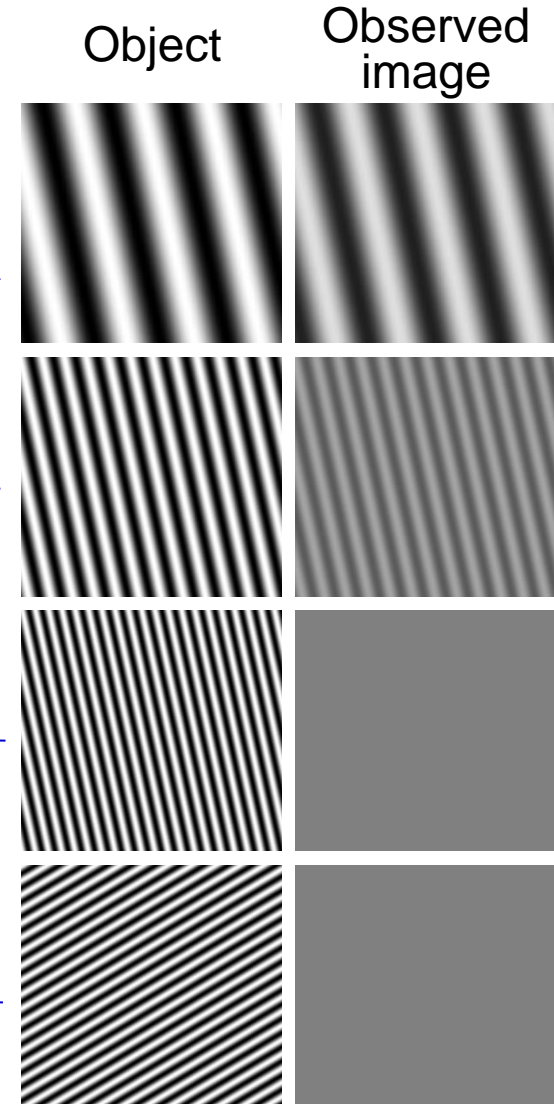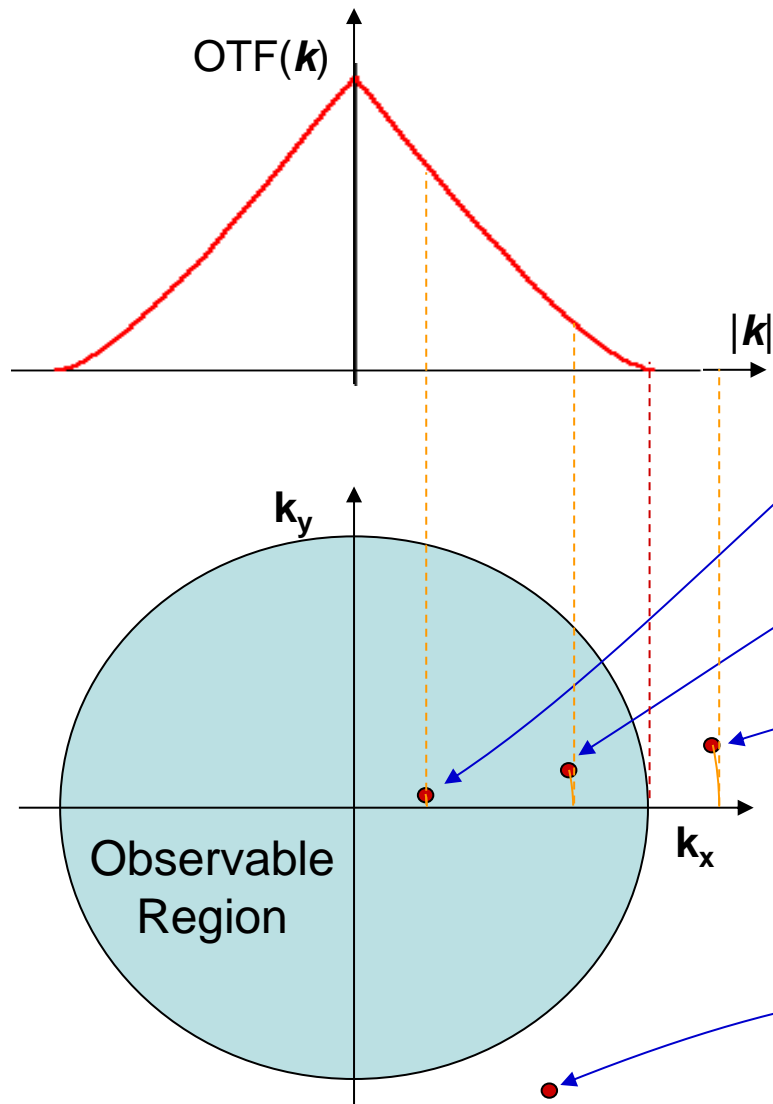
Kurt Thorn, University of California, San Francisco

The OTF and Imaging

True
Object

convolution

PSF

Observed
Image

⊗

=

Fourier
Transform

OTF

•

=

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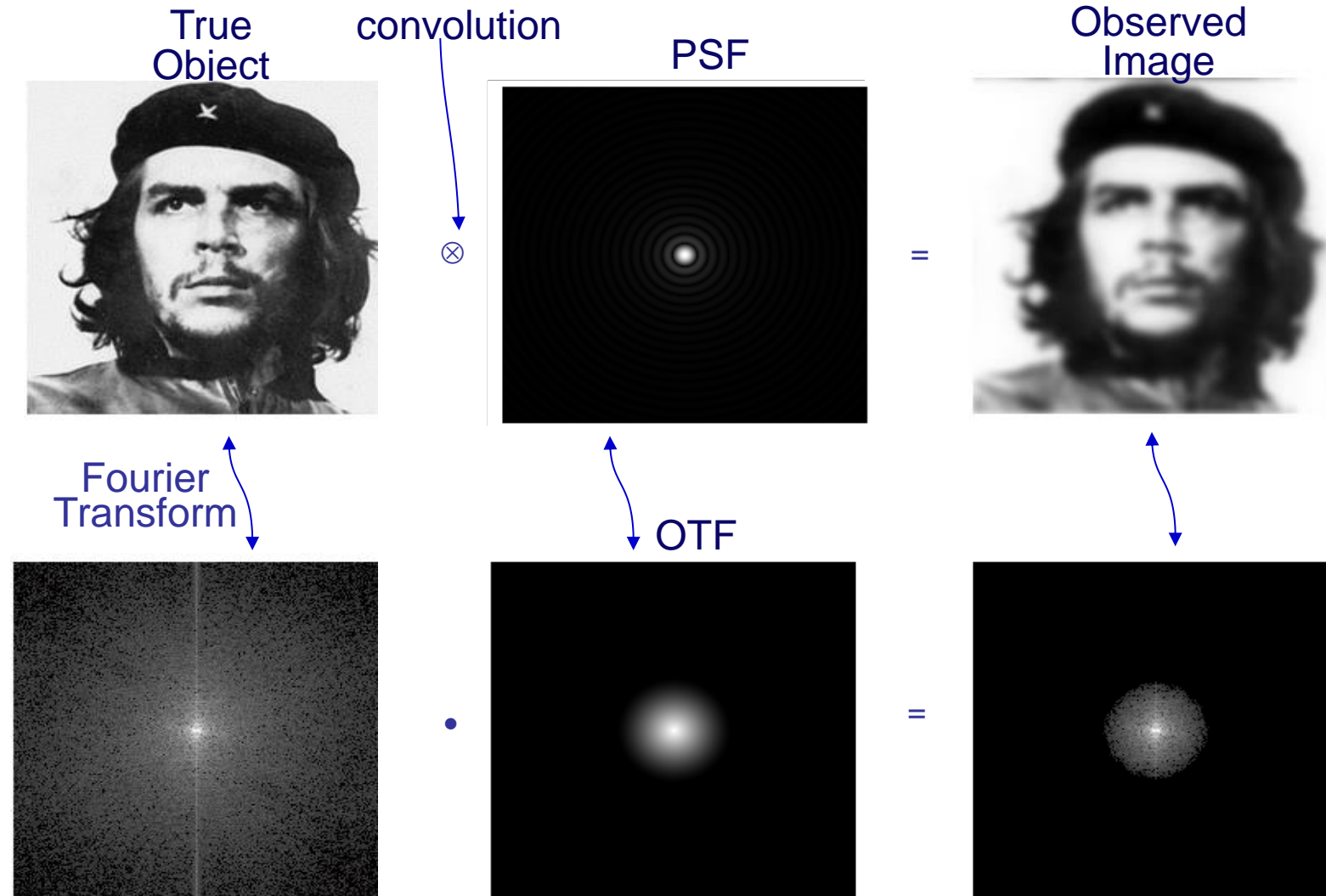
Nomenclature

Optical transfer function, OTF
Wave transfer function, WTF
Contrast transfer function, CTF

Similar concepts:
Complex values (amplitude and phase)

Weak-phase object
very thin sample: no absorption (no change in amplitude) and only weak phase shifts induced in the scattered beams

Contrast Transfer Function in HRTEM, CTF
For weak-phase objects only the phase is considered

Principle of HRTEM formation

incident plane wave
$\Psi_0(\vec{r})$

atomic potential of specimen
$\phi(x, y)$

Object Exit Wave **+** *CTF* of lens **=** HRTEM image

A **+** B **=**

TEM experiment
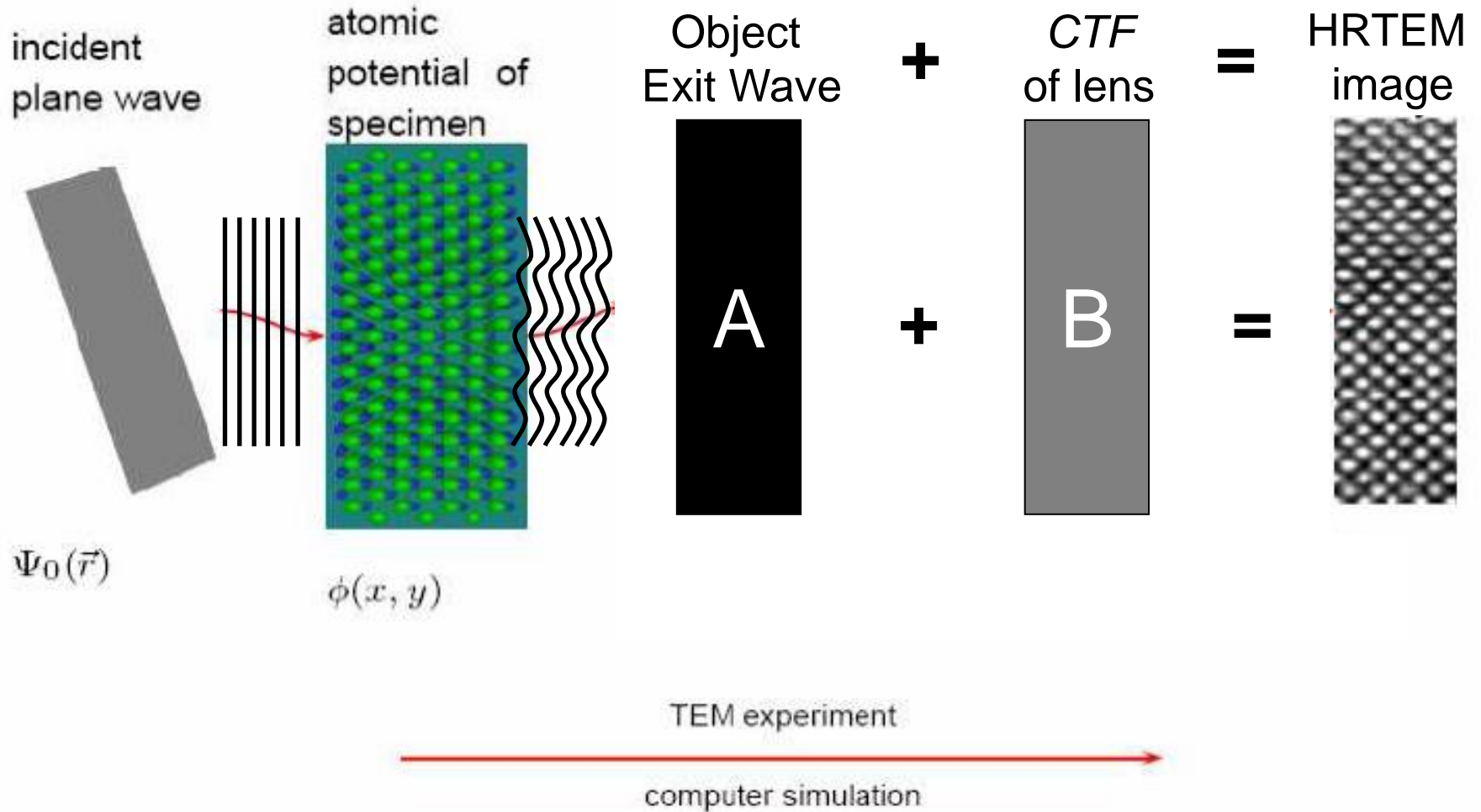
computer simulation

Principle of HRTEM formation

Figure 3.6: Schematic of HRTEM image formation of an individual SWCNT. It also represents the principle of HRTEM image simulation process.

Resolution in HRTEM

The resolution of an electron microscope is more complex than simple Rayleigh Criterion (Independent of Object nature) used in Light Optics.

Image "resolution" is a measure of the spatial frequencies transferred from the image amplitude spectrum (exit-surface wave-function) into the image intensity spectrum (the Fourier transform of the image intensity). This transfer is affected by several factors:

- the phases of the diffracted beams exiting the sample surface,
- additional phase changes imposed by the objective lens defocus and spherical aberration,
- the physical objective aperture,
- coherence effects that can be characterized by the microscope spread-of-focus and incident beam convergence.

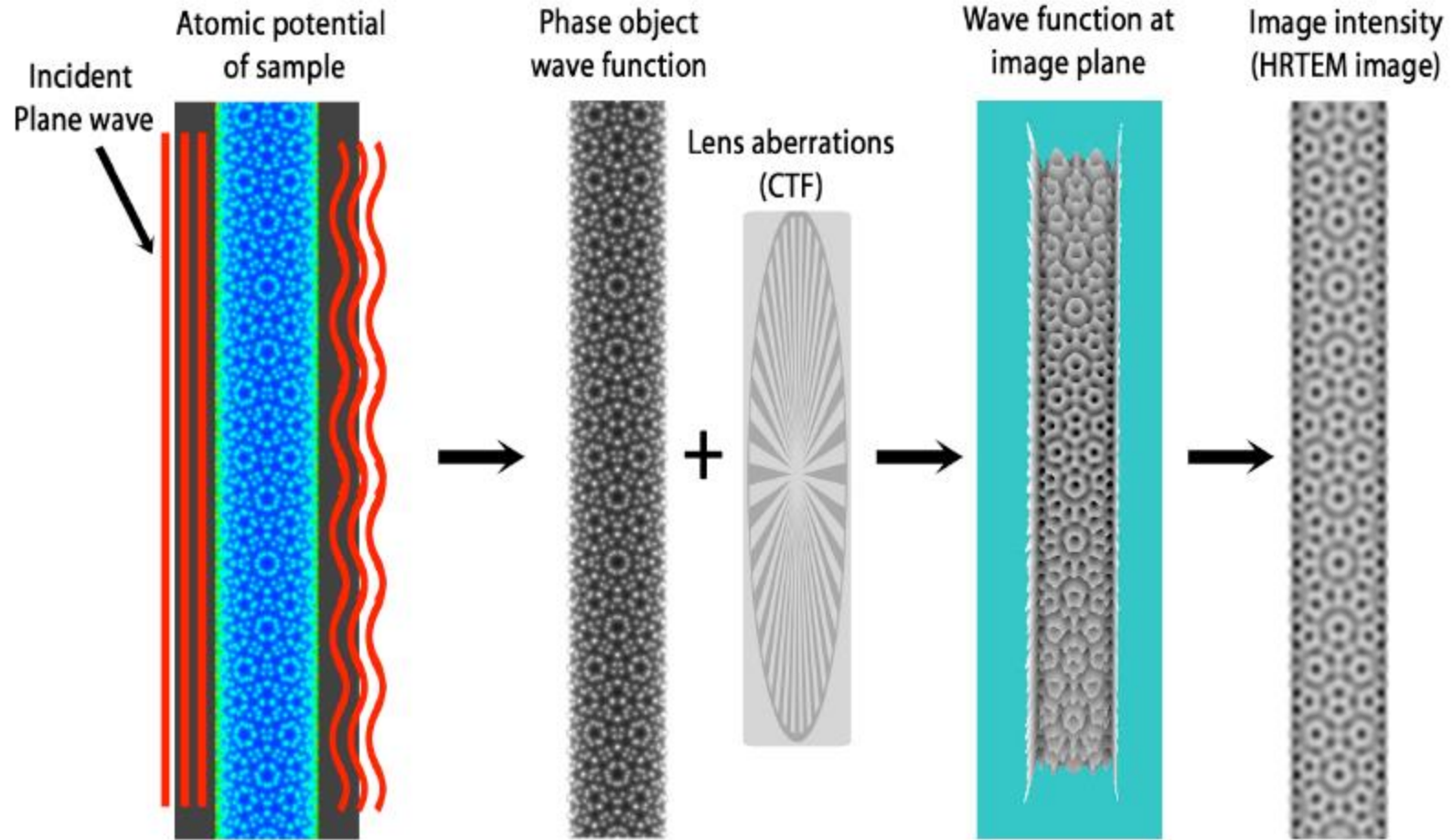For thicker crystals, the frequency-damping action of the coherence effects is complex but for a thin crystal, i.e., one behaving as a weak-phase object (WPO), the damping action can best be described by quasi-coherent imaging theory in terms of envelope functions imposed on the usual phase-contrast transfer function.

The concept of HRTEM resolution is only meaningful for thin objects and, furthermore, one has to distinguish between **point resolution** and **information limit**.

Contrast transfer function

In the Fraunhofer approximation to image formation, the intensity in the back focal plane of the objective lens is simply the Fourier transform of the wave function exiting the specimen. Inverse transformation in the back focal plane leads to the image in the image plane.

If the phase-object approximation holds (no absorption), the image of the specimen by a perfect lens shows no amplitude modulation. In reality, a combination with the extra phase shifts induced by defocus and the spherical aberration of the objective lens generates suitable contrast.

The influence of these extra phase shifts can be taken into account by multiplying the wavefunction at the back focal plane with functions describing each specific effect. The phase factor used to describe the shifts introduced by defocus and spherical aberration is:

$$\chi(q) = \pi \lambda \Delta f q^2 + 1/2 \pi Cs \lambda^3 q^4$$

with Δf the defocus value and Cs the spherical aberration coefficient. The function that multiplies the exit wave is then:
$$B(q) = \exp(i\chi(q))$$

If the specimen behaves as a weak-phase object, only the imaginary part of this function contributes to the contrast in the image, and one can set:

$$B(q) = 2\sin(\chi(q))$$

The phase information from the specimen is converted into intensity information by the phase shift introduced by the objective lens and this equation determines the weight of each scattered beam transferred to the image intensity spectrum. **For this reason, sin(χ) is known as the contrast transfer function (CTF) of the objective lens or Phase Contrast Transfer Function.**

WEAK PHASE OBJECT APPROXIMATION

- Object is very thin
- induces no amplitude modulation of the incident wave (no absorption)
- Only induces very weak phase shift on the scattered wave

Then, the contrast in the image is only due the additional phase shift on this exit scattered wave induced by Objective Lens

(a) Defocus Δf
(b) Spherical Aberration C_s

Contrast Transfer Function:

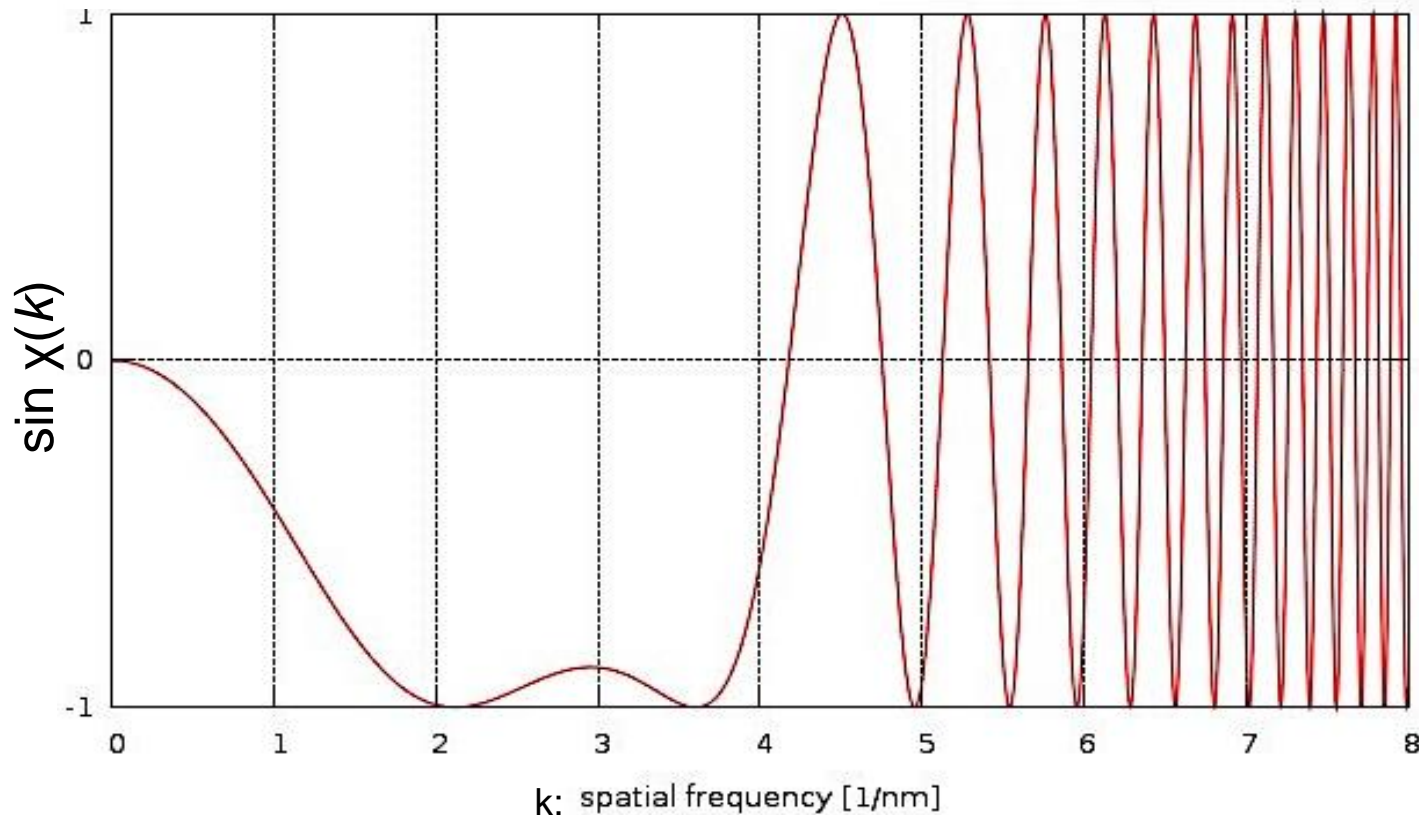$$B(q) = \sin(\phi(q)) = \sin(\frac{1}{2}\pi\lambda^3 q^4 C_s + \pi\lambda\Delta f \cdot q^2)$$

q = Spatial Frequency (In Fourier space or Reciprocal scape), corresponding distance in image plane is 1/q

Contrast transfer function

$$\sin \chi(k) = \sin(\pi\lambda\Delta f k^2 + 1/2\pi C s\lambda^3 k^4)$$

k: spatial frequency [1/nm]

parameters: $\lambda = 0.0025$ nm (200 kV), $c_s = 1.1$ mm, $\Delta f = -60$ nm

The CTF oscillates between -1 (negative contrast transfer) and +1 (positive contrast transfer). The exact locations of the zero crossings (where no contrast is transferred, and information is lost) depends on the defocus.

Scherzer defocus

Every zero-crossing of the graph corresponds to a contrast inversion in the image.

Up to the first zero-crossing k_0 the contrast does not change its sign.

The reciprocal value $1/k_0$ is called Point Resolution.

The defocus value which maximizes this point resolution is called the Scherzer defocus.

Optimum defocus: At Scherzer defocus, by choosing the right defocus value Δf one flattens $\chi(u)$ and creates a wide band where low spatial frequencies **k** are transferred into image intensity with a similar phase.

Optimum defocus (Scherzer Defocus)

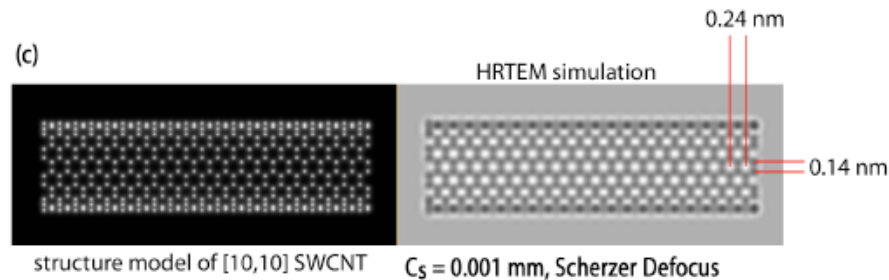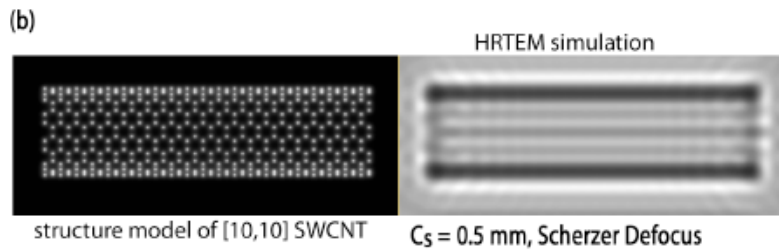$$\Delta f_{sch} = -1.2 \cdot \sqrt{(C_s \lambda)}$$

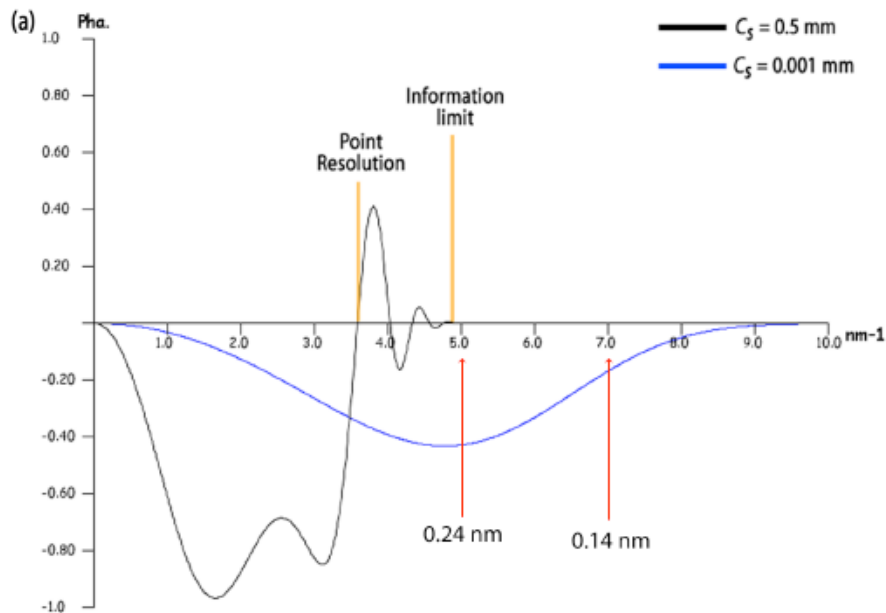**Working at Scherzer defocus ensures the transmission of a broad band of spatial frequencies with constant contrast and allows an unambiguous interpretation of the image.**

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Centre for Materials Science and Nanotechnology (SMN)

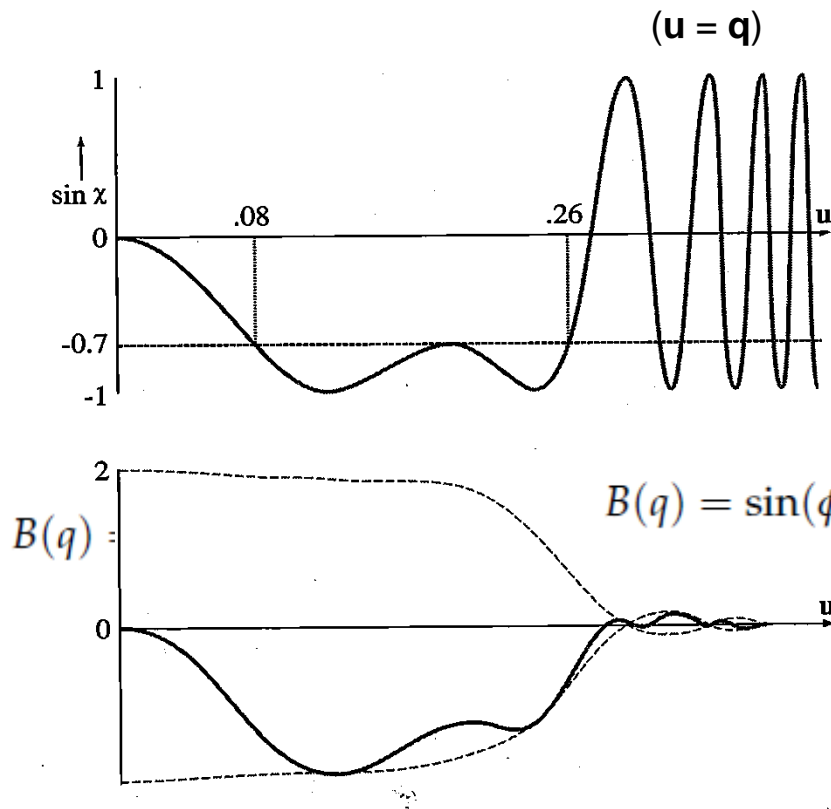# Point resolution

Optimum defocus (Scherzer Defocus)

$$\Delta f_{sch} = -1.2 \cdot \sqrt{(C_s \lambda)}$$

$$r_{sch} = 0.66 \cdot (C_s \lambda^3)^{\frac{1}{4}}$$

The Envelope functions

The resolution is also limited by the spatial coherence of the source and by chromatic effects (changes of electron energy in time):

(u = q)

Phase Contrast Transfer Function:

For Weak Phase Object Approximation:

$$B(q) = \sin(\phi(q)) = \sin(\frac{1}{2}\pi\lambda^3 q^4 C_s - \pi\lambda\Delta f \cdot q^2)$$

Envelope Functions

$$B(q) = \sin(\phi(q)) = \sin(\frac{1}{2}\pi\lambda^3 q^4 C_s - \pi\lambda\Delta f \cdot q^2) \cdot [E_s(q) \cdot E_t(q) \cdot E_i(q) \cdot A(q)]$$

Envelope functions related to incoherencies in electron beam dampen out the CTF

The envelope function imposes a "virtual aperture" in the back focal plane of the objective lens.
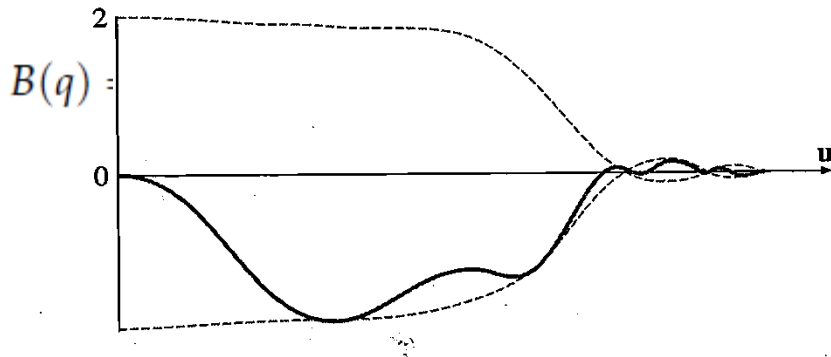
The Envelope functions

$$B(q) = \sin(\phi(q)) = \sin(\frac{1}{2}\pi\lambda^3 q^4 C_s - \pi\lambda\Delta f \cdot q^2) \cdot [E_s(q) \cdot E_t(q) \cdot E_i(q) \cdot A(q)]$$

E_s and E_t represent the envelope functions of spatial and temporal coherence, E_i the instrumental instabilities and A represents the effect of objective aperture size.

E_s is *spatial coherency envelope* (caused by the finite incident beam convergence, i.e., the beam is not fully parallel)

E_t is the *temporal coherency envelope* (caused by chromatic aberrations, focal and energy spread, instabilities in the high tension and objective lens current)

Envelope functions related to incoherencies in electron beam dampen out the CTF

The envelope function imposes a "virtual aperture" in the back focal plane of the objective lens.

Phase contrast and information limit

Point Resolution (or Point-to-Point, or Directly Interpretable Resolution) of a microscope corresponds to the to the point when the CTF first crosses the k-axis:

$$k = 0.67 C^{1/4} \lambda^{3/4}$$

Phase contrast images are directly interpretable only up to the point resolution (Scherzer resolution limit). If the information limit is beyond the point resolution limit, one needs to use image simulation software to interpret any detail beyond point resolution limit.

Information limit goes well beyond point resolution limit for FEG microscopes (due to high spatial and temporal coherency).

For the microscopes with thermionic electron sources (LaB6 and W), the info limit usually coincides with the point resolution.
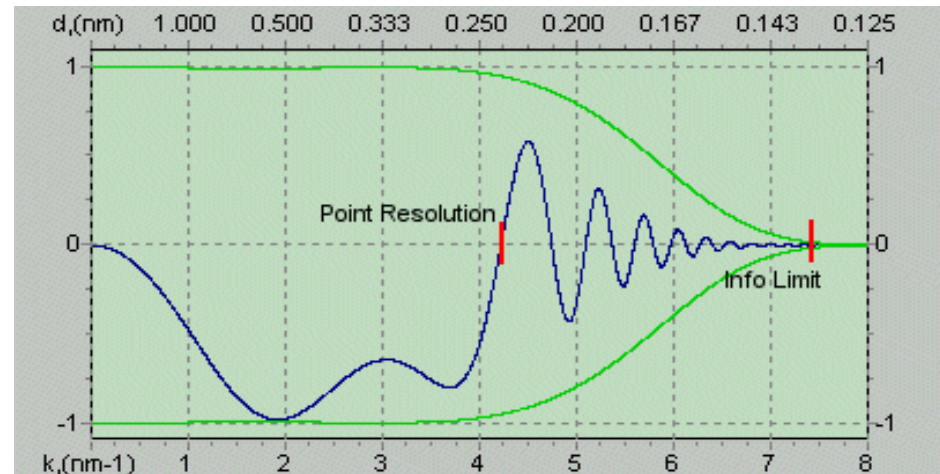
http://www.maxsidorov.com/ctfexplorer/webhelp/effect_of_defocus.htm

Important points to notice

- CTF is oscillatory: there are "passbands" where it is NOT equal to zero (good "transmittance") and there are "gaps" where it IS equal (or very close to) zero (no "transmittance").

- When it is negative, positive phase contrast occurs, meaning that atoms will appear dark on a bright background.

- When it is positive, negative phase contrast occurs, meaning that atoms will appear bright on a dark background.

- When it is equal to zero, there is no contrast (information transfer) for this spatial frequency.

- At Scherzer defocus CTF starts at 0 and decreases, then

- CTF stays almost constant and close to -1 (providing a broad band of good transmittance), then

- CTF starts to increase, and

- CTF crosses the u-axis, and then

- CTF repeatedly crosses the u-axis as u increases.

- CTF can continue forever but, in reality, it is modified by envelope functions and eventually dies off.

Ray Diagrams

University of Oslo
Centre for Materials Science and Nanotechnology (SMN)

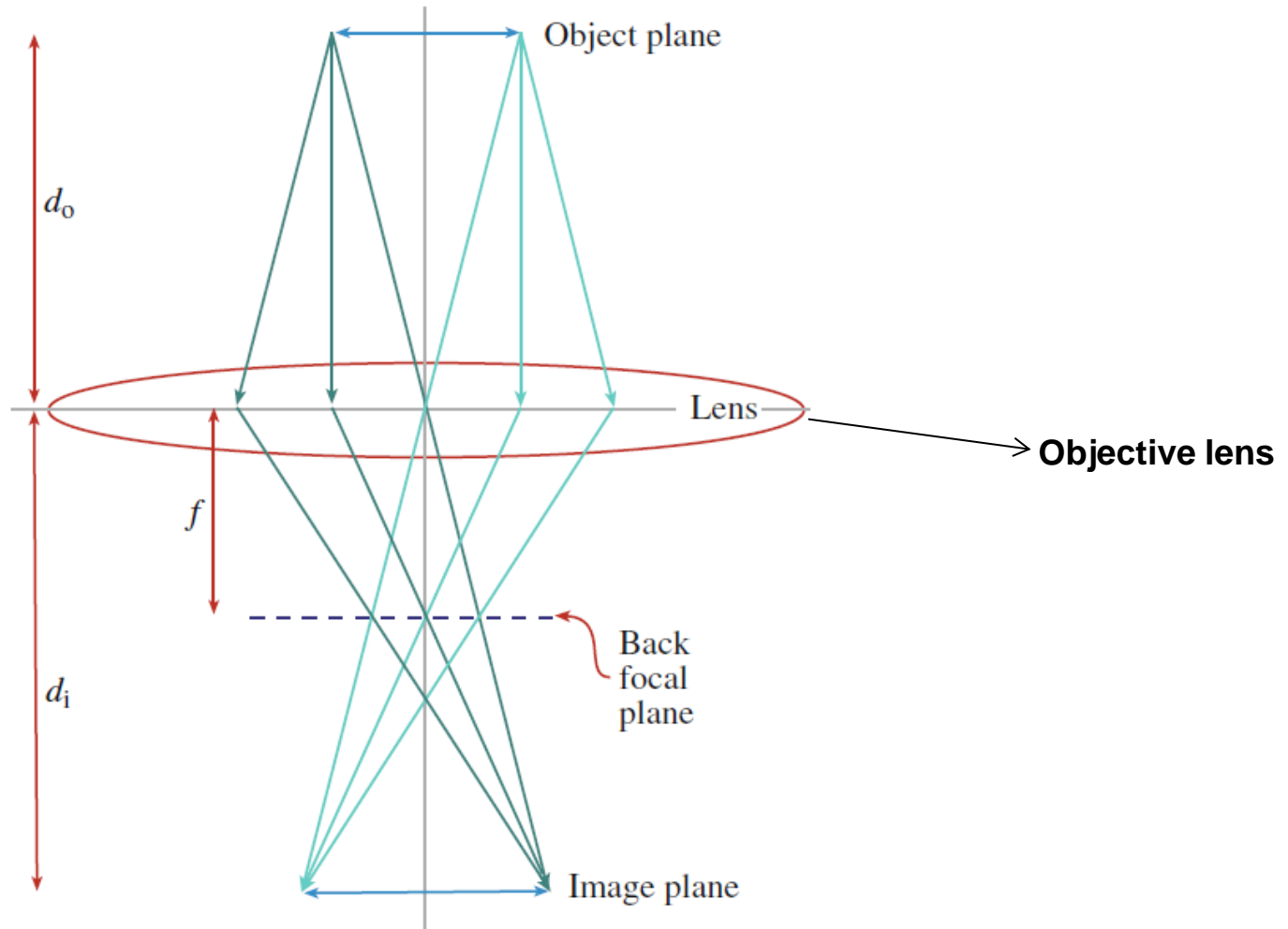# Simplified ray diagram of conventional TEM – Practise for Exam

FIGURE 6.3. A complete ray diagram for a finite object, symmetrically positioned around the optic axis. All rays emerging from a point in the object (distance d_o from the lens) that are gathered by the lens converge to a point in the image (distance d_i from the lens) and all parallel rays coming from the object are focused in the focal plane (distance f from the lens).
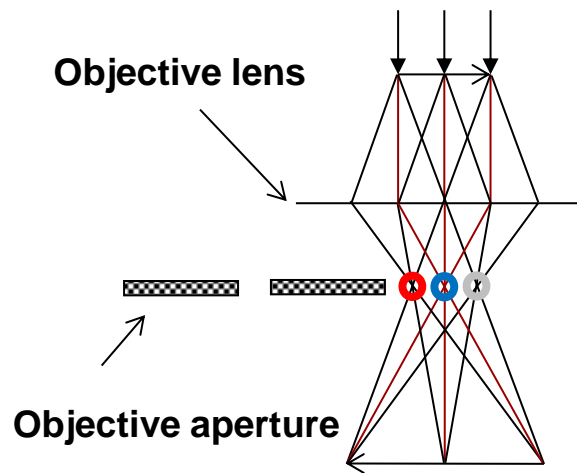
Courtesy: William & Carter TEM Text Book

Simplified ray diagram of TEM Diffraction and Imaging modes – Practise for Exam
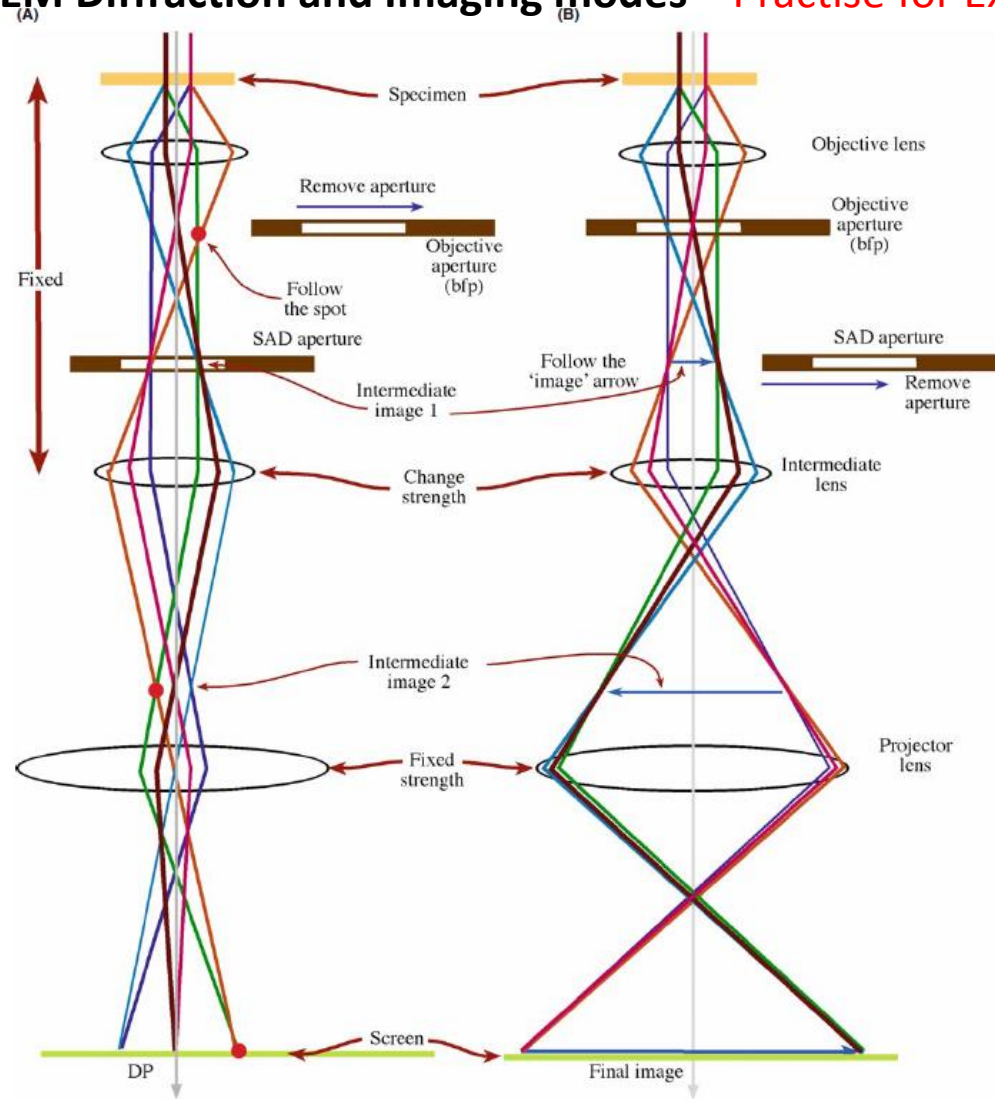

FIGURE 9.12. The two basic operations of the TEM imaging system involve (A) diffraction mode: projecting the DP onto the viewing screen and (B) image mode: projecting the image onto the screen. In each case the intermediate lens selects either the BFP (A) or the image plane (B) of the objective lens as its object. The imaging systems shown here are highly simplified. Most TEMs have many more imaging lenses, which give greater flexibility in terms of magnification and focusing range for both images and DPs. The SAD and objective diaphragms are also shown appropriately inserted or retracted. NOTE: This is a highly simplified diagram showing only three lenses. Modern TEM columns have many more lenses in their imaging systems.

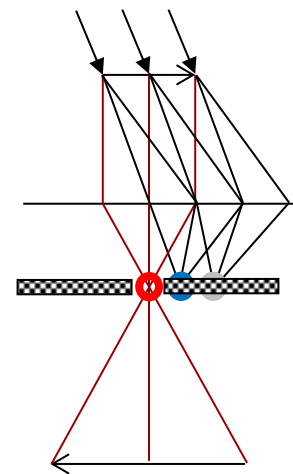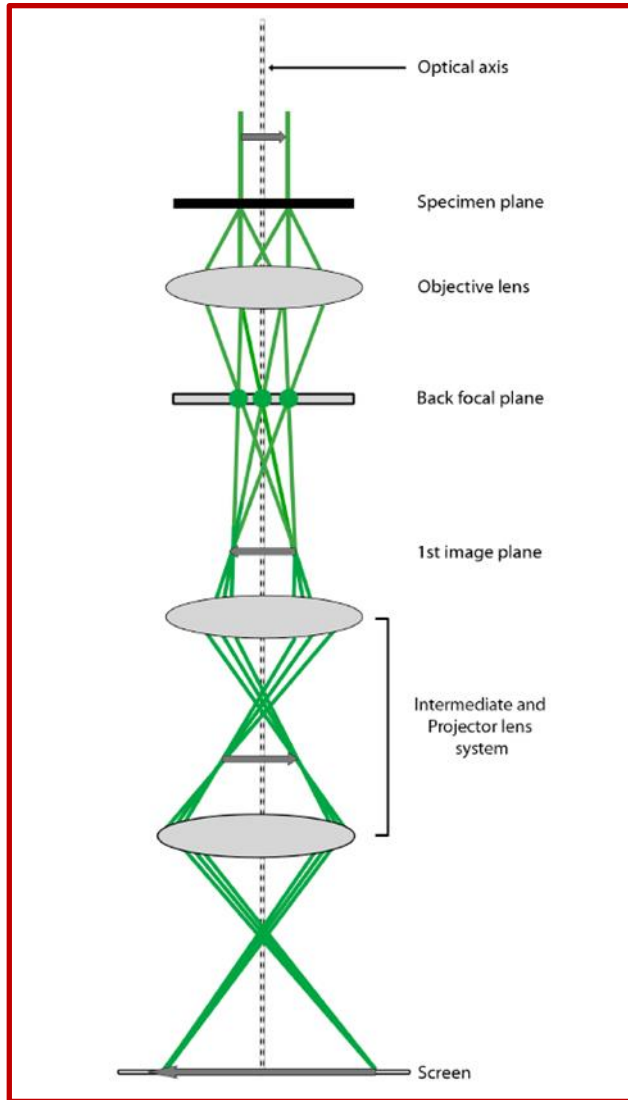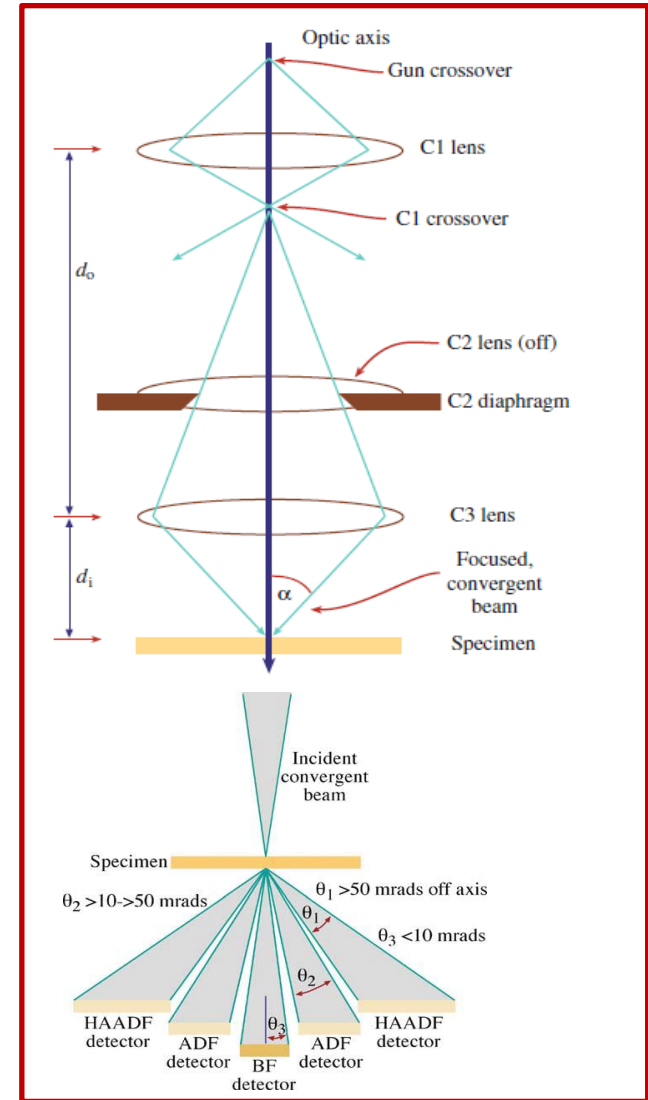*Courtesy: William & Carter TEM Text Book*

Bright Field TEM image

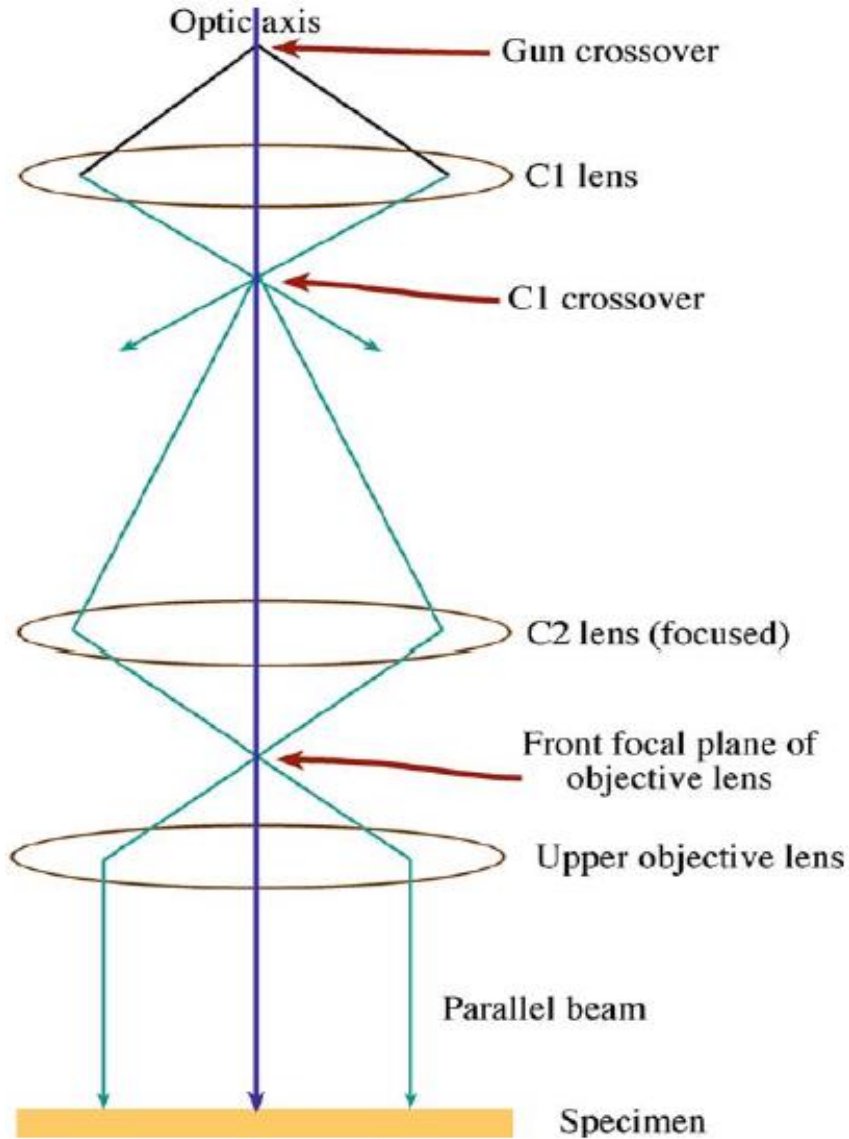**Dark Field TEM image**

Objective lens
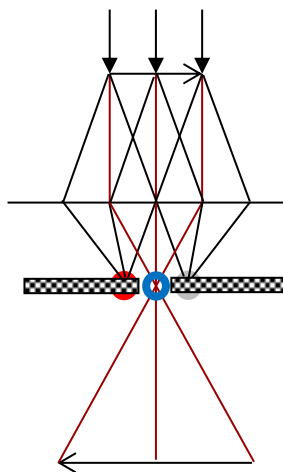
Objective aperture

Simplified ray diagram of conventional TEM

Simplified ray diagram of conventional STEM

TEM imaging – Illumination optics path

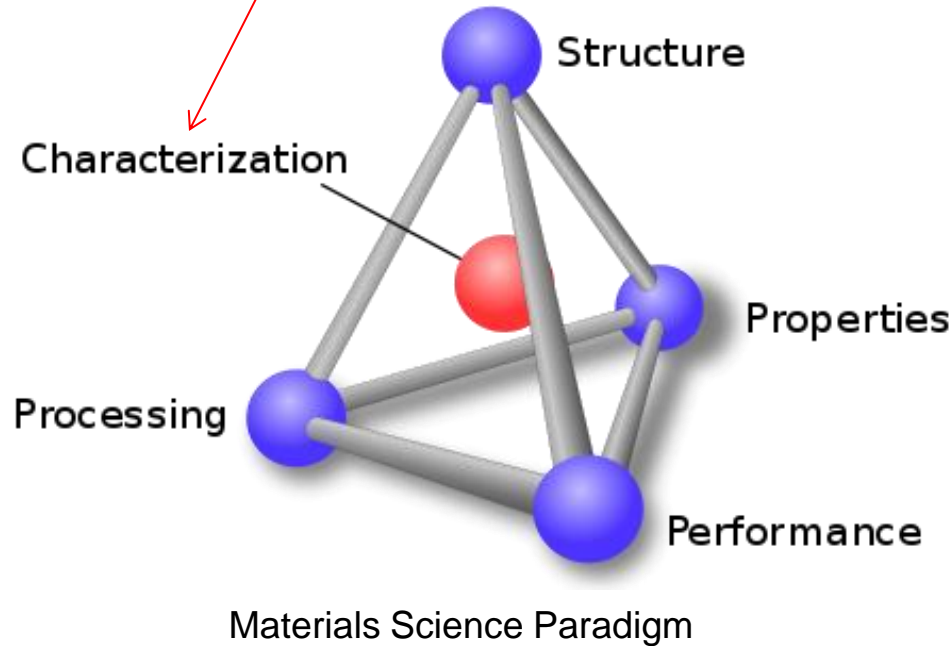Optic axis — Gun crossover

C1 lens

C1 crossover

C2 lens (focused)

Front focal plane of objective lens

Upper objective lens

Parallel beam

Specimen

STEM imaging– Illumination optics path

Optic axis — Gun crossover

C1 lens

C1 crossover

d_o

C2 lens (off)

C2 diaphragm

C3 lens

Focused, convergent beam

α

d_i

Specimen

Courtesy: William & Carter TEM Text Book

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Centre for Materials Science and Nanotechnology (SMN)
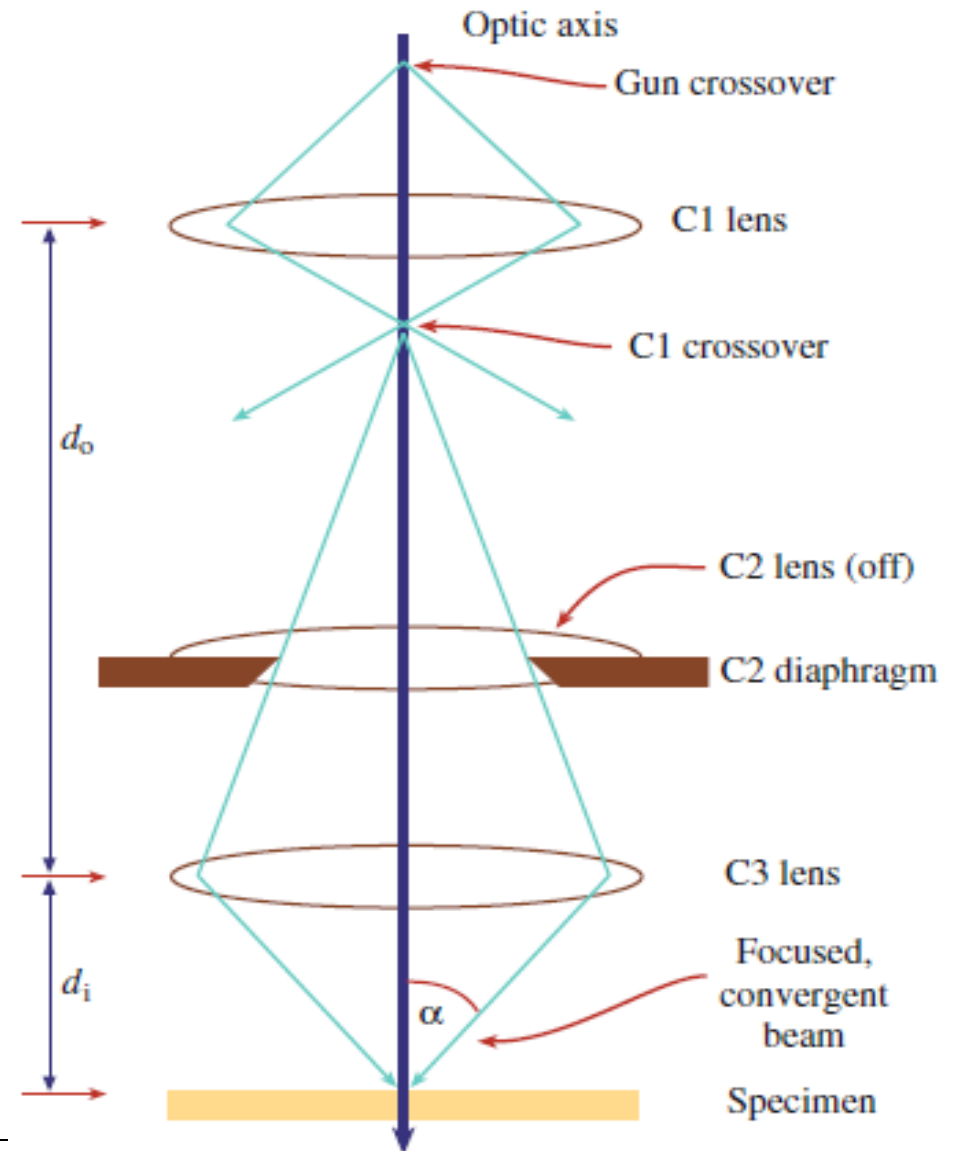
Role of TEM in Materials Science Research and Development

Materials Science Paradigm

Solving Materials Science problems/mysteries by probing analytically and understanding structure-property relationships at atomic scale level

Courtesy: www.wikipedia.com

Thank you!

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