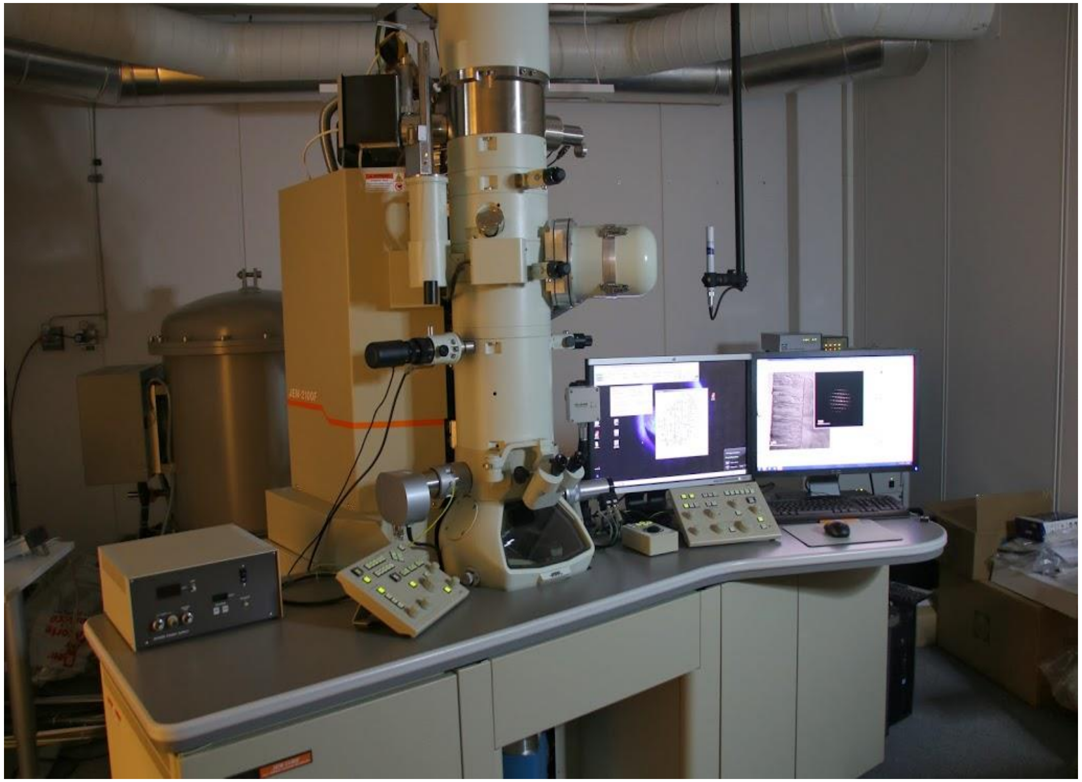
Transmissions Electron Microscopy (TEM)



Basic principles

Diffraction

Imaging

Specimen preparation

TEM is based on three possible set of techniqes

Diffraction

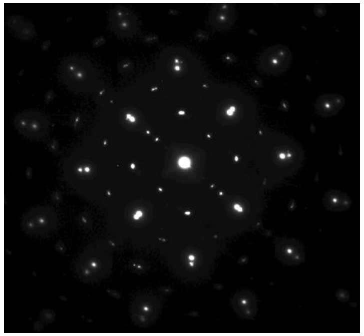
From regions down to a few nm (CBED).

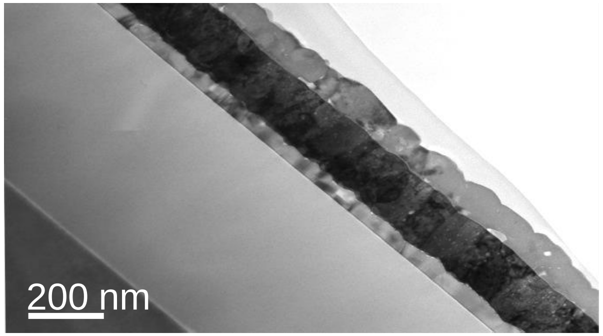
Imaging

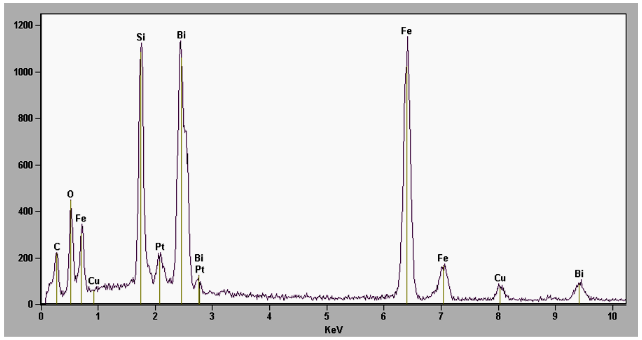
With spatial resolution down to the atomic level (HREM and STEM)

Spectroscopy

Chemistry and elecronic states (EDS and EELS). Spatial and energy resolution down to the atomic level and ~0.1 eV.

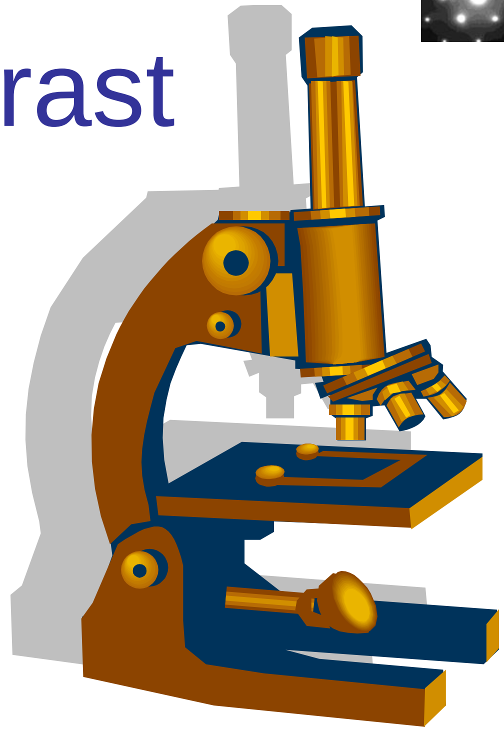






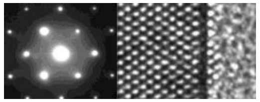
Electrons interacts 100-1000 times stronger with matter than X-rays

Imaging and contrast



Resolution of the eyes:~ 0.1-0.2 mm
Resolution in a visible light microscope: ~300 nm

Modern TEMs with Cs correctors have sub Å resolution!



Introduction

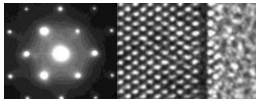
EM and materials

The interesting objects for TEM is not the average structure or homogenous materials but local structure and inhomogeneities

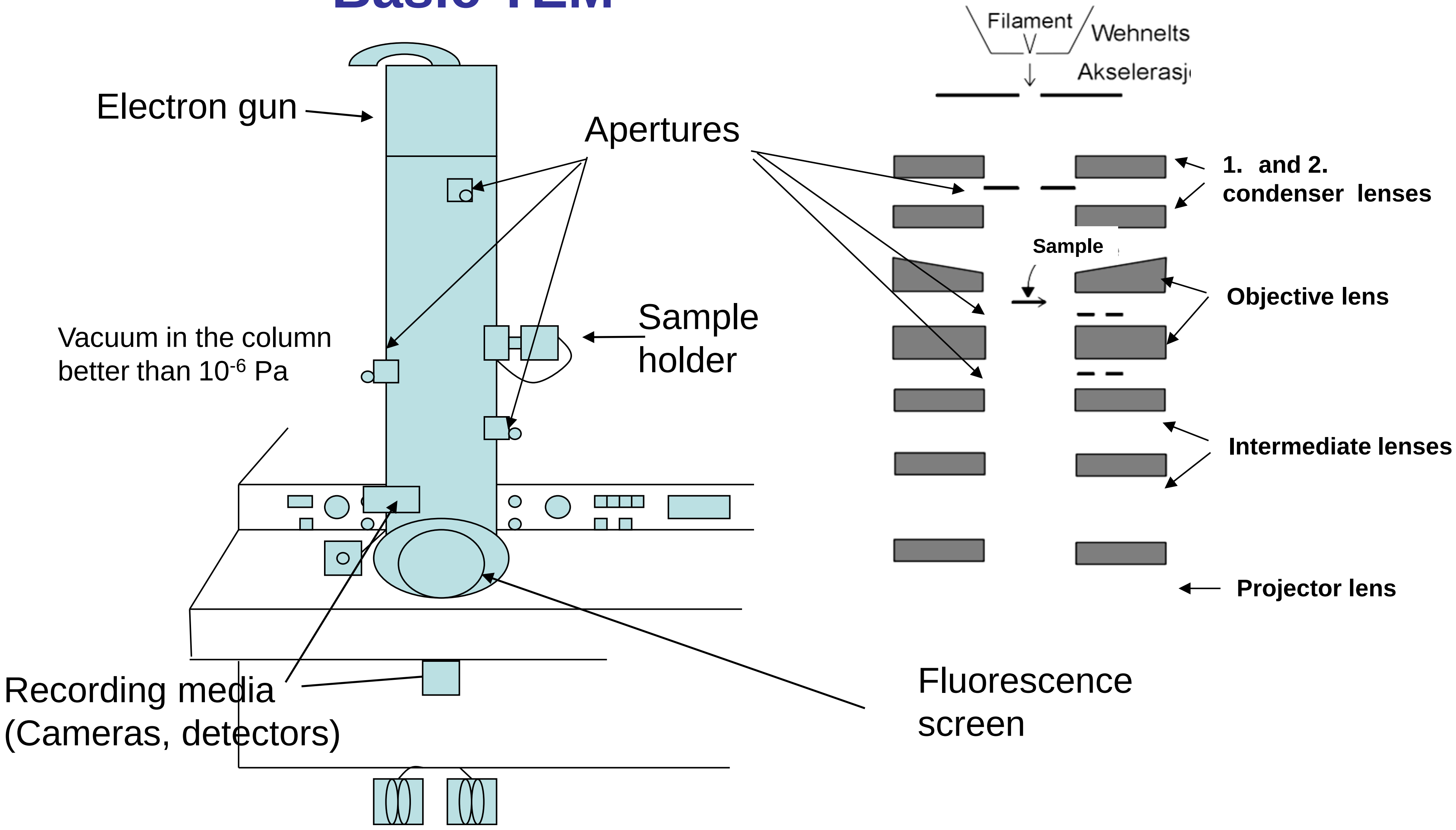
Defects

Interfaces

Precipitates



Basic TEM



Electron gun

Apertures

Vacuum in the column better than 10^{-6} Pa

Sample holder

Recording media (Cameras, detectors)

Fluorescence screen

1. and 2. condenser lenses

Sample

Objective lens

Intermediate lenses

Projector lens

Similar components as a transmission light microscope

The electron source

- **Two types of emission guns:**
  - **Thermionic emission**
    - W or LaB_6

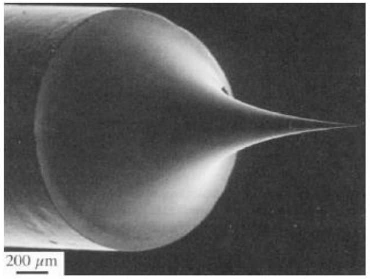
  - **Field emission**

Cold FEG

W

Schottky FEG

ZrO/W



Thermionic guns

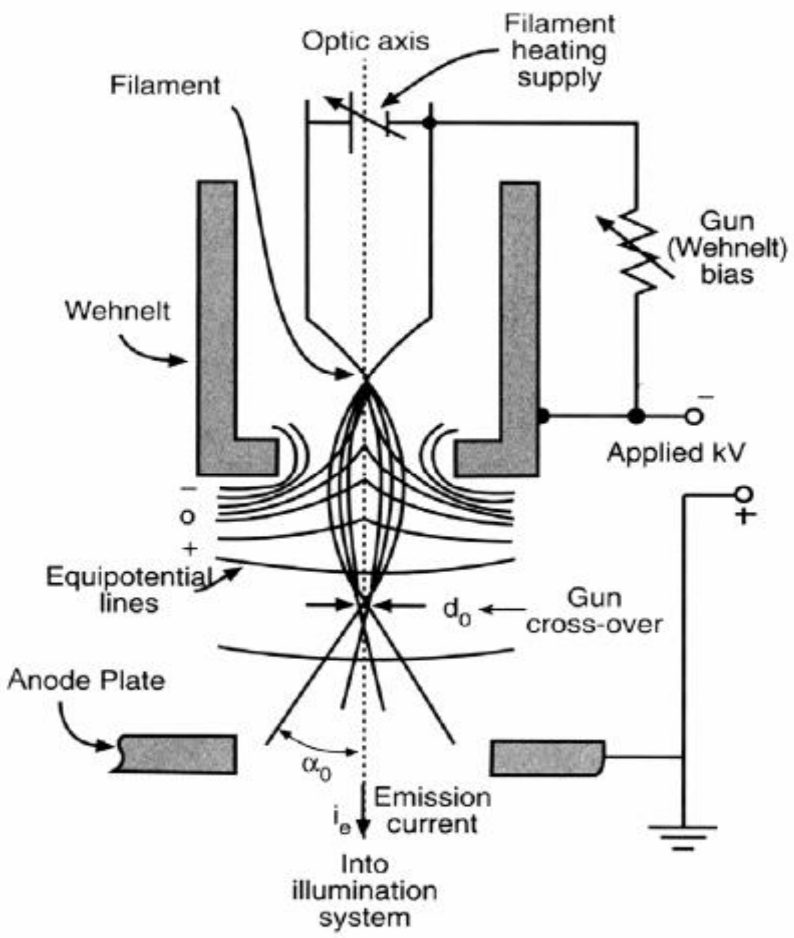
Filament heated to give thermionic emission

-Directly (W) or
 indirectly (LaB_6)

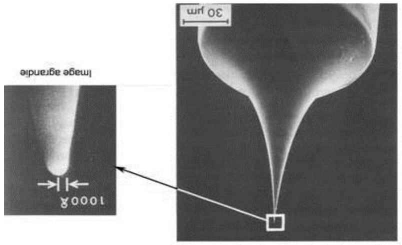
Filament negative potential to ground

Wehnelt produces a small negative bias

-Brings electrons to
 cross over



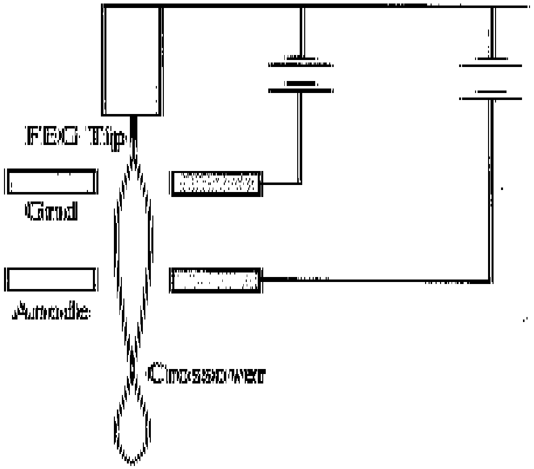
Field emission gun



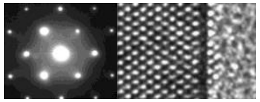
- The principle:
  - The strength of an electric field E is considerably increased at sharp points.

$$E=V/r$$

- $r_W < 0.1$ μm, V=1 kV → $E = 10^{10}$ V/m
  - Lowers the work-function barrier so that electrons can tunnel out of the tungsten.



- Surface has to be pristine (no contamination or oxide)
  - Ultra high vacuum condition (Cold FEG) or poorer vacuum if tip is heated (”thermal” FE; ZrO surface tratments → Schottky emitters).



Resolution

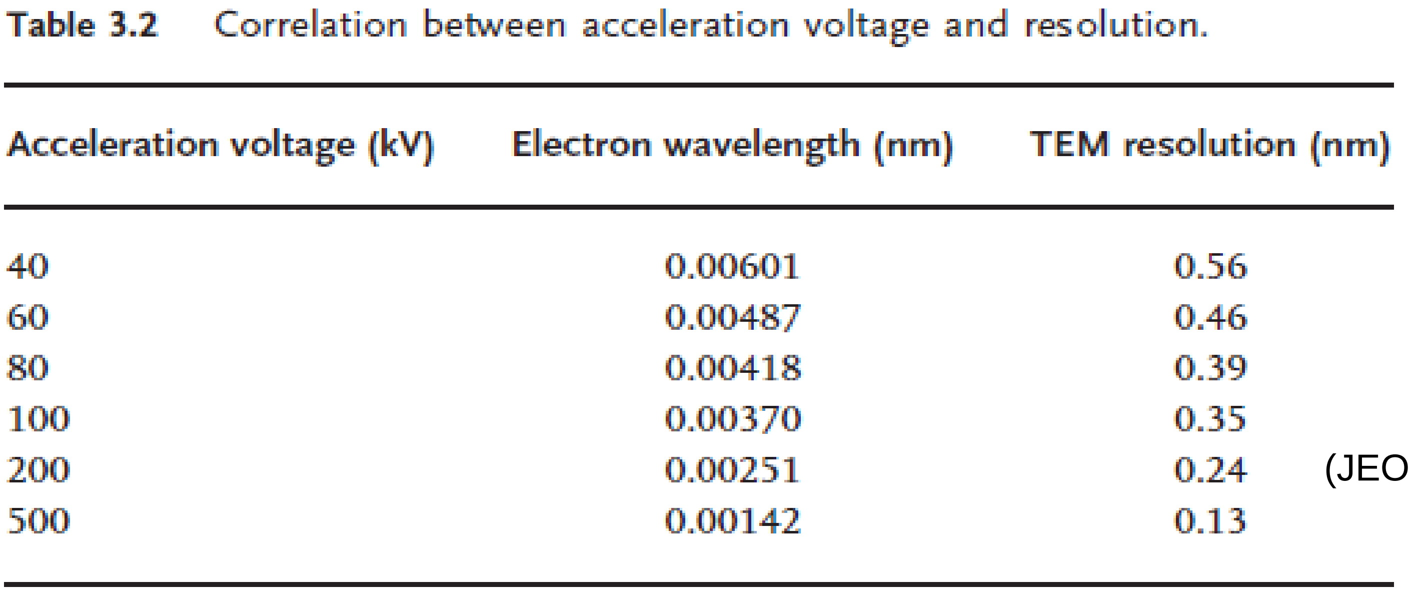


Table 3.2 Correlation between acceleration voltage and resolution.

Acceleration voltage (kV)	Electron wavelength (nm)	TEM resolution (nm)
40	0.00601	0.56
60	0.00487	0.46
80	0.00418	0.39
100	0.00370	0.35
200	0.00251	0.24
500	0.00142	0.13

(JEOL2010F: 0.19 nm)

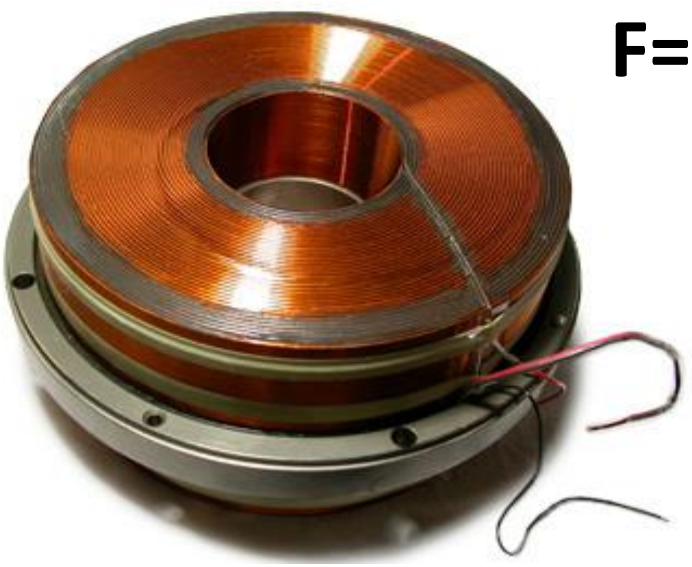
$$\lambda = \frac{h}{[2m_0 eV(1+\frac{eV}{2m_0c^2})]^{1/2}}$$

The point resolution in a TEM is limited by the aberrations of the lenses.

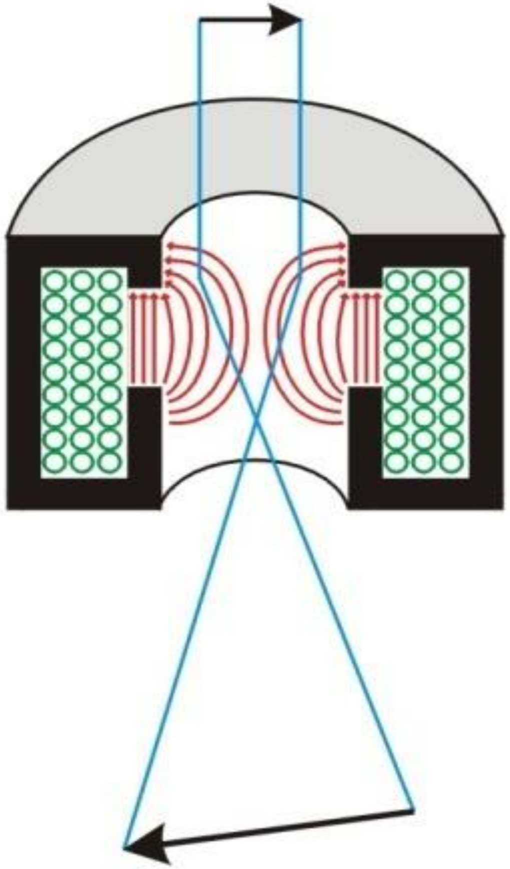
-Spherical
- Chromatic
-Astigmatism

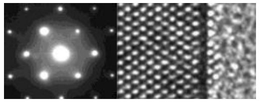
Electromagnetic lenses

A charged particle such as an electron, is deflected by a magnetic field. The direction and magnitude of the force **F**, on the electron is given by the vector equation:

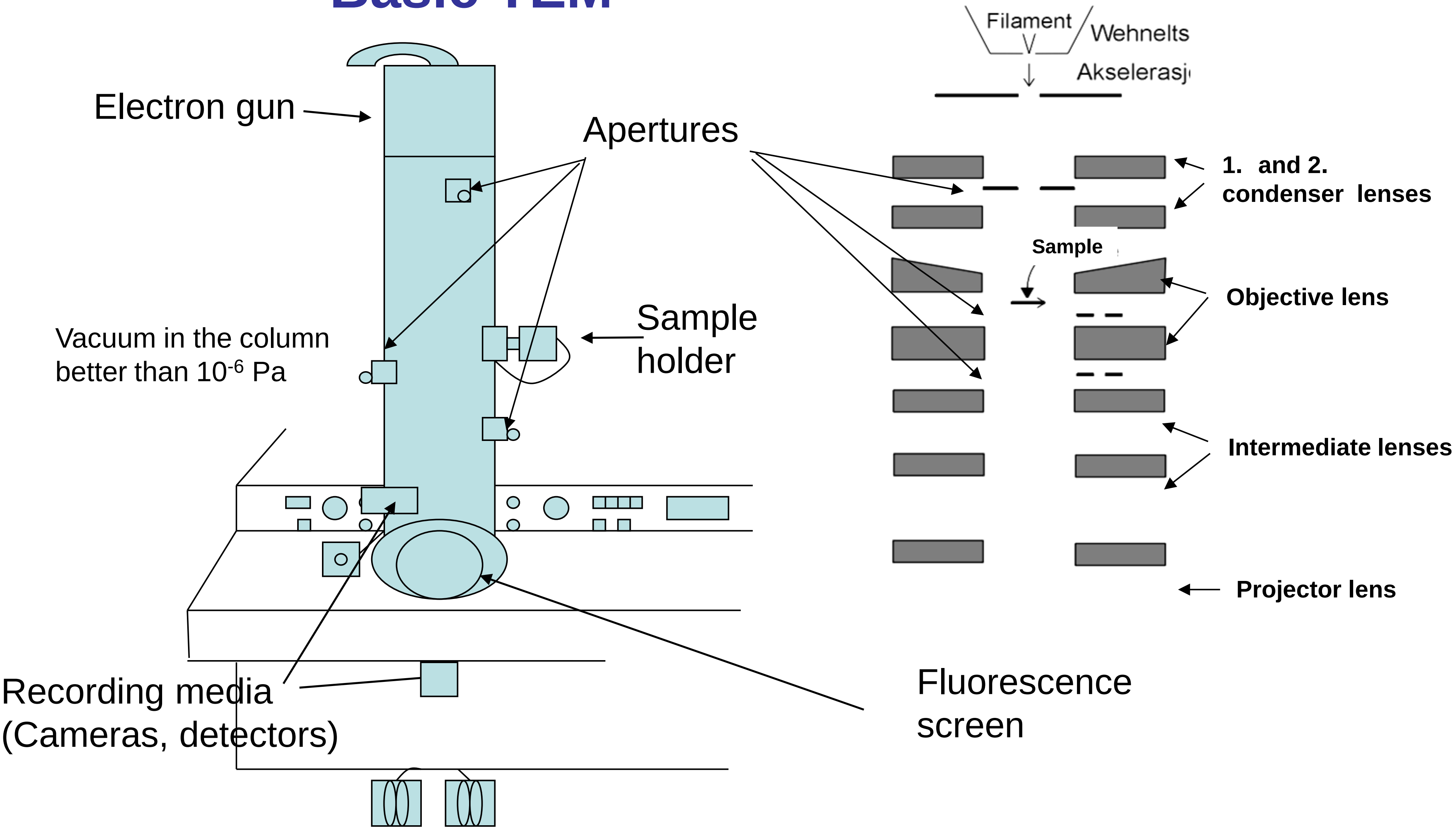


$$F = -e(v \times B)$$





Basic TEM



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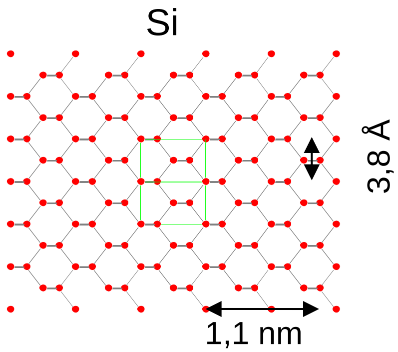
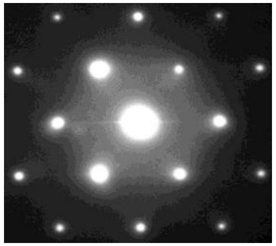
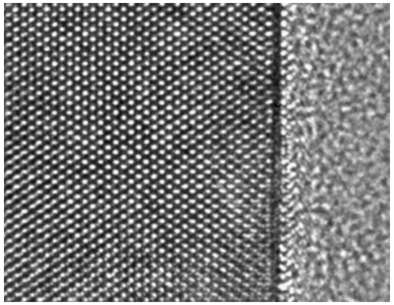
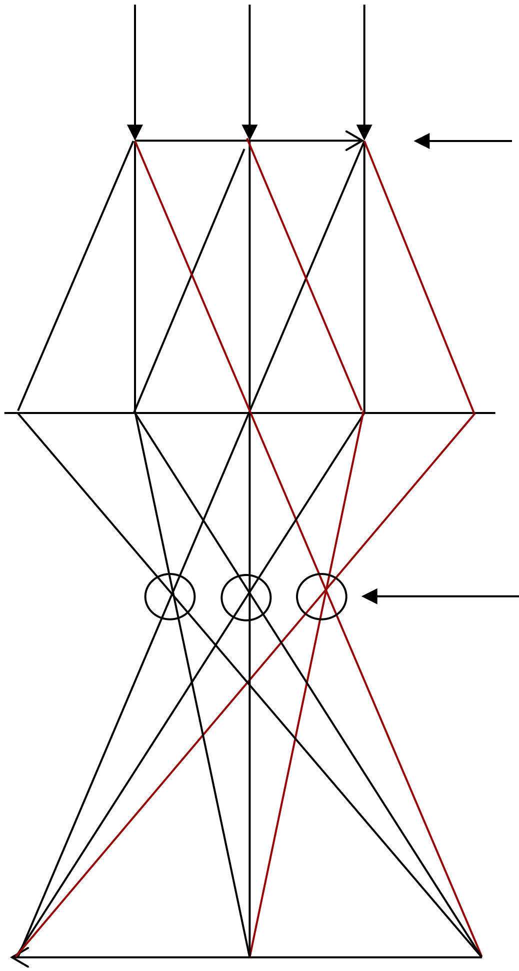
Simplified ray diagram







Parallel incoming electron beam



Sample

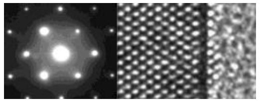
Objective lense

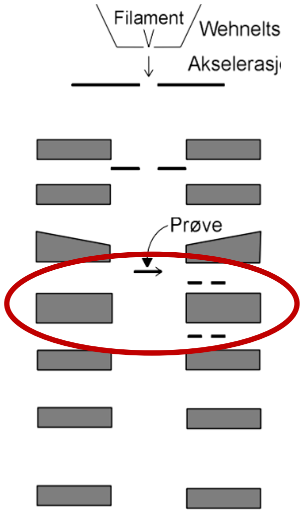
Diffraction plane (back focal plane)

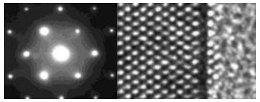
Objective aperture

Image plane

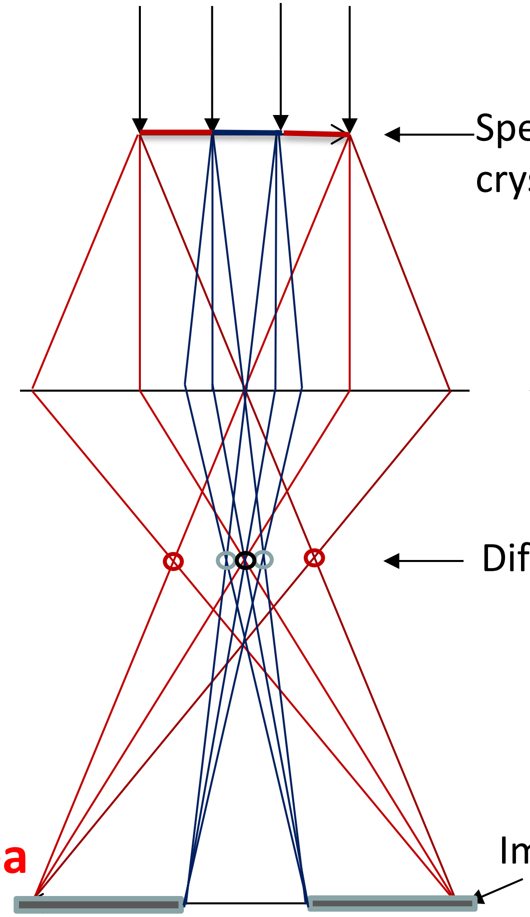
Selected area aperture







Selected area diffraction



Parallel incoming electron beam

Specimen with two crystals (red and blue)

Objective lense

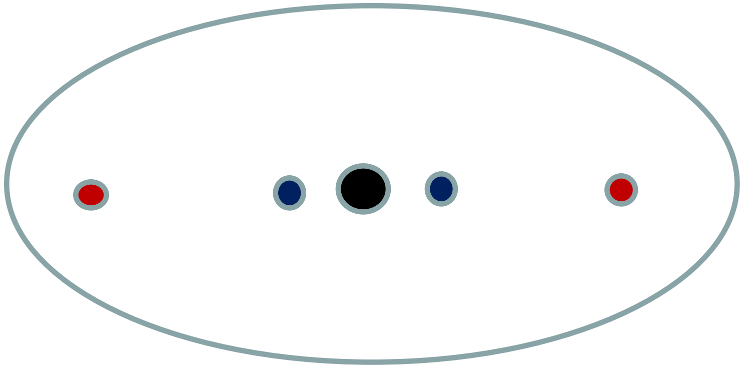
Diffraction pattern

Image plane

Selected area aperture

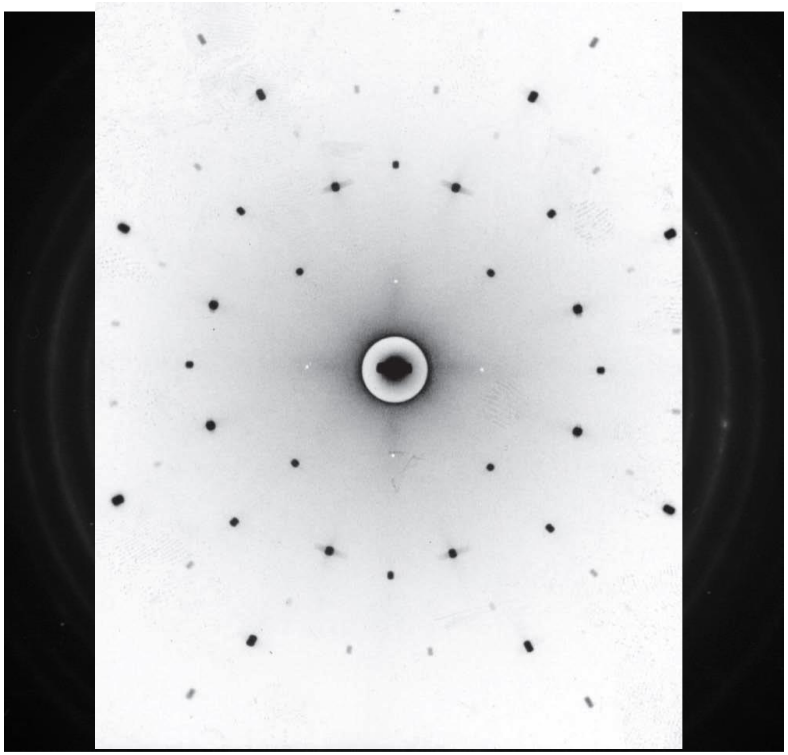
- Diffraction from a single crystal in a polycrystalline sample if the aperture is small enough/crystal large enough.
- Orientation relationships can be determined.
- ~2% accuracy of lattice parameters
  - XRD is much more accurate

Pattern on the screen

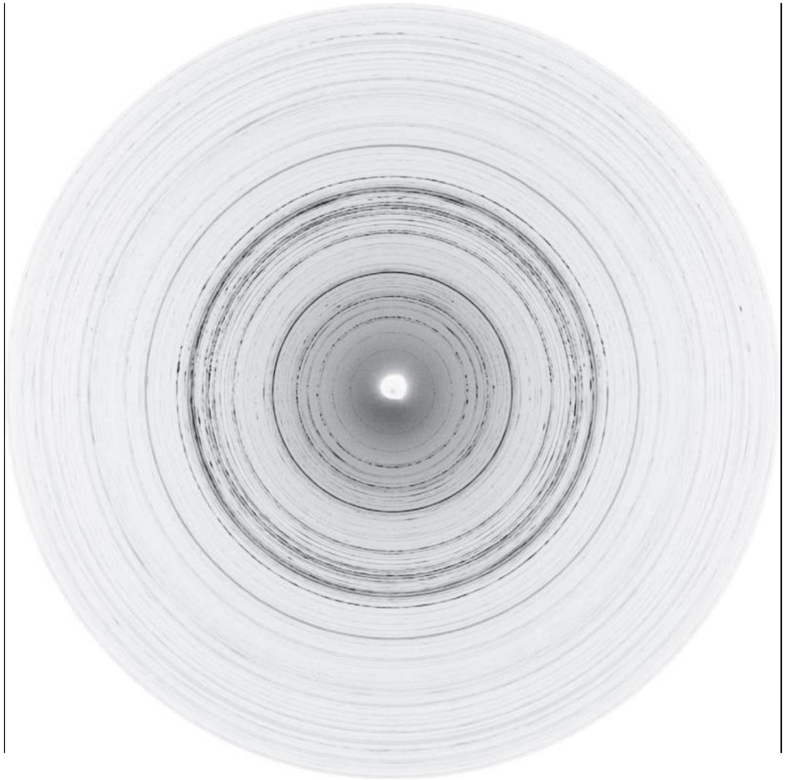


Diffraction with large SAD aperture, ring and spot patterns

Poly crystalline sample

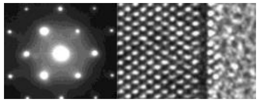


Four epitaxial phases



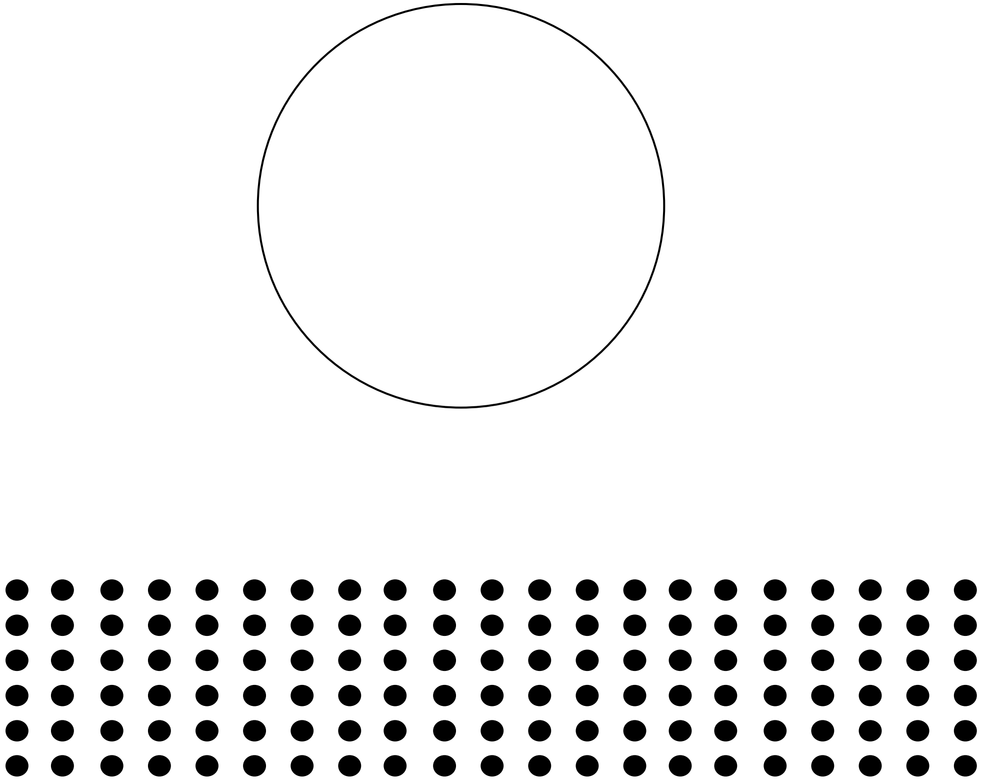
Similar to XRD from polycrystalline samples.

The orientation relationship between the phases can be determined with ED.



Why do we observe many reflections in one diffraction pattern?

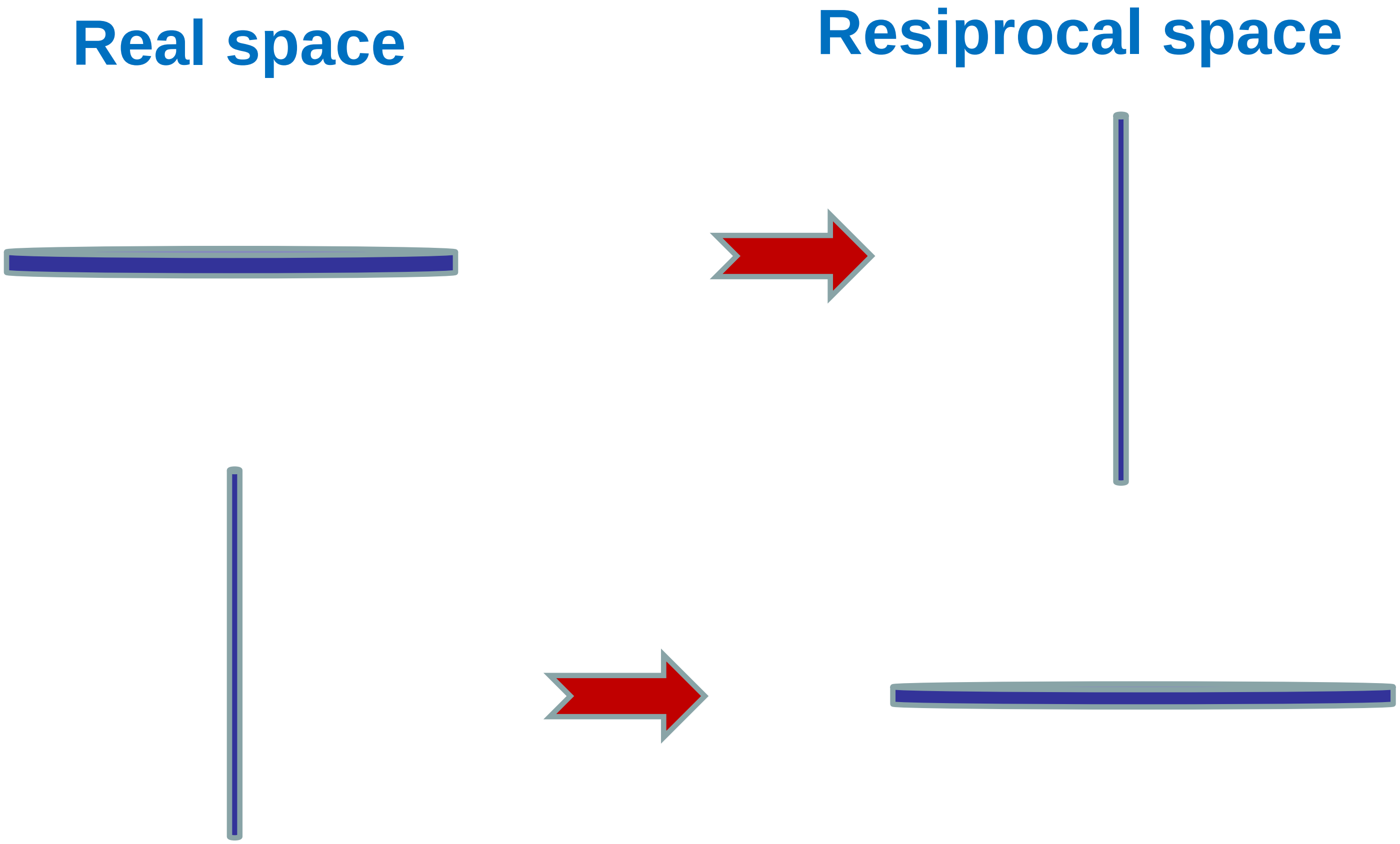
The Ewald Sphere is flat (almost)



Cu K_{alpha} X-ray: λ = 150 pm => small k

Electrons at 200 kV: λ = 2.5 pm => large k

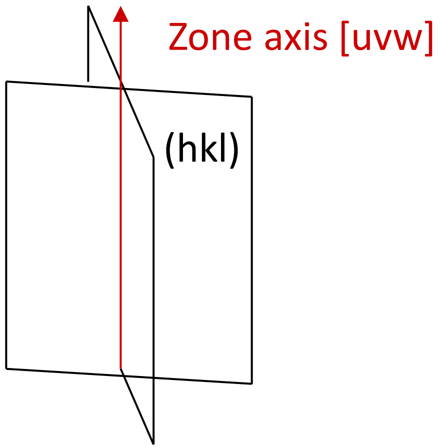
ED and form effects



Real space

Resiprocal space

Zone axis and Laue zones



$uh+vk+wl= 0$

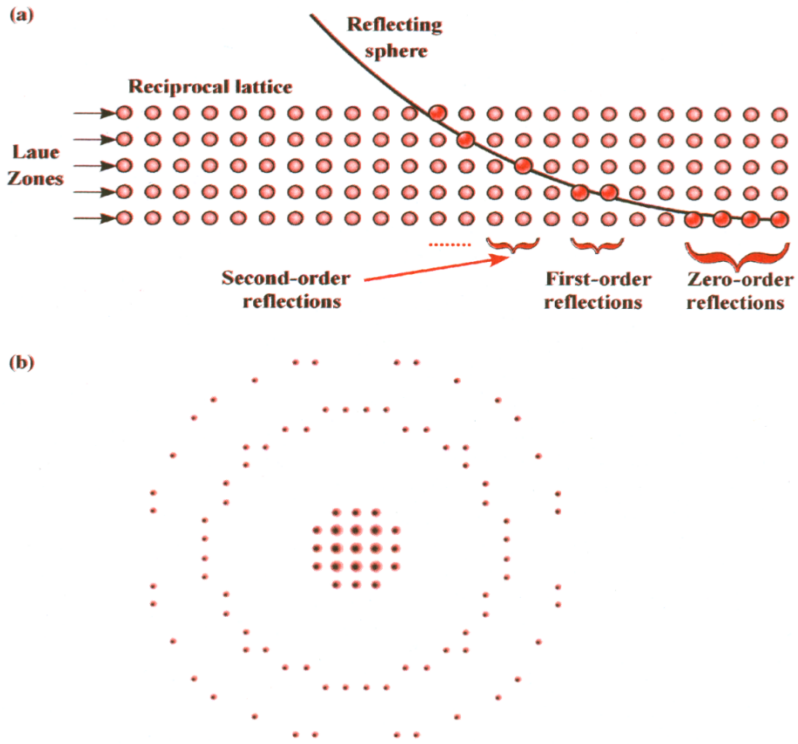
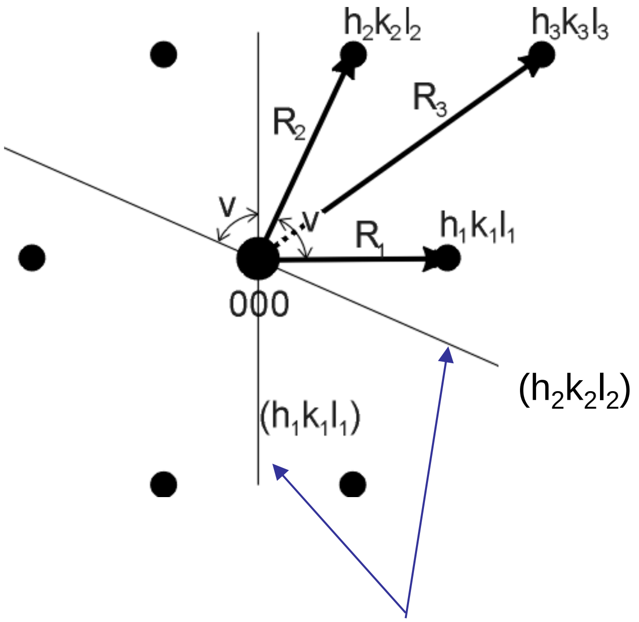


Figure 2.35 Single crystal electron diffraction from more than one Laue zone: (a) mechanism of formation; (b) a diffraction pattern from a [100] oriented aluminium single crystal film.

Indexing diffraction patterns

The **g** vector to a reflection is normal to the corresponding (h k l) plane and IgI=1/$d_{nh\ nk\ nl}$

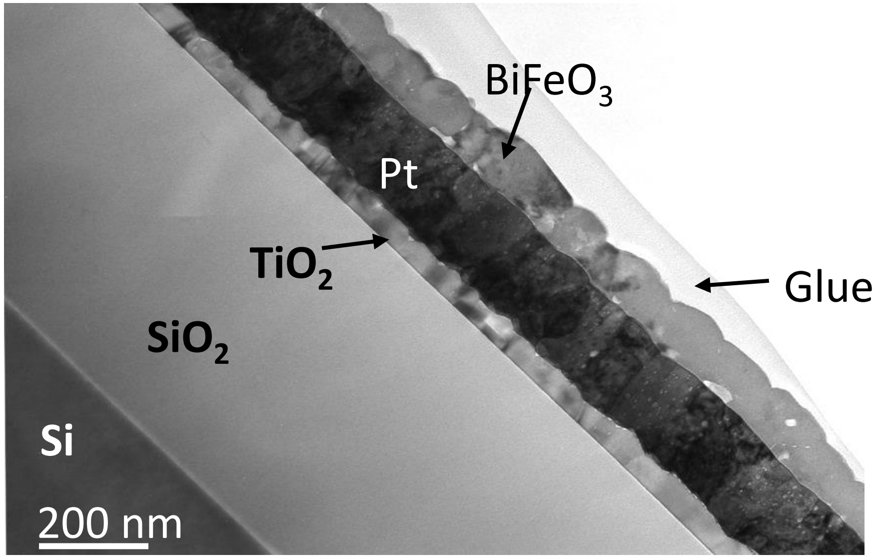


Orientations of corresponding planes in the real space

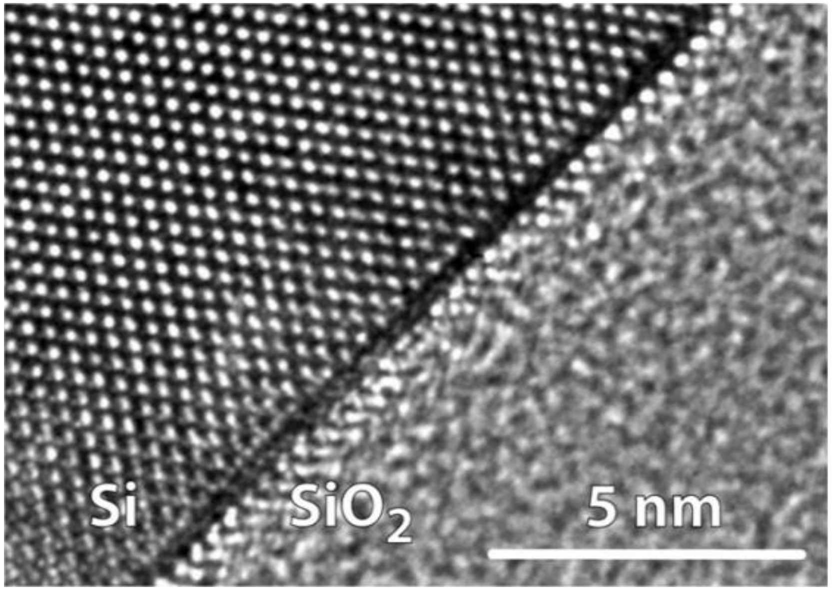
- Measure R_i and the angles between the reflections
- Calculate d_i , i=1,2,3 (=K/R_i)
- Compare with tabulated/theoretical calculated d-values of possible phases
- Compare R_i/R_j with tabulated values for cubic structure.
- $\mathbf{g}_{1,hkl}+\mathbf{g}_{2,hkl}=\mathbf{g}_{3,hkl}$ (vector sum must be ok)
- Perpendicular vectors: $\mathbf{g}_i \bullet \mathbf{g}_j = 0$
- Zone axis: $\mathbf{g}_i \times \mathbf{g}_j = [HKL]_z$
- All indexed **g** must satisfy: $\mathbf{g} \bullet [HKL]_z=0$

Imaging / microscopy

Amplitude contrast



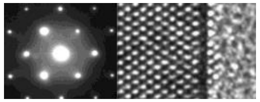
Phase contrast



The elctron wave can change both its amplitude and phase as it traverses the specimen

Give rise to contrast

We select imaging conditions so that one of them dominates.



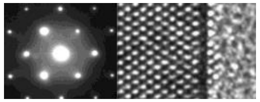
Contrast

- Difference in intensity of to adjacent areas:

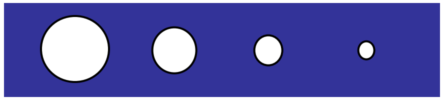
$$C = \frac{(I_2 - I_1)}{I_1} = \frac{\Delta I}{I_1}$$

The eyes can not see intensity chanes that is less then 5-10%, however, contrast in images can be enhanced digitally.

NB! It is correct to talk about strong and week contrast but not bright and dark contrast



Use of apertures



Condenser aperture:

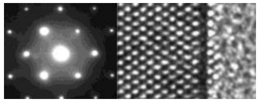
Limits the number of electrons reaching the specimen (reducing the intensity),
Affecting the convergent of the electron beam.

Selected area aperture:

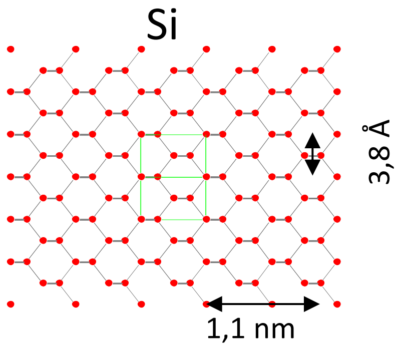
Allows only electrons going through an area on the sample that is limited by the SAD aperture to contribute to the diffraction pattern (SAD pattern).

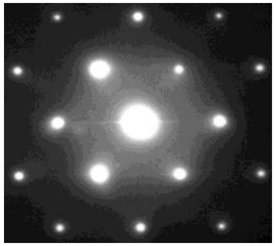
Objective aperture:

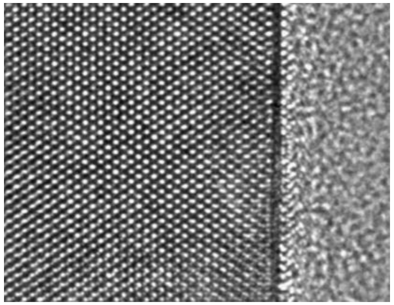
Allows certain reflections to contribute to the image. Increases the contrast in the image.
Bright field imaging (central beam, 000), Dark field imaging (one reflection, **g**), High resolution Images (several reflections from a zone axis).

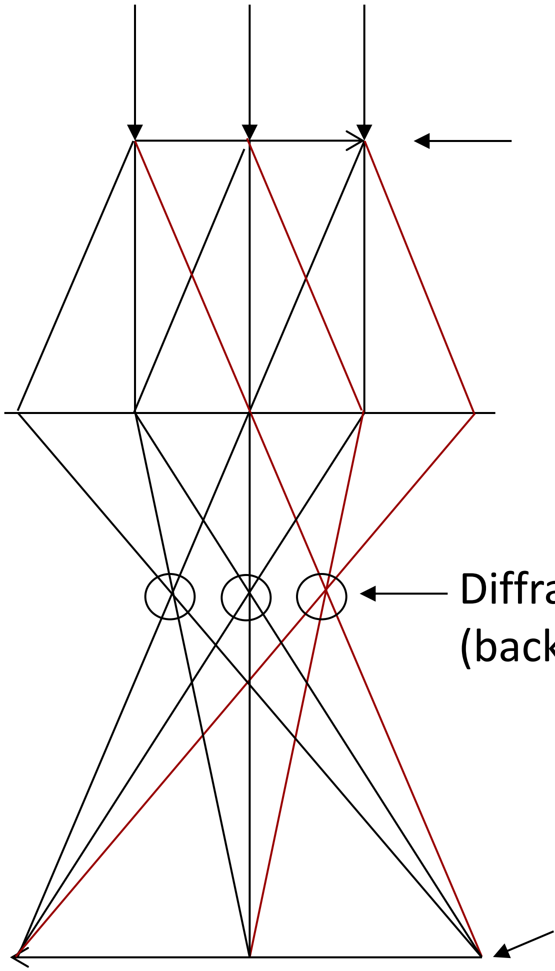


Simplified ray diagram









Parallel incoming electron beam

Sample

Objective lense

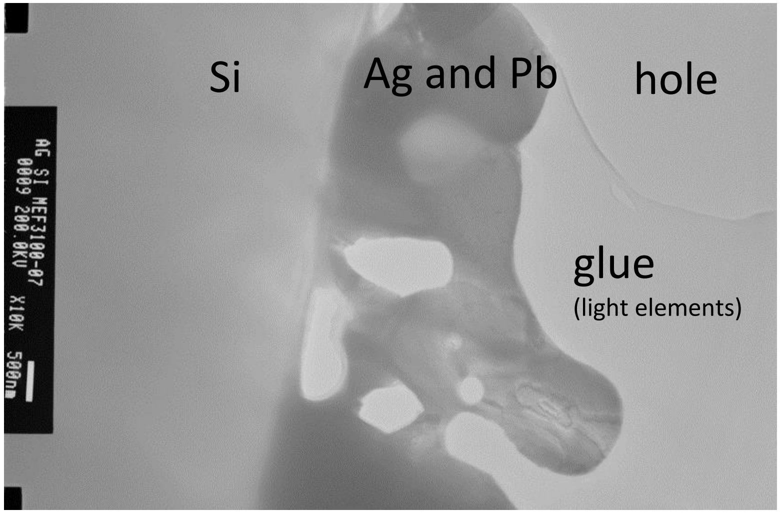
Diffraction plane
(back focal plane)

Objective aperture

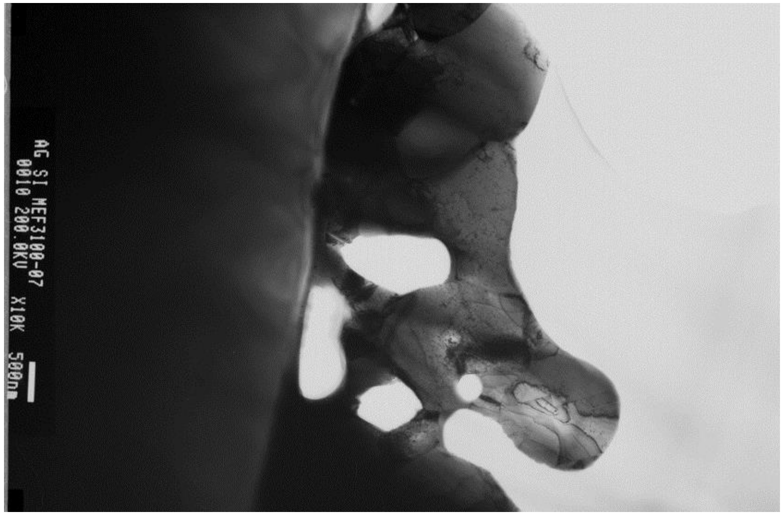
Image plane

Selected area aperture

Objective aperture: Contrast enhancement



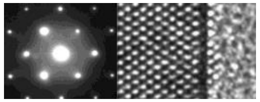
No aperture used



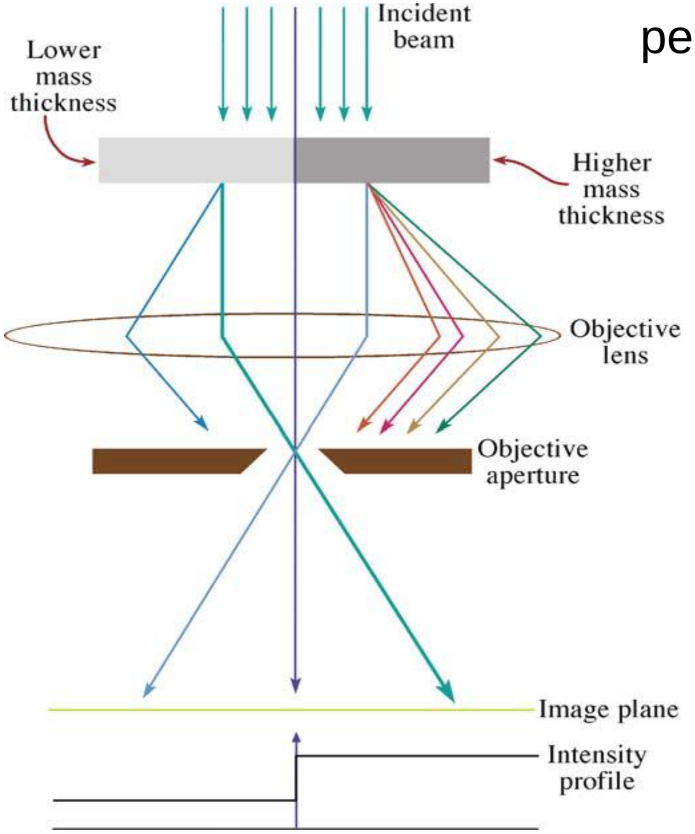
Central beam selected

Amplitude contrast:

Mass-Density contrast and Diffraction contrast



Mass-Density contrast in TEM



Williams and Carter, TEM, Part 3
Springer 2009

Incoherent elastic scattering (Rutherford scattering): peaked in the forward direction, t and Z-dependent

Areas of greater Z and/or t scatter electrons more strongly (in total).

TEM variables that affect the contrast:

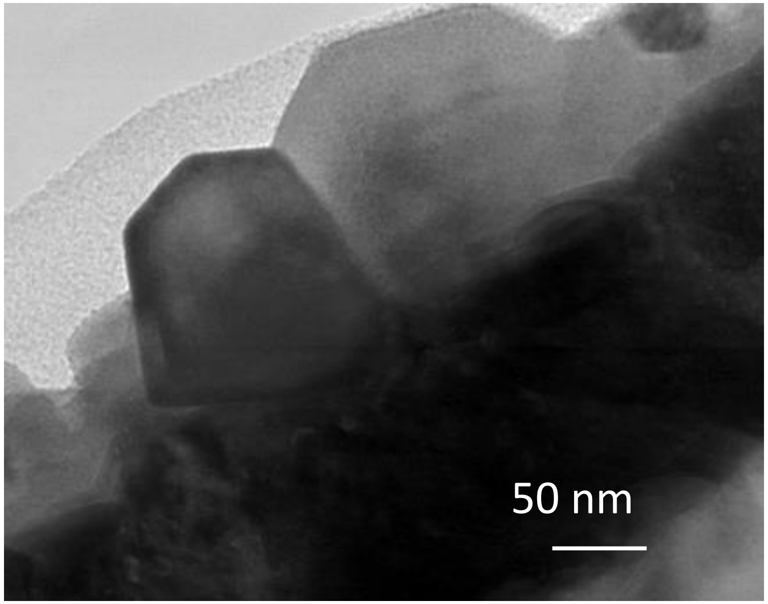
-The objective aperture size .

-The high tension of the TEM.

Objective aperture: Contrast enhancement

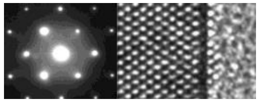
Intensity:
Dependent on grain orientation

Diffraction contrast

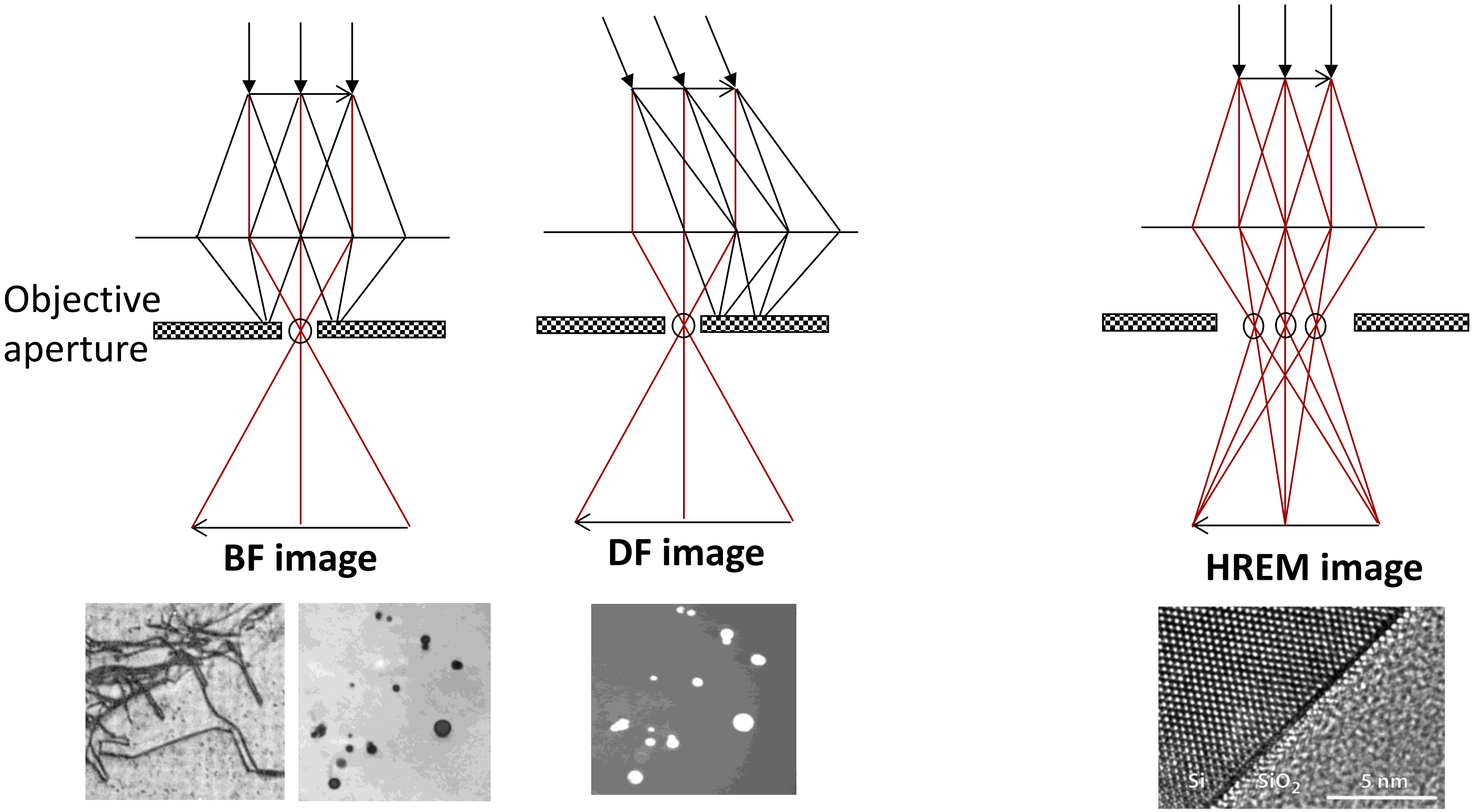


Try to make an illustration to explain why we get this enhanced contrast when only the central beam is selected by the optical aperture.

Size of objective aperture



Bright field (BF), dark field (DF) and High resolution EM (HREM)

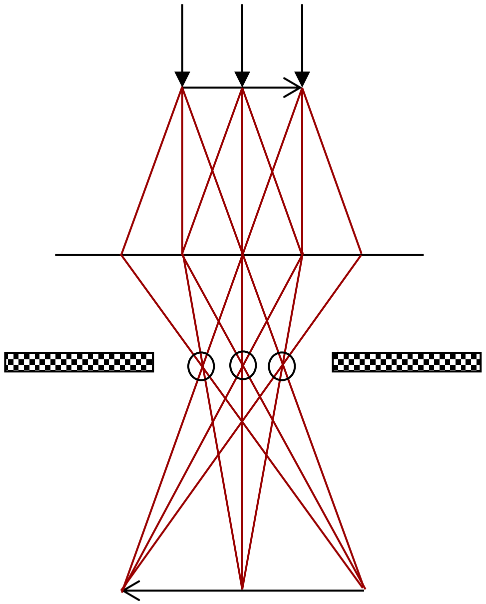


Amplitude/Diffraction contrast

Phase contrast

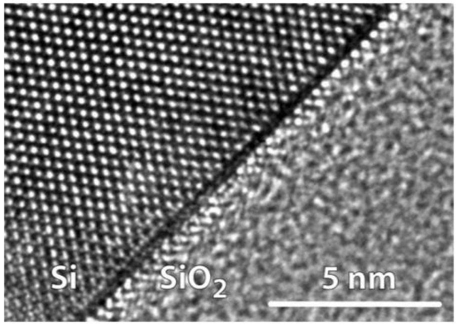
Phase contrast:

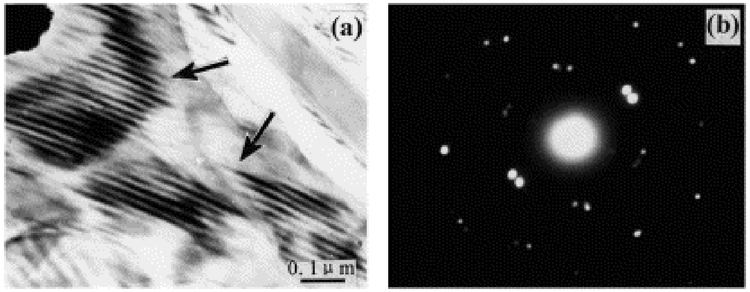
HREM and Moire’ fringes



HREM image

Interference pattern



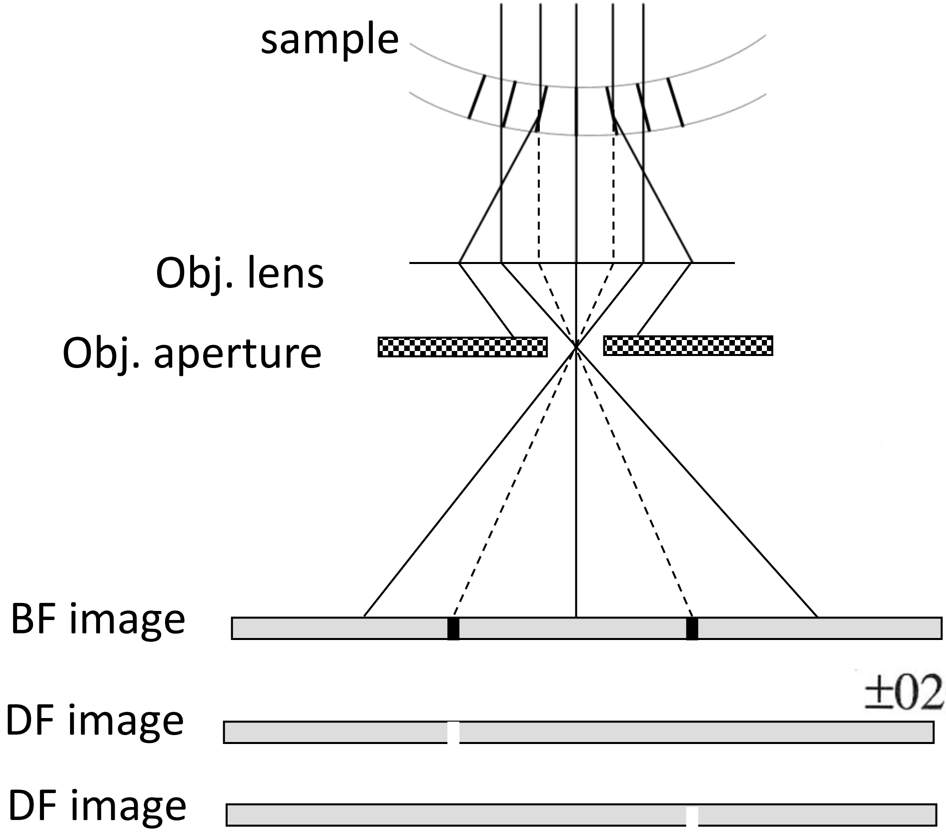


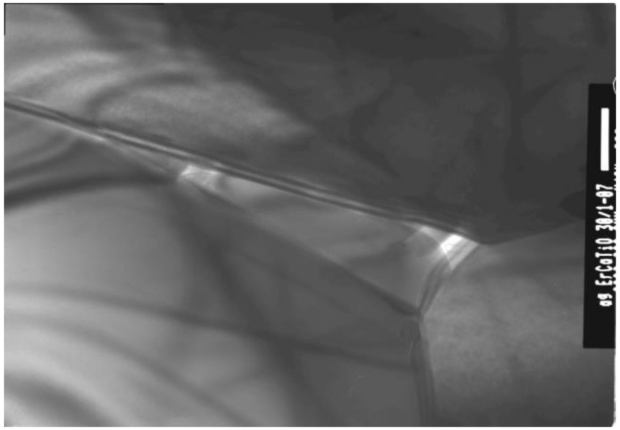
Long-Wei Yin et al., Materials Letters, 52, p.187-191

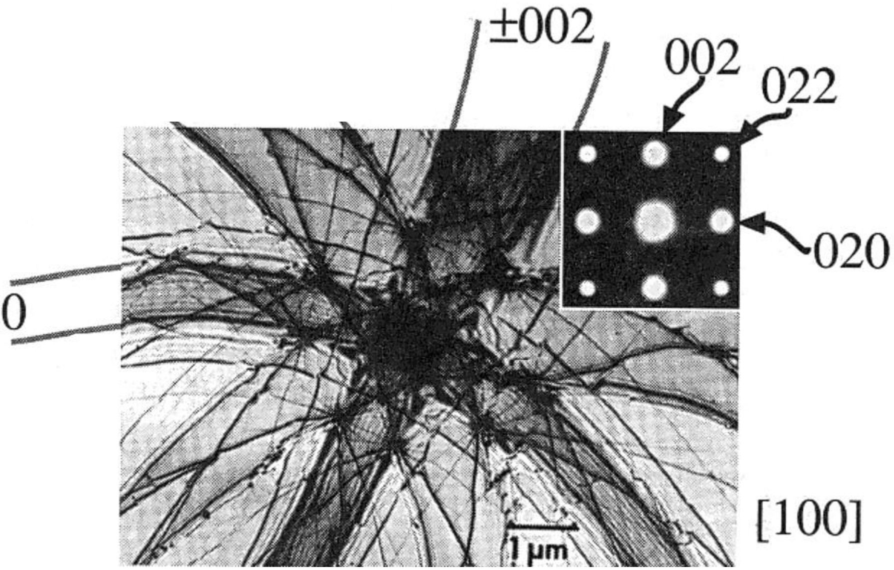
A **Moiré pattern** is an interference pattern created, for example, when two grids are overlaid at an angle, or when they have slightly different mesh sizes (rotational and parallel Moire’ patterns).

http://www.mathematik.com/Moire/

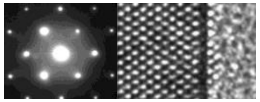
Bending contours





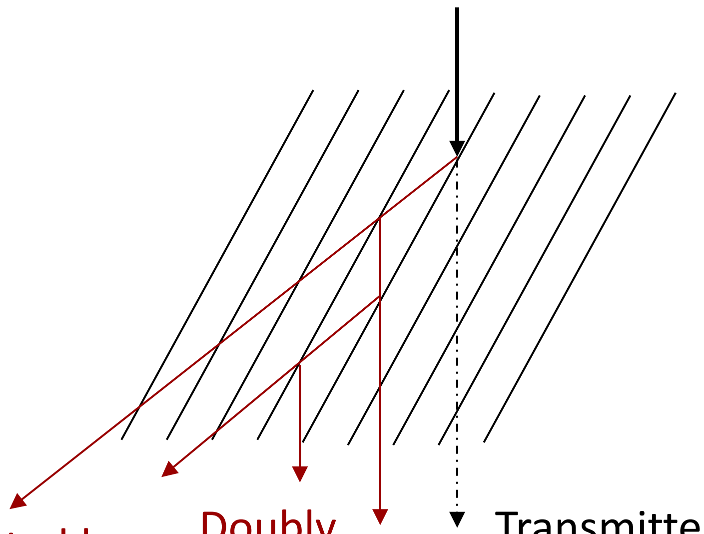


Solberg, Jan Ketil & Hansen, Vidar (2001).
Innføring i transmisjon elektronmikroskopi



Double diffraction, extinction thickness

- **Double electron diffraction** leads to oscillations in the diffracted intensity with increasing thickness of the sample
  - No double diffraction with XRD, kinematical intensities
  - Forbidden reflection may be observed
- **t_0: Extinction thickness**
  - Periodicity of the oscillations
  - $t_0=\pi V_c/\lambda|F(hkl)|$



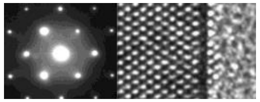
Incident beam

Diffracted beam

Doubly diffracted beam

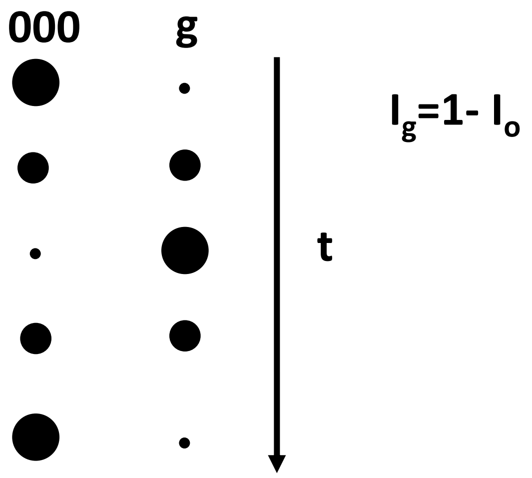
Transmitted beam

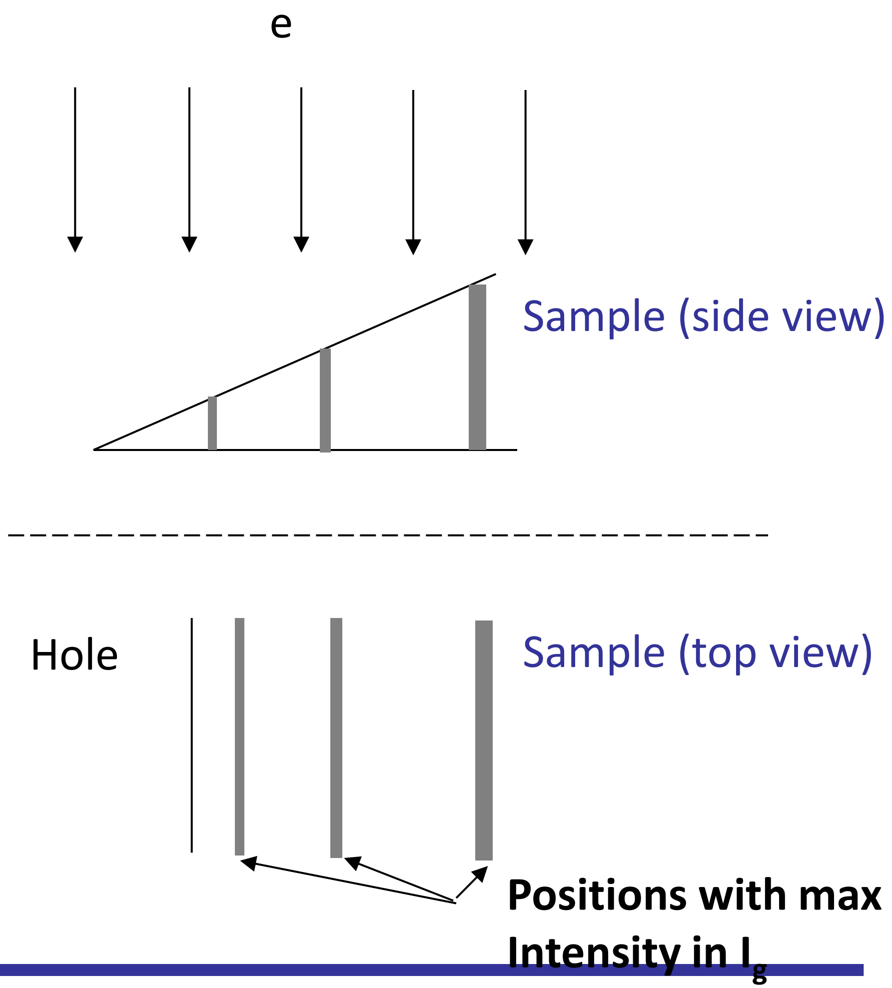
Wedge shaped TEM sample



Thickness fringes/contours

In the two-beam situation the intensity of the diffracted and direct beam is periodic with thickness ($I_g=1- I_o$)



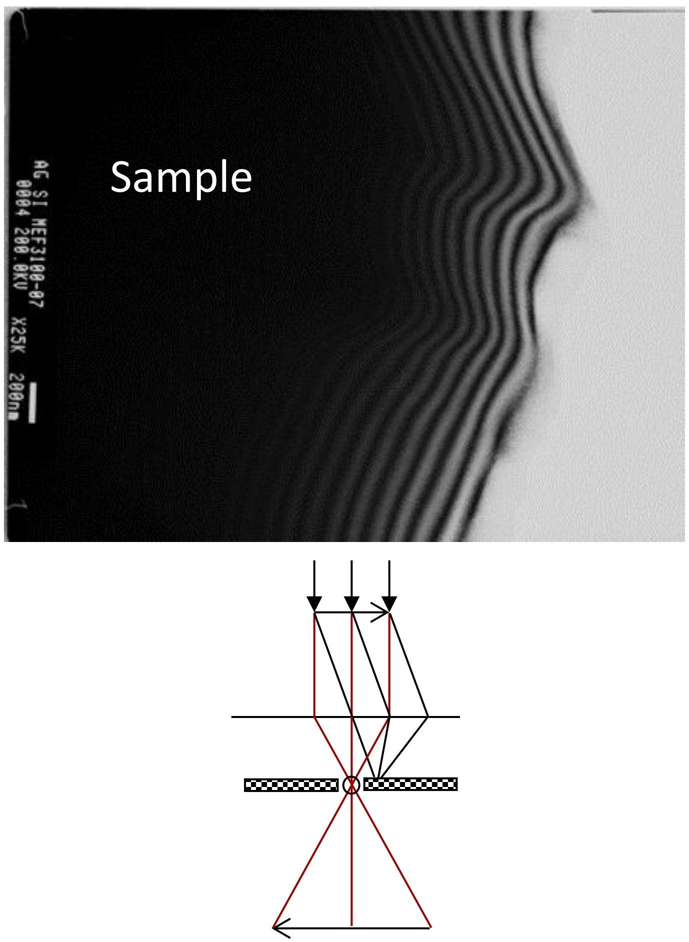


$I_g=(\pi t/\xi_g)^2(\sin^2(\pi t s_{eff})/(\pi t s_{eff})^2)$

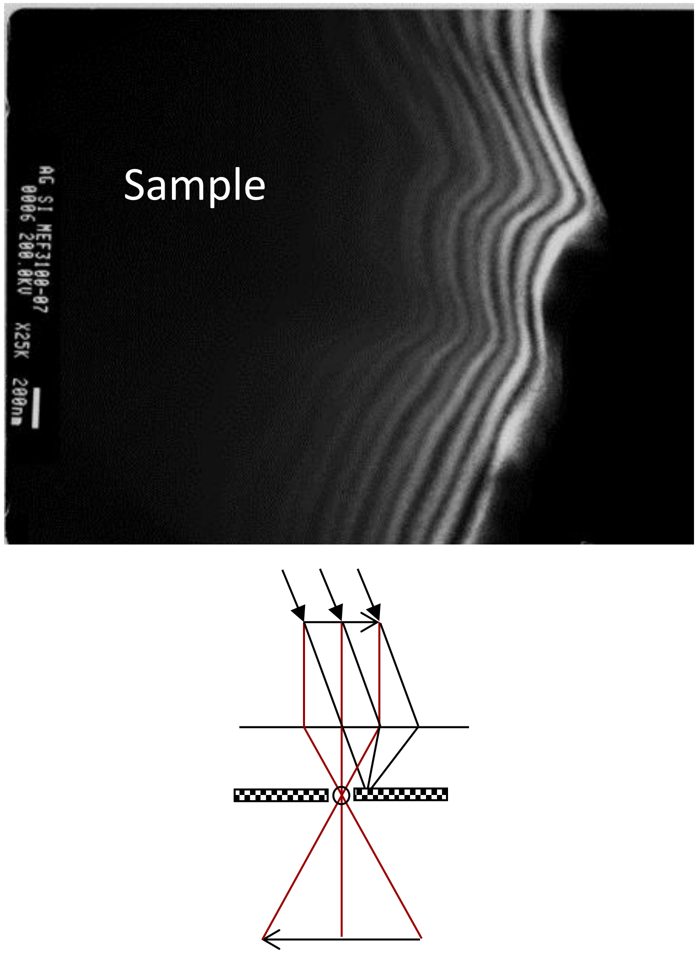
t = distance ”traveled” by the diffracted beam.
ξ_g = extinction distance

Thickness fringes

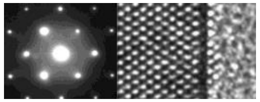
bright and dark field images



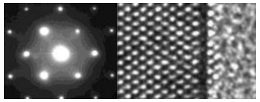
BF image



DF image



TEM specimen preparation



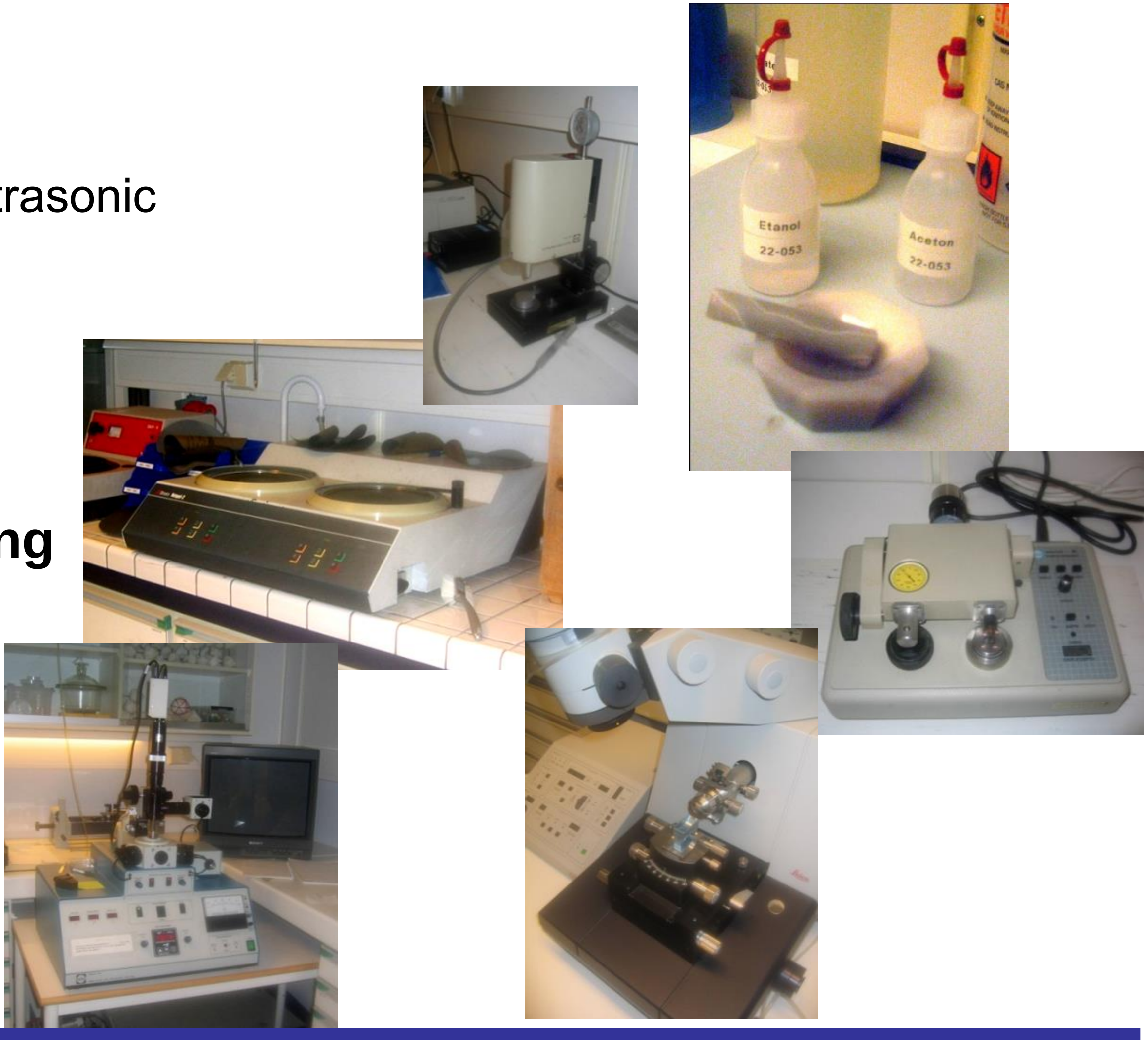
What to considder before preparing a TEM specimen

- Ductile/fragile
- Bulk/surface/powder
- Insulating/conducting
- Heat resistant
- Single phase/multi phase
- Etc, etc…….

What is the objectiv of the TEM work?

Specimen preparation for TEM

- **Crushing**
- **Cutting**
    - saw, “diamond” pen, ultrasonic drill, FIB
- **Mechanical thinning**
    - Grinding, dimpling,
    - Tripod polishing
- **Electrochemical thinning**
- **Ion milling**
- **Coating**
- **Replica methods**
- **Etc.**



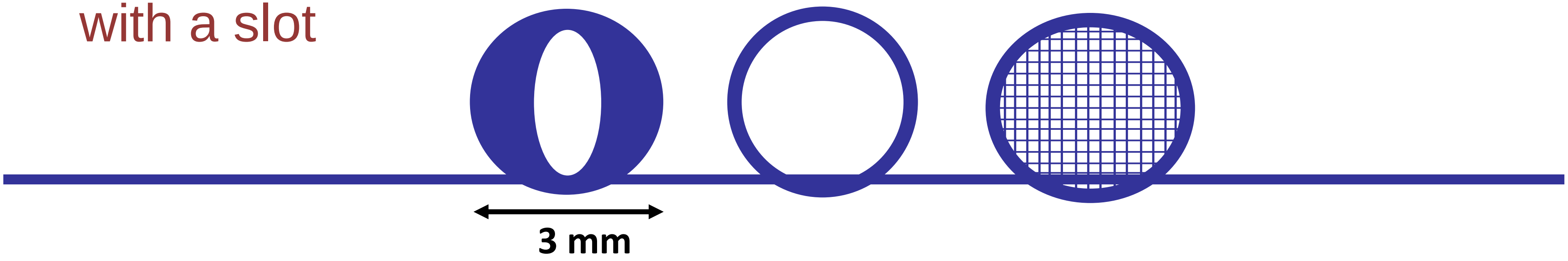
Self-supporting disk or grid

- Self supporting disk
  - Consists of one material
    - Can be a composite
  - Can be handled with a tweezers
    - Metallic, magnetic, non-magnetic, plastic, vacuum

If brittle, consider Cu washer with a slot

- Grid
  - Several types
  - Different materials (Cu, Ni…)
  - Support brittle materials
  - Support small particles

The grid may contribute to the EDS.

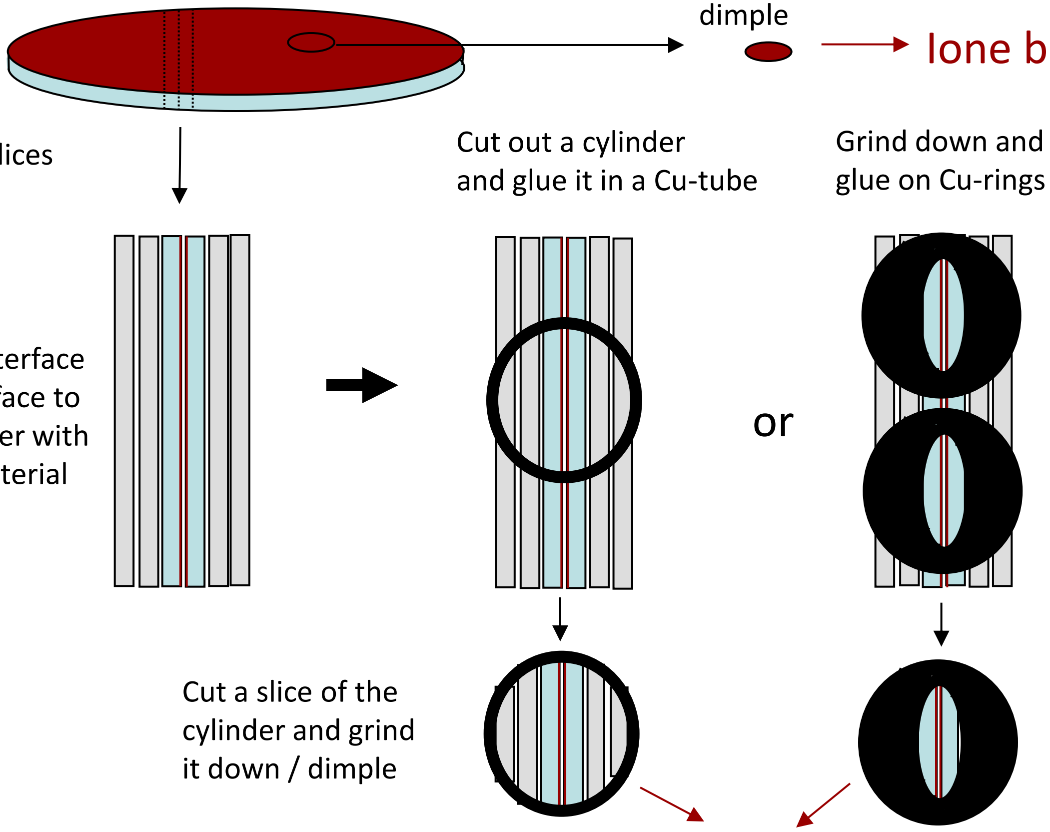


Preparation of self-supporting discs
Top view

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

Cross section TEM sample preparation: Thin films

- **Top view**
- **Cross section**
- **Focused Ion Beam (FIB)**



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

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

or

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

Cut off excess
material

Ione beam thinning