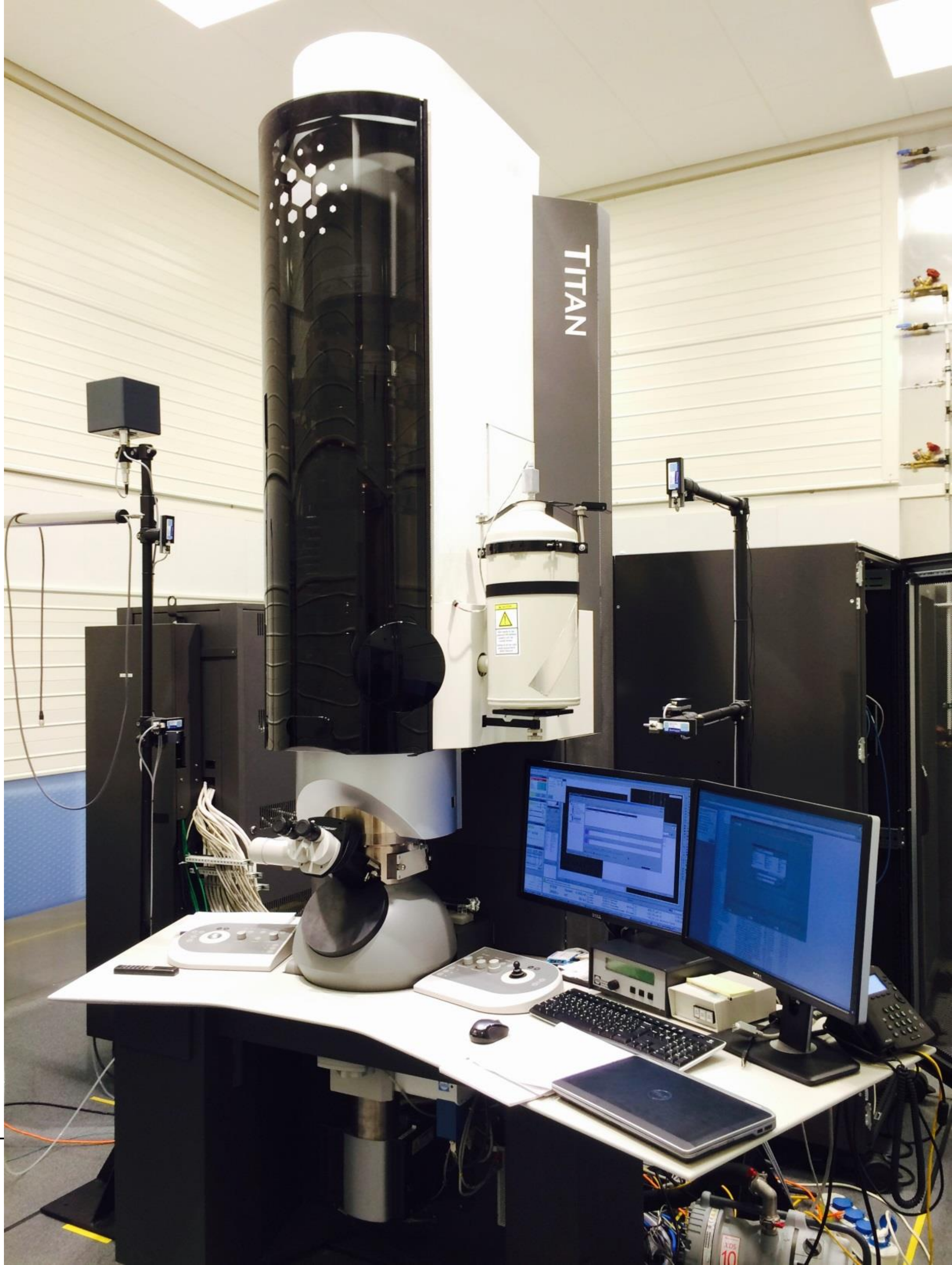
FYS 4340/FYS 9340

Diffraction Methods
& Electron Microscopy

Lecture 3

Sandeep Gorantla

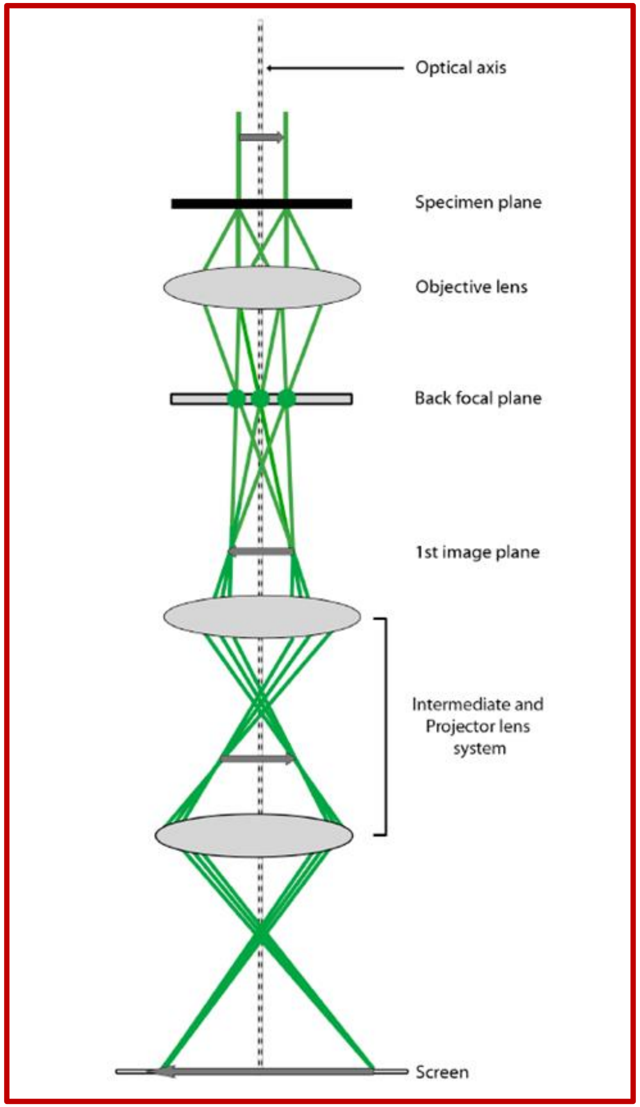


UiO University of Oslo
Centre for Materials Science and Nanotechnology (SMN)

Lab Groups

THURSDAY TEM COURSE (FYS 4340/FYS 9340) LAB GROUPS PLAN		
Group 1	Group 2	Group 3
9:00-11:00	12:00-14:00	14:00-16:00
Annika Utz	**Amalie Berg**	**Hans Jakob Sivertsen Mollatt**
Andrei Karzhou	**Nikita Thind**	**Heine Ness**
Martin Løvøy	**Hengyi zhu**	**Henrik Riis**
Martin Jensen/Anne Klemm	**PrasantaDhak**	

Simplified ray diagram of conventional TEM



Simplified ray diagram of conventional STEM

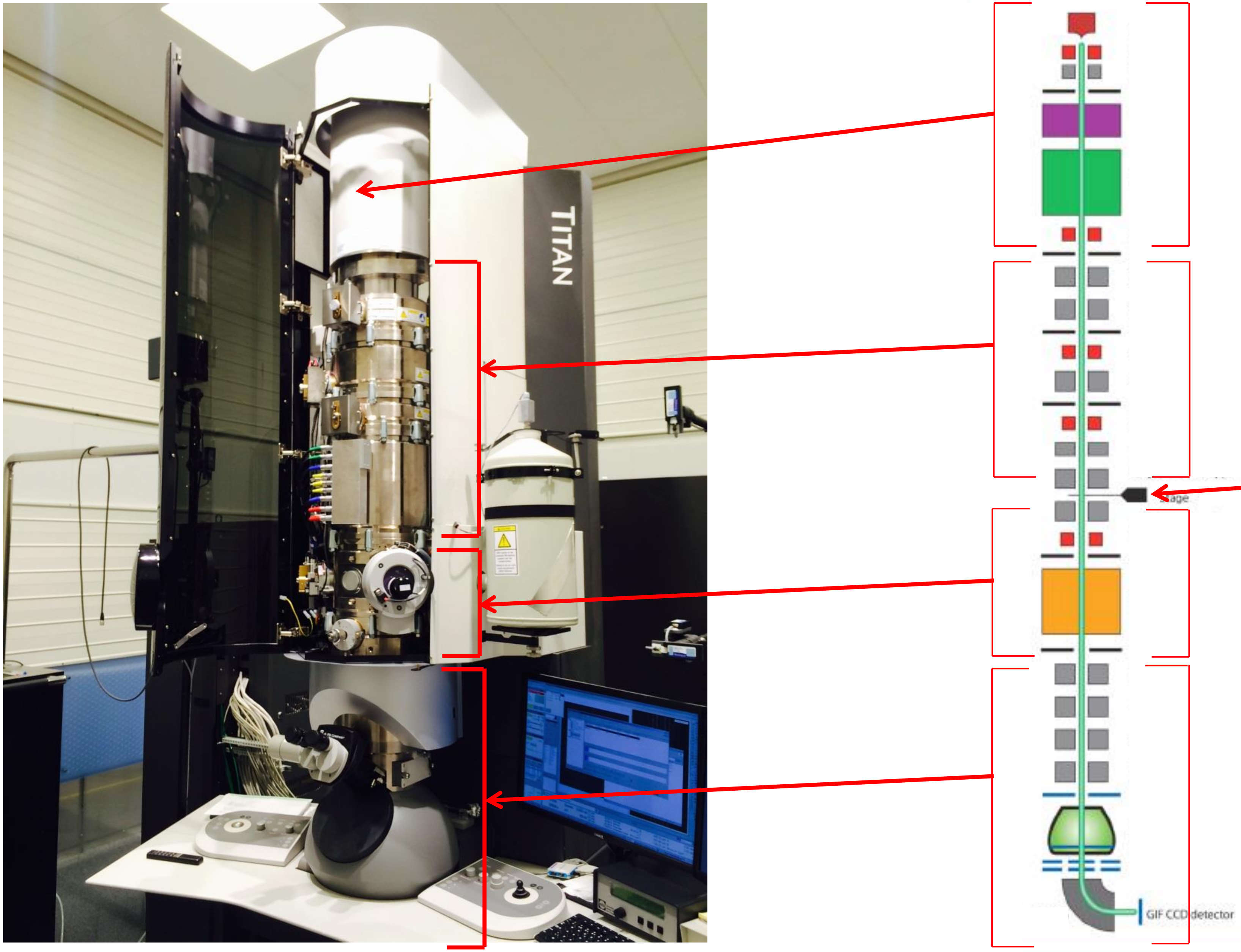
This Lecture

- TEM Instrumentation – Part 2

  (Text book Chapters: 5 – 9)

- TEM Specimen Preparation

  (Text book Chapters: 10)



Electron gun

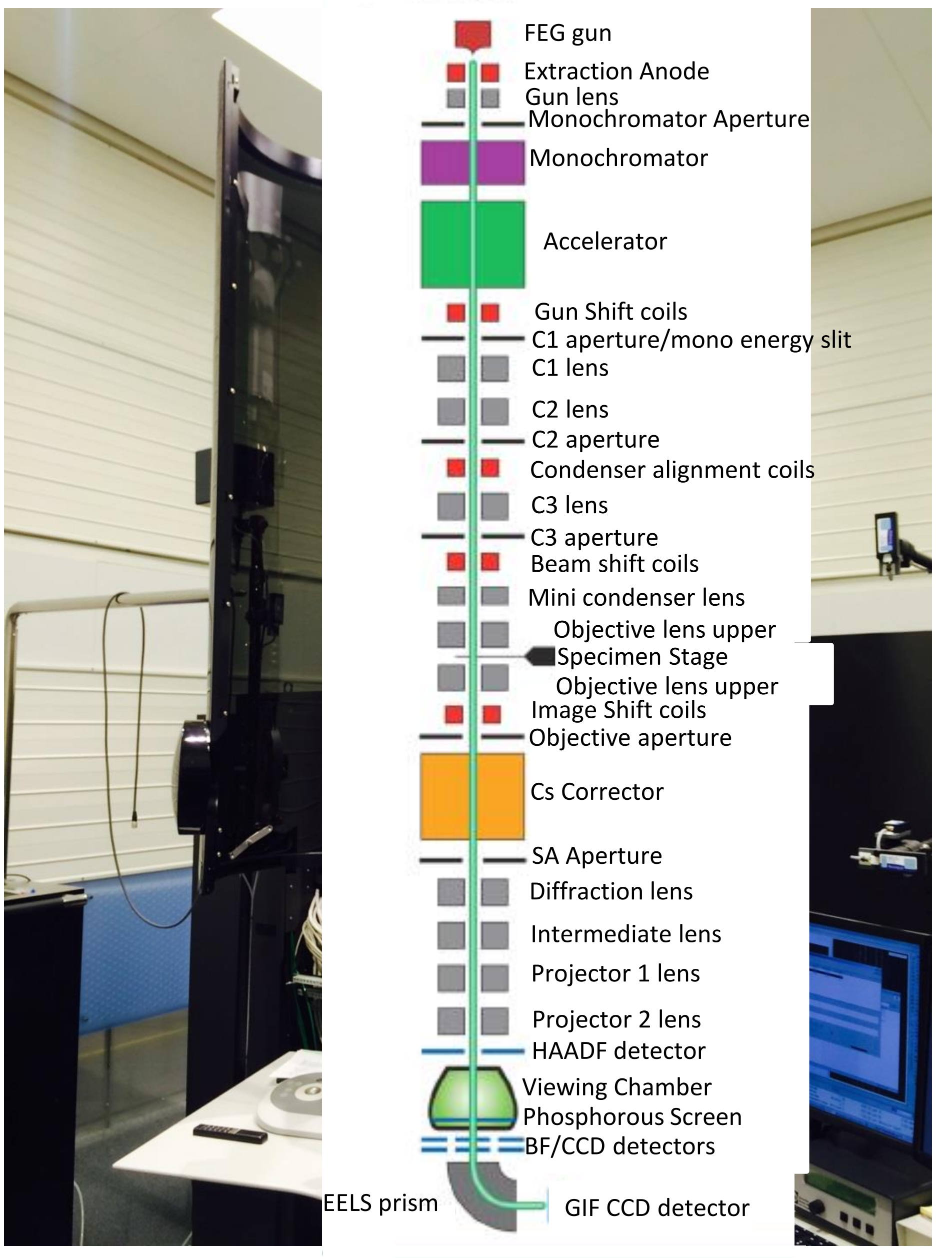
Illumination system

Specimen stage

Imaging system

Projection and Detection system

Courtesy: David Rassouw



- **Electron Gun**
- **Electron Lens**
- **Apertures**
- **Stigmators, scan coils and beam deflecting coils**
- **Specimen Stage/Holders**
- **Lq. N_2 Coldtrap**
- **Image Viewing/Recording system**
- **Spectrometers**

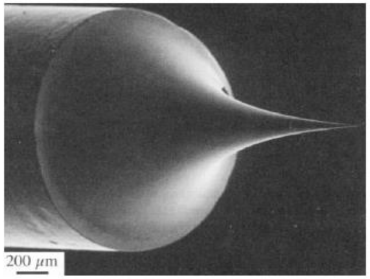
Courtesy: David Rassouw, CCEM, Canada

The requirements of the illumination system

- High electron intensity
  - Image visible at high magnifications
- Small energy spread
  - Reduce chromatic aberrations effect in obj. lens
- High brightness of the electron beam
  - Reduce spherical aberration effects in the obj. lens
- Adequate working space between the illumination system and the specimen

The electron source

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



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

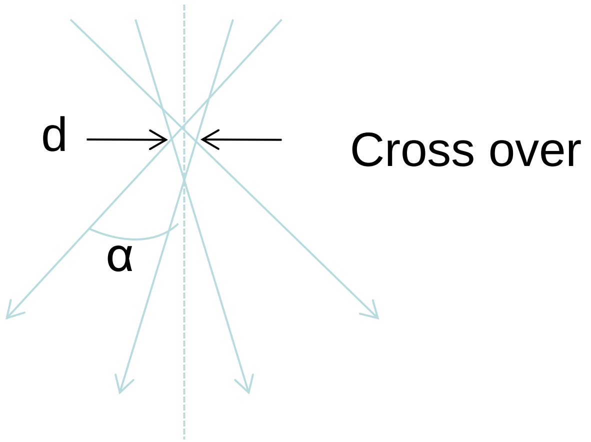


Image of source

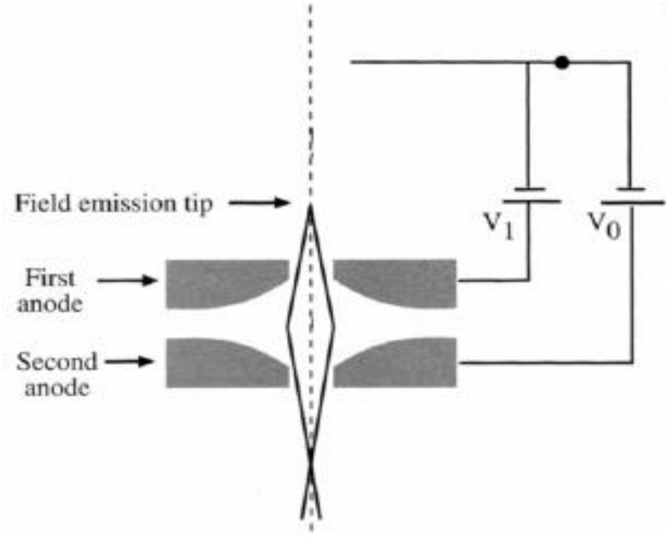
Brightness

- Brightness is the current density per unit solid angle of the source
- $\beta = i_{cr}/(\pi d_{cr}\alpha_{cr})^2$

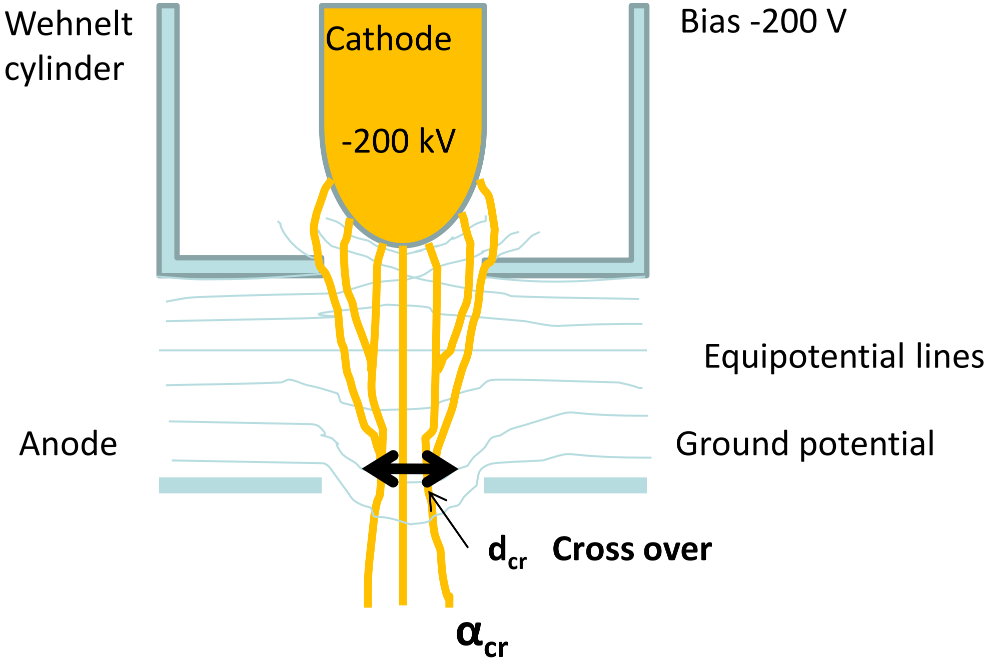
Beam diameter, d_{cr}
Divergence angle, α_{cr}
Beam current, I_{cr}
Beam brightness, β_{cr}
at the cross over

The electron gun

FEG



Thermionic gun



Wehnelt cylinder

Cathode

-200 kV

Bias -200 V

Equipotential lines

Anode

Ground potential

d_{cr} **Cross over**

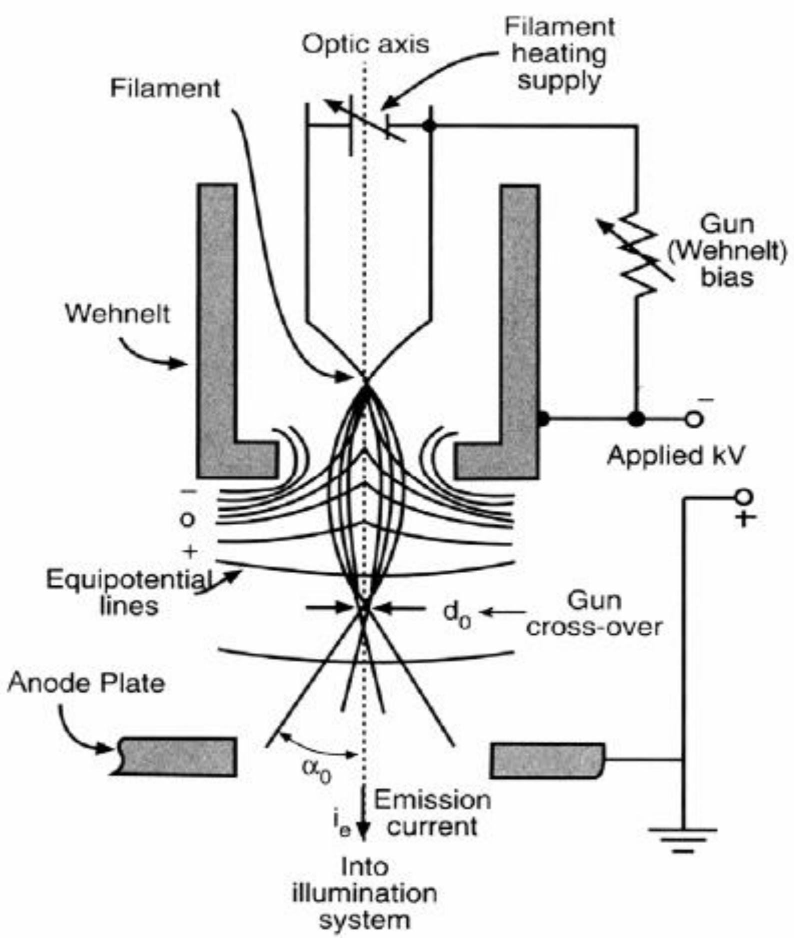
α_{cr}

Thermionic guns

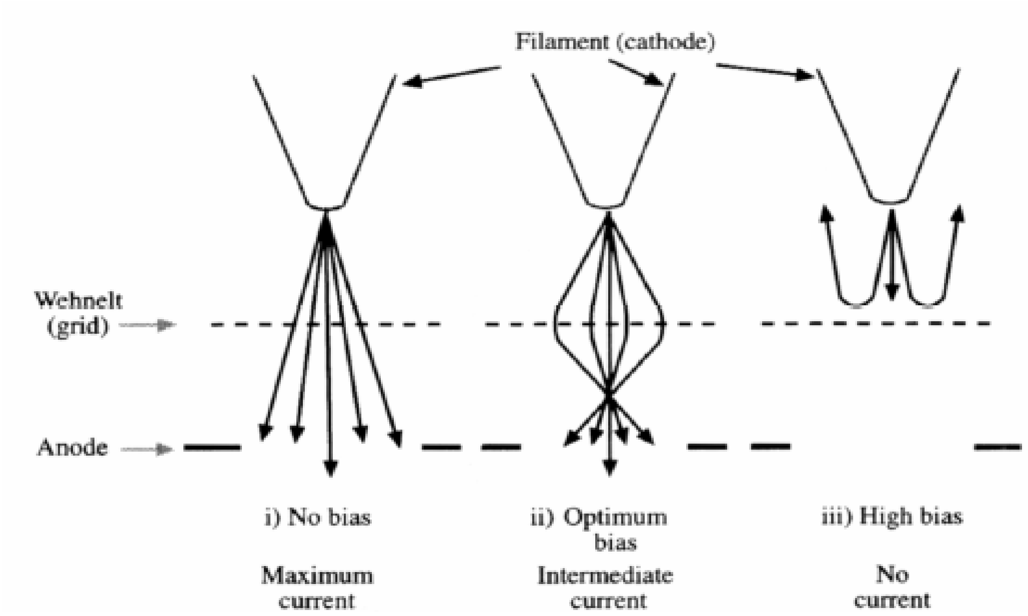
Filament heated to give Thermionic emission
-Directly (W) or
indirectly (LaB_6)

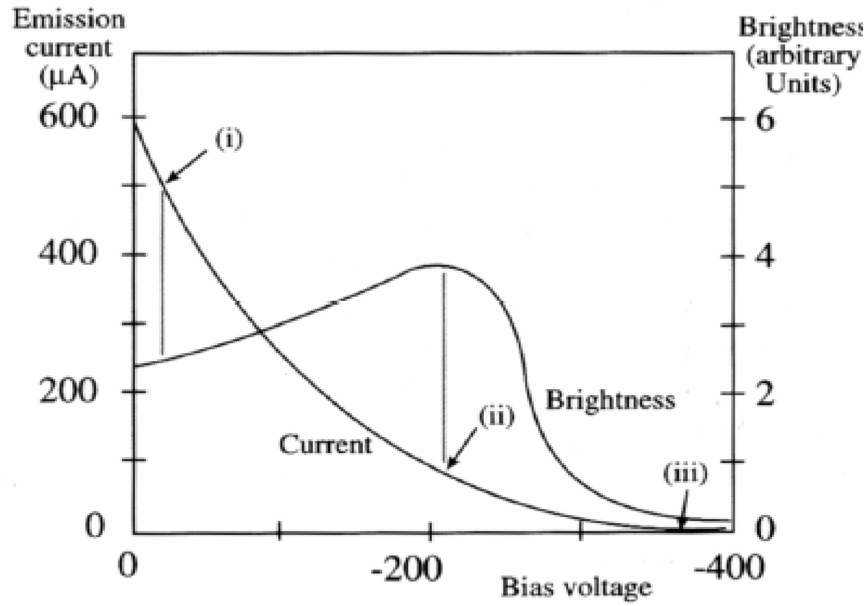
Filament negative potential to ground

Wehnelt produces a small negative bias
-Brings electrons to
cross over



Thermionic guns





Thermionic emission

- Current density: $J_c = A_c T^2 \exp(-\phi_c/kT)$

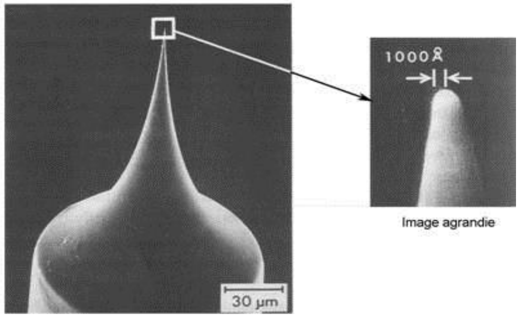
Richardson-Dushman

  - Ac: Richardson's constant, material dependent
  - T: Operating temperature (K)
  - ϕ: Work function (natural barrier to prevent electrons to leak out from the surface)
  - k: Boltzmann's constant

Maximum usable temperature T is determined by the onset of the evaporation of material.

Field emission

- 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).

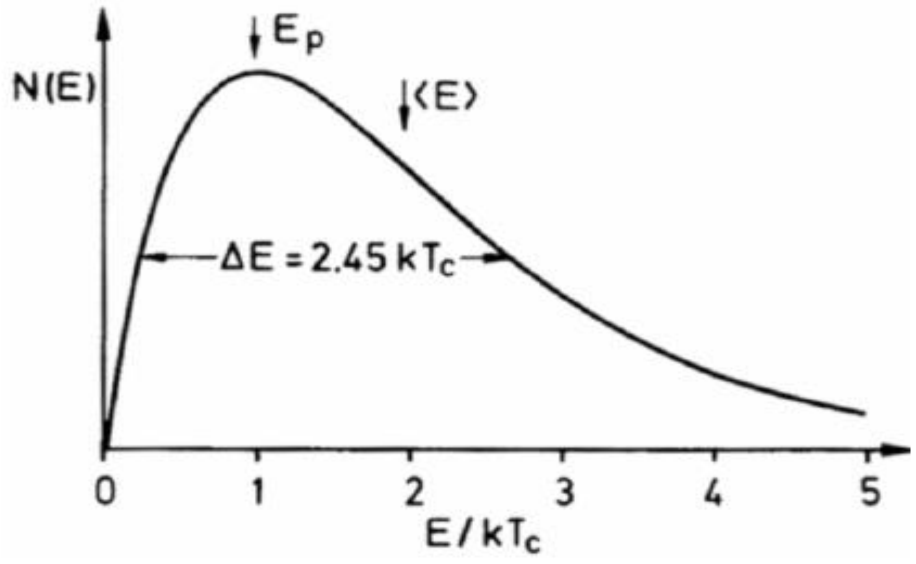
Field emission

- Current density:

$$j_c = \frac{k_1 |E|^2}{\phi} \exp\left(\frac{k_2 \phi^{3/2}}{|E|}\right)$$

Fowler-Norheim

Maxwell-Boltzmann
energy distribution
for all sources



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^2sr)	$5*10^9$	$5*10^{10}$	$5*10^{12}$	$5*10^{12}$
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

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

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
  - Not used as imaging lenses, but are used in modern monochromators

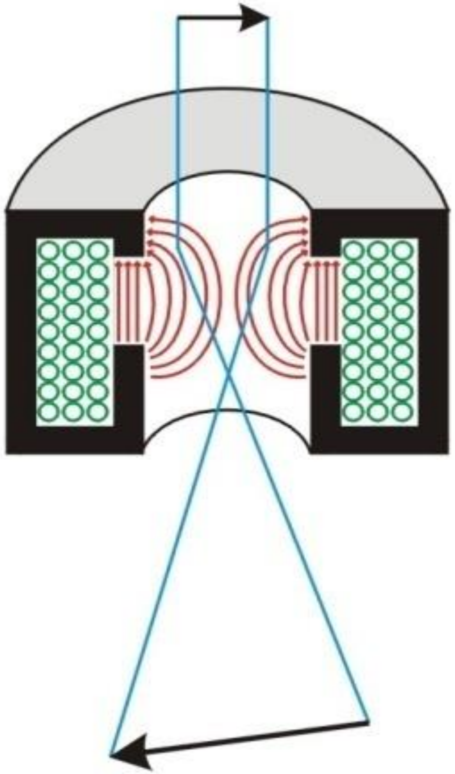
$$\mathbf{F} = -e\mathbf{E}$$

- ElectroMagnetic
  - Can be made more accurately
  - Shorter focal length

$$\mathbf{F} = -e(\mathbf{v} \times \mathbf{B})$$

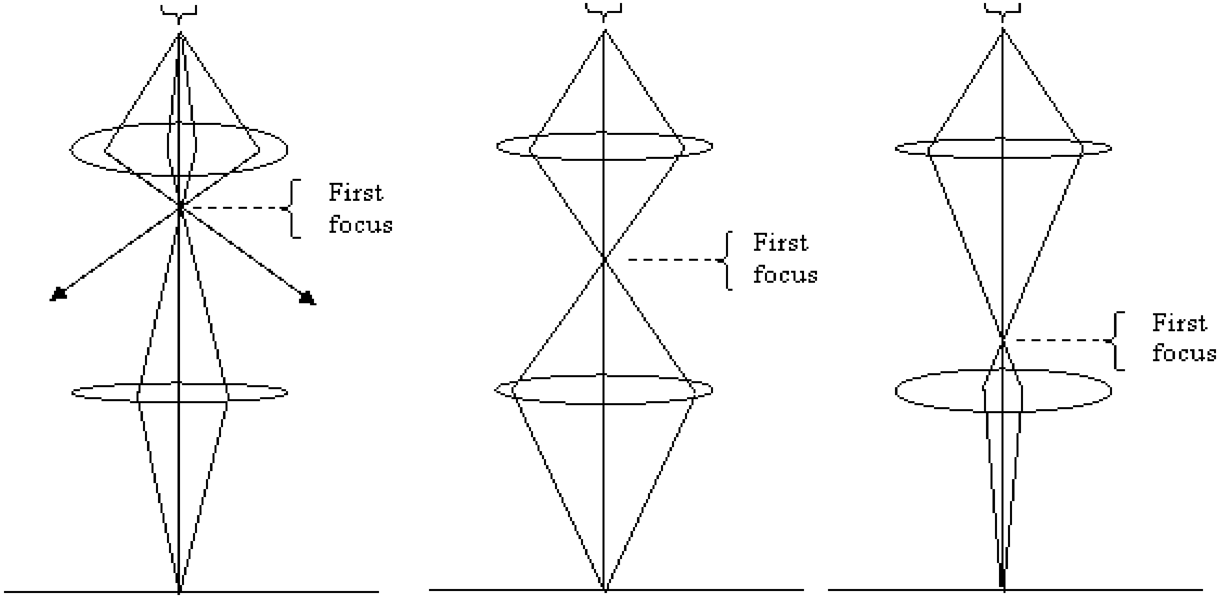
General features of magnetic lenses

- 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

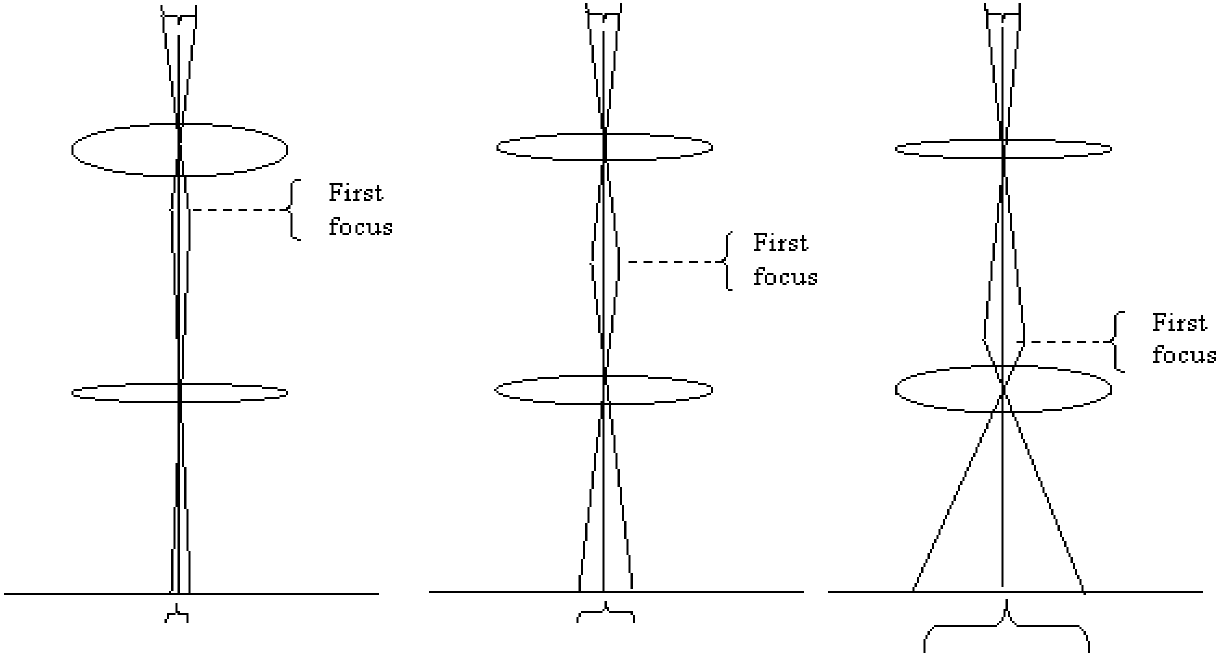
Strengths of lenses and focused image of the source



http://www.rodenburg.org/guide/t300.html

If you turn up one lens (i.e. make it stronger, or 'over- focus' then you must turn the other lens down (i.e. make it weaker, or 'under-focus' it, or turn its knob anti-clockwise) to keep the image in focus.

Magnification of image,
Rays from different parts of the object



http://www.rodenburg.org/guide/t300.html

If the strengths (excitations) of the two lenses are changed, the magnification of the image changes

The Objective lens

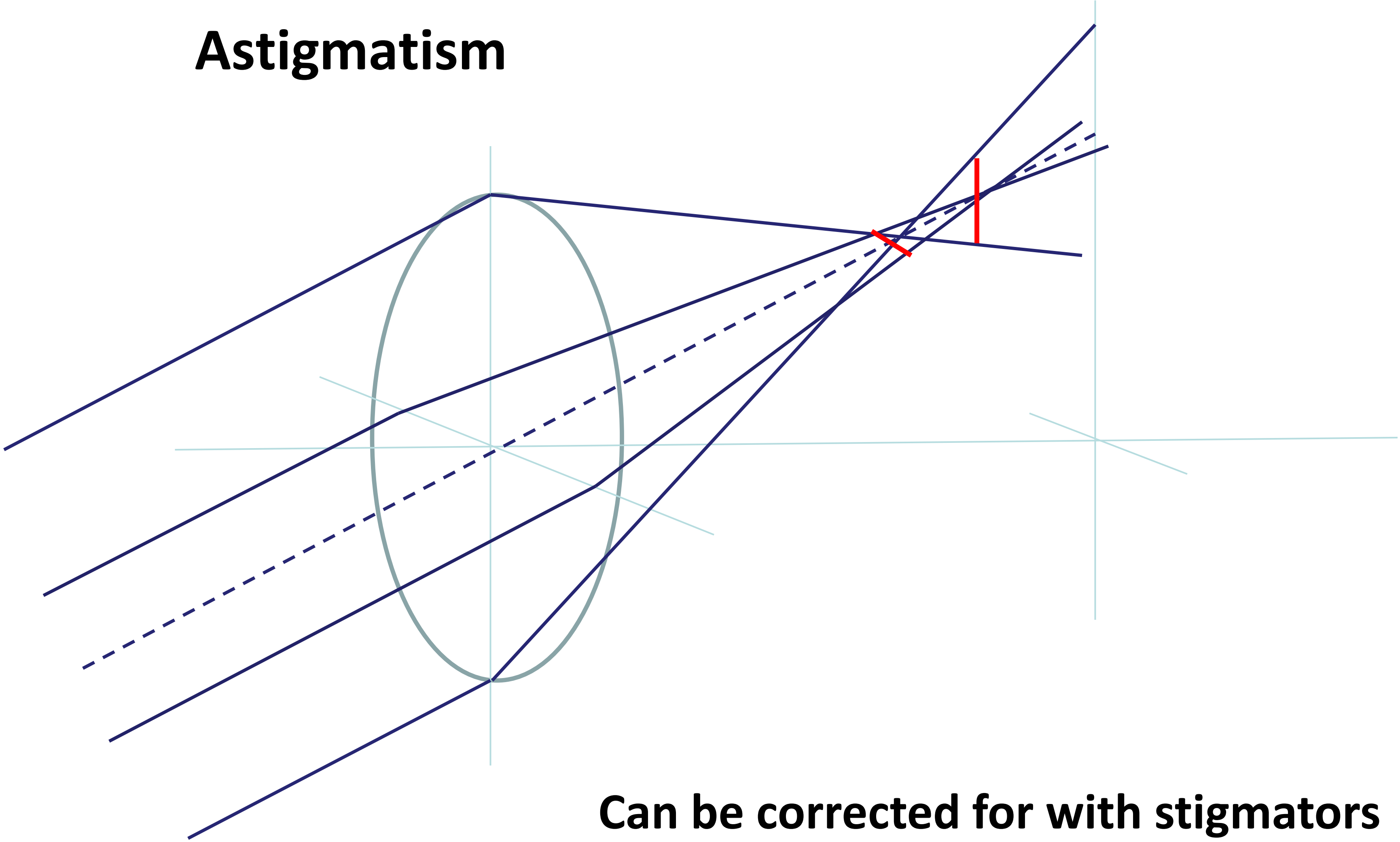
- Often a double or twin lens

- 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
  - Chromatical

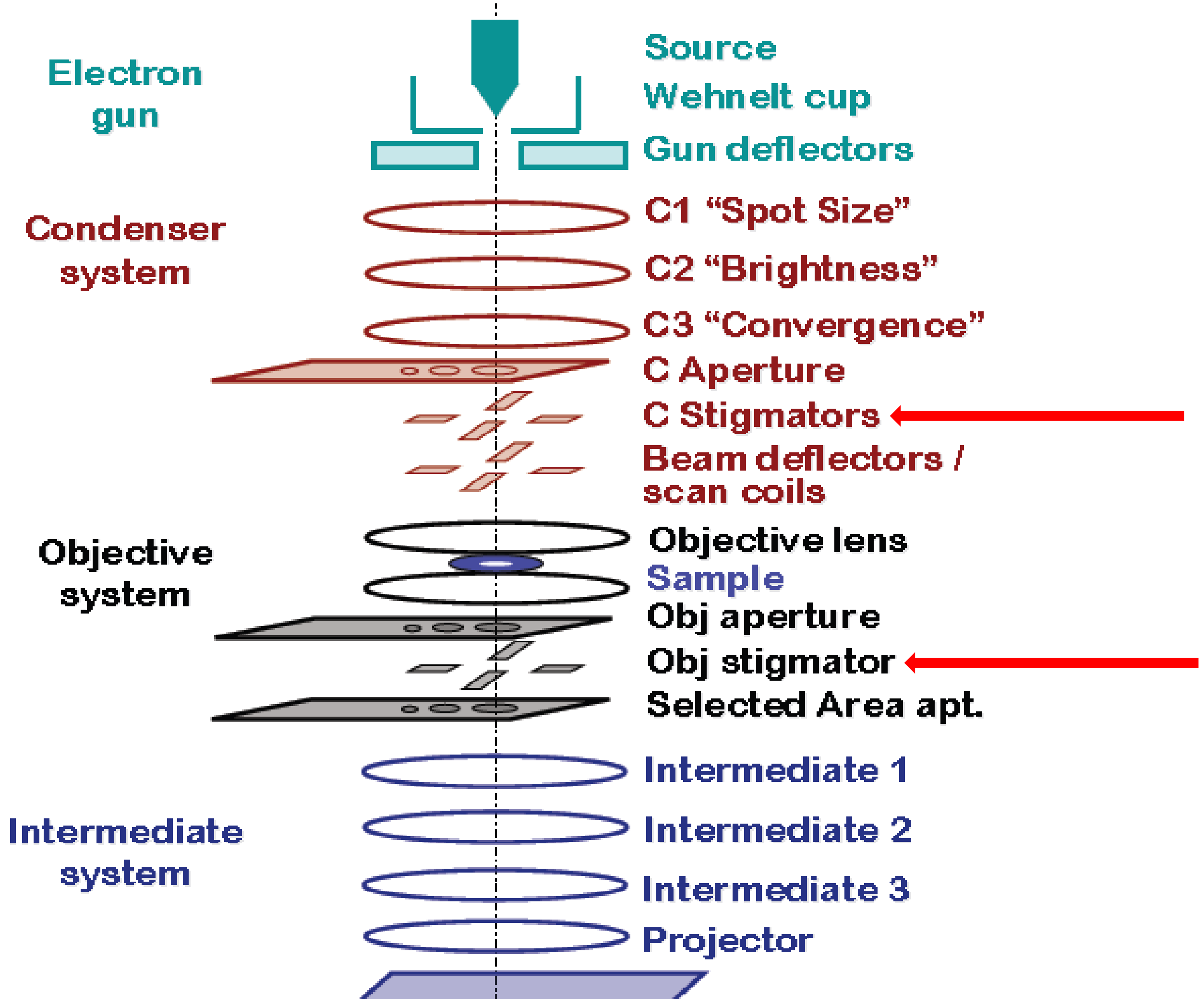
Stigmators

Astigmatism

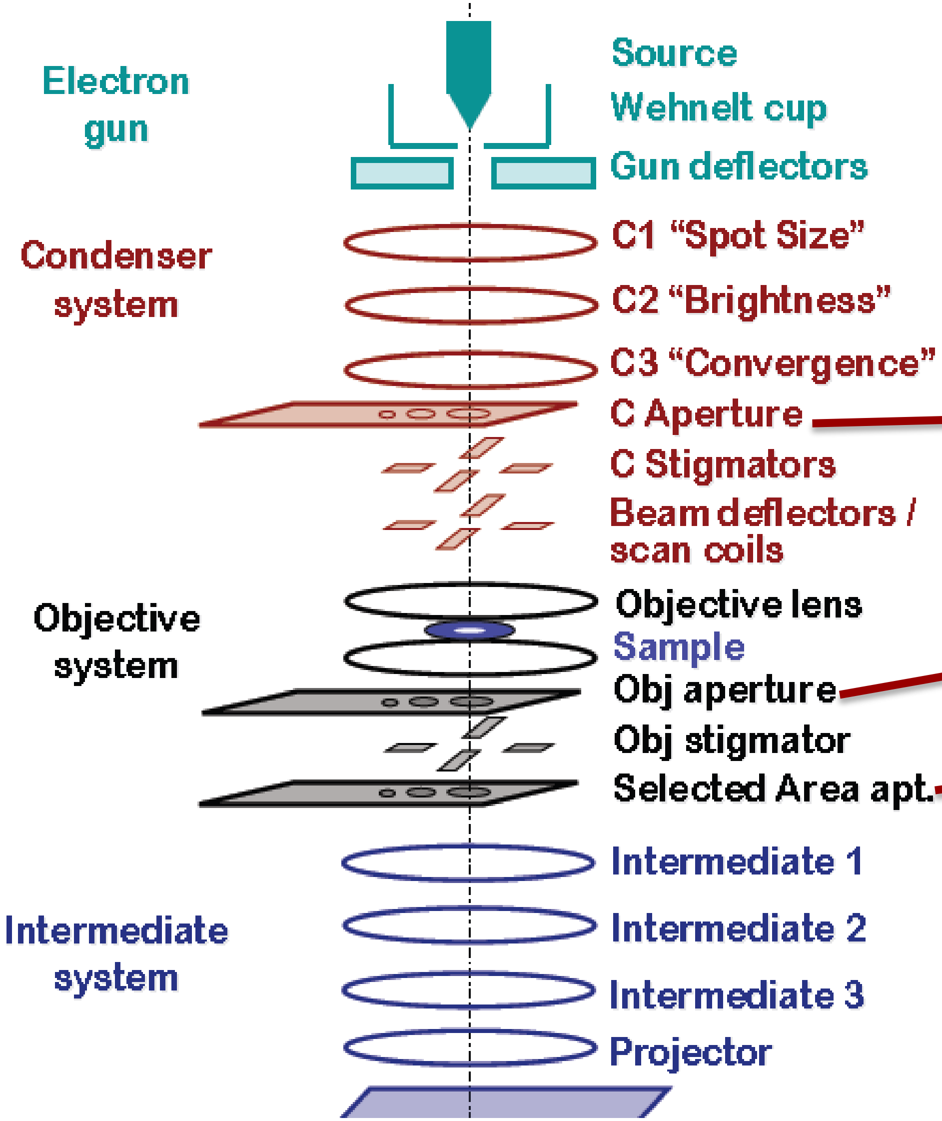


Can be corrected for with stigmators

Stigmators


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

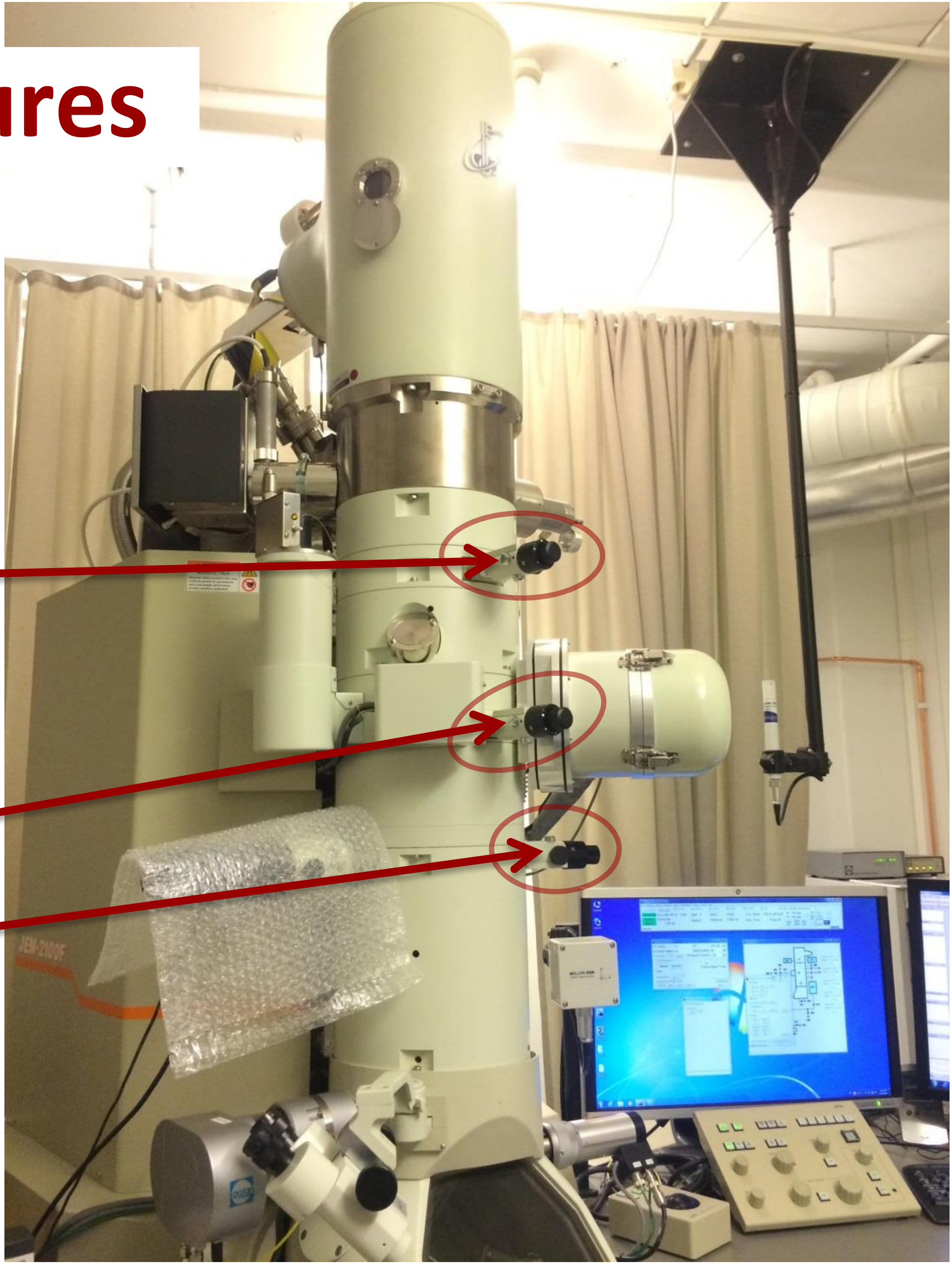


Electron gun
- Source
- Wehnelt cup
- Gun deflectors

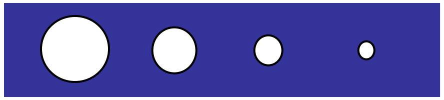
Condenser system
- C1 “Spot Size”
- C2 “Brightness”
- C3 “Convergence”
- C Aperture
- C Stigmators
- Beam deflectors / scan coils

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:

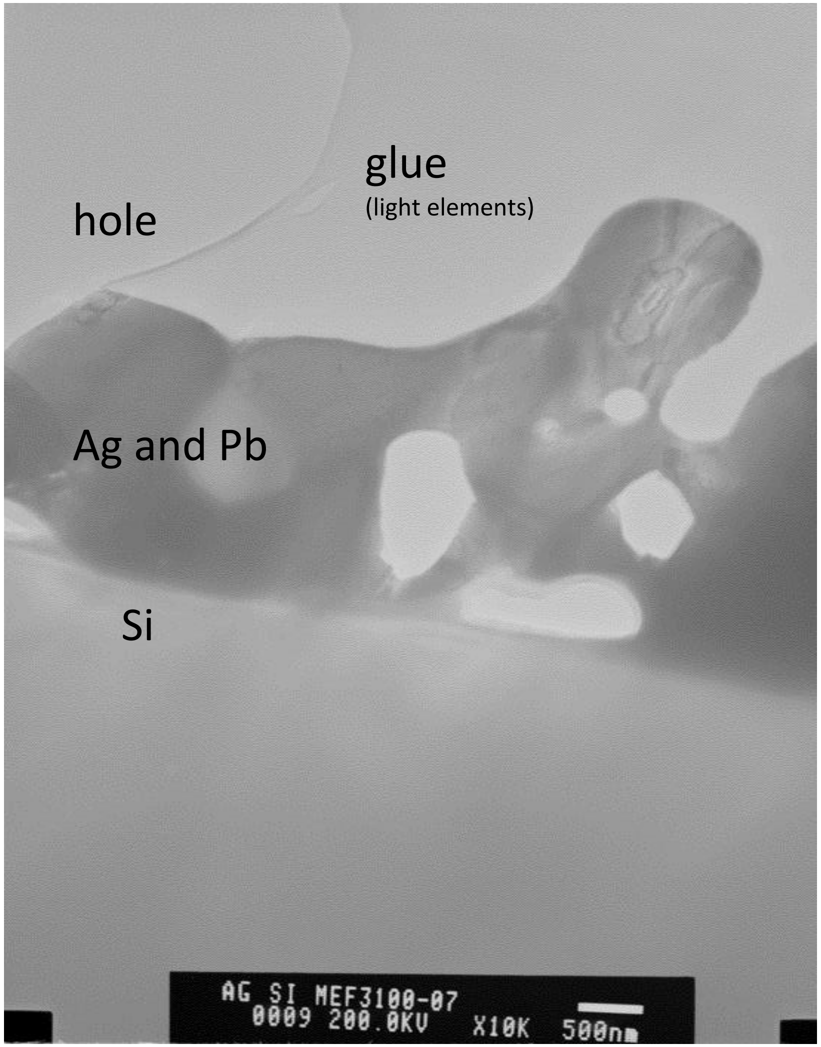
Control the contrast in the image. Allow certain reflections to contribute to the image. Bright field imaging (central beam, 000), Dark field imaging (one reflection, **g**), High resolution Images (several reflections from a zone axis).

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



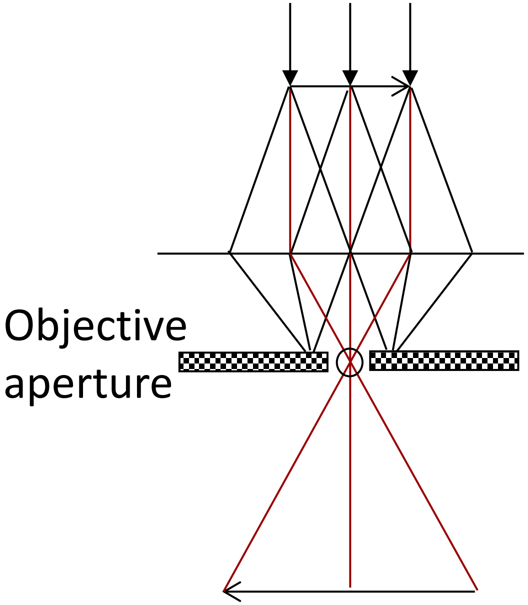
All electrons contributes to the image.

Only central beam contributes to the image.

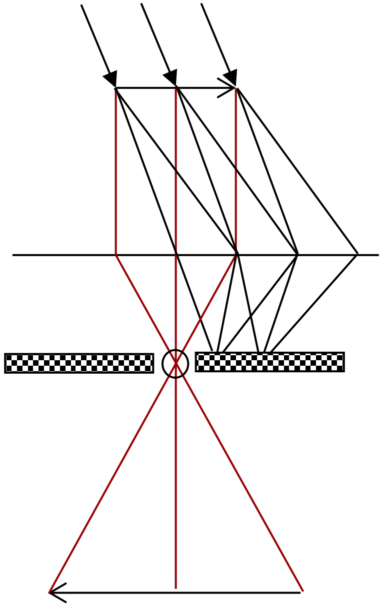
Small objective aperture

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

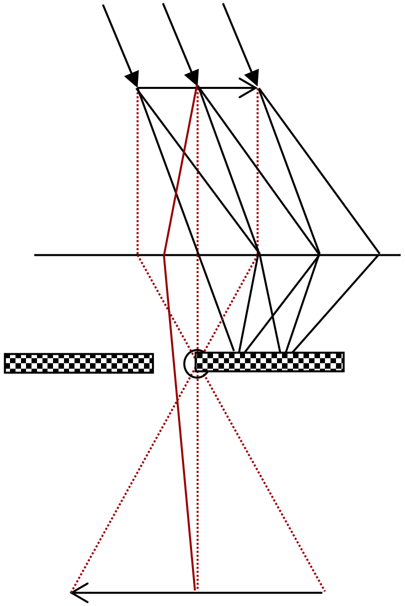
(Diffraction contrast)



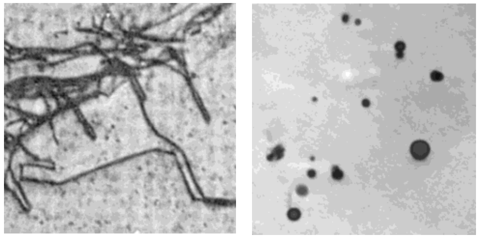
BF image

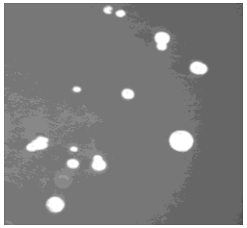


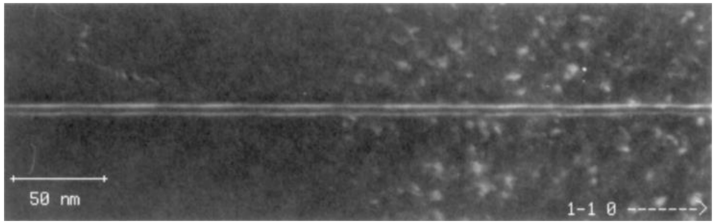
DF image



Weak-beam



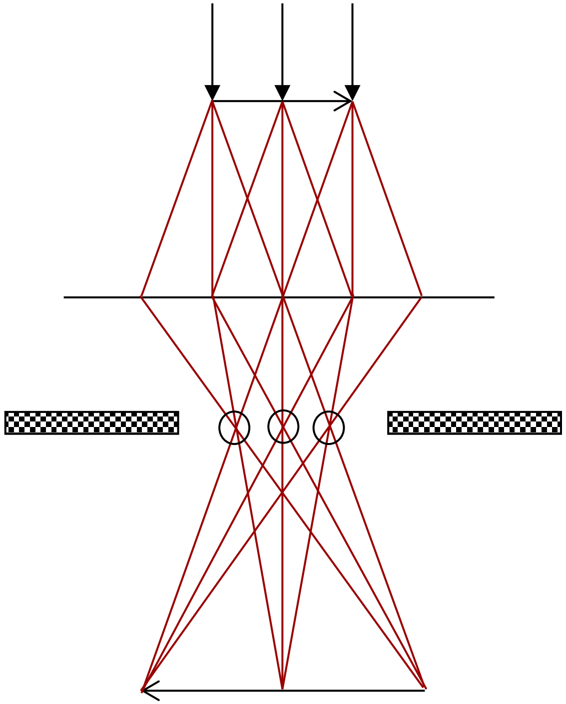




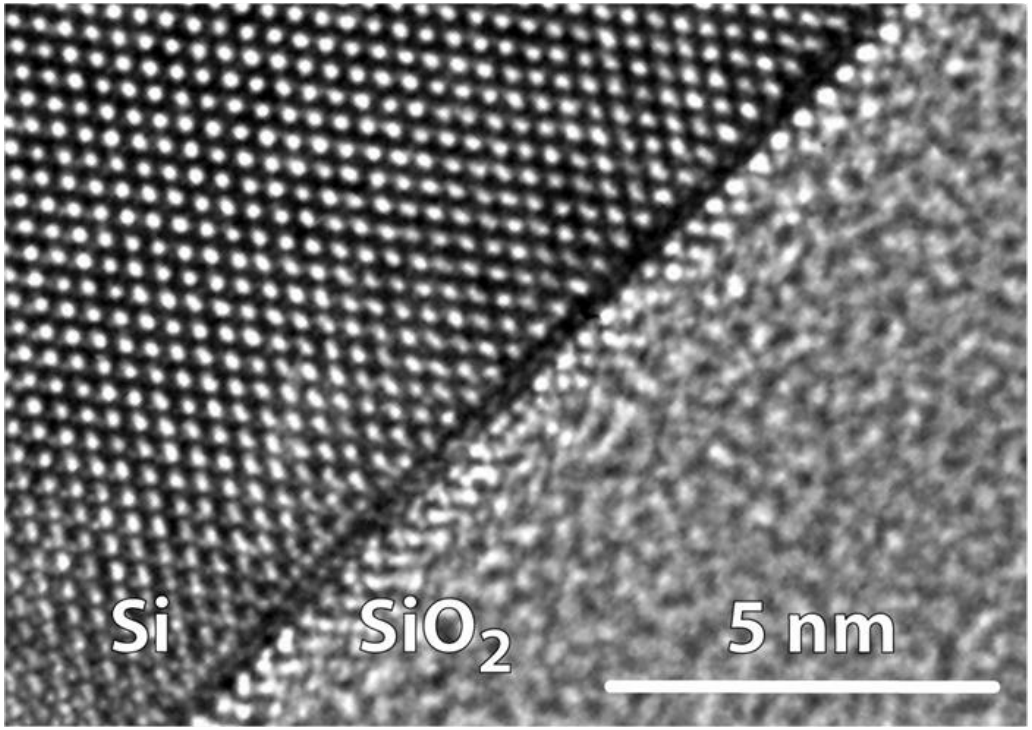
Dissociation of pure screw dislocation In Ni_3Al, Meng and Preston, J. Mater. Scicence, 35, p. 821-828, 2000.

Large objective aperture

High Resolution Electron Microscopy (HREM)



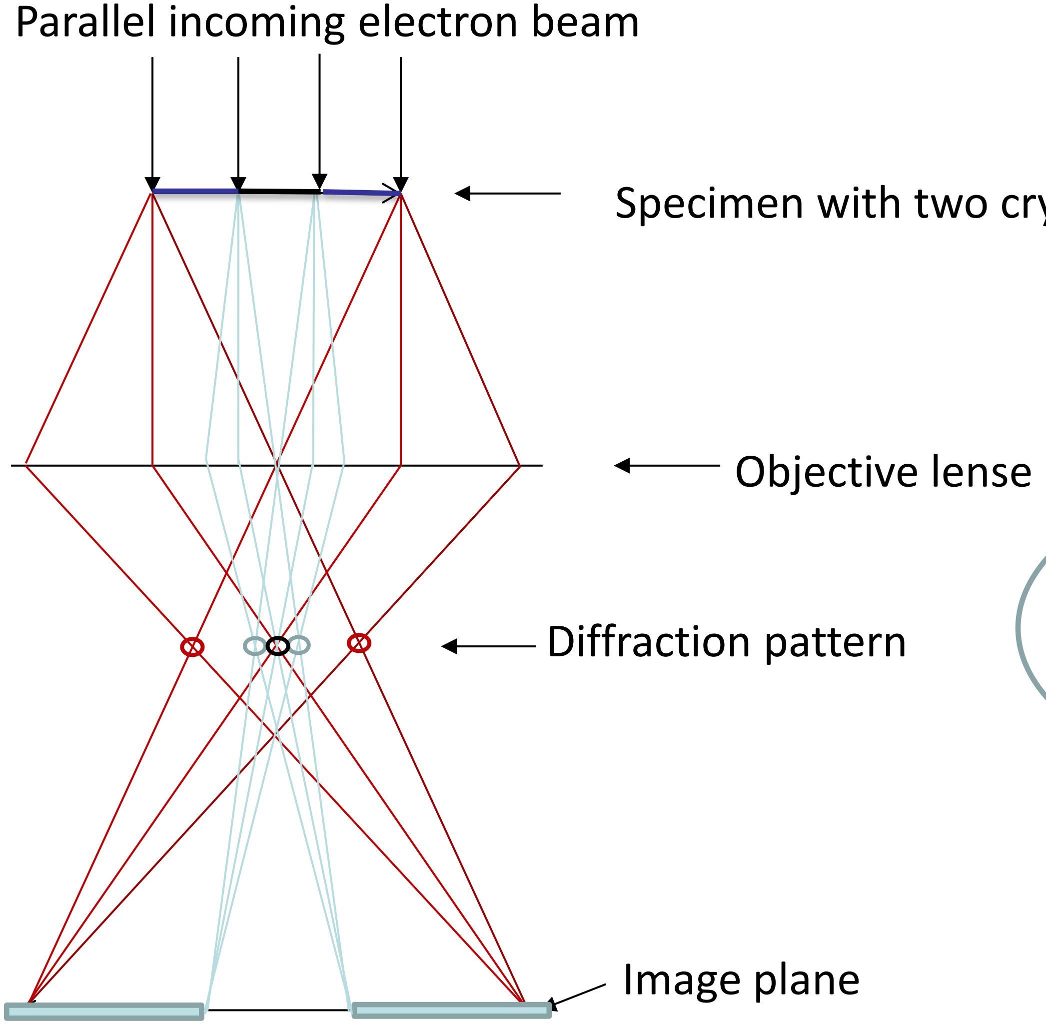
HREM image

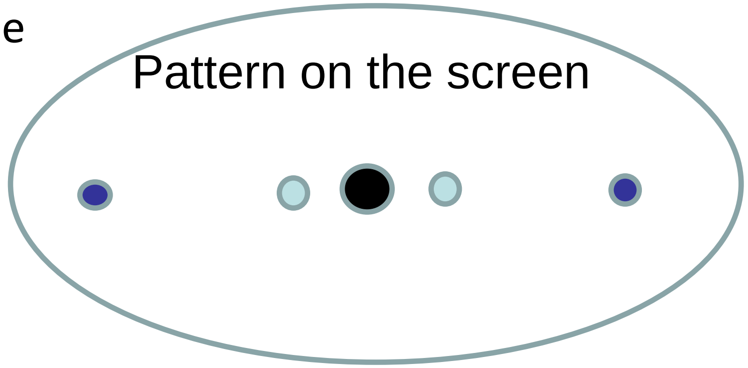


Phase contrast

Selected Area Diffraction Aperture

Selected area diffraction



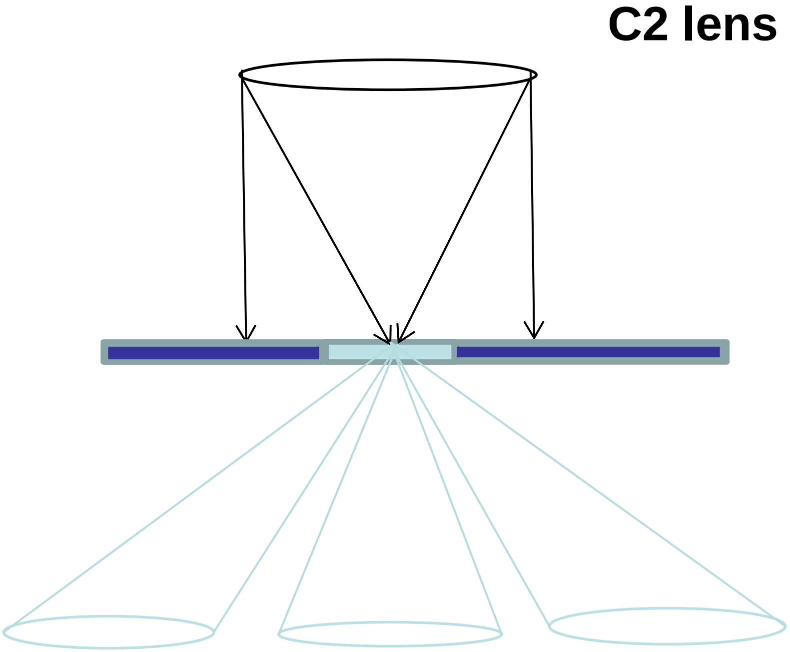


Selected area aperture

Diffraction with no apertures

Convergent beam and Micro diffraction (CBED and µ-diffraction)

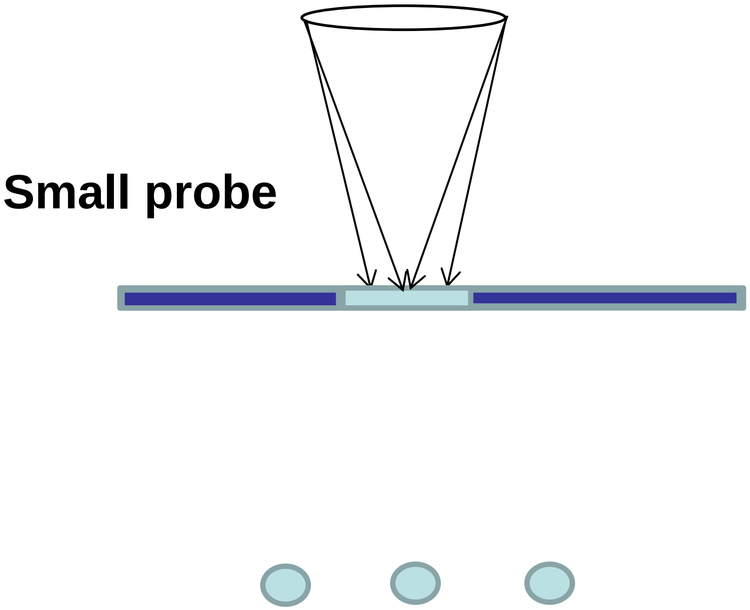
Convergent beam
Focused beam



CBED pattern

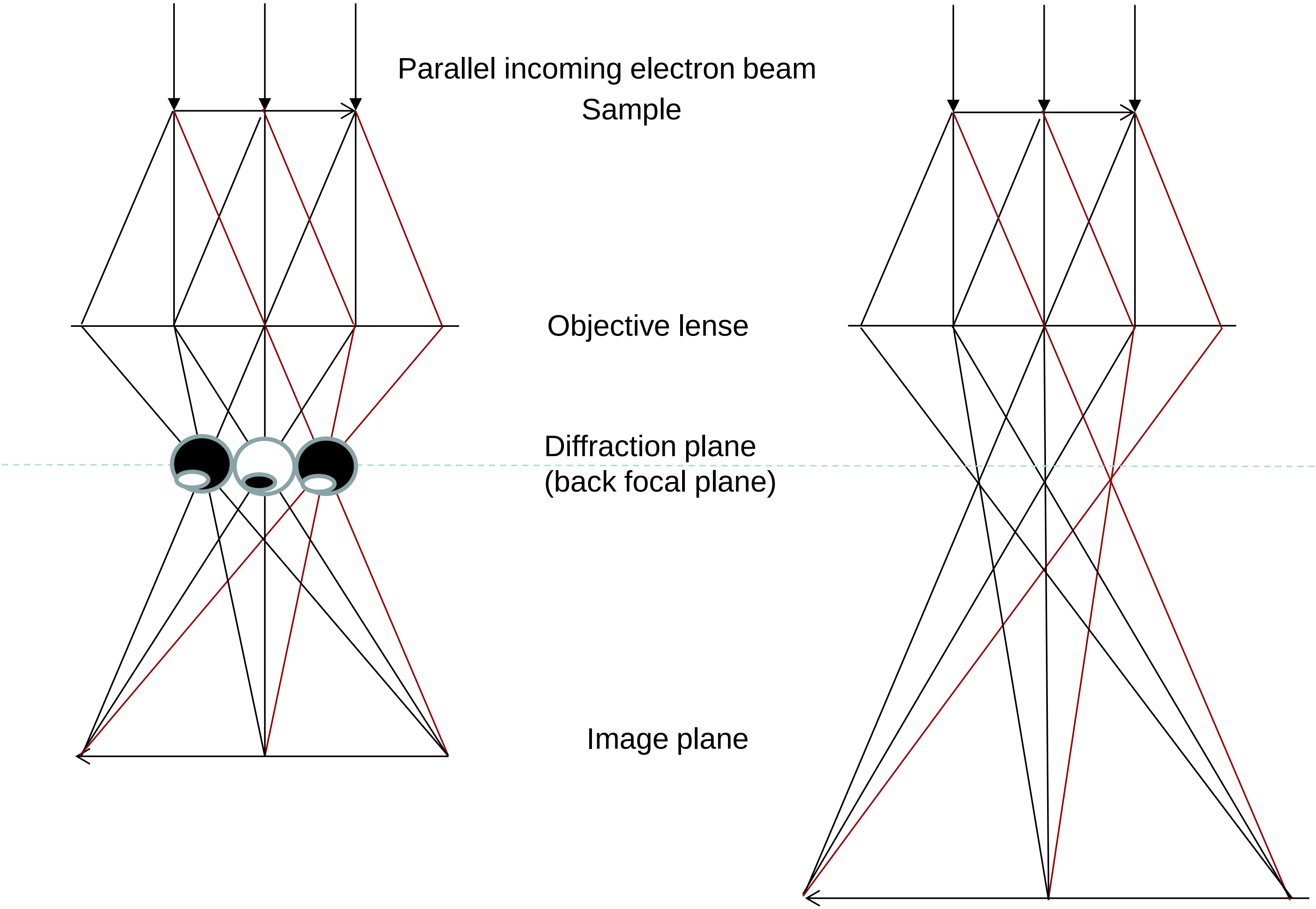
Diffraction information from an area with
~ same thickness and crystal orientation

Convergent beam
Illuminated area less than
the SAD aperture size.



µ-diffraction pattern

Shadow imaging (diffraction mode)



Parallel incoming electron beam

Sample

Objective lense

Diffraction plane
(back focal plane)

Image plane

Specimen holders and goniometers

- **Specimen holders**
  - Single tilt holders
  - Double tilt holders
  - Rotation holders
  - Heating holders
    - Up to 800ºC
  - Cooling holders
    - N: -100 - -150ºC
    - He: 4-10K
  - Strain holders
  - Environmental cells

- **Goniometers:**

- Side-entry stage

- Most common type
- Eucentric

- Top-entry stage

- Less obj. lens aberrations
- Not eucentric
- Smaller tilting angles

Next Lecture

- TEM Specimen Preparation

(Text book Chapters: 10)

Learning outcome

- HMS awareness
- Overview of common techniques
- Possible artifacts
- You should be able to evaluate which technique to use for a given sample

- Lab will give you some practical skills

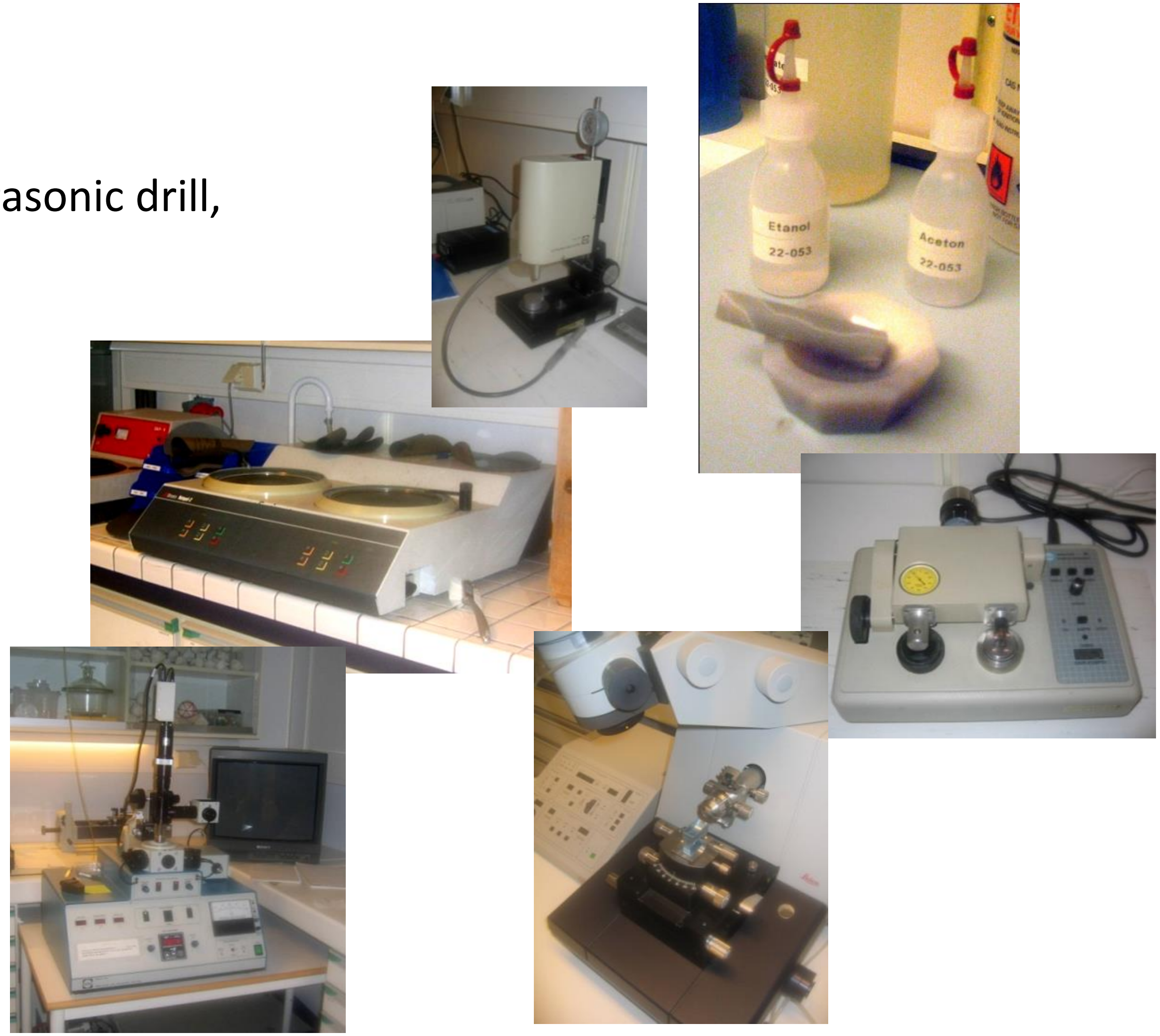
What to consider before preparing a TEM specimen

- 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 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.**



SAFETY!!!!

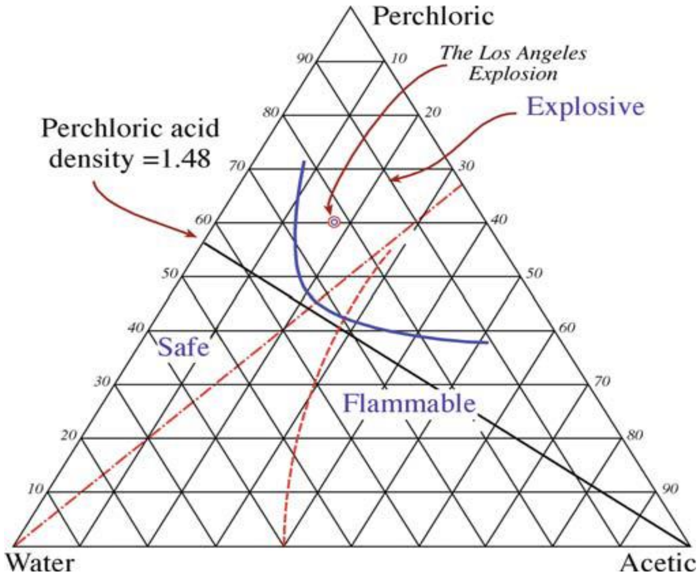
- Know what you handling.
  - MSDS
- Protect your self and others around you.
  - Follow instructions
- If an accident occurs, know how to respond.

Safety rules

- Be sure that you can safely dispose of the waste product before you start.
- Be sure you have the ‘antidote’ at hand.
- Never work alone in the specimen-preparation laboratory.
- Always wear safety glasses when preparing specimens and/or full protective clothing, including face masks and gloves, if so advised by the safety manual.
- Only make up enough of the solution for the one polishing session. Never use a mouth pipette for measuring any component of the solution. Dispose of the solution after use.
- Always work in a fume hood when using chemicals.
- Check that the extraction rate of the hood is sufficient for the chemical used.

Some acids for specimen preparation

- Cyanide solutions:
  - DO NOT USE
- Perchloric acid in ethanol or methanol
  - Ole Bjørn will make the solution if needed



- Nitric acid (HNO_3)
  - Can produce explosive mixtures with ethanol.
- Hydrofluoric acid (HF)
  - Penetrates flesh and dissolves bones rapidly!

You need to have approval by supervisors and Ole Bjørn first!

Work in the Stucture Physics lab

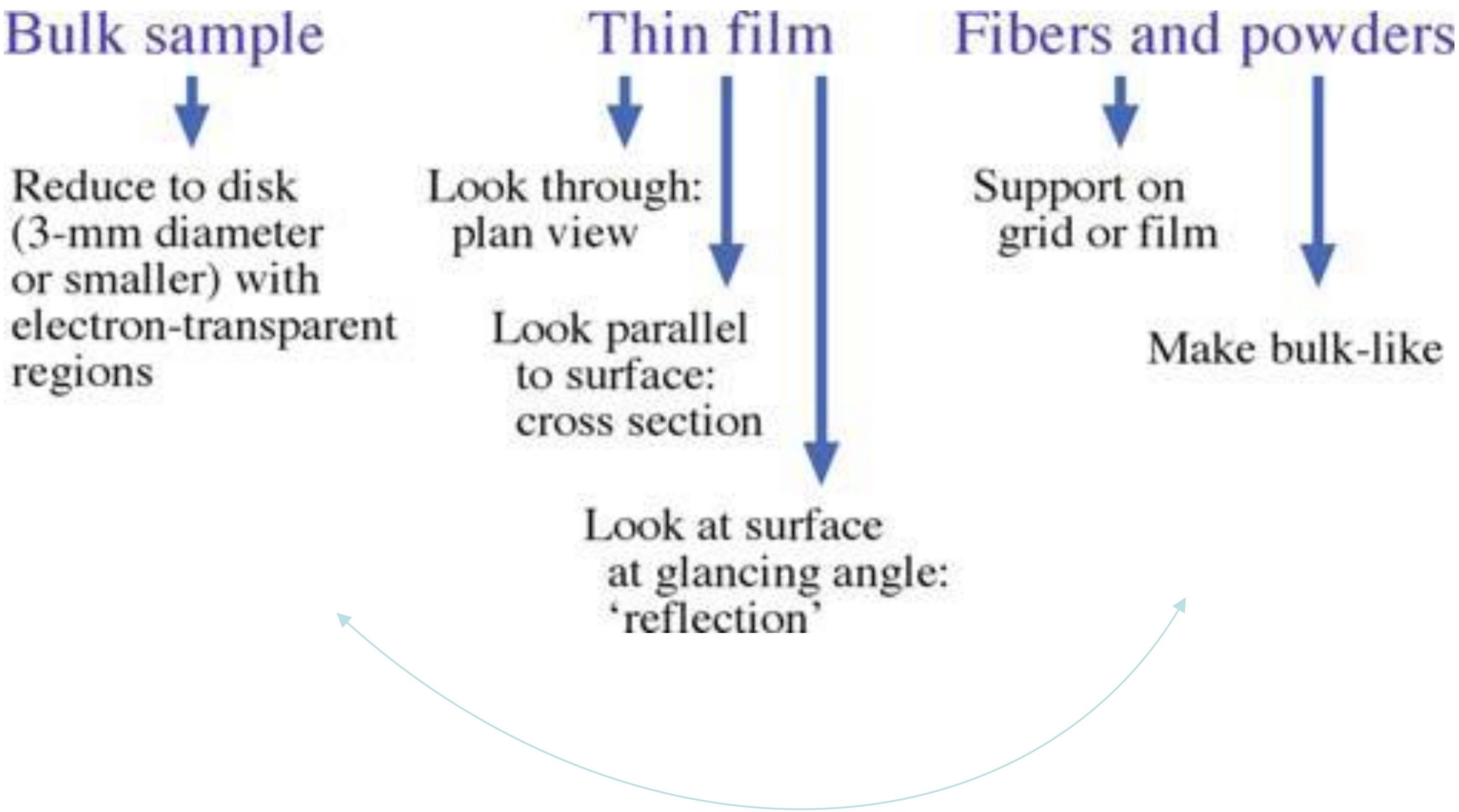
- Get the local HMS instructions from

  Ole Bjørn Karlsen

Ask

Sign a form confirming that you have got the information

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

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

Self-supporting disk or grid

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

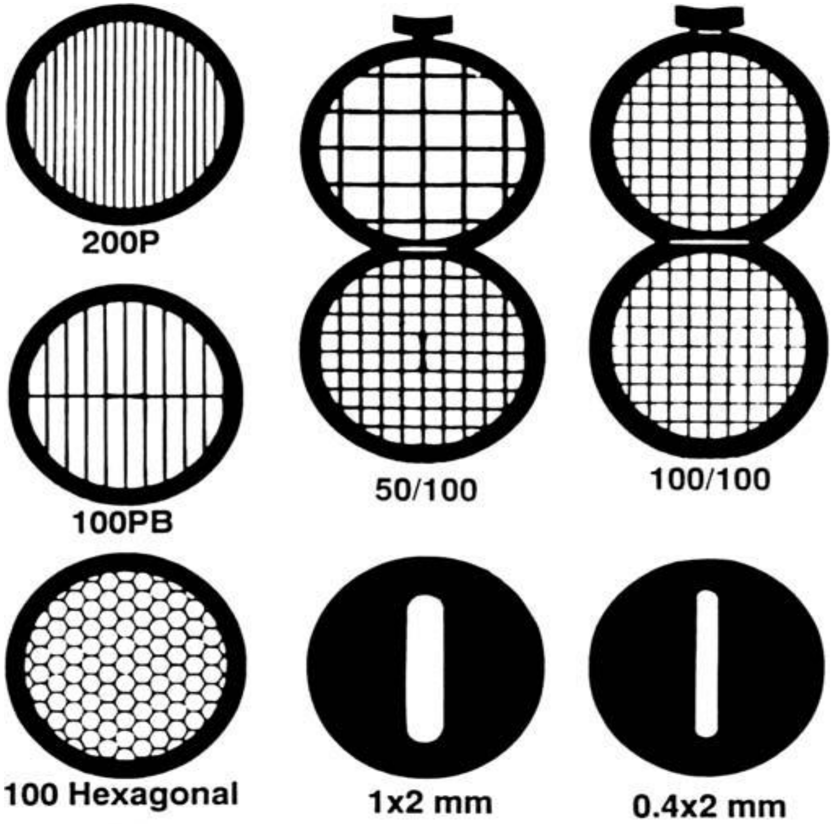
If brittle, consider Cu washer with a slot

- Grid
  - Several types (Fig. 10.3)
  - Different materials (Cu, Ni…)
  - Support brittle materials
  - Support small particles

The grid may contribute to the EDS.

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

Grids and washers used as specimen support



May contribute to the EDS signal.

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

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

Self-supporting disk or grid

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

If brittle, consider Cu washer with a slot

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

Preparation of self-supporting discs

- Cutting/cleaving
  - Ductile material or not?

Cutting and cleaving

Cutting with a saw:

Soft or brittle material?

Brittle materials with well-defined cleavage plane

- Si
- GaAs
- NaCl
- MgO

Razor blade or ultramicrotome

Preparation of self-supporting discs

- Cutting/cleaving
  - Ductile material or not?
- Grinding
  - 100-200 µm thick
  - polish
- Cut the 3mm disc

Cutting a 3 mm disc

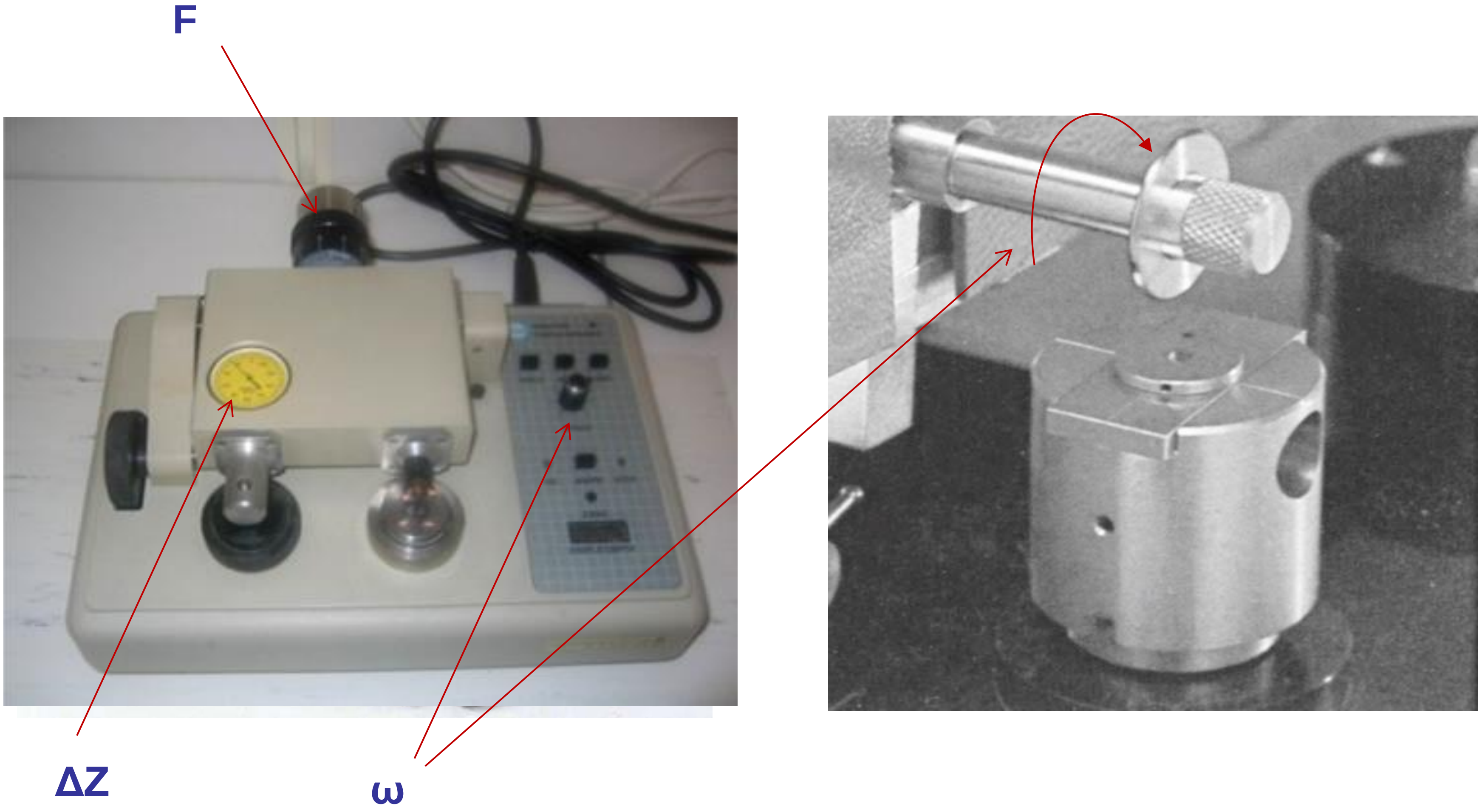
Soft or brittle material?
Mechanical damage OK?

Brittle: Spark erosion, ultrasonic drill, grinding drill

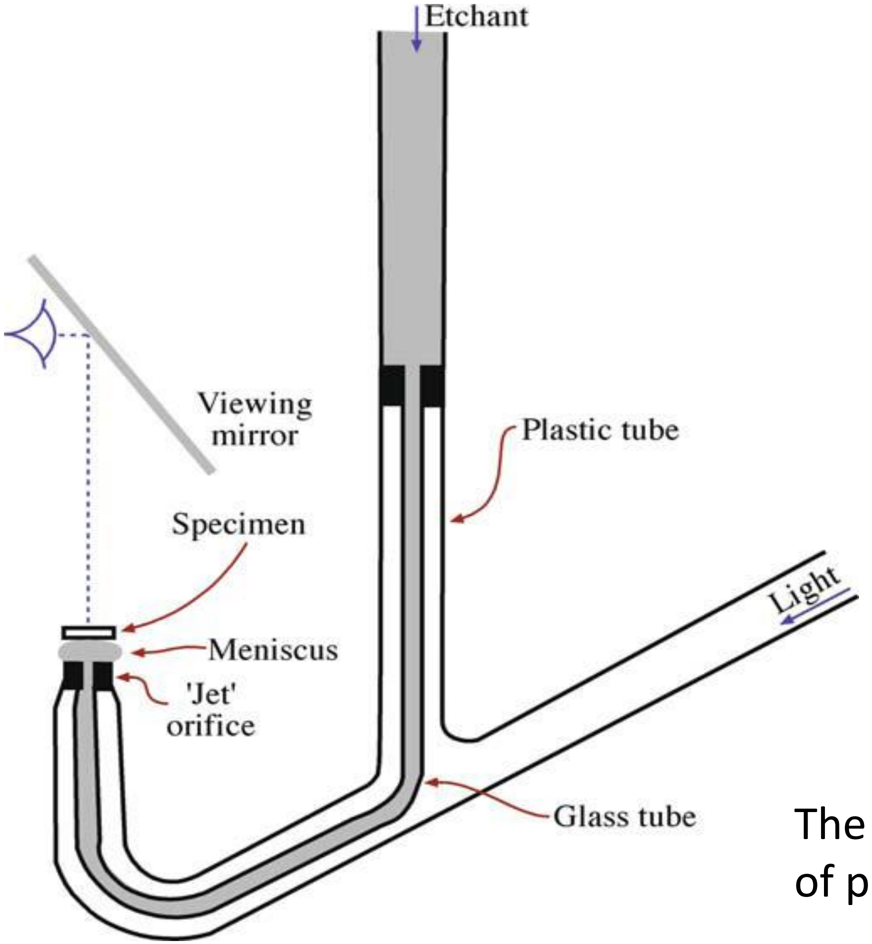
Preparation of self-supporting discs

- Cutting
  - Ductile material or not?
- Grinding
  - 100-200 μm thick
  - polish
- Cut the 3mm disc
- Prethinning
  - Dimpling
  - Tripod polishing

Dimpling

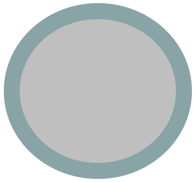


Surface dimpling using a chemical solution



Si: HF + HNO3

GaAs: Br + methanol

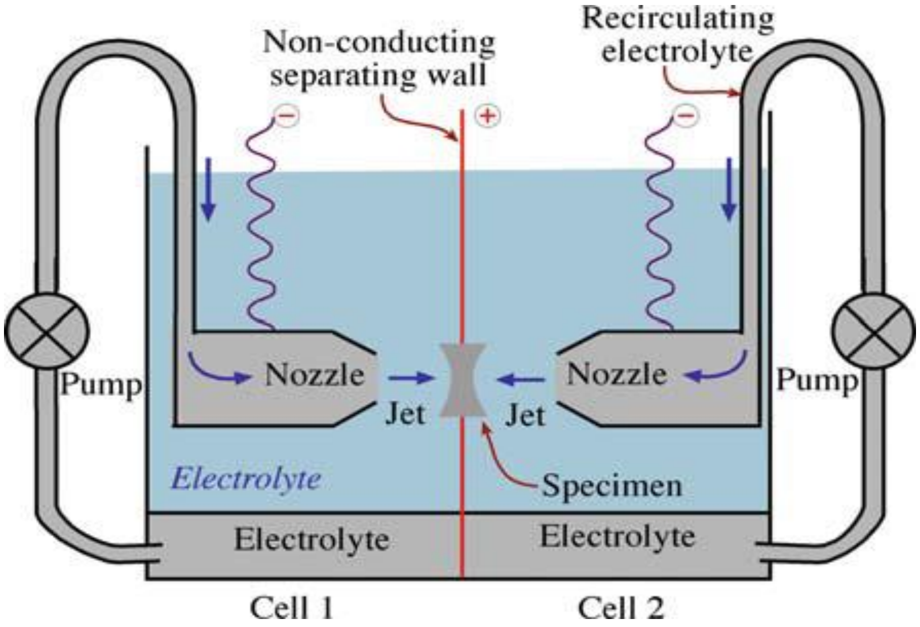


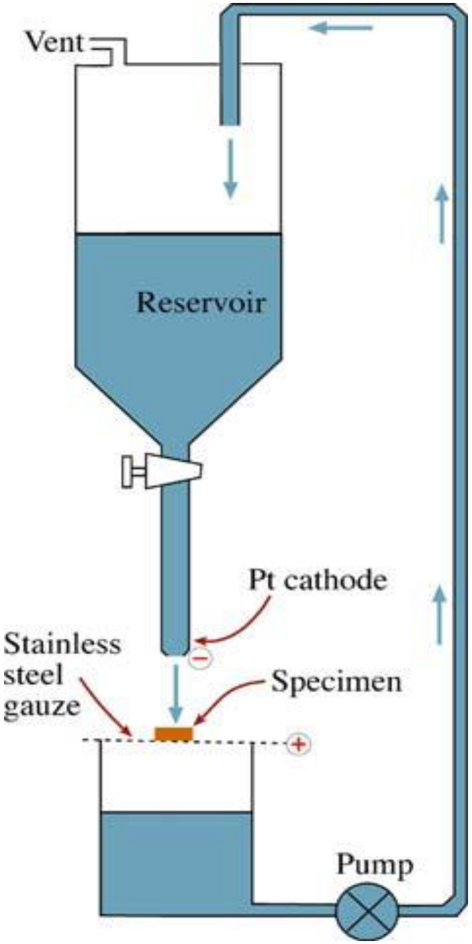
The light pipe permits visual detection of perforation using the mirror.

Final thinning of the discs

- Electropolishing
- Ionmilling

Jet polishing

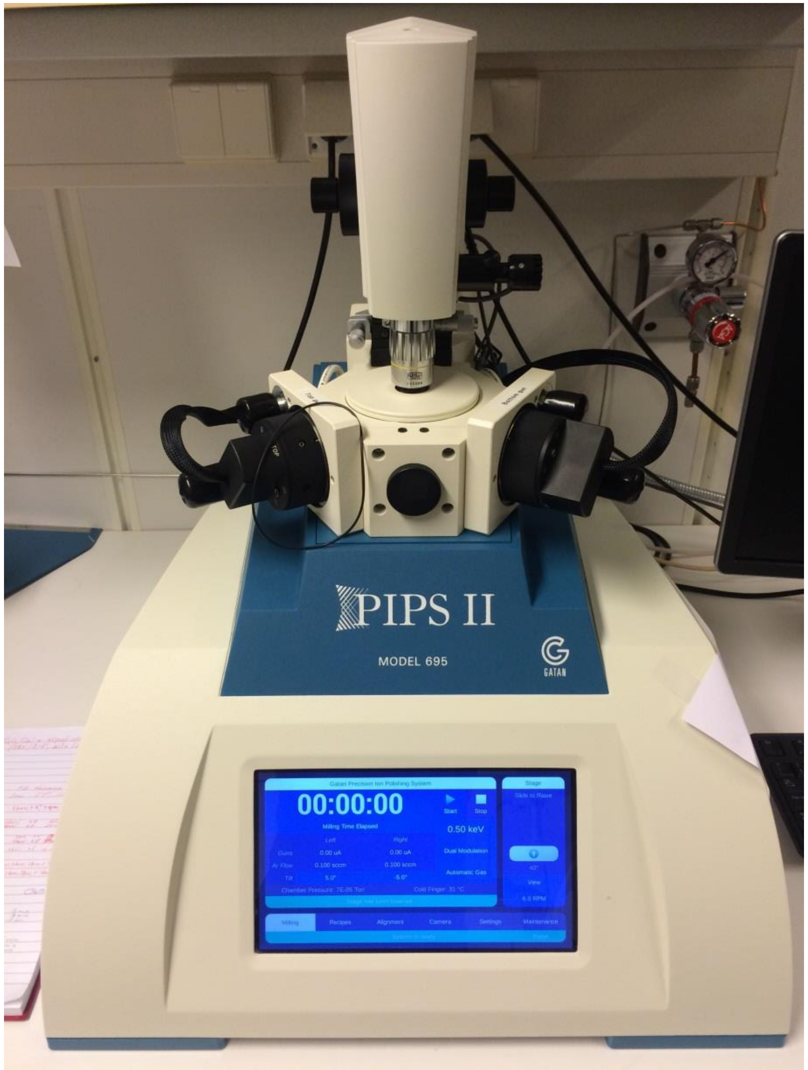


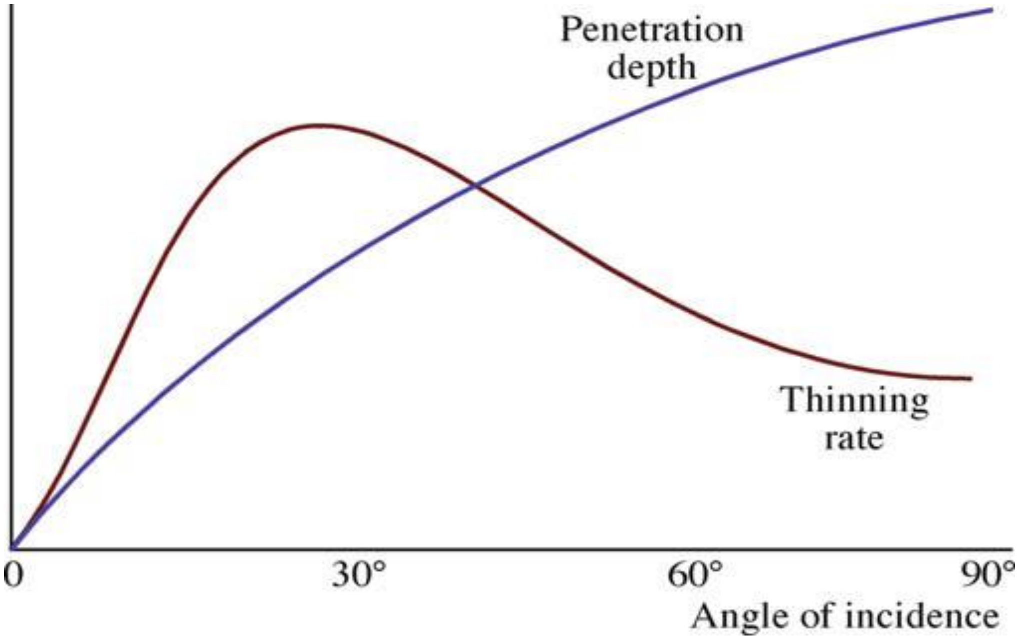


Twin-jet electropolishing apparatus.
The positively charged specimen is held in a Teflon holder between the jets. A light pipe (not shown) detects perforation and terminates the polishing.

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

Ar ion beam thinning



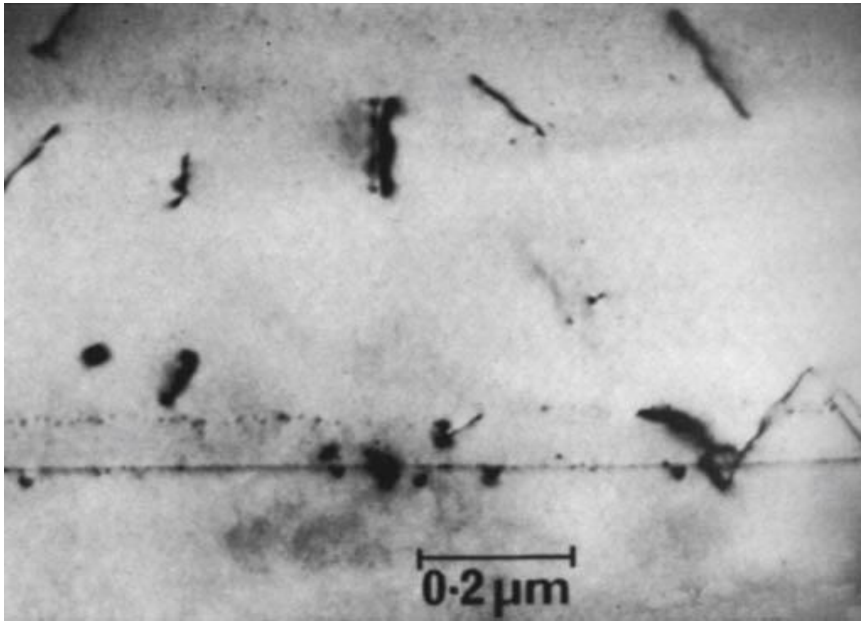


Variation in penetration depth and thinning rate with the angle of incidence.

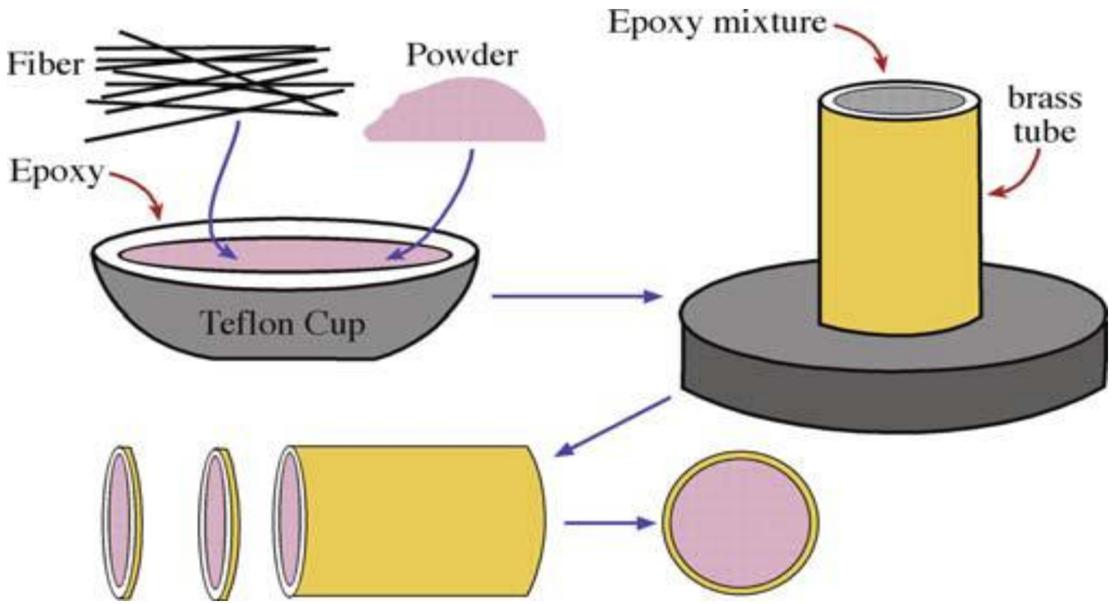
Effect of Ar-thinning on CdTe

Defects (dark spots) in Ar-thinned specimen

Crystal thinned by reactive iodine
ion milling.



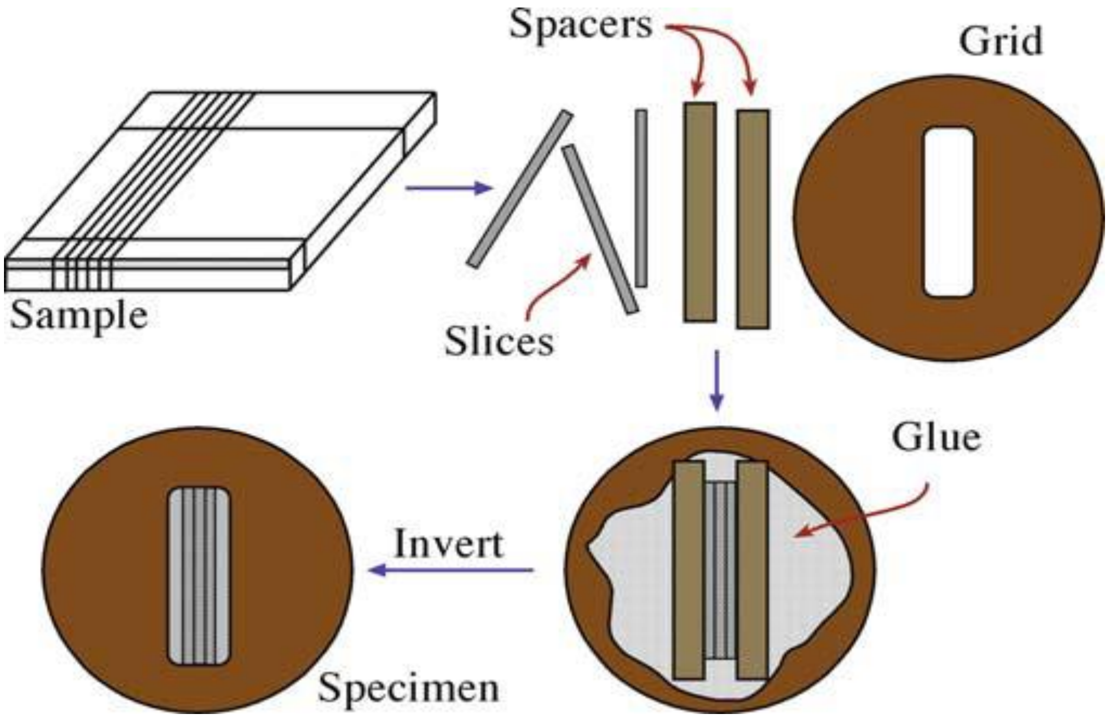
Preparation of **particles and fibers**



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.

THIN FILMS TEM specimen preparation

Initial preparation steps



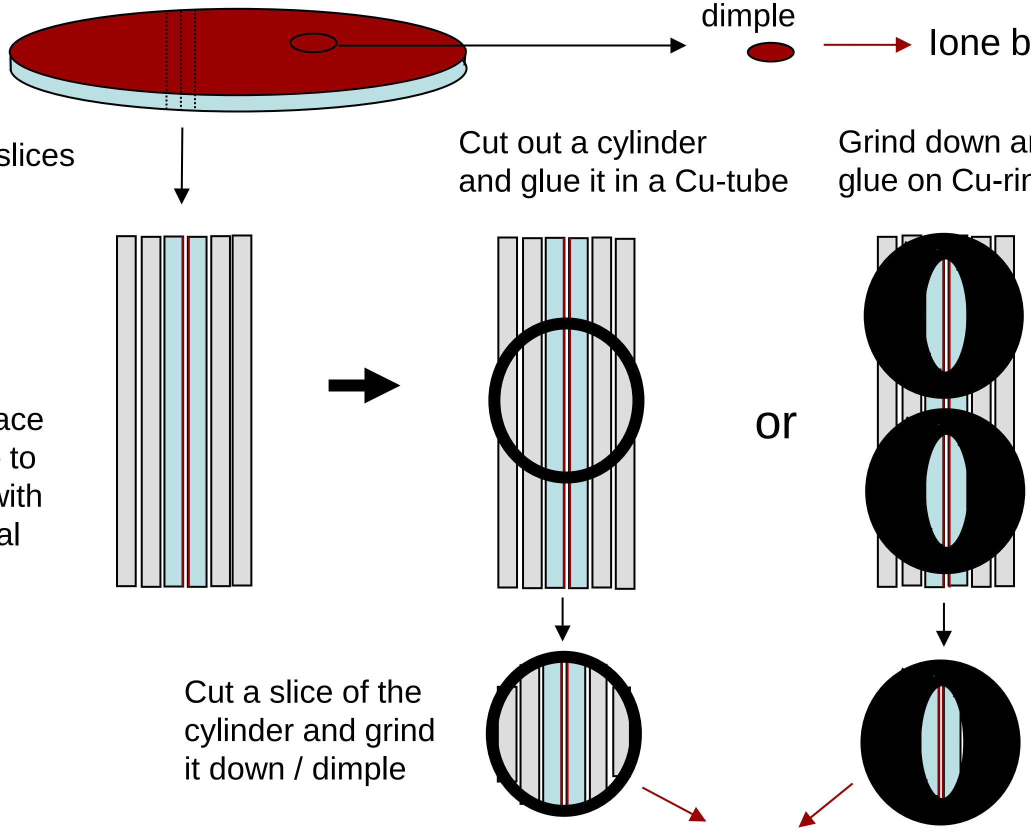
Spacers : Si, glass, or some other inexpensive material.

THIN FILMS TEM specimen preparation

- **Top view**
- **Cross section**

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

- **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

or

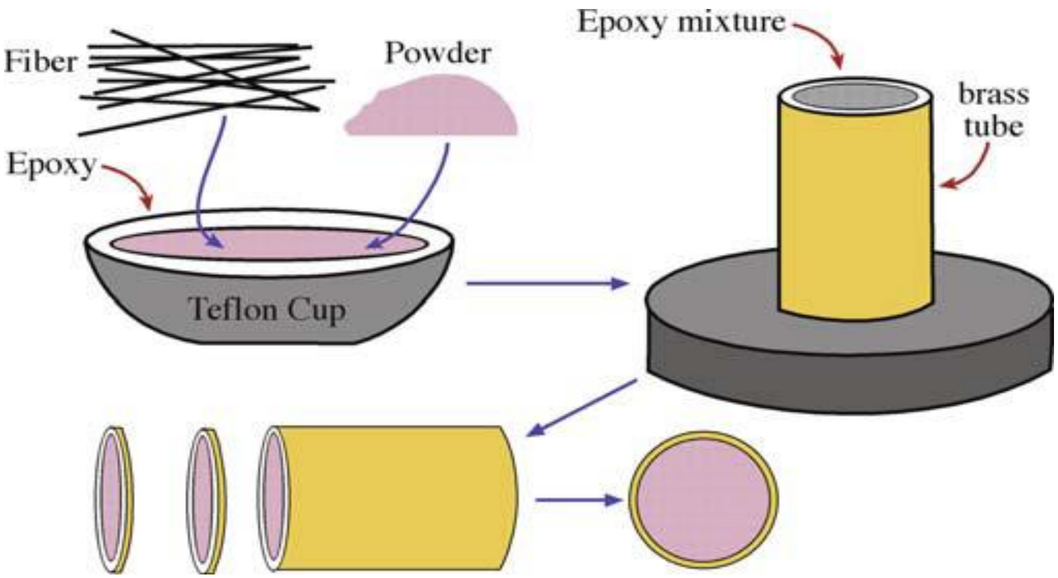
Cut a slice of the cylinder and grind it down / dimple

Cut off excess material

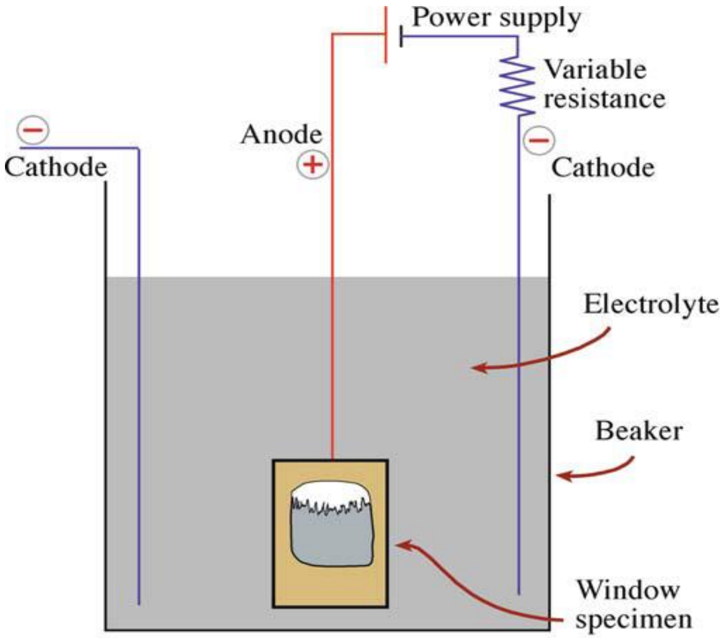
Ione beam thinning

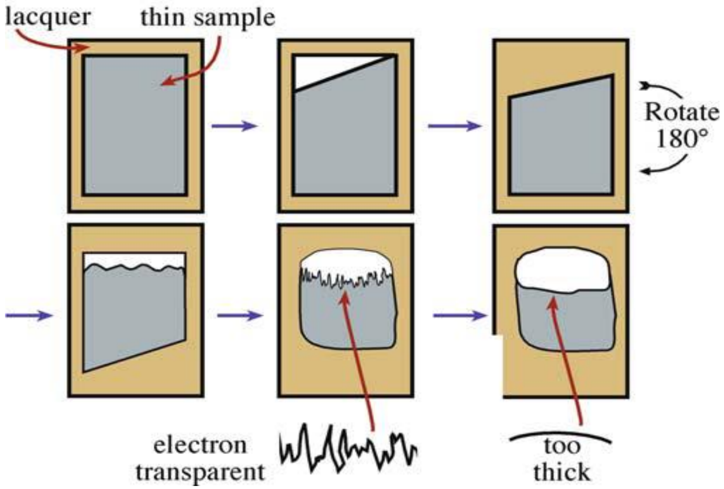
Specimens on grids/washers

- Electropolishing
  - The window method
- Ultramicrotomy
- Crushing
  - In ethanol
  - Mix in an epoxy
- Replication and extraction
- Cleaving and SACT
- The 90^o wedge
- Lithography
- Preferensial chemical etching



Window polishing

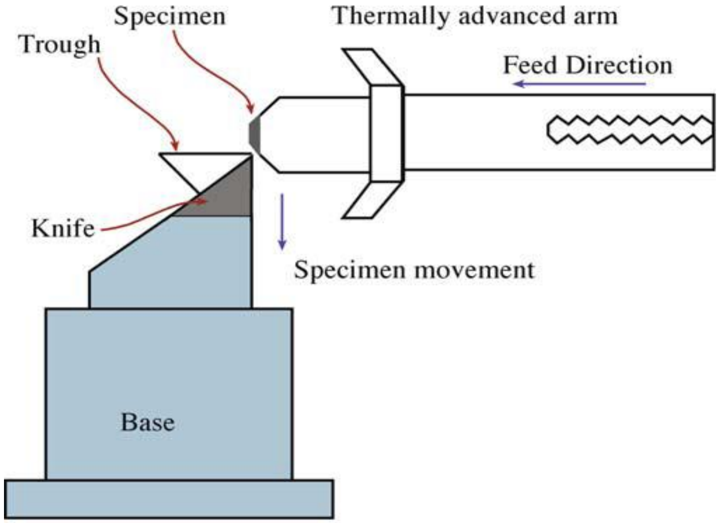




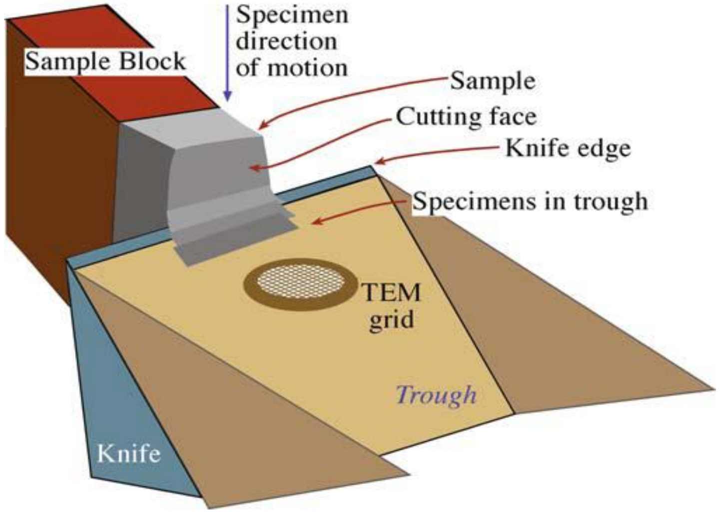
- A sheet of the metal 100mm^2 is lacquered around the edges and made the anode of an electrolytic cell.

- Progress during thinning: the initial perforation usually occurs at the top of the sheet; lacquer is used to cover the initial perforation and the sheet is rotated 180^o and thinning continues to ensure that final thinning occurs near the center of the sheet.

Ultramicrotomy

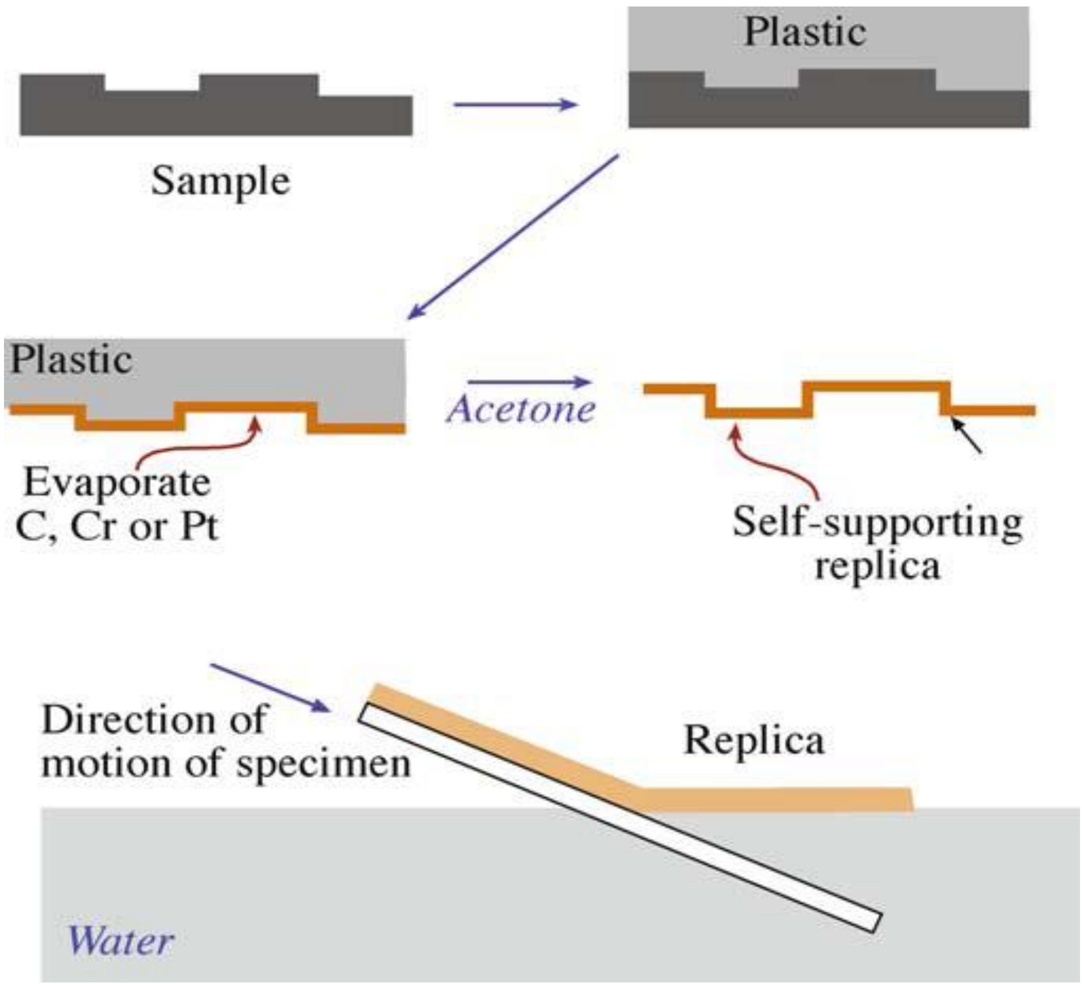


The sample is first embedded in epoxy or some other medium or the whole sample is clamped and moved across a knife edge.



The thin flakes float off onto water or an appropriate inert medium, from where they are collected on grids.

Replication of a surface



1) Spray acetone on the surface to be replicated before pressing a plastic
(usually cellulose acetate)

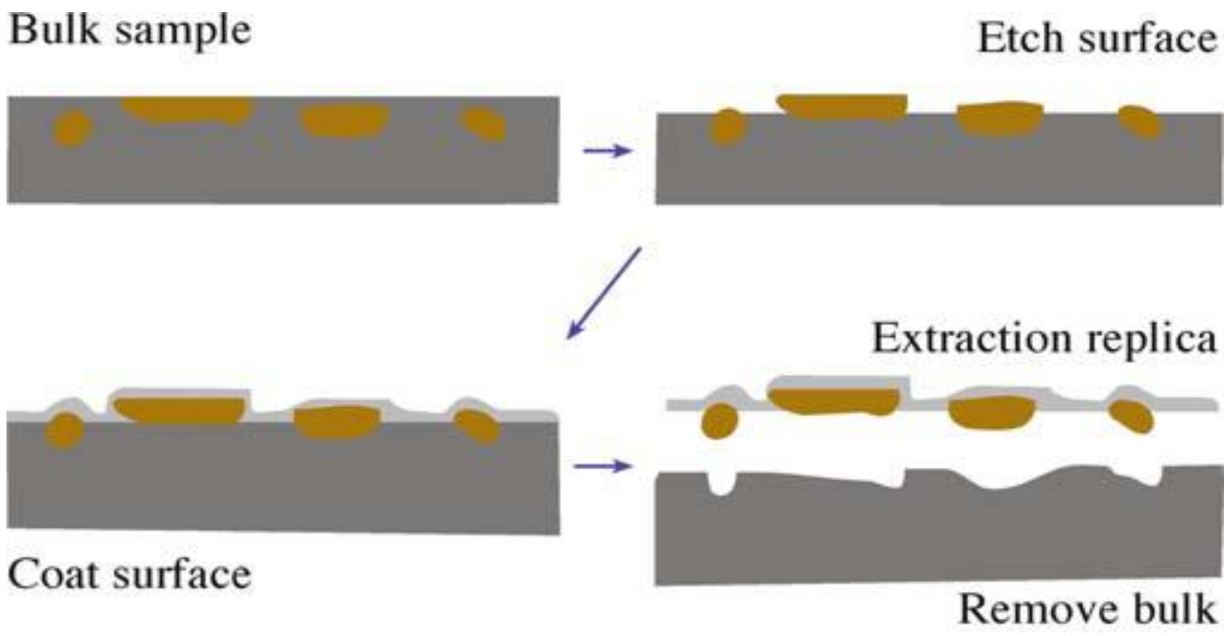
2) Removed the plastic from the surface when hardened

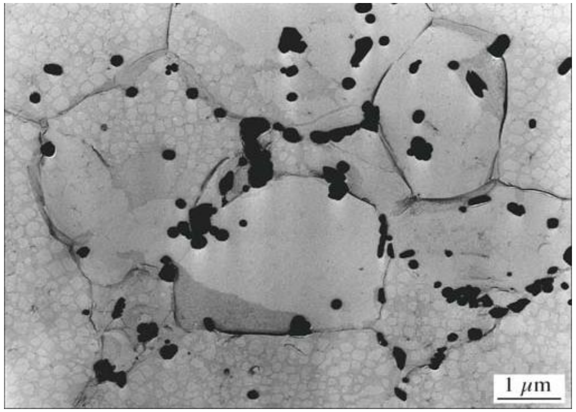
3) Evaporate a C, Cr, or Pt film onto the replicated plastic surface.

4) Dissolve the plastic with acetone

Alternatively: the direct carbon replica.

Extraction replication

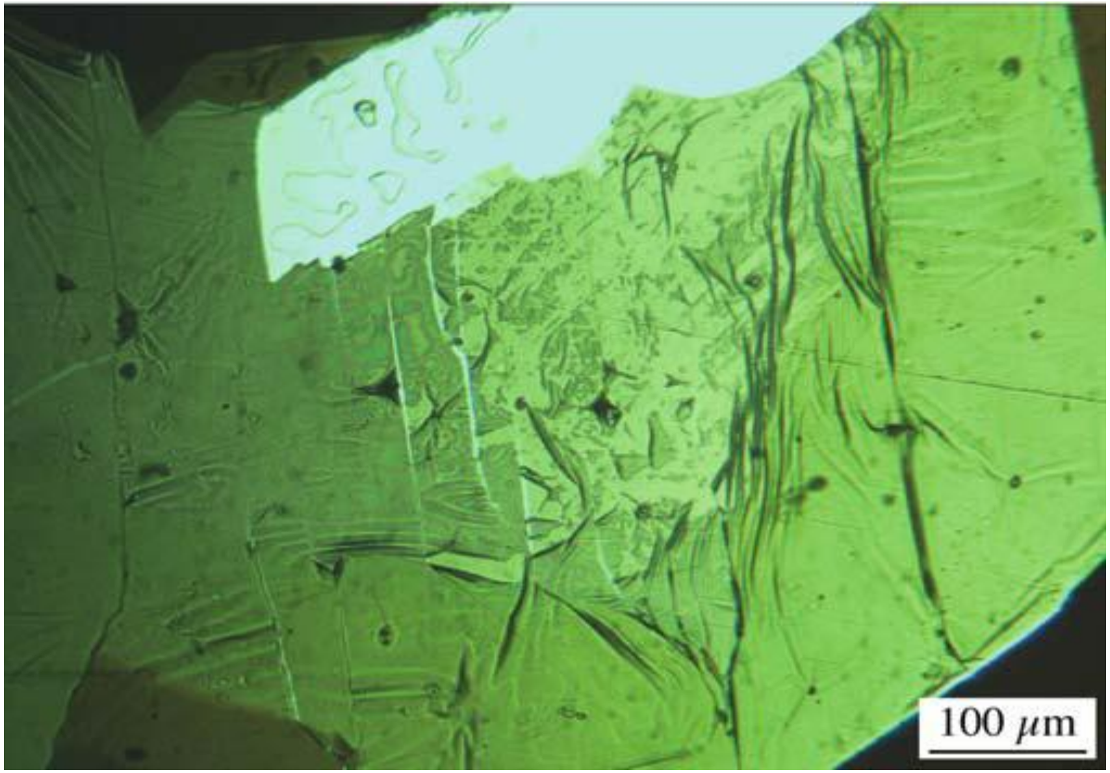




A thin amorphous carbon film is evaporated over the particles

The rest of the matrix is etched

Cleaving



1) Use tape

2) Dissolve tape in a solvent

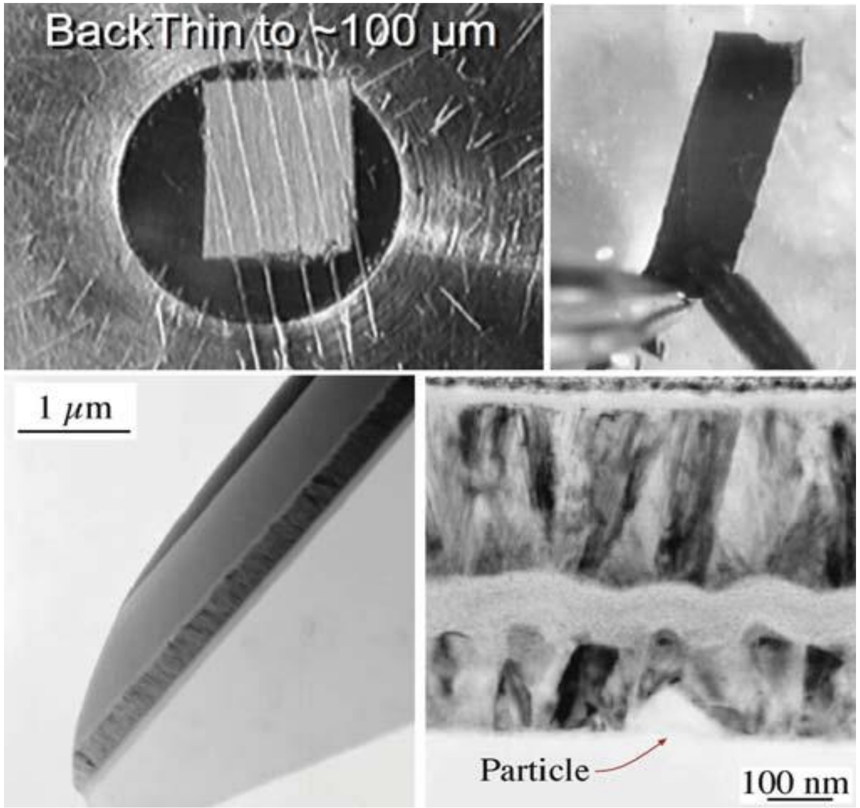
Cleaved MoS_2 showing regions of different shades of green, which correspond to different thicknesses.

SACT

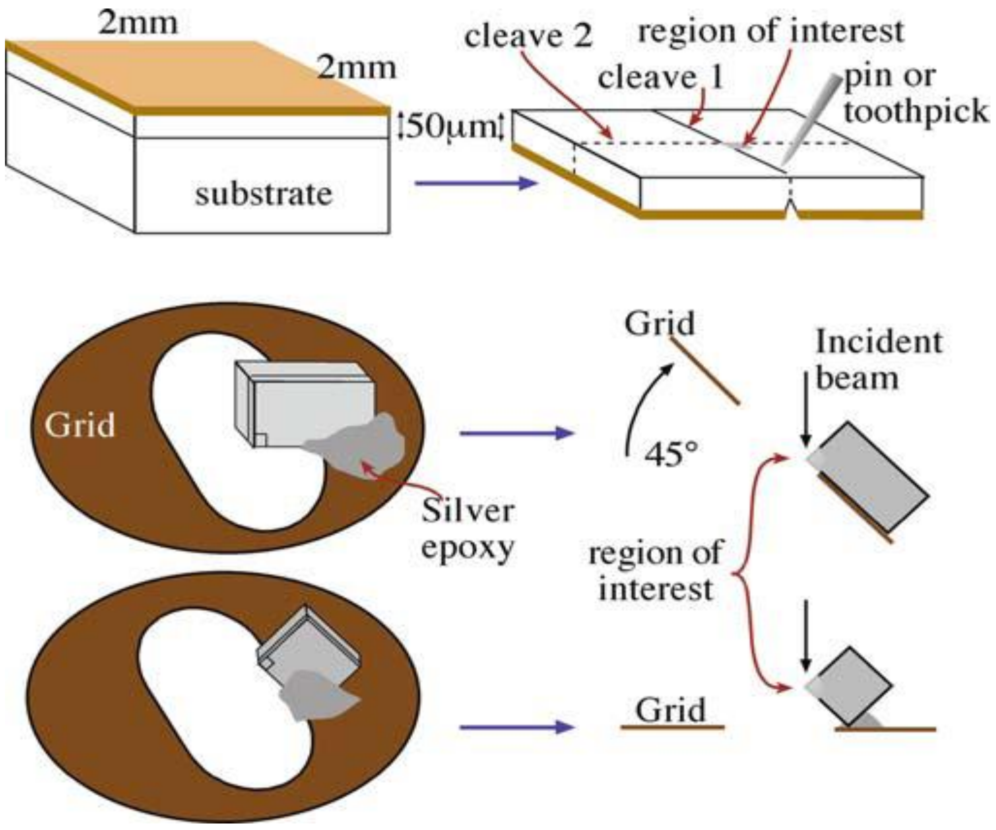
The small-angle cleaving technique

Invaluable for films on Si or glass where there is no crystal structure

1. Scratch the sample;
2. Cleaving along the scratch;



LACT- The 90° wedge

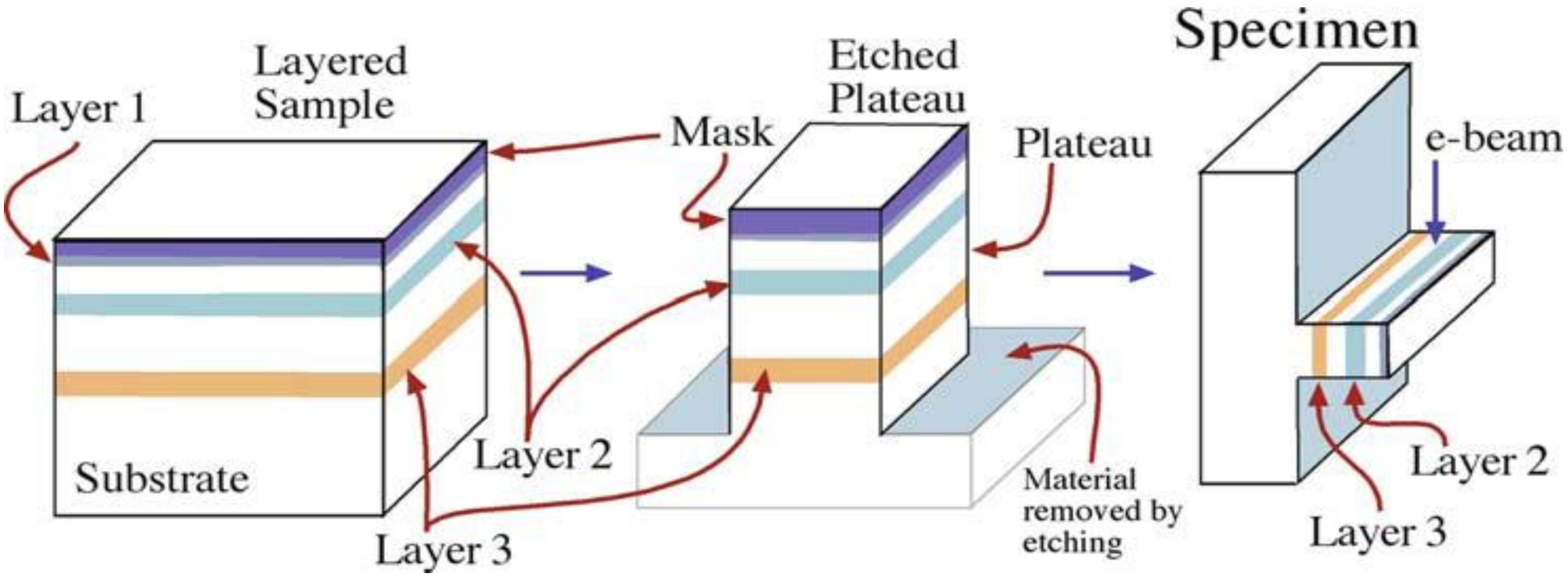


1) Prethin: 2-mm square of the multilayers on a Si substrate

2) Scribe the Si through the surface layers, turn over, and cleave
Need: a sharp 90° edge;

3) Mount the 90° corner

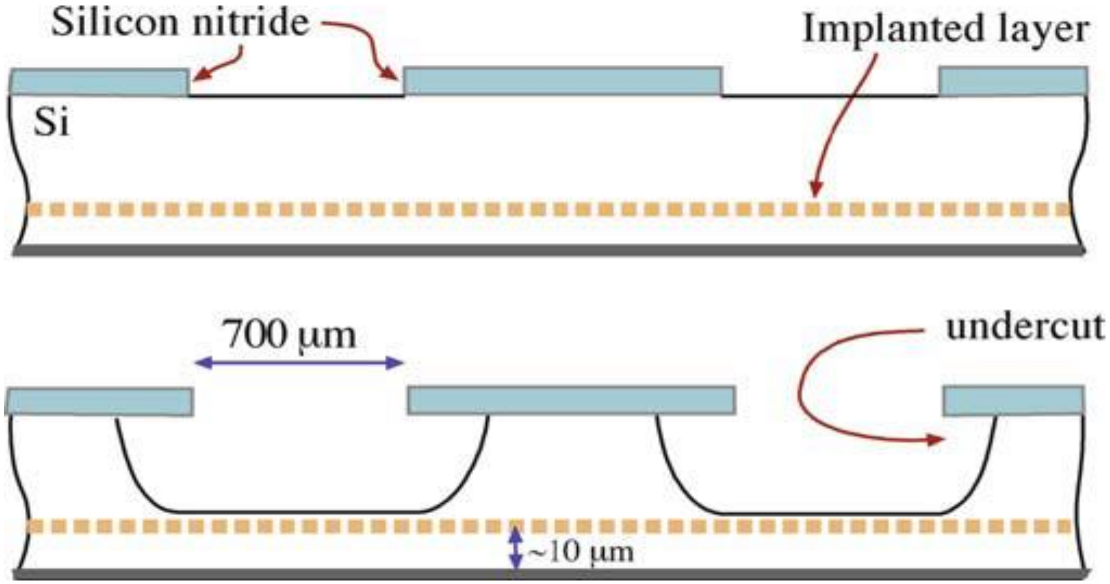
Preferential chemical etching

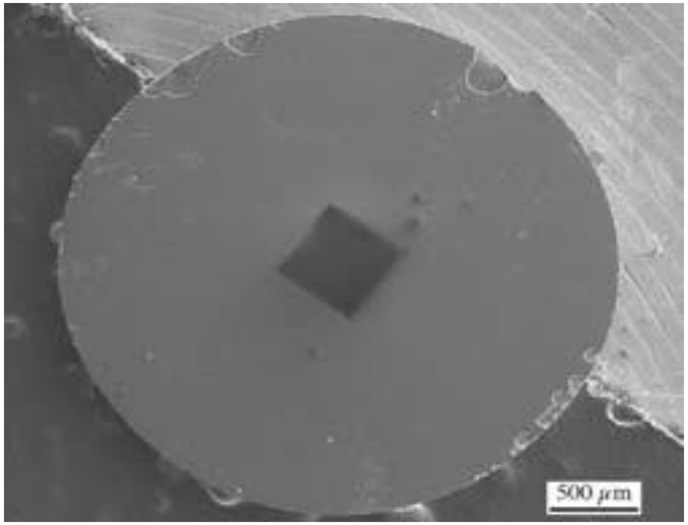


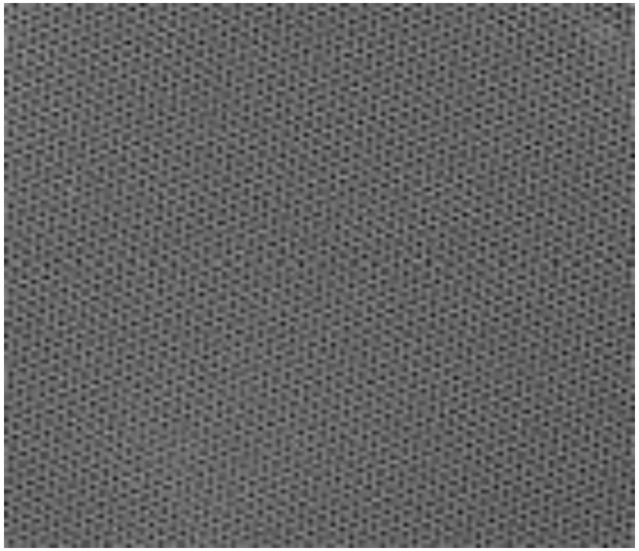
Etch away most of the sample, leaving a small etched plateau
Mask a region <50 nm across and etch away the majority of the surrounding plateau.

Turn 90o and mounted in a specimen holder

Lithographic techniques

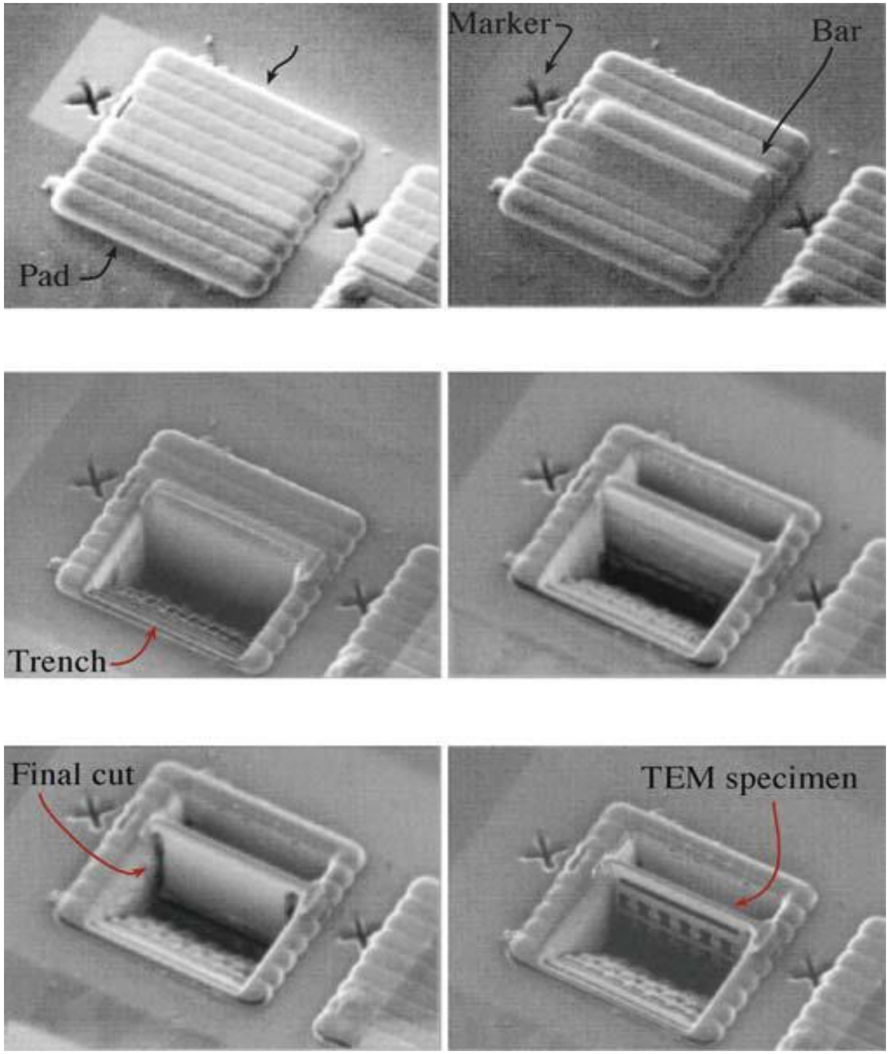


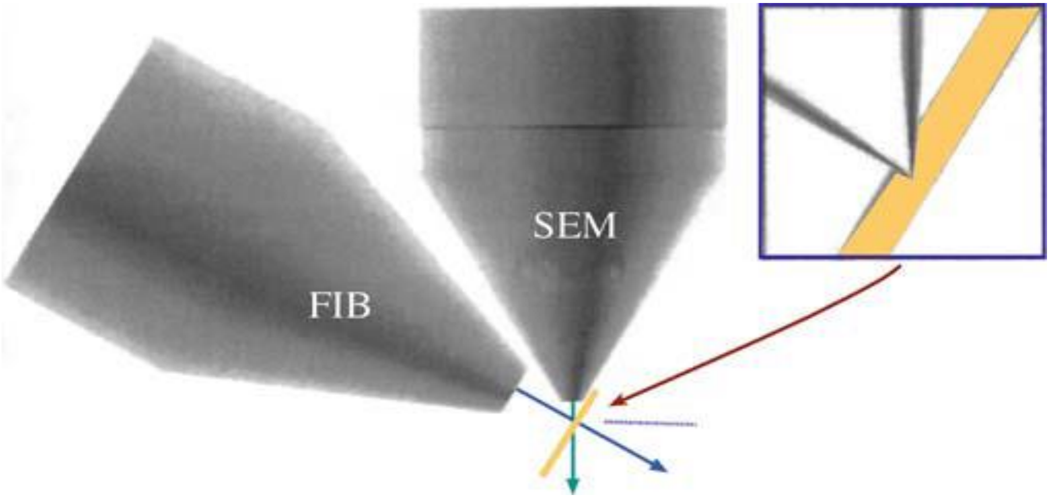




Etching between the barrier layers
Produces an undercutting down to
the implanted layer which acts as
an etch stop, producing a uniform
layer 10 mm thick.

FIB

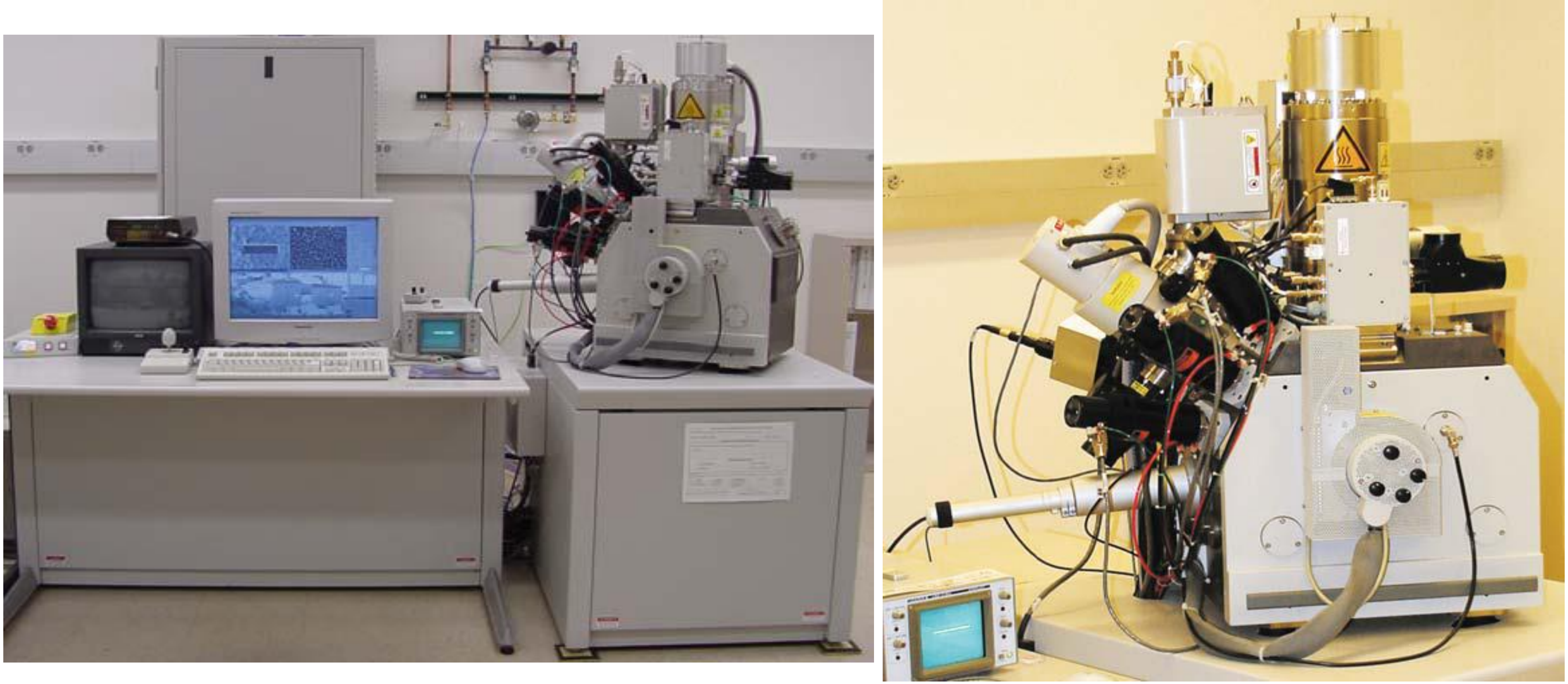




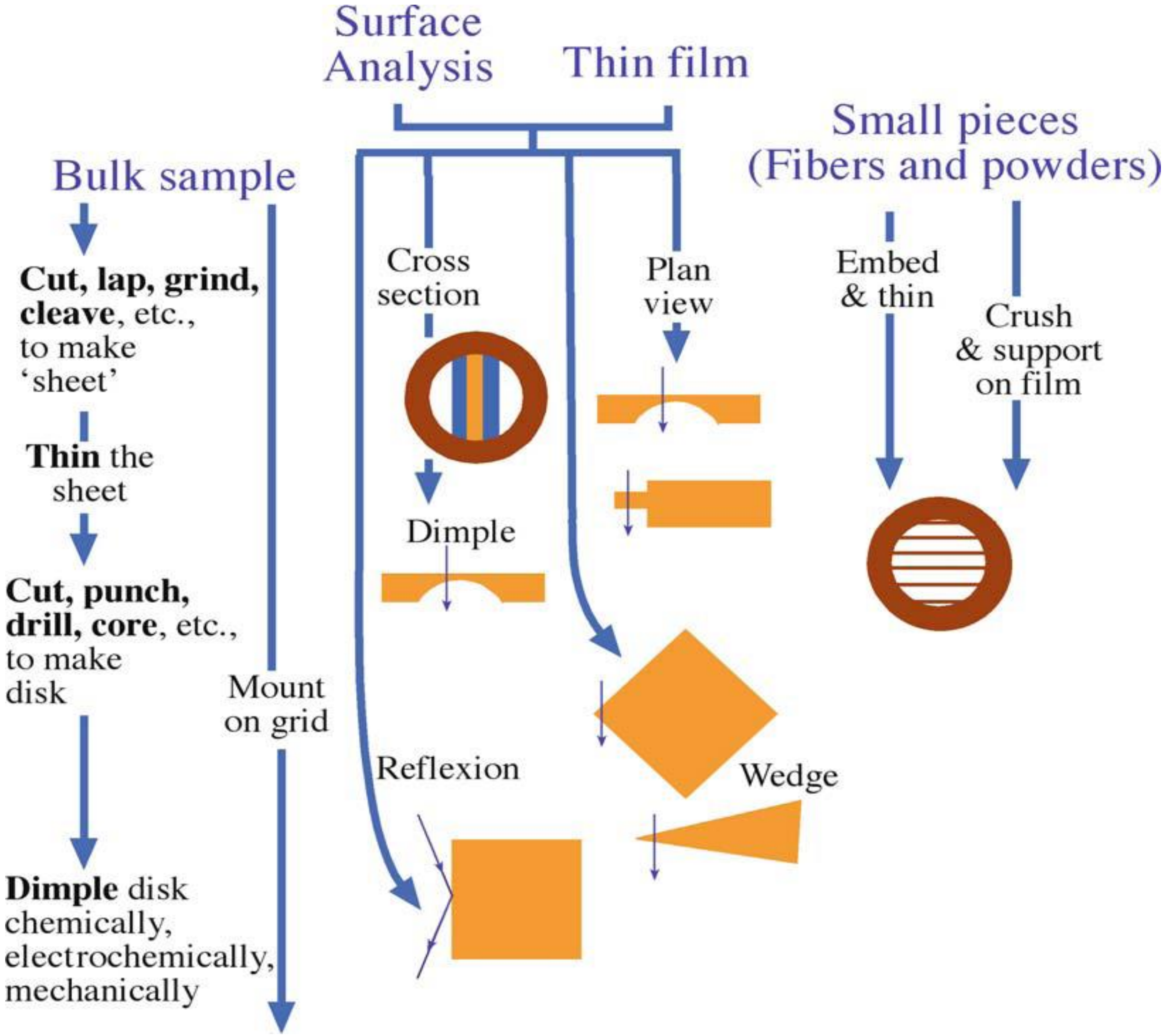
Schematic of a two-beam (electron and ion) FIB instrument.

-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.

A dual-beam FIB instrument.



Summary flow chart for specimen preparation



Surface Analysis

Thin film

Small pieces (Fibers and powders)

Bulk sample

Cut, lap, grind, cleave, etc., to make 'sheet'

Thin the sheet

Cut, punch, drill, core, etc., to make disk

Dimple disk chemically, electrochemically, mechanically

Mount on grid

Cross section

Dimple

Reflexion

Plan view

Wedge

Embed & thin

Crush & support on film

THERE WILL BE TEM COURSE LAB THIS THURSDAY

***

Next Lecture

- Introduction to Crystallography

by

Patricia Almeida Carvalho

Senior Research Scientist

SINTEF