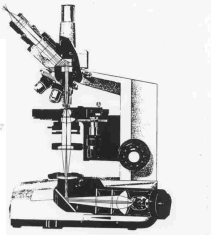
Transmission Electron Microscopy

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2.1. DEFINITIONS



a) "Biological" type of compound microscope.[1b] Courtesy of Olympus Cor. Diagram pertaining to Olympus microscope shown in Figure 1.1a.[1b] Courtesy o Corporation.[1c]

Microscopy is the interpretive use of microscopes.[1,1a,2] No matter what kind of microscope, employing whatever medium, in whatever manner, with whatever kind of specimen, microscopy also requires a *primary* observer to interpret the image. Accordingly, microscopy can be interpreted as science,[2a] art,[3,3a] or a game—what is it?

Interpretation requires both a sharp eye and an active brain.[1a] To-

A *microscope* is an instrument that increases resolution over that of the human eye alone (about 150 μm between two points or lines). *Resolution* is the distance between two specific points or parts of the object as viewed by the eye, microscope, camera, or video. Actually resolution is the revelation of the two points or lines on two individual receptors (rods and cones),[4,5]

Resolving power is the ability to distinguish two points of an object as separate in an image (with their diffraction discs not overlapping more

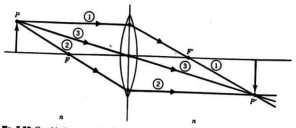


Fig. 3.30 Graphical construction for a thin lens with the refractive index on the incident side equal to that on the transmitting side.

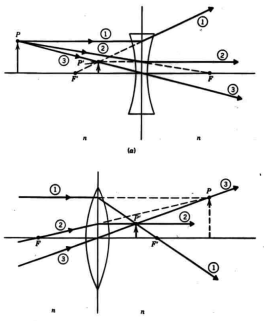


Fig. 3.31 (a) Virtual image construction. (b) Virtual object construction.

Lens Equation

$$\frac{1}{x} + \frac{1}{y} = \frac{1}{F}$$

Magnification

$$M = P / P'$$

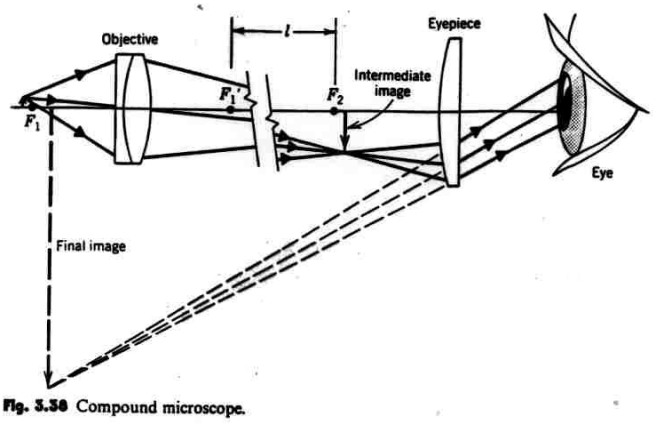


Fig. 3.38 Compound microscope.

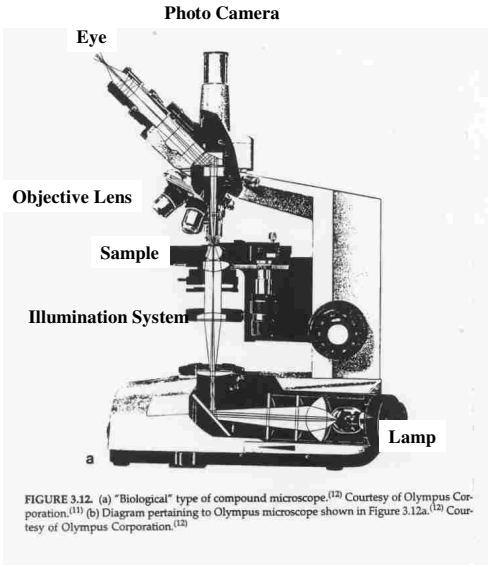
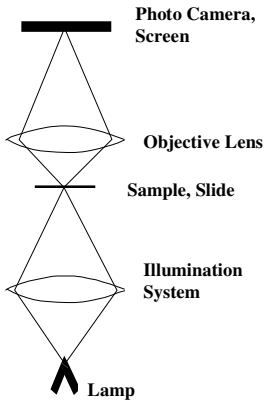
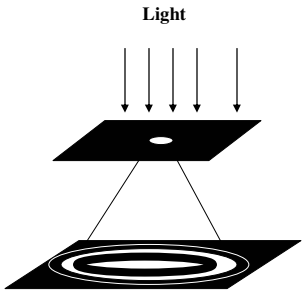


FIGURE 3.12. (a) "Biological" type of compound microscope.[12] Courtesy of Olympus Corporation.[11] (b) Diagram pertaining to Olympus microscope shown in Figure 3.12a.[12] Courtesy of Olympus Corporation.[12]



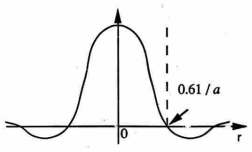


For a circular aperture of diameter a

$t(xy) = (\pi a^2 / 2)\ J_1(\pi a r) / (\pi a r)$ J_1 first order Bessel f.

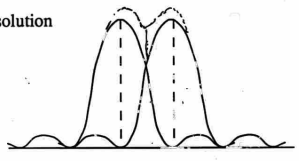
$r = (x^2 + y^2)^{1/2}$

for a point source, the image would be a disk (airy-disk) of radius $1.22 / a$





Rayleigh Criterion for resolution



The least resolvable distance

$$\Delta = 1.22\ \lambda / \alpha$$

Optical microscopy

Theoretical value of resolution $\approx \lambda / 2$

for visible light 2000-3000 Å

Electron microscopy

for electrons $\lambda \approx h / p$

$\lambda \approx (150 / E)^{1/2}$ λ in Å , E in V

For 100 keV electrons $\lambda = 0.037$ Å

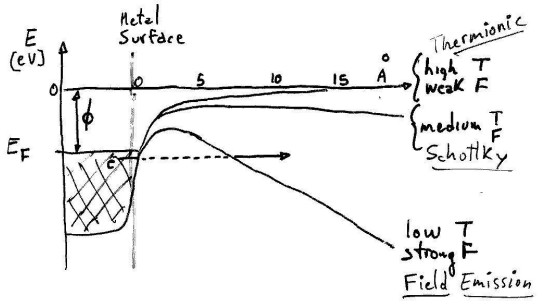
Typical resolution in EM 1-5 Å due to severe aberration from electron lenses.

angles, $\alpha \approx \lambda / d$

interatomic distances ≈ 1-2 Å

$\alpha \approx 2\ 10^{-2}$ mrad (≈ 1°)

Lamp -> Electron Gun



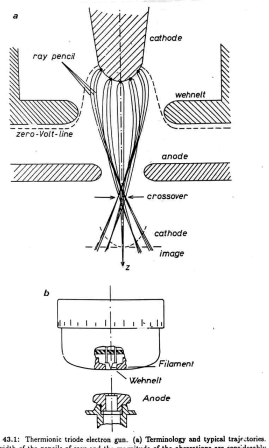


Fig. 42.1: Thermionic triode electron gun. (a) Terminology and typical trajectories. The width of the pencils of rays and the magnitude of the aberrations are considerably exaggerated. (b) A more realistic view (courtesy of Philips, Eindhoven).

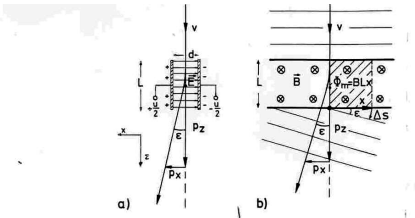
Gun Parameters

I	**current**
ΔE	**Energy Width (1.5 eV THER, 0.5 FEG)**

$\beta = I / (\Delta S\ \Delta\Omega)$ **Brightness**

Stability, Life Time, Vacuum
Price

Controlling Electron Movement



Example 100 kV e^- deflect $\varepsilon = 0.1$ rad $\sim 5°$
$L = 1$ cm $d = 2$ mm
$\Rightarrow |E| = 2 \cdot 10^4$ V/cm
$\Rightarrow$ $U = 4000$ V

magnet N turns, current I, slit d
$B = \mu_0 \frac{NI}{d} \Rightarrow 5° \rightarrow B = 10^{-2}$ T
$N = 100 \Rightarrow I = 0.2$ A
$NI = 20$ A

$$p_x = \int_0^T eE\,dt = \frac{e}{v}\int_0^L E\,dz = \frac{eEL}{v}$$

$$p_x = \int_0^T evB\,dt = eBL$$

Deflection ε

$$\varepsilon = \frac{p_x}{p_z} = \frac{eEL}{mv \cdot v}$$

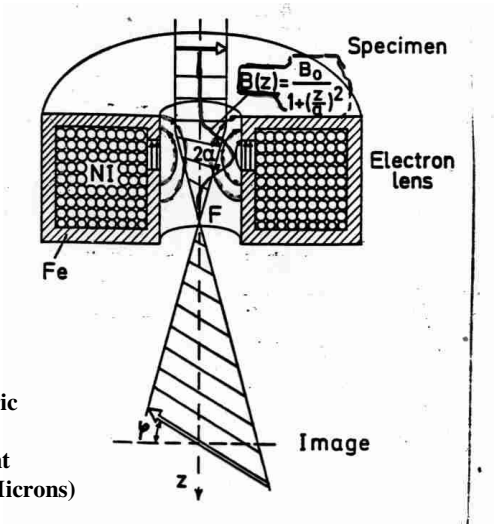
$$\varepsilon = \frac{eBL}{mv}$$

Relativistic

$$\varepsilon = \frac{eUL}{2Ed}\,\frac{1 + E/E_0}{\left(1 + \frac{E}{2E_0}\right)}$$

$$\varepsilon = \frac{eBL}{\left[2 m_0 E \left(1 + \frac{E}{2E_0}\right)\right]^{1/2}}$$

Magnetic Lens



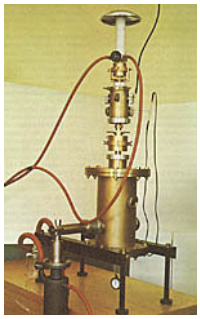
Lenses

Cylindrically symmetric

Are always convergent
Cs is rather large (mm vs. Microns)



Figure 1.1. The electron microscope built by Ruska and Knoll in Berlin in the early 1930s.



The Nobel Prize in Physics 1986

"for his fundamental work in electron optics, and for the design of the first electron microscope"



"for their design of the scanning tunneling microscope"

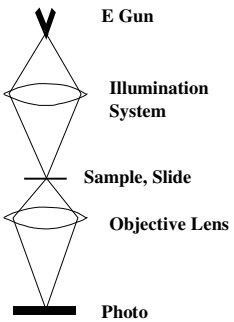
Ernst Ruska	**Gerd Binnig**	**Heinrich Rohrer**
1/2 of the prize	1/4 of the prize	1/4 of the prize
Federal Republic of Germany	Federal Republic of Germany	Switzerland
Fritz-Haber-Institut der Max-Planck-Gesellschaft Berlin, Federal Republic of Germany	IBM Zurich Research Laboratory Rüschlikon, Switzerland	IBM Zurich Research Laboratory Rüschlikon, Switzerland
b. 1906 d. 1988	b. 1947	b. 1933

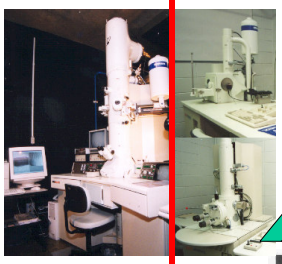
Press Release:
15 October 1986

The significance of the electron microscope in different fields of science such as biology and medicine is now fully established: it is one of the most important inventions of this century

Electron Microscopes

Type 1
Projection System
(Transmission Elec. Mic., TEM)

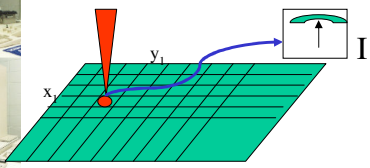


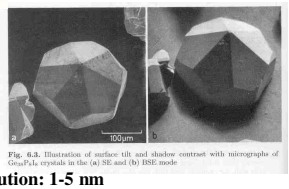


Resolution: 0.1-0.5 nm

Type 2
Scanning Elec. Mic., SEM

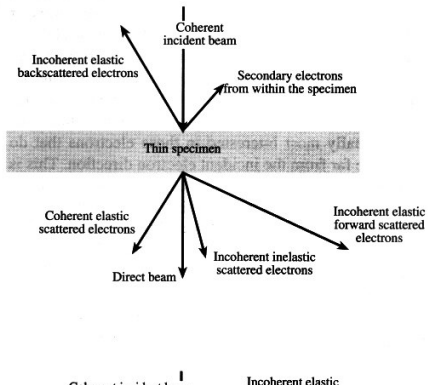
Instead of Projecting with a BROAD electron beam SCAN a NARROW e- beam





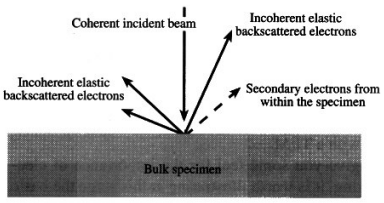
Resolution: 1-5 nm

Also a combination: Scanning Transmission Electron Microscope (STEM), Resolution ~0.1 nm

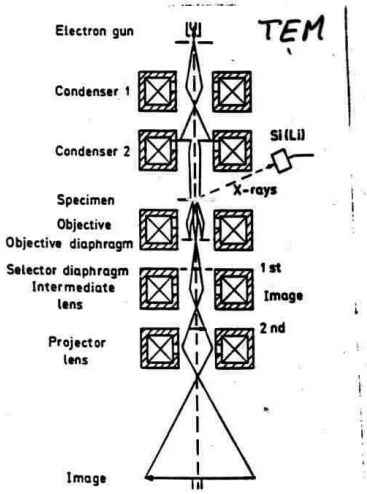




B



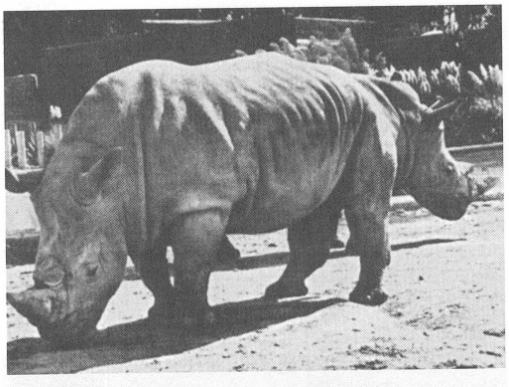
Transmission Electron Microscopy, (TEM)
Projection System

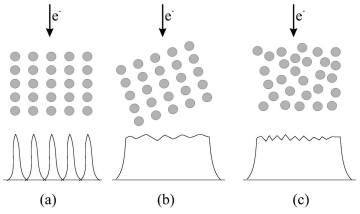


TEM

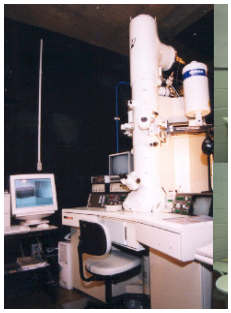
100 – 1500 kV

0.1 – 0.5 nm





Projection: requires sample must be transparent .....
for high energy electrons
Sample thickness 20-80 nm



3. COMPOSITION AND CONSTRUCTION

3.3.2 Column Interior

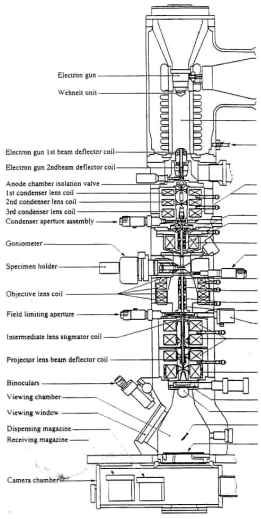


Fig. 3.3–2 Cross section of col

Coils and Lenses

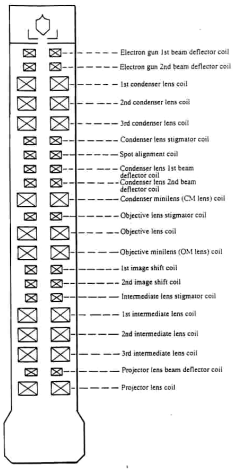
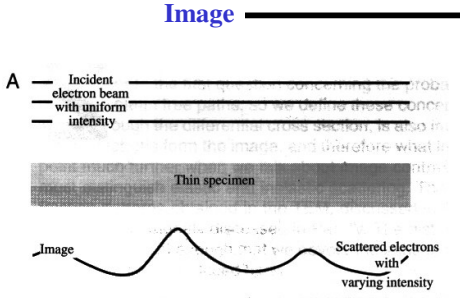
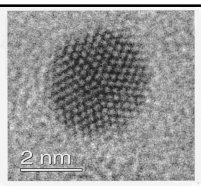
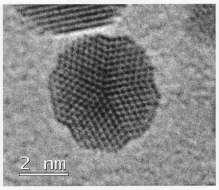


Fig. 3.3–3 Location of coils and lenses

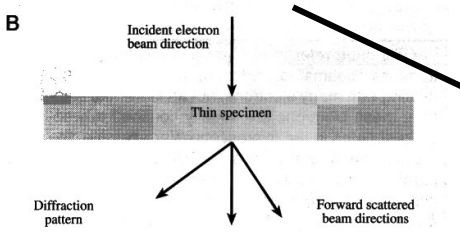


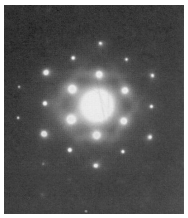
Image





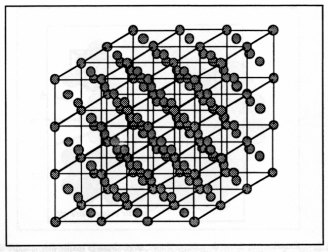
Diffraction





Decahedral Gold Nanoparticles

D. Zanchet, D. Ugarte LME/LNLS



7) One-dimensional point lattice

$f_7(x) = \sum_{n=1}^{N} \delta(x - x_n)$ $\quad F_7(q) = \frac{\sin \pi q N d}{\sin \pi q d}$



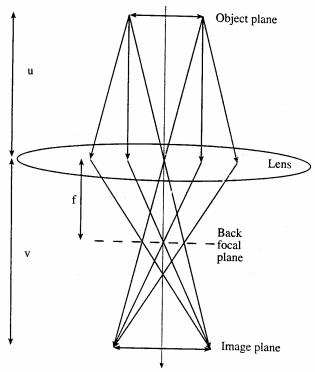


8) N slits of width a

$f_8(x) = f_1(x) \otimes f_7(x)$ $\quad F_8(q) = F_1(q) \cdot F_7(q) = a \frac{\sin \pi a q}{\pi a q} \frac{\sin \pi q N d}{\sin \pi q d}$



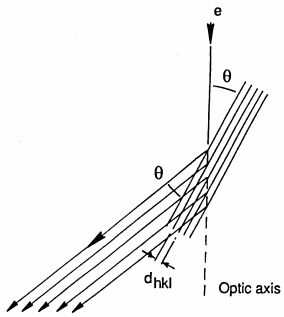


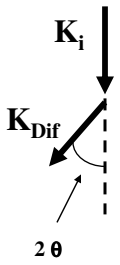


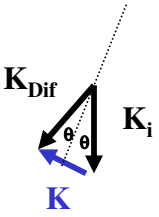
Lens Eq. $\quad \frac{1}{u} + \frac{1}{v} = \frac{1}{f}$

Magnification $\quad M = \frac{v}{u}$

Diffraction







Bragg'||s Law

2 d senθ = m λ

m = 0,1, 2...

K_{Dif}-K_i= K $\qquad$ **$|K_i| = |K_{Dif}| = 1/\lambda$**

Sin θ = |K|/2 / $|K_i|$ $\Rightarrow$ $\quad |K| = 2 \text{ Sin } \theta / \lambda$

|K| = 1/ d $\quad \Rightarrow \quad$ **Bragg's Law**

|K| = g $\quad$ **(Recip. Space Vector)**

Determing g we know all allowed difracted beams

Reciprocal lattice construction

Lattice $\mathbf{r} = n\,\mathbf{a} + m\,\mathbf{b} + p\,\mathbf{c}$

Reciprocal Lattice

$\mathbf{r}^* = h\,\mathbf{a}^* + k\,\mathbf{b}^* + l\,\mathbf{c}^*$

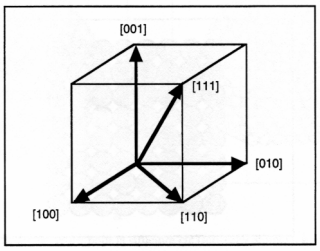
$\mathbf{a}^*.\mathbf{b} = \mathbf{a}^*.\mathbf{c} = \mathbf{b}^*.\mathbf{a} = \mathbf{b}^*.\mathbf{c} = \mathbf{c}^*.\mathbf{a} = \mathbf{c}^*.\mathbf{b} = 0$

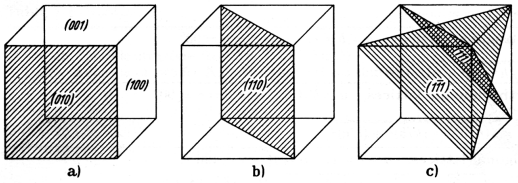
$\mathbf{a}^*.\mathbf{a}^* = \mathbf{b}^*.\mathbf{b}^* = \mathbf{c}^*.\mathbf{c}^* = 1$

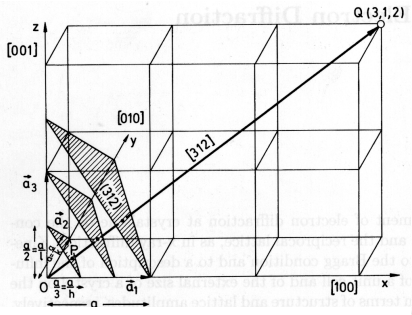
$\mathbf{a}^* = \mathbf{b} \wedge \mathbf{c} / V_c \qquad (V_c = \mathbf{a}.\mathbf{b} \wedge \mathbf{c})$

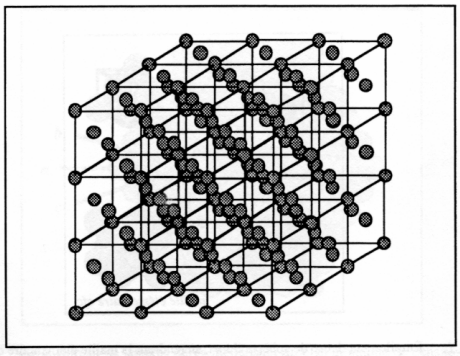
$\mathbf{g}_{hkl} = h\,\mathbf{a}^* + k\,\mathbf{b}^* + l\,\mathbf{c}^* \qquad |g_{hkl}| = 1/d_{hkl}$

Reciprocal Space $\qquad |\mathbf{g}_{hkl}| = 1 / |\mathbf{d}_{hkl}|$









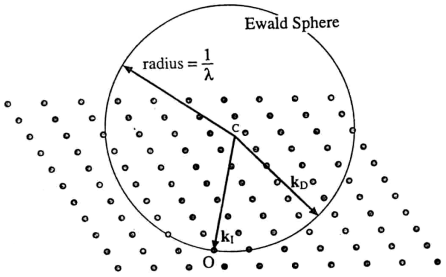
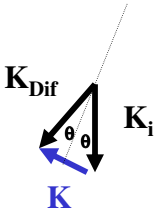


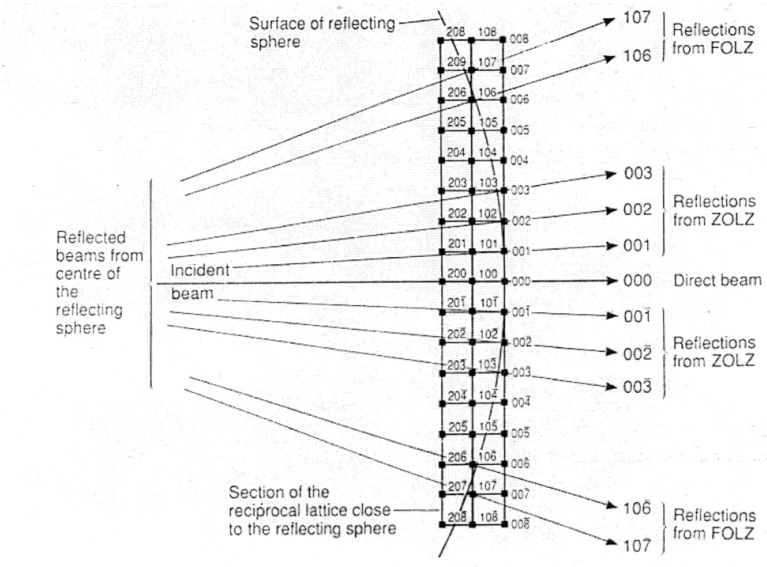
Figure 12.3. The Ewald sphere of reflection is shown intersecting a noncubic array of reciprocal-lattice points. The vector **CO** represents $\mathbf{k}_i$, the wave vector of the incident wave, and O is the origin of the reciprocal lattice. $\mathbf{k}_D$ is any radius vector. When the radius of the sphere is similar to the spacing between the points in the reciprocal lattice, as is the case for X-rays, the sphere can only intersect a few points, as shown. When λ is much smaller, as for 100-keV electrons, the radius is much larger, the sphere is flatter, and it intersects many more points.



$|K_i| = |K_{Dif}| = 1/\lambda$

$K_{Dif} - K_i = K$

$|K| = g$ **diffracted beam**



Efeito da Lámina Fina

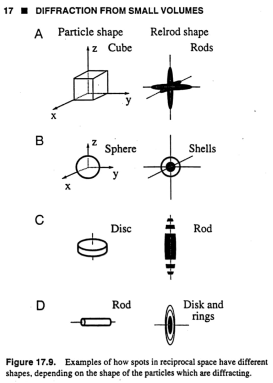
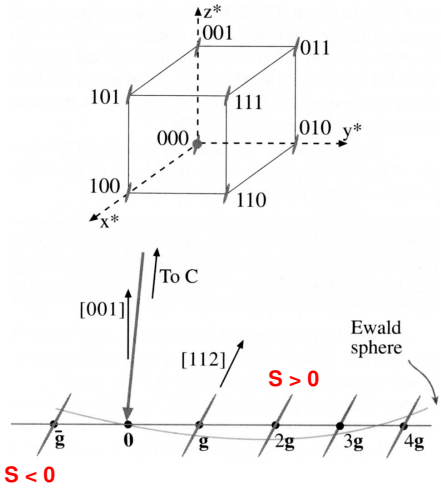
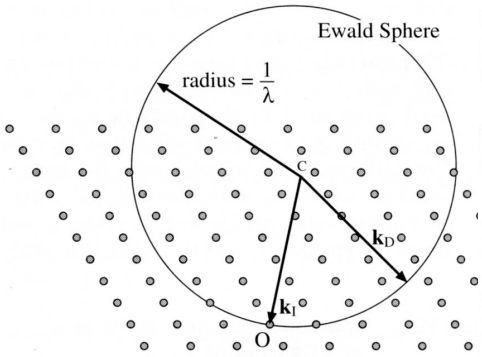
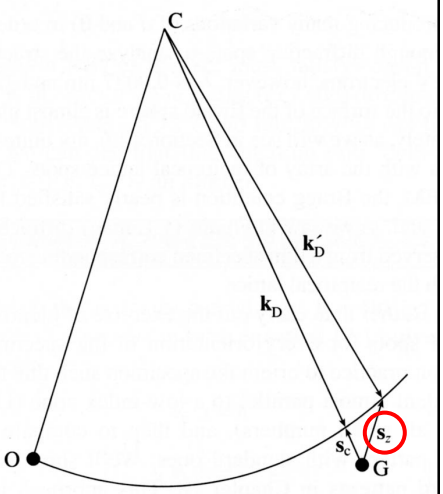


Figure 17.9. Examples of how spots in reciprocal space have different shapes, depending on the shape of the particles which are diffracting.

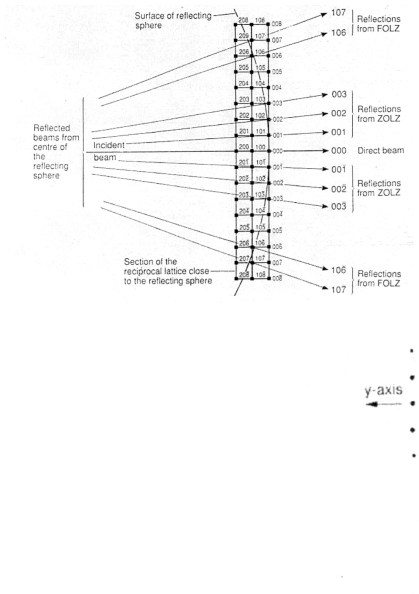


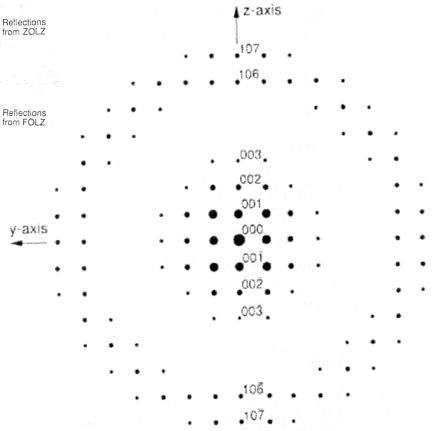
Erro de Exitação

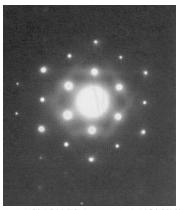




$$K = g + s$$







17 ■ DIFFRACTION FROM SMALL VOLUMES

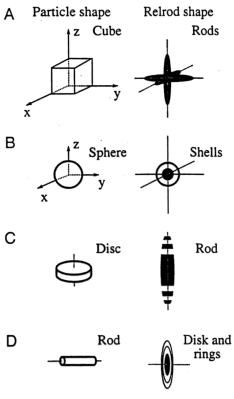


Figure 17.9. Examples of how spots in reciprocal space have different shapes, depending on the shape of the particles which are diffracting.

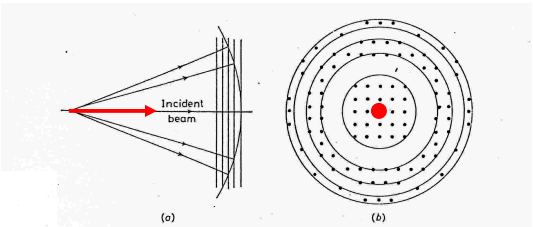


Figure 5.4. The effect of curvature of the reflecting sphere, without a tilted beam. (a) Intersection between sphere and reciprocal lattice planes. (b) Corresponding schematic diffraction pattern—symmetrical Laue zones

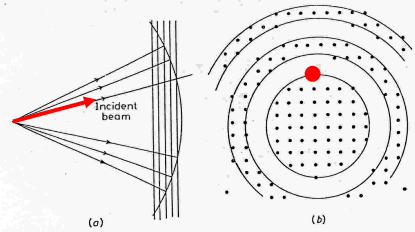
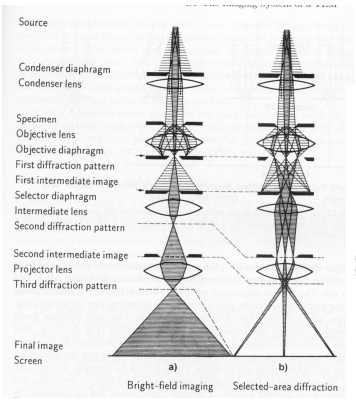


Figure 5.5. The effect of curvature of the reflecting sphere, with a tilted beam. (a) Intersection between sphere and reciprocal lattice planes. (b) Corresponding schematic diffraction pattern—asymmetrical Laue zones



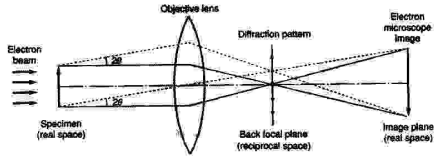


Fig. 1.1. Optical ray diagram with an optical objective lens showing the principle of the imaging process in a transmission electron microscope

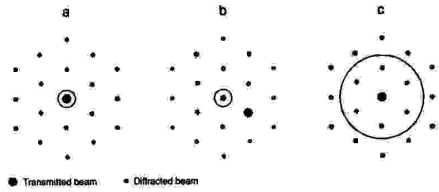


Fig. 1.2. Three observation modes in electron microscopy using an objective aperture. The center of the objective aperture is assumed to be set to the optical axis. a Bright-field method; b dark-field method; c high-resolution electron microscopy (axial illumination)

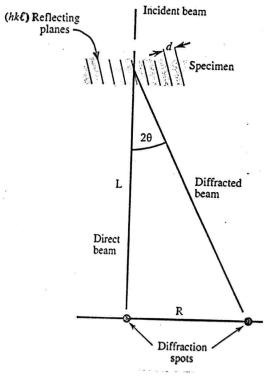
WE can obtain:

Spatial information -> images

(but selected diff information included, Bright Field, Dark Field)

Crystallographic information (but select area, SAD)

Selected Area Difraction (SAD)



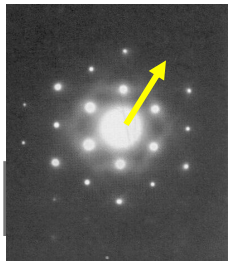
L - camera length

↑ depende das lentes projetoras

$\frac{R}{L} = tg2\theta \approx 2\theta$

$Rd = \lambda L$

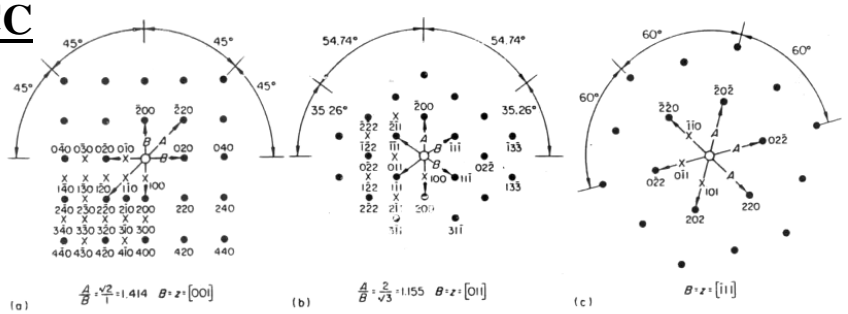
→ calibração



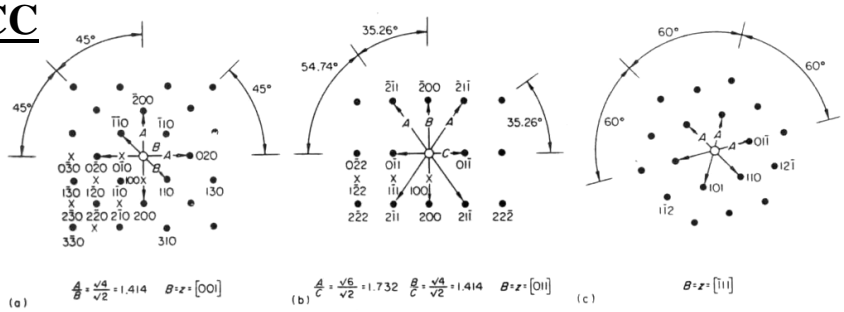
Dif. Spots -> Atomic plane distances -> lattice parameters

Angles -> symmetries

FCC

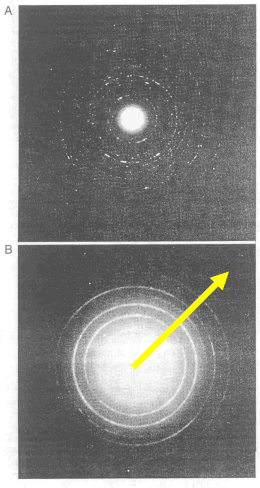


BCC

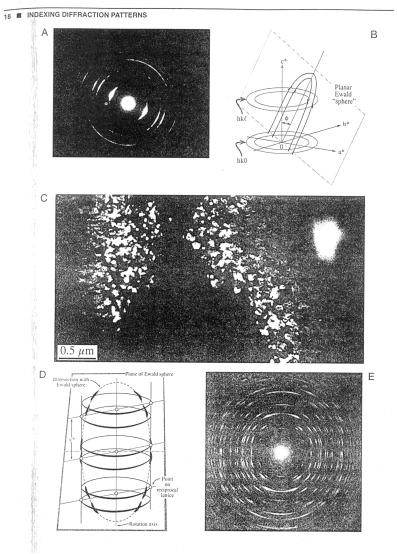


Selected Area Difraction (SAD)

Poly-Cristalline Sample



Rings Diameters: Lattice Spacings
Radial Profile: Powder Difraction (X-ray)

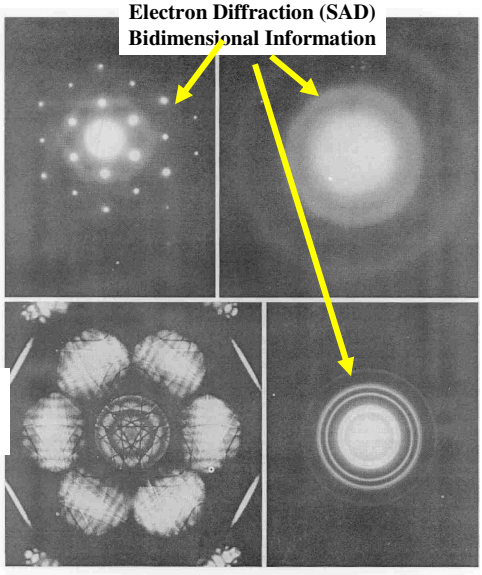


TEXTURE

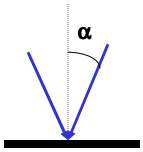
Selected Area
Electron Diffraction (SAD)
Bidimensional Information

SAD



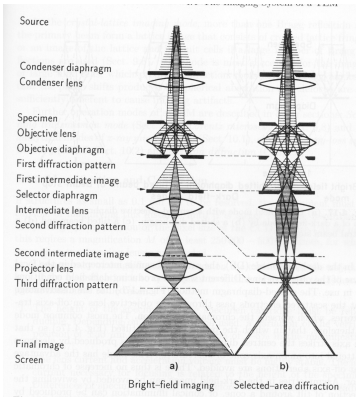


Convergent Beam
Electron Diffr.(CBED)
Some 3D Information



Lattice parameter determination **RX vs electron difraction**
3D vs mostly 2D informtion
But high spatial information (100 nm)

Image: Bidimensional (x,y) intensity distribution
We must calculate intensities!!!!!!



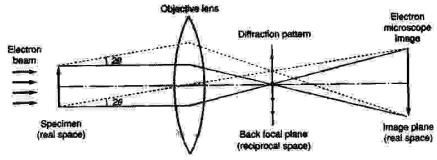


Fig. 1.1. Optical ray diagram with an optical objective lens showing the principle of the imaging process in a transmission electron microscope

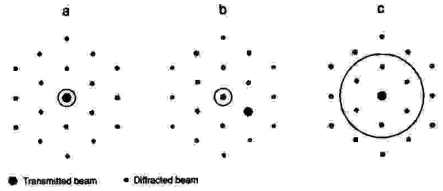
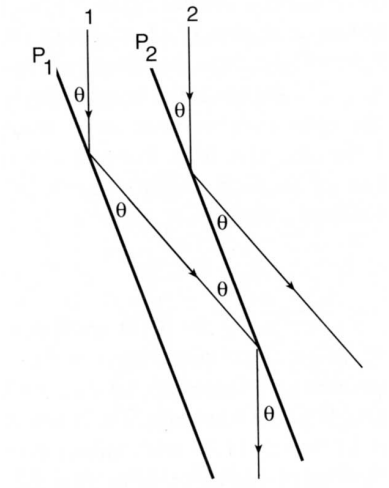


Fig. 1.2. Three observation modes in electron microscopy using an objective aperture. The center of the objective aperture is assumed to be set to the optical axis. a Bright-field method: b dark-field method: c high-resolution electron microscopy (axial illumination)

Bragg´s Law: no intensitiy normalization
Many excited diffracted beams
Intensities must be coupled

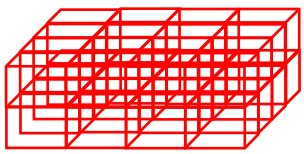
Dynamical Diffraction



Difração Dinâmica



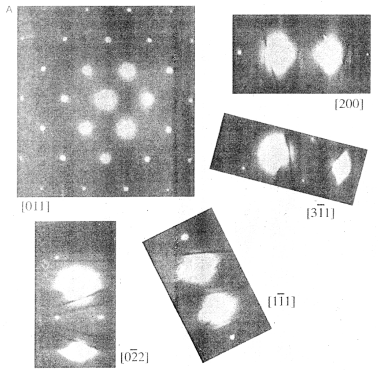
$$A_{\text{cell}} = \frac{e^{2\pi i \mathbf{k}\cdot\mathbf{r}}}{r} F(\theta)$$

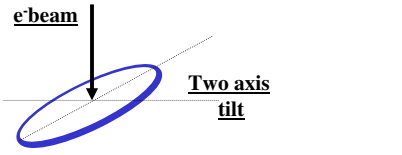


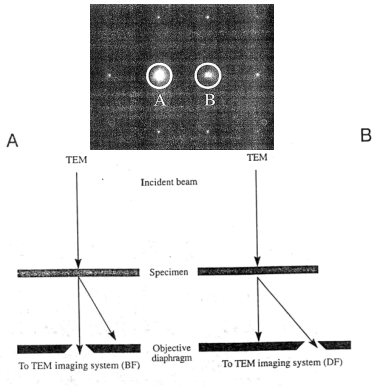
Difração Dinâmica

$$\psi^{T} = \phi_0 e^{2\pi i \chi_O \cdot \mathbf{r}} + \phi_{\mathbf{g}_1} e^{2\pi i \chi_{G_1} \cdot \mathbf{r}} + \phi_{\mathbf{g}_2} e^{2\pi i \chi_{G_2} \cdot \mathbf{r}} + \cdots$$

Setting <u>Two Beam Condition:</u>
Controlling orientation of the sample







WE can obtain:

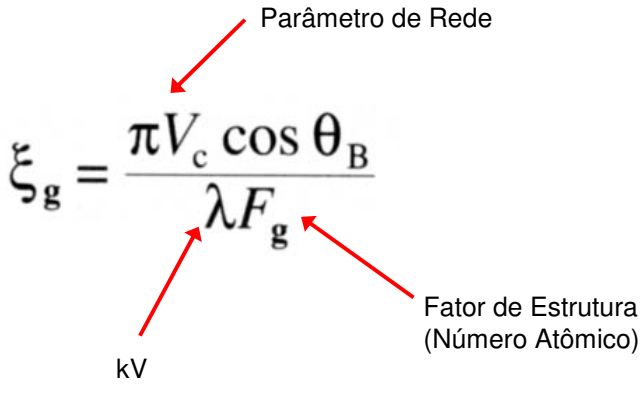
Spatial information -> images
(but diff information included, Bright Field, Dark Field)
Crystallographic information (but select area, SAD)

Condição de 2 Feixes – Two Beam

$$d\phi_g = \left\{ \frac{\pi i}{\xi_g}\phi_0 e^{2\pi i(\chi_O - \chi_D)\cdot \mathbf{r}} + \frac{\pi i}{\xi_0}\phi_g \right\} dz$$

$$d\phi_0 = \left\{ \frac{\pi i}{\xi_0}\phi_0 + \frac{\pi i}{\xi_g}\phi_g e^{2\pi i(\chi_D - \chi_O)\cdot \mathbf{r}} \right\} dz$$

Distância de Extinção [nm]



Distancia de Extinção [nm]

Examples of Extinction Distances (in nm)

Material $hk\ell$ =	110	111	200	220	400
Al	-	56.3	68.5	114.4	202.4
Cu	-	28.6	32.6	47.3	76.4
Au	-	18.3	20.2	27.8	43.5
MgO	-	272.6	46.1	66.2	103.3
Fe	28.6	-	41.2	65.8	116.2
W	18.0	-	24.5	35.5	55.6
Diamond	-	47.6	-	66.5	121.5
Si	-	60.2	-	75.7	126.8
Ge	-	43.0	-	45.2	65.9

Condição de 2 Feixes – Dif. Dinâmica

Howie-Whelan eqs.

$$\frac{d\phi_g}{dz} = \frac{\pi i}{\xi_g}\phi_0 e^{-2\pi i s z} + \frac{\pi i}{\xi_0}\phi_g$$

$$\frac{d\phi_0}{dz} = \frac{\pi i}{\xi_0}\phi_0 + \frac{\pi i}{\xi_g}\phi_g e^{2\pi i s z}$$

Similar to Coupled oscillators

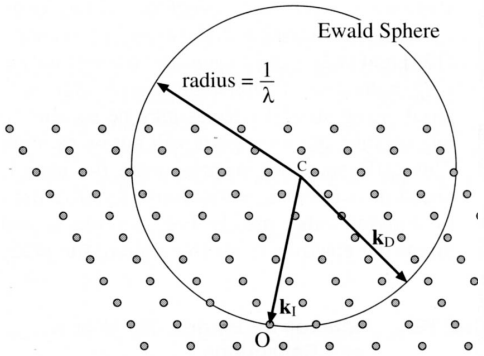
Erro de excitação Efetivo

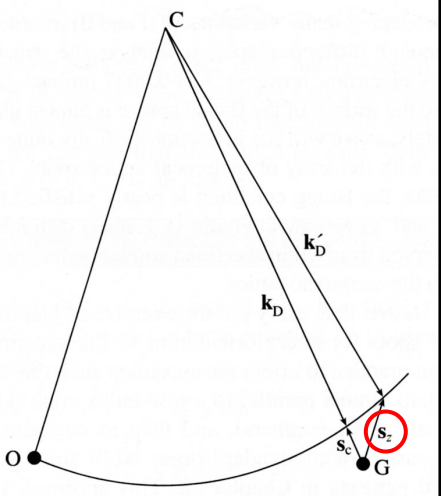
$$s_{\text{eff}} = \sqrt{s^2 + \frac{1}{\xi_g^{\,2}}} = \frac{\sqrt{w^2+1}}{\xi_g}$$

Intensidade do ponto de difração

$$|\phi_g|^2 = \left(\frac{\pi t}{\xi_g}\right)^2 \frac{\sin^2(\pi t s_{\text{eff}})}{(\pi t s_{\text{eff}})^2}$$

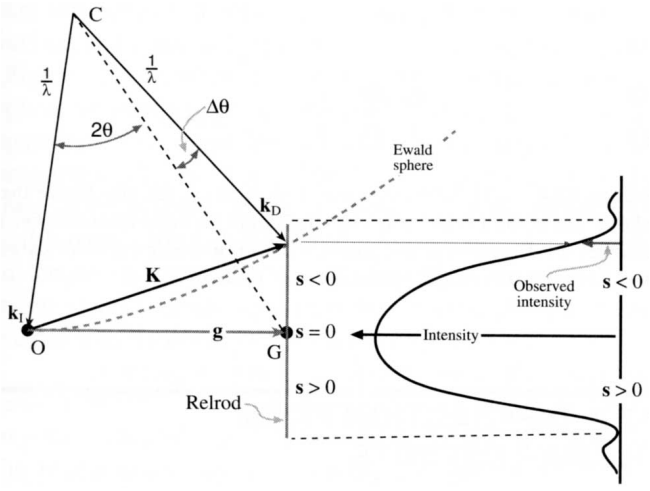
Erro de Exitação



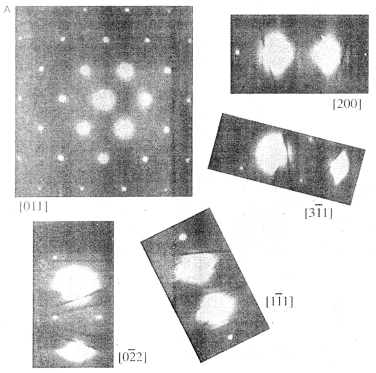


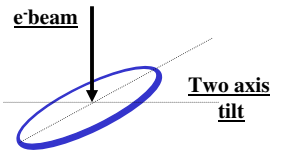
$$K = g + s$$

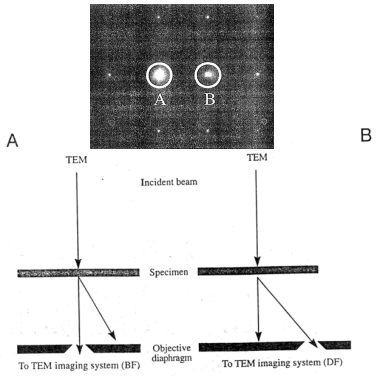
Erro de Excitação (s) - Intensidade



Setting <u>Two Beam Condition:</u>
Controlling orientation of the sample







WE can obtain:

Spatial information -> images
(but diff information included, Bright Field, Dark Field)
Crystallographic information (but select area, SAD)

Franjas de Espessura

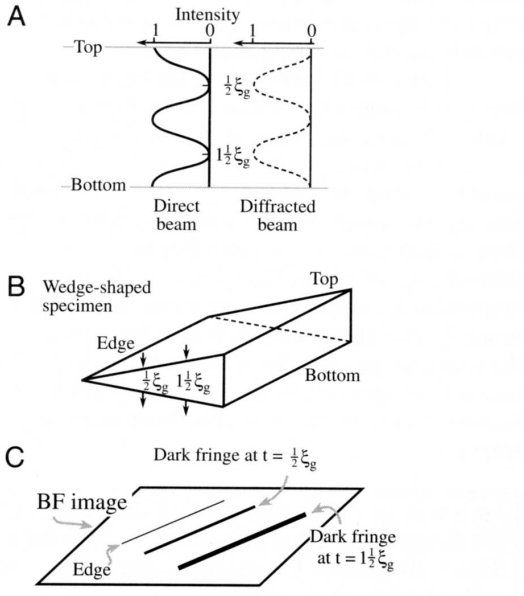


Figure 23.2. (A) At the Bragg condition (**s** = 0), the intensities of the direct and diffracted beams oscillate in a complementary way. (B) For a wedge specimen, the separation of the fringes in the image (C) is determined by the angle of the wedge and the extinction distance, ξ_g.

Franjas de Espessura

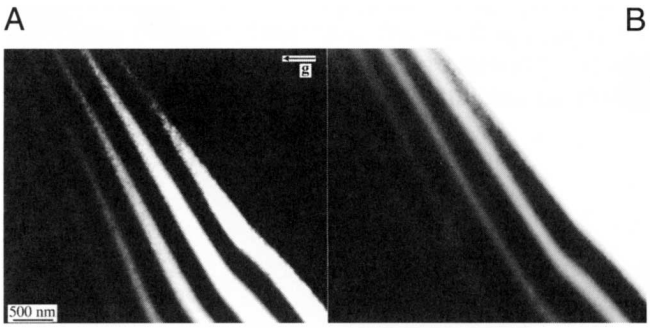
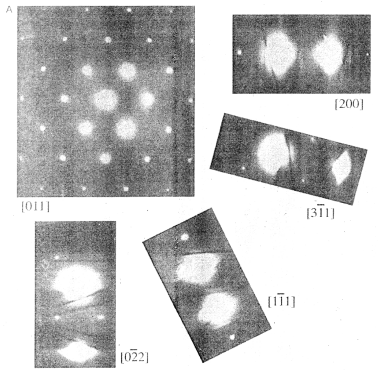
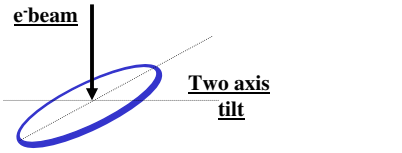
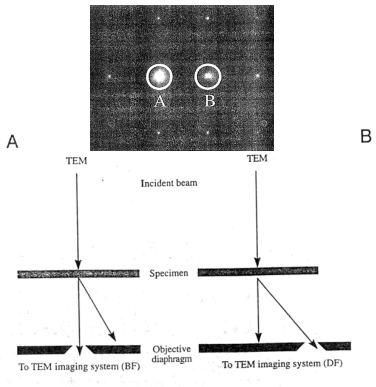


Figure 23.1. (A) DF and (B) BF images from the same region of a wedge-shaped specimen of Si at 300 kV tilted so that **g**(220) is strong. The periodicity and contrast of the fringes is similar and complementary in each image.

Setting <u>Two Beam Condition:</u>
Controlling orientation of the sample



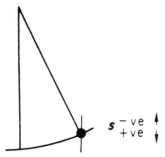




WE can obtain:

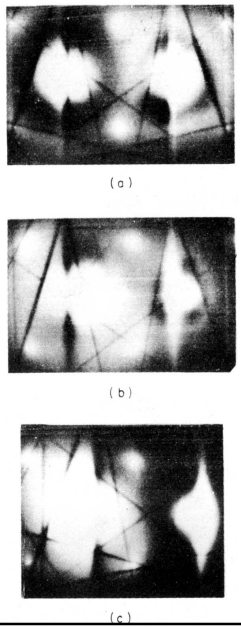
Spatial information -> images
(but diff information included, Bright Field, Dark Field)
Crystallographic information (but select area, SAD)

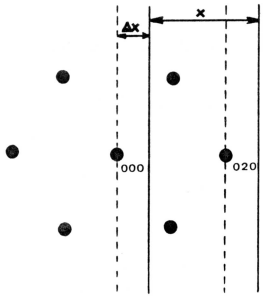
Erro de Excitação (s)



S<0

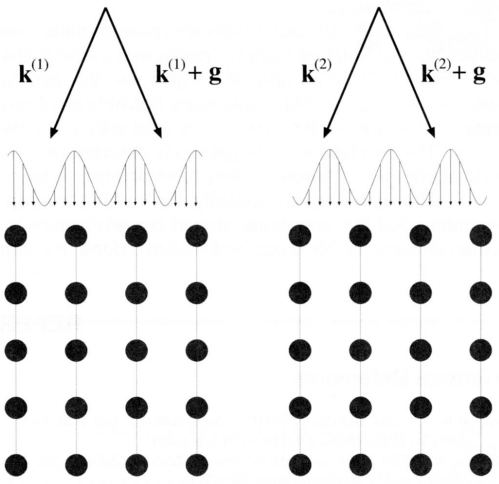
S>0



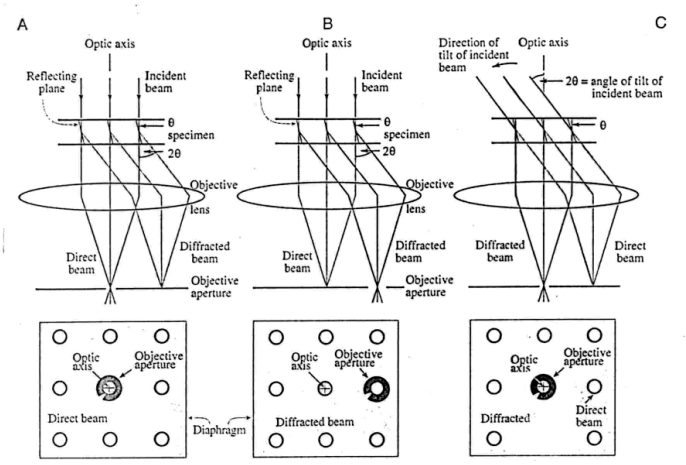


Ondas de Bloch

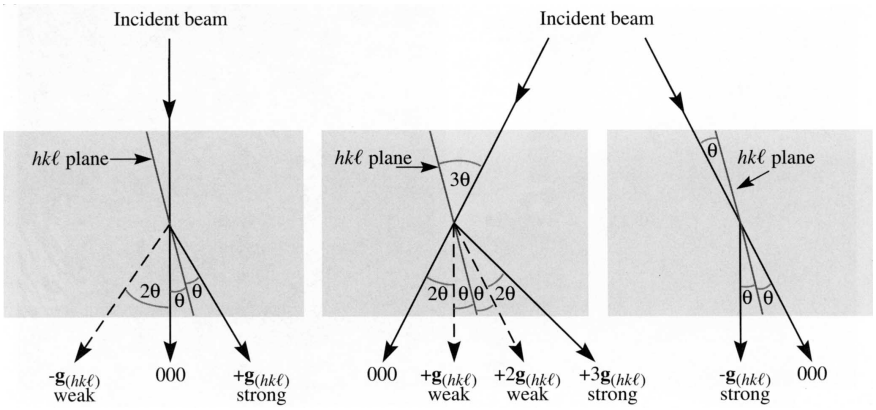
Predomina S<0



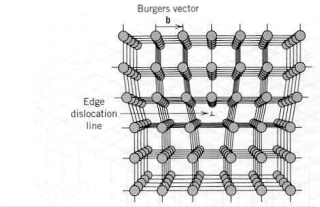
Predomina S>0

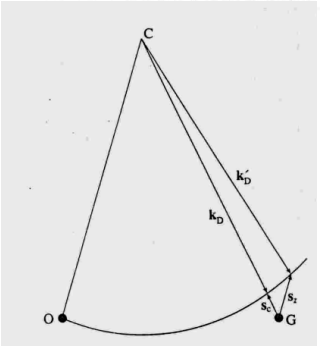


Condição de 2 Feixes – Two Beam



Dislocations





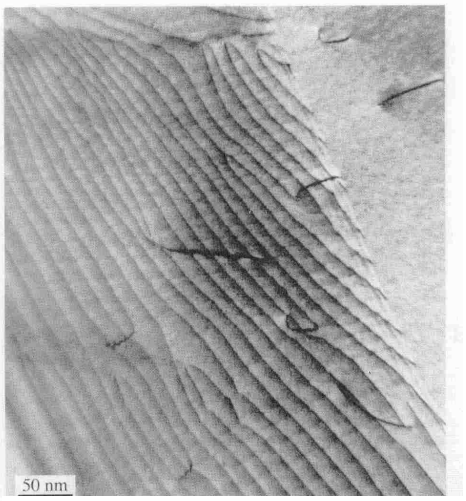
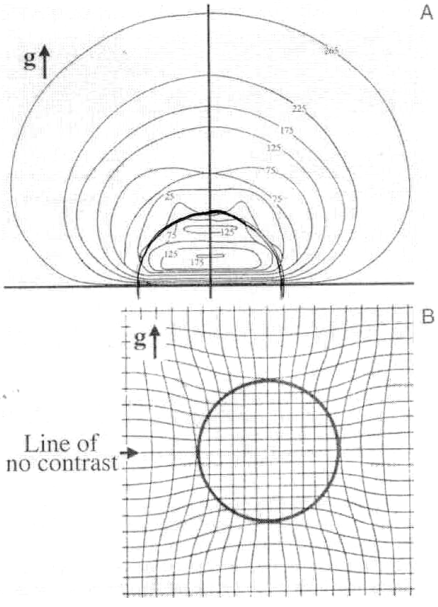
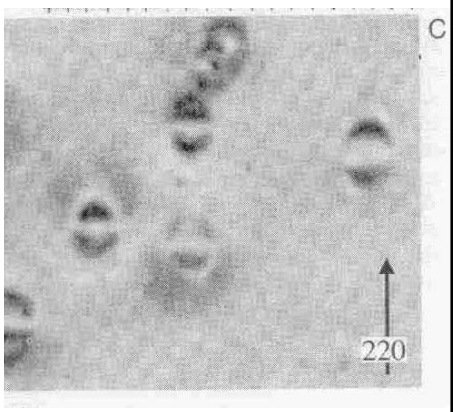


Image contrast is induced by local changes of crystallographic order (expansion/contraction, orientation, etc.)

Dislocation: line defect (a line of atoms)
Resolution **10-20 nm!!!! (subtle changes of the Crystal)**

Inclusions





Recommended Books
TEXTBOOK for TEM
I recommend the Williams & Carter for somebody starting with TEM.
It has been specially written for graduate courses on TEM of USA.
The other ones are rather more adapted for getting deeper knowledge once you have gained some experience.
Transmission Electron Microscopy: A textbook for Materials Science
D.B. Williams, C.B. Carter
Plenum Press 1996
ISBN 0-306-45247-2 (hardbound) ISBN 0-306-45324-X (pbk)

Conventional TEM (the Bible)
Electron Microscopy of Thin Crystals
P. Hirsh, A. Howie, R. Nicholson, D.W. Pashley, M.J. Whelan
Robert Krieger Pub. Co., Huntington NY (1977).

General (and deeper) knowledge TEM (Gun, lens, etc)
Transmission Electron Microscopy
L. Reimer
Springer Verlag
High Resolution Transmission Electron Microscopy
High Resolution Transmission Electron Microscopy and Associated Techniques
P. Buseck, J. Cowley, L. Eyring
Oxford Science Pub. 1992
Electron Diffraction
Electron Microdiffraction
J.C.H. Spence, J.M. Zuo
Plenum Press NY 1992
TEM Sample Preparation
Thin Foil Preparation for Electron Microscopy
P.J. Goddhew
Elsevier 1985, ed. Audrey M. Galuert

Focussed beams, and analytical capabilities
RESOLUTION

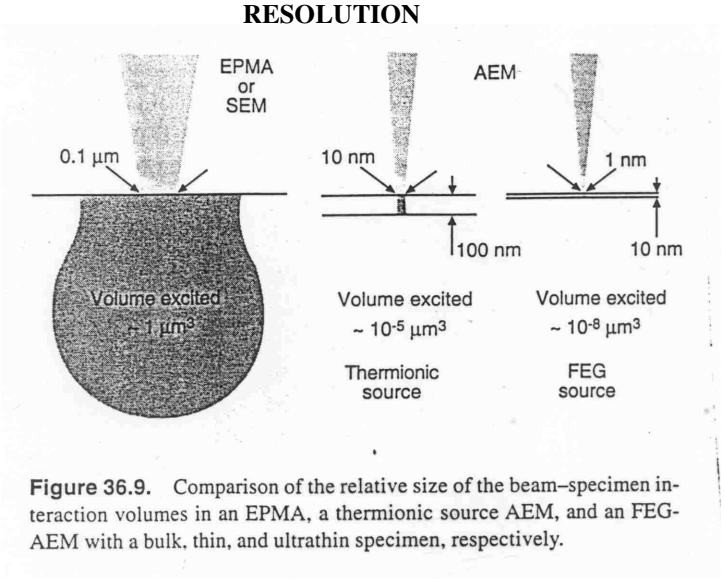
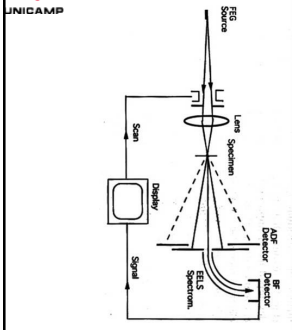


Figure 36.9. Comparison of the relative size of the beam–specimen interaction volumes in an EPMA, a thermionic source AEM, and an FEG-AEM with a bulk, thin, and ultrathin specimen, respectively.

Scanning Transmission Electron Microscope



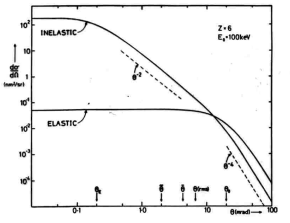


Figure 3.7. Angular dependence of the differential cross sections for elastic and inelastic scattering of 100-keV electrons from a carbon atom, calculated using the Lenz model, Eqs. (3.5), (3.7), and (3.15). Shown along the horizontal axis are (from left to right) the characteristic, median, mean, root-mean-square, and effective-cutoff angles for total-inelastic scattering, evaluated using Eqs. (3.53)–(3.56).

Annular Dark Field

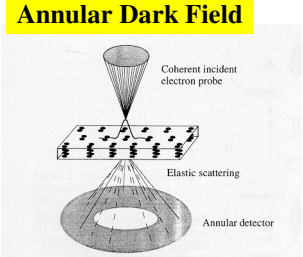


Figure 4. Schematic of the STEM optics for incoherent imaging.

Elestic scattering: 1–10°

Inelastic scattering: < 1°

Z-Contrast

$\frac{\sigma_{in}}{\sigma_{el}} \simeq \frac{Z^2}{Z}$ (Z contrast)

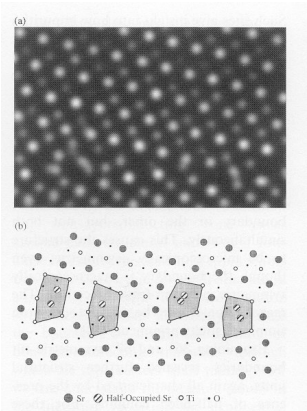


Figure 19. (a) Reconstructed image and (b) atomic structure for a $\Sigma = 85$ {920}(001) symmetric tilt boundary in $SrTiO_3$ deduced using the 100 kV STEM. Strontium columns (seen bright) are spaced 3.9 Å apart in the bulk crystal.

Minor Crystallographic effects (Incoherent scattering),
RememberTEM constrast is based on Diffraction and phase contrast!!!

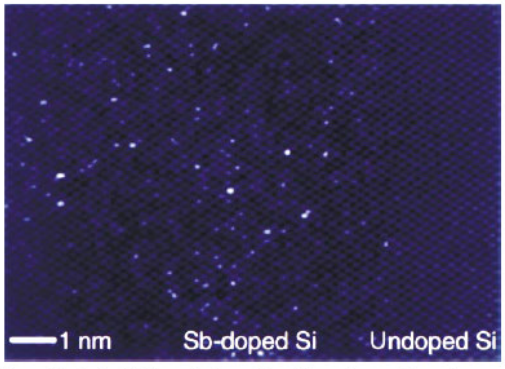


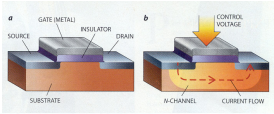
Figure 1 Annular dark-field scanning transmission electron microscopy image of a cross-section of highly Sb-doped Si. The brightest dots (left) are atomic columns containing one or more Sb atoms, absent in substrate (right), where there is no Sb. Thickness variations along the wedge have been subtracted using a second-order polynomial fit. The image has been low-pass filtered to remove scan noise, and is displayed with a nonlinear intensity scale to highlight bright features.

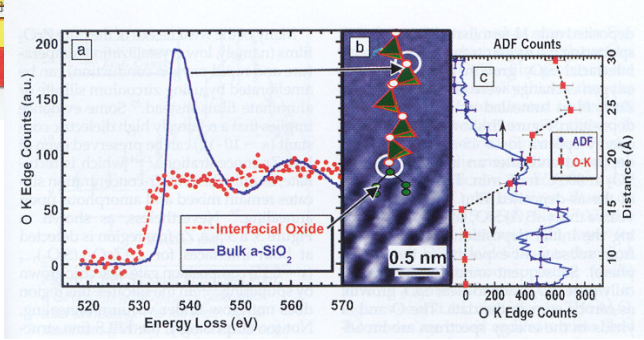
Voyles et al, Nature 416, 826 (2002)



Microelectronics:

miniaturization and intrinsic materials limits





Electron Energy Loss Spectrum (EELS)

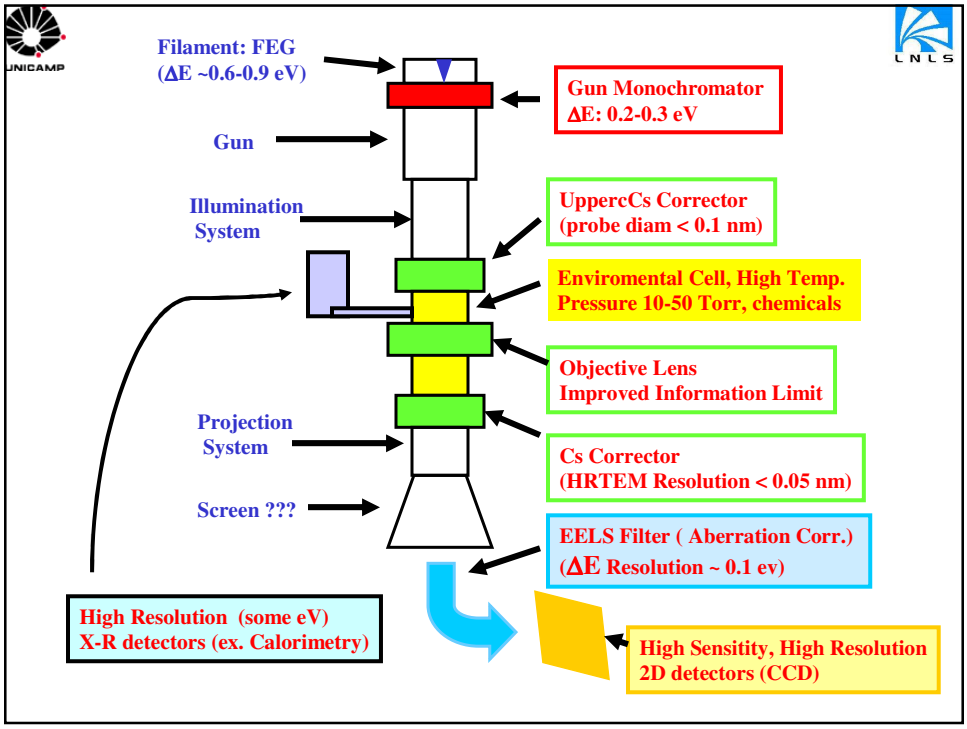
Muller et al, Nature 399, 758 (1999)

Future???

Now: **Automatization, informatization**
(TEM as routine analysis in Industry)
Many softwares and interfaces
(similar hardware, better User Interface)
Still request "good feeling and fingers"

Next: **New Hardware Generation**
entering commercial microscopes
Correctors & Monochromators
Data treatment
In-situ experiments

Requirements:
0.5 Å resol at 100-300 keV, sub- Å probe, 0.2 eV energy spread


Filament: FEG
(ΔE ~0.6-0.9 eV)
Gun
Illumination System
Projection System
Screen ???
Gun Monochromator
ΔE: 0.2-0.3 eV
UppercCs Corrector
(probe diam < 0.1 nm)
Enviromental Cell, High Temp.
Pressure 10-50 Torr, chemicals
Objective Lens
Improved Information Limit
Cs Corrector
(HRTEM Resolution < 0.05 nm)
EELS Filter (Aberration Corr.)
(ΔE Resolution ~ 0.1 ev)
High Resolution (some eV)
X-R detectors (ex. Calorimetry)
High Sensitity, High Resolution
2D detectors (CCD)