ELECTRON MICROSCOPY

14:20 – 17:20, Mar. 18, 2008
14:20 – 17:20, Mar. 25, 2008
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References:

Optics, in any standard freshman or high school physics course.

"Transmission Electron Microscopy" D.B. Williams and C. B. Carter, 1996, Plenum.

"Scanning Electron Microscopy and X-ray Microanalysis" J.I. Goldstein, D.E. Newbury, P. Echin, D.C. Joy, C.E. Lyman, E. Lifshin, L. Sawyer, and J.R. Michael, 3rd ed, 2003, Kluwer/Plenum.

"Diffraction Physics" J.M. Cowley, 3rd ed, 1995, North-Holland.

"Electron Microscopy of Thin Crystals" P. Hirsch, A. Howie, R.B. Nicholson, D.W. Pashley, and M.J. Whelan; 2nd ed., 1977, Robert E. Krieger.

"Practical Electron Microscopy in Materials Science" J. W. Edington, l976, Van Nostrand Reinhold.

"Procedures in Electron Microscopy", eds. A.W. Robards and A.J. Wilson, 1996 (or later), Wiley.

"Atlas of Optical Transforms" G. Harburn, C.A. Taylor, and T. R. Welberry; 1967, Cornell University.

“DigitalMicrograph”, Gatan, Inc.

Outline:

Introduction

The Electron microscope

Principle of image formation

Diffraction

Specimen preparation

Contrast/Applications

Scanning electron microscopy

Electron microprobe / Analytical electron microscopy

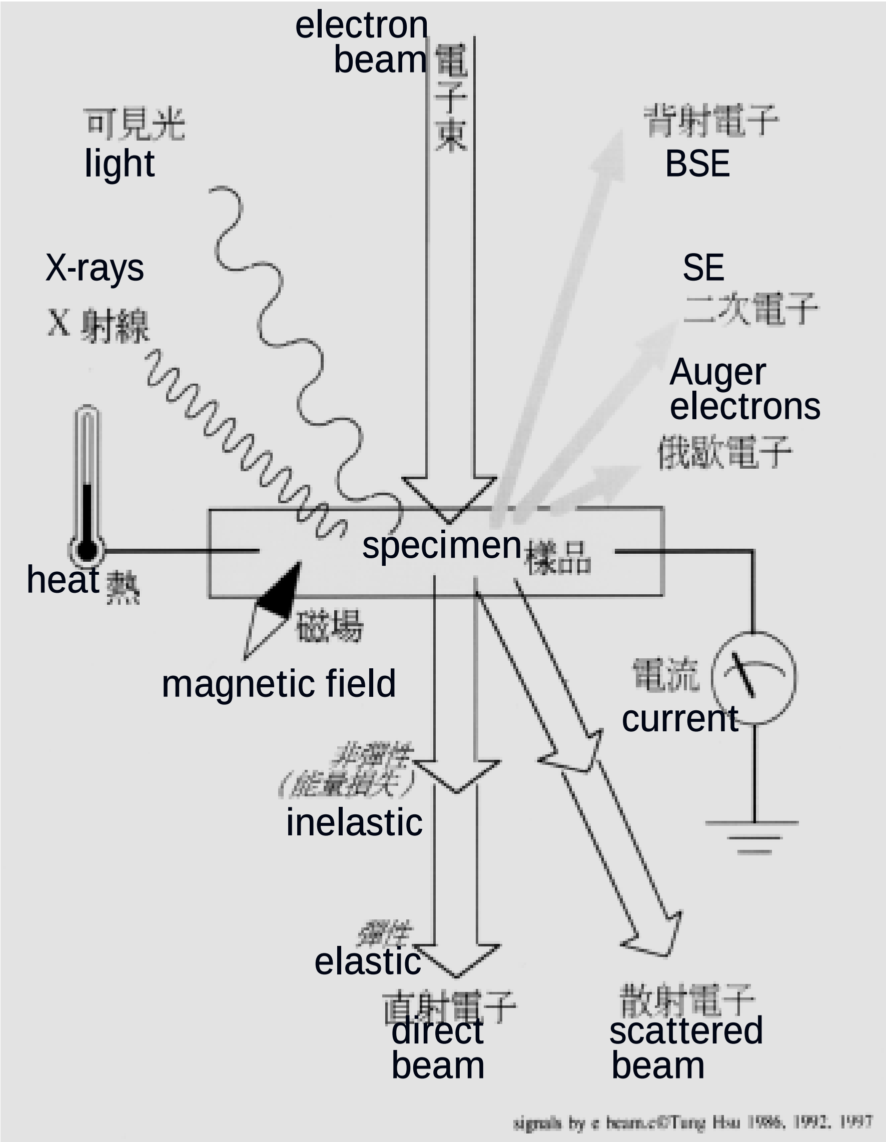
Introduction:

Why electron microscopy?

- Sensitivity:
  - Beam/solid (specimen) interaction
- (Spatial) Resolution:
  - Microscopy vs. microprobe
  - Wavelength, properties of lens
  - Beam/solid interaction
- Information other than the image

A brief history of electron microscopy

Traditional materials characterization:
incidence beam (probe): photon
exit beam (signal): photon
detector: eye
processor/storage: brain
(ref. Taiyo)



Why electron microscopy (EM)?

Information obtainable from EM

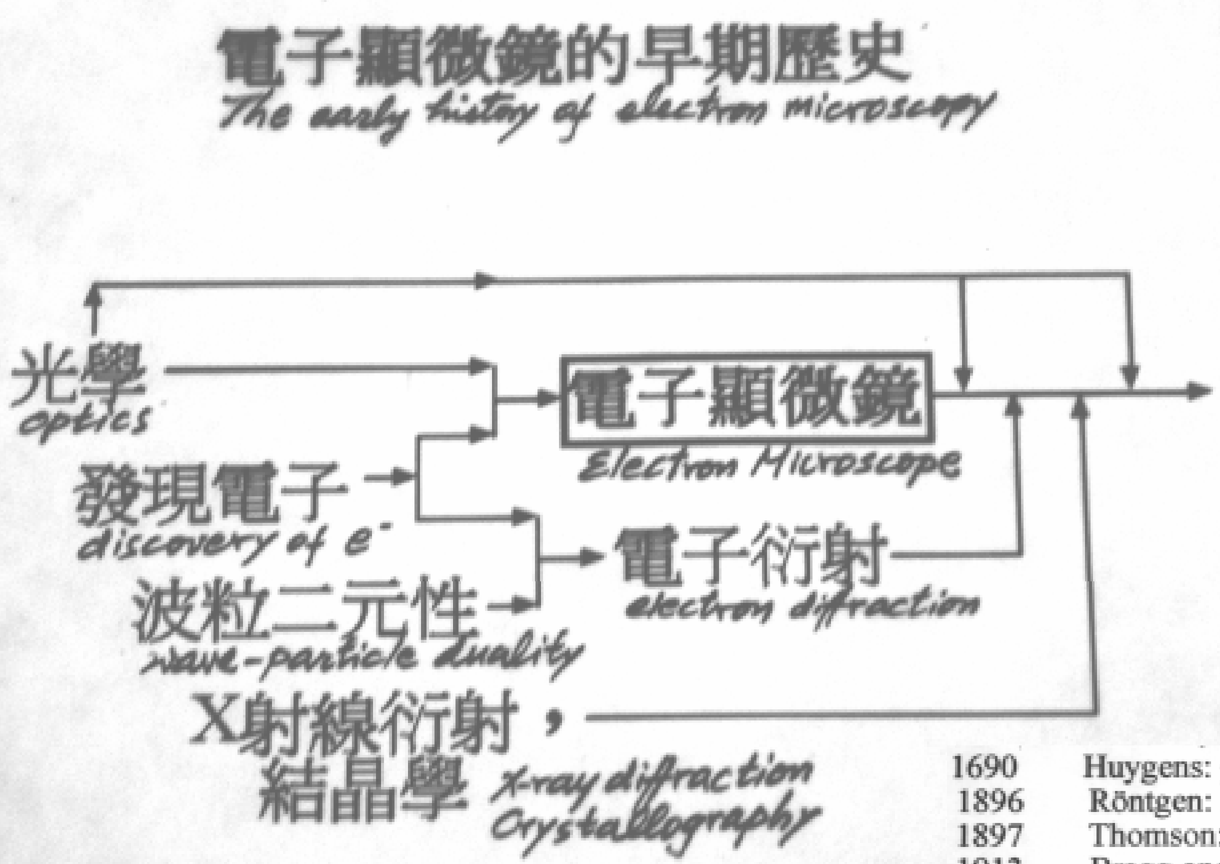
Beam/solid interaction

- image: morphology
  - scattering power
  - crystal structure
  - crystal defects
  - atomic structure
- other than the image:
  - (chemical) elemental composition
  - electronic structure

(Spatial) Resolution:

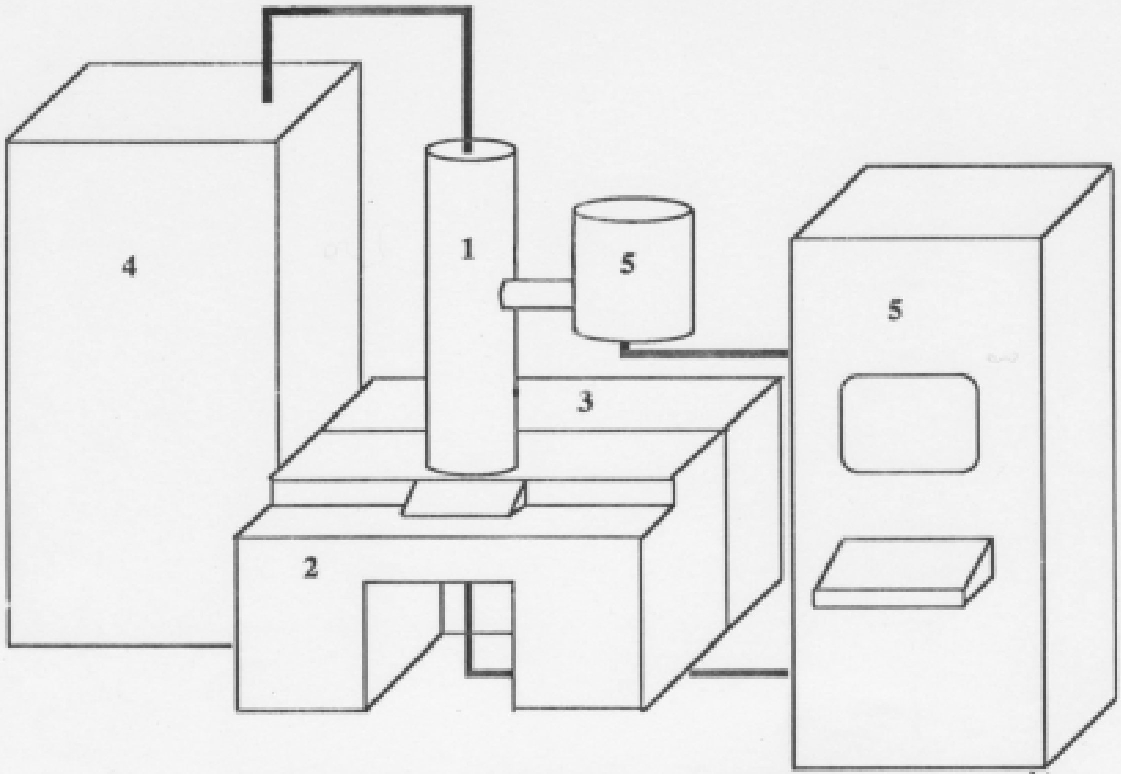
- Microscopy vs. microprobe
- Wavelength, properties of lens

A brief history of electron microscopy



1690	Huygens: 光波，衍射
1896	Röntgen: 發現X射線
1897	Thomson: 發現電子
1913	Bragg and Bragg, von Laue: X射線衍射
1924	de Broglie 波
1926	Schödinger 方程式
	Busch: 電子束聚焦
1927	Davisson & Germer, Thomson: 電子衍射
1931	Ruska & Knoll: 鐵心磁鏡
1934	完成電子顯微鏡

Various Electron Microscopes



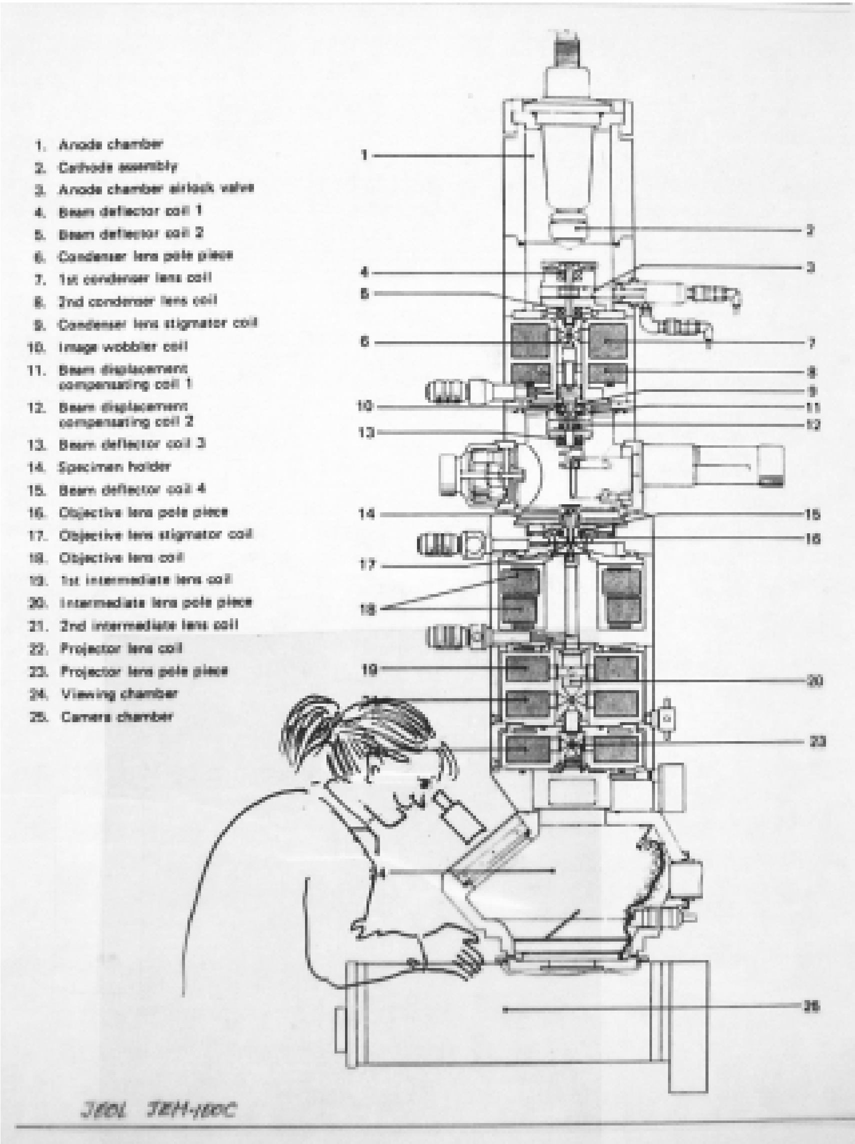
穿透式电子显微镜的主要部件

MAJOR COMPONENTS OF A TEM

1. electron optics column 镜筒
2. electronics and controls 电子系统
3. vacuum system 真空系统
4. high voltage power supply 高压电源
5. accessories 附件.

The Electron microscope

Structure and major components



The Electron Optics Column of JEOL JEM-100C

The Lens System:

Condenser Lens:

- Controls beam intensity, density, convergence, coherence.

Objective Lens:

- Magnification, introducing contrast.

Intermediate Lens:

- Further magnification, imaging or diffraction.

Projector Lens:

- Final magnification

Apertures

Specimen chamber

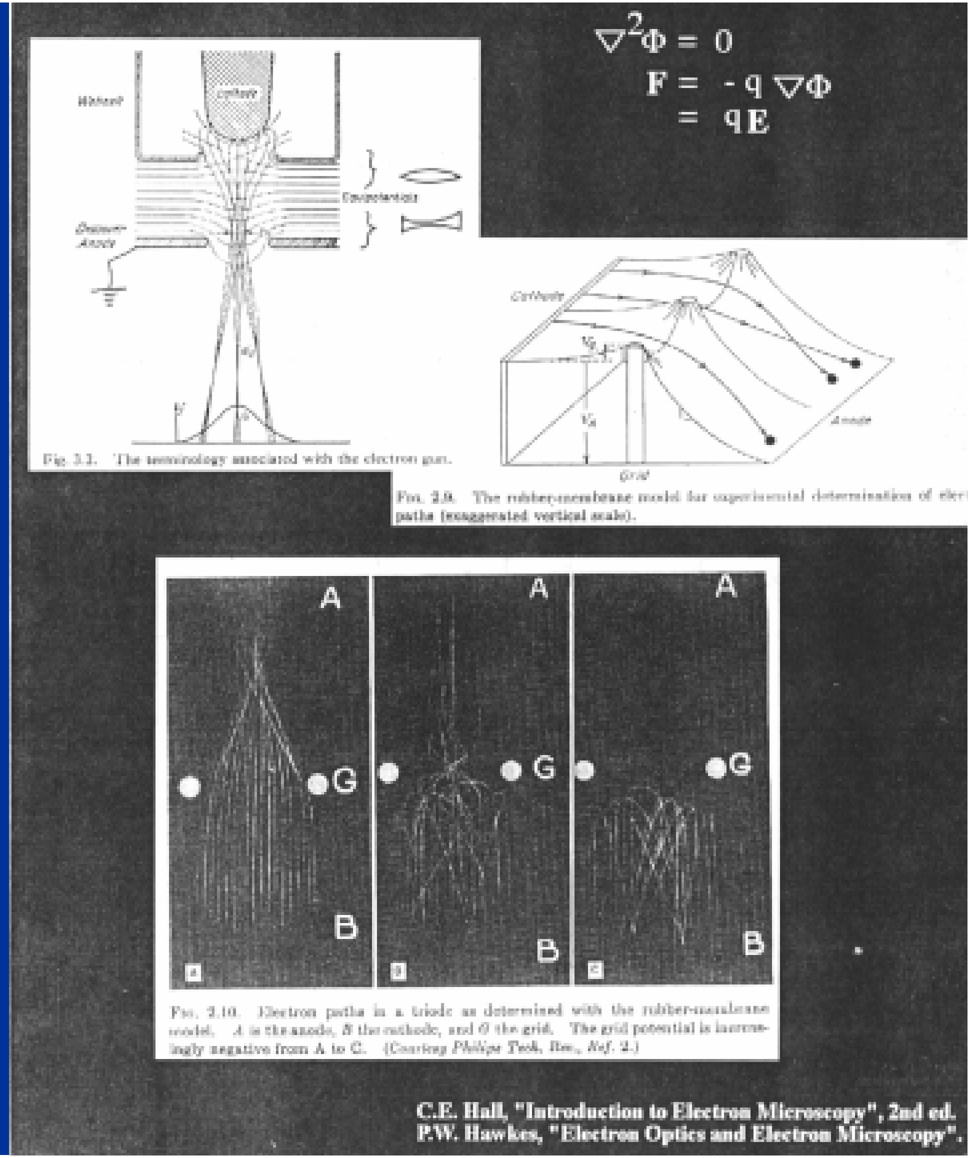
Camera

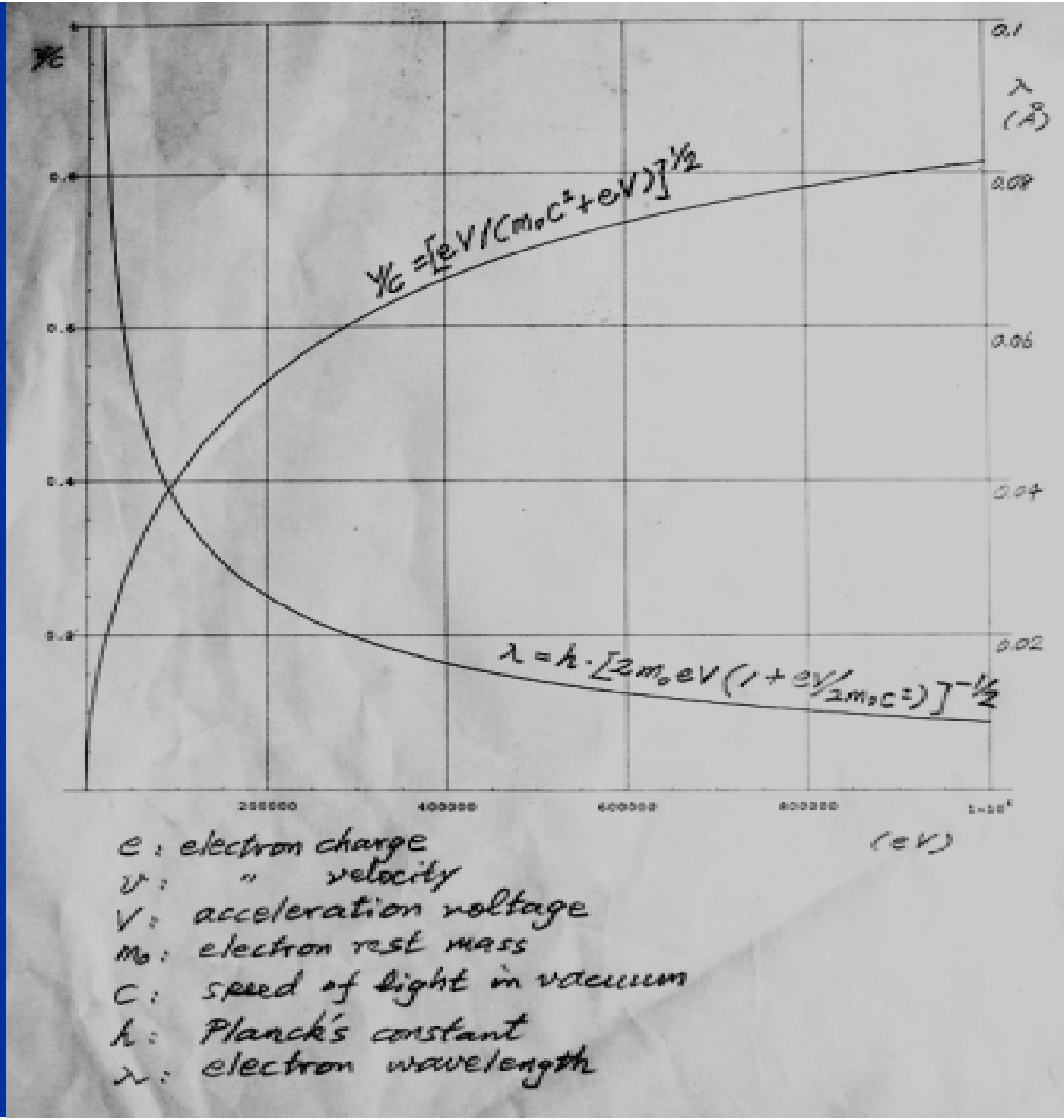
The electron gun:

An electrostatic lens +
an electron accelerator

Filament: Tungsten
LaB_6
Field emission

Acceleration voltage:
(HV or HT)
100kV – 1MV





v/c
λ (Å)
0.1
0.08
0.06
0.04
0.02
v/c = [eV/(m₀c² + eV)]^½
λ = h · [2m₀eV(1 + eV/2m₀c²)]^−½
200000
400000
600000
800000
(eV)
e : electron charge
v : " velocity
V : acceleration voltage
m₀ : electron rest mass
c : speed of light in vacuum
h : Planck's constant
λ : electron wavelength

The electromagnetic lens

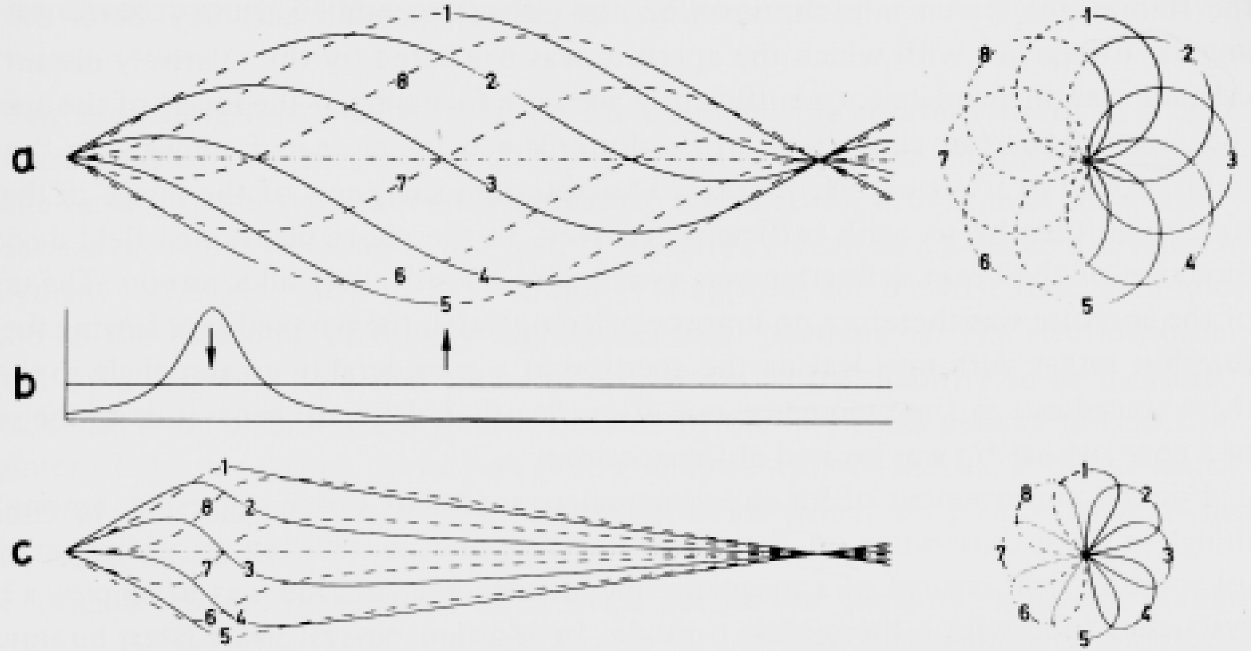
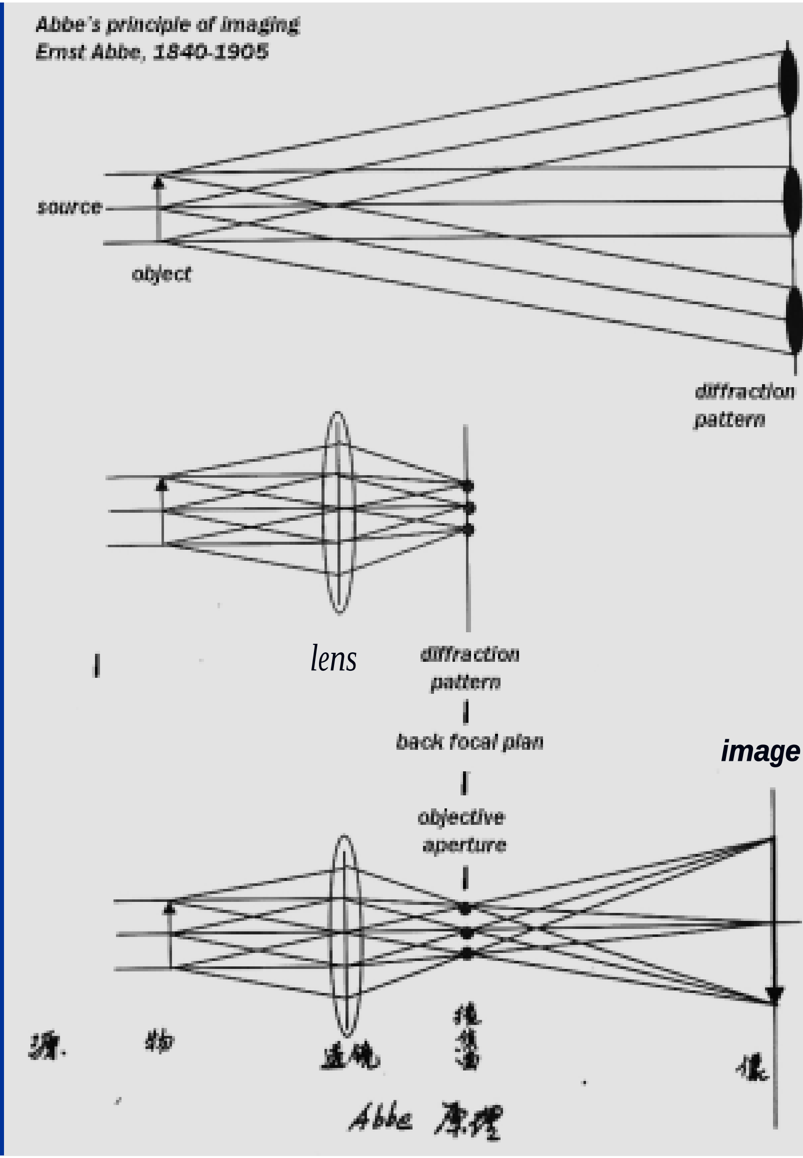


Fig. 1. Electron trajectories in a uniform (a) and in a non-uniform (c) magnetic field, issuing from an axial point of the specimen for different azimuth angles, but making the same angle with the lens axis. (b) Field distributions corresponding to (a) and (c).

"The early development of electron lenses and electron microscopy", Ernst Ruska, 1980, S. Hirzel Verlag Stuttgart

OPTICAL MICROSCOPY

ABBE'S PRINCIPLE



Abbe's Principle of image formation

Principle of Fundamental geometrical and physical optics

Abbe's principle and the back focal plan (BFP)

Contrast: Beam/solid interaction

BFP and the objective aperture:

- Bright field (BF)
- Dark field (DF) images.

Principle of image formation

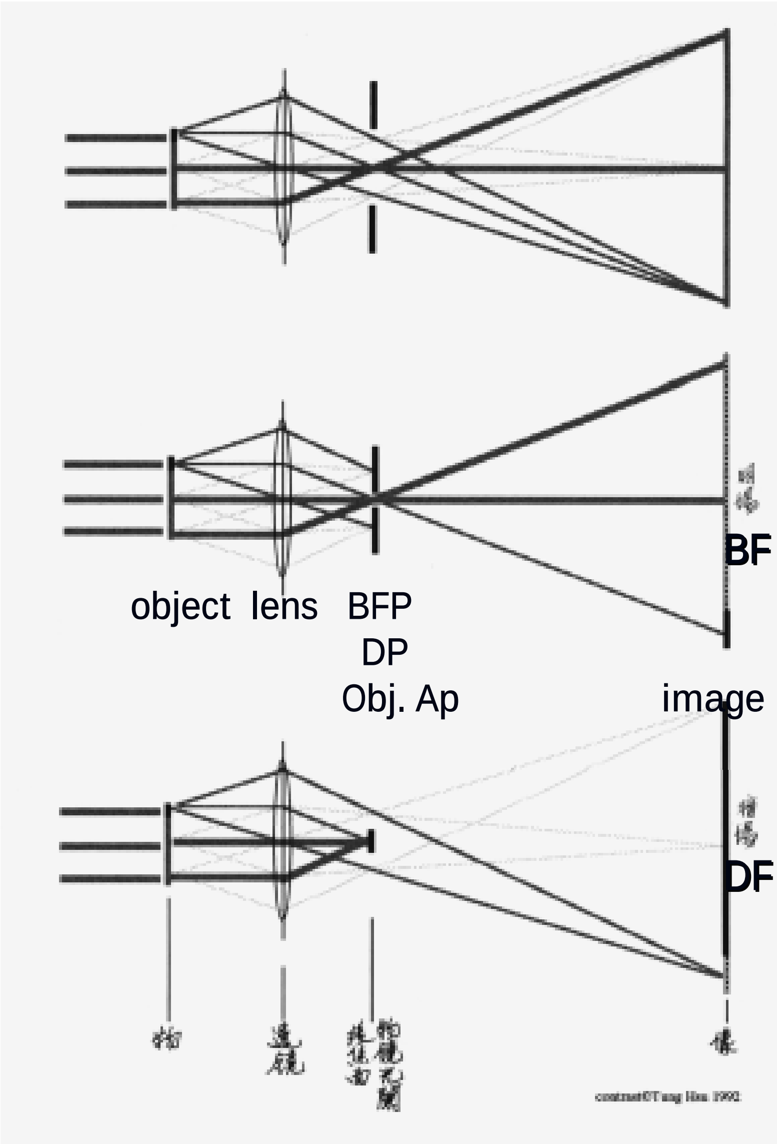
Fundamental geometrical and physical optics

Abbe’s principle and the back focal plan (BFP)

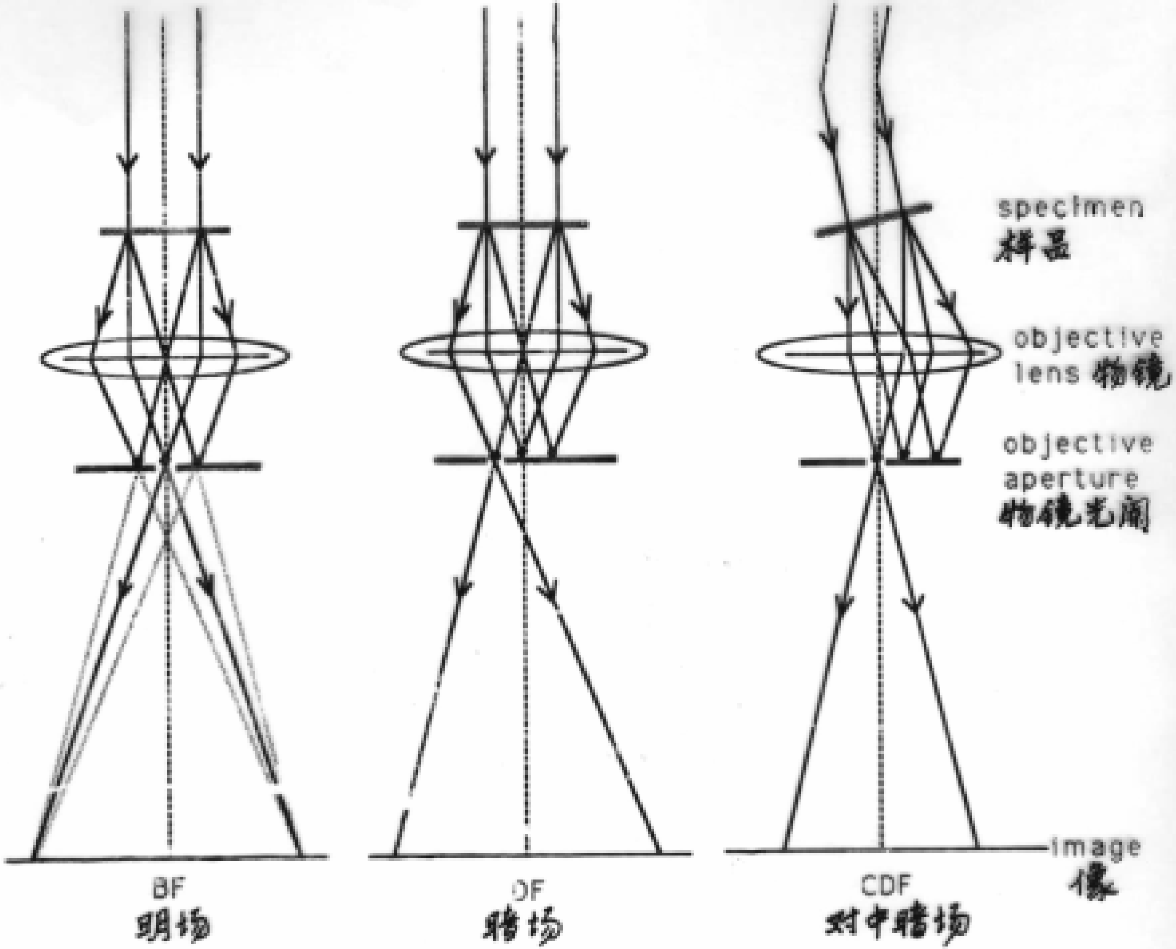
Contrast: Beam/solid interaction

BFP and the objective aperture:

Bright field (BF) and dark field (DF) images.

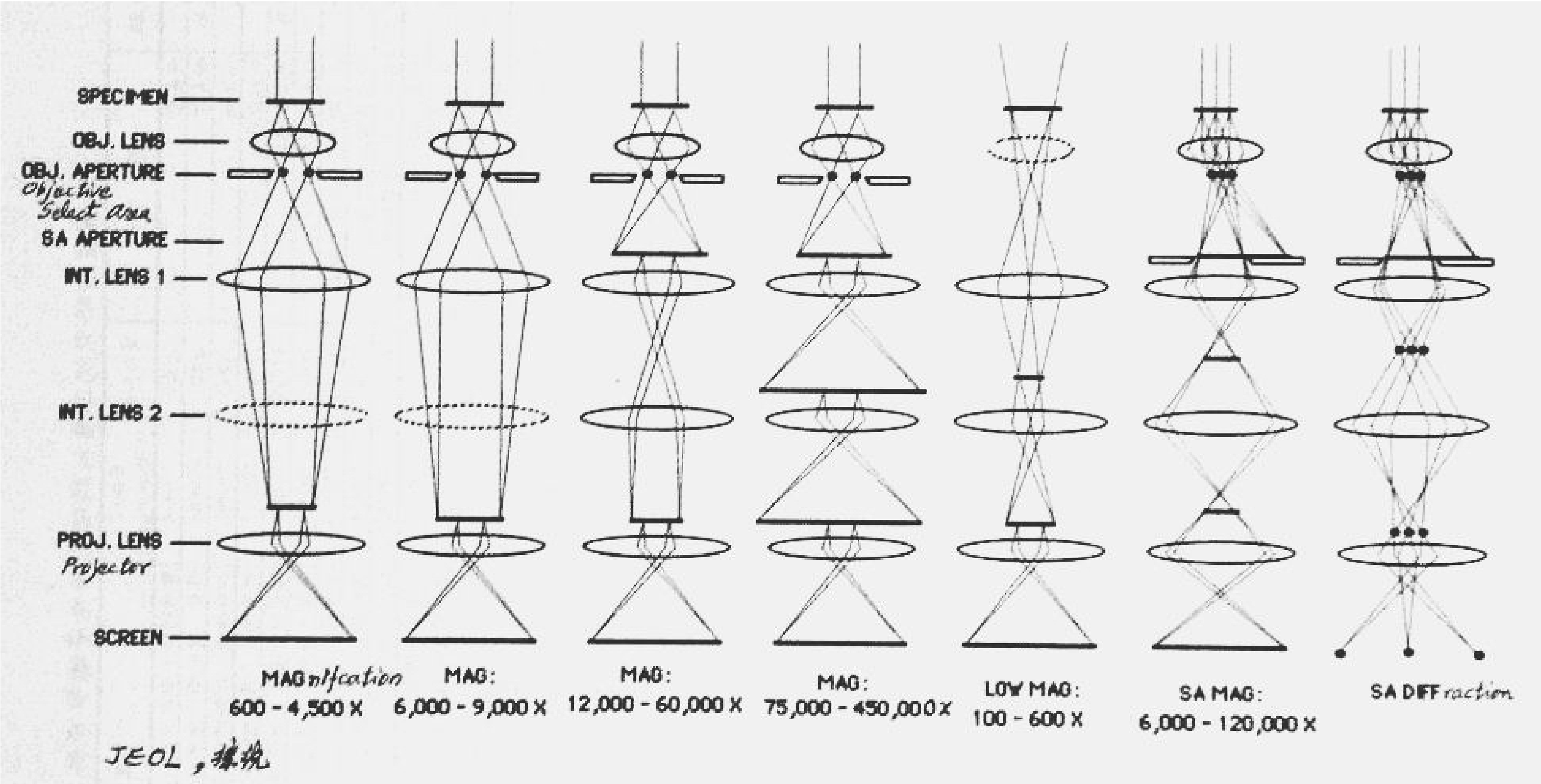


Contrast: Beam/solid interaction
BFP and the objective aperture:
Bright field (BF) and dark field (DF) images.



DIFFRACTION CONTRAST

繞射襯度（衍衬）



The Electron microscope operation

diffraction pattern ↑

Electron micrographs
(EM, TEM images)

And

(Transmission) electron diffraction patterns
(TED patterns, DP)

Diffraction Pattern

Diffraction Contrast

What is Diffraction?

What is DIFFRACTION?

Encyclopedia Britannica 1994-2002

Diffraction the spreading of waves around obstacles. Diffraction takes place with sound; with electromagnetic radiation..., and electrons, which show wavelike properties. One consequence of diffraction is that sharp shadows are not produced. The phenomenon is the result of interference...

Wikipedia 2006-2-2

Diffraction is the bending and spreading of waves when they meet an obstruction. It can occur with any type of wave...

Diffraction also occurs when any group of waves of a finite size is propagating; for example...

Diffraction is one particular type of wave interference, caused by the partial obstruction or lateral restriction of a wave; another example...

Grant R. Fowles, “Introduction to Modern Optics”, 2nd ed., 1975, Dover, p. 106

5.1. General Description of Diffraction

If an opaque object is placed between a point source of light and a white screen, it is found that the shadow that is cast by the object departs from the perfect sharpness predicted by geometrical optics.

Born and Wolf, “Principles of Optics”, 4th ed., 1970.
Ch. VIII. Elements of the theory of diffraction

In carrying out the transition from the general electromagnetic field to the optical field, which is characterized by very high frequencies (short wavelengths), We found that in certain regions the simple geometrical model of energy propagation was inadequate. In particular, we saw that deviation from this model must be expected in the immediate neighborhood of the boundaries of shadows and in regions where a large number of rays meet. These deviations are manifested by the appearance of dark and bright bands, the diffraction fringes.

Hecht “Optics” 2nd ed, 1989

p.3. The phenomenon of diffraction, i.e., the deviation from rectilinear propagation that occurs when light advances beyond an obstruction, was first noted ...

pp. 128-129. ... an optical device is ... unable to collect all the emitted light; the system accepts only a segment of the wavefront... there will always be an apparent deviation from rectilinear propagation even in homogeneous media – the wave will be ***diffracted***.

J.M. Cowley, “Diffraction physics”

(No definitions given)

Feynman “Lectures on Physics” Ch. 30. Diffraction

This chapter is a direct continuation of the previous one, although the name has been changed from ***Interference*** to ***Diffraction***. <u>***No one has ever been able to define the difference between interference and diffraction satisfactorily***</u>. It is just a question of usage, and there is no specific, important physical difference between them. The best we can do, roughly speaking, is to say that when there are only a few sources, say two, interfering, then the result is usually called interference, but if there is a large number of them, it seems that the word diffraction is more often used. So, we shall not worry about whether it is interference or diffraction, but continue directly from where we left off in the middle of the subject in the last chapter.

What else?

We don’t even need the word “diffraction”. What we observe experimentally is the result of wave propagation. When there is an object in the way of the propagating waves, a pattern (intensity distribution) associated with the shape and nature of the object and the nature of the wave is formed. This pattern may be displayed on a screen or recorded with other devices.

This pattern may be called the Fresnel pattern or the Fraunhofer pattern, depending upon the approximations used in describing it.

Related terms:

Scattering (of particles)

Reflection (by atom plans in a solid)

WAVE PROPAGATION, SCATTERING, AND SUPERPOSITION

Electrons fly through the vacuum = electron wave propagating through the vacuum.

Electrons (electron waves) can be scattered by electrostatic potential of atoms.

When two or more electron waves meet, their amplitudes are added.

How to add waves:

Direct method

Amplitude-phase diagram (vector method)

Fourier transform

Optical bench (Atlas)

Computer

Diffraction Patterns from 3D objects

Bragg's Law $n\lambda = 2d \sin\theta$

Examples of electron micrographs and (transmission) electron diffraction (TED) patterns

Contrast mechanism:

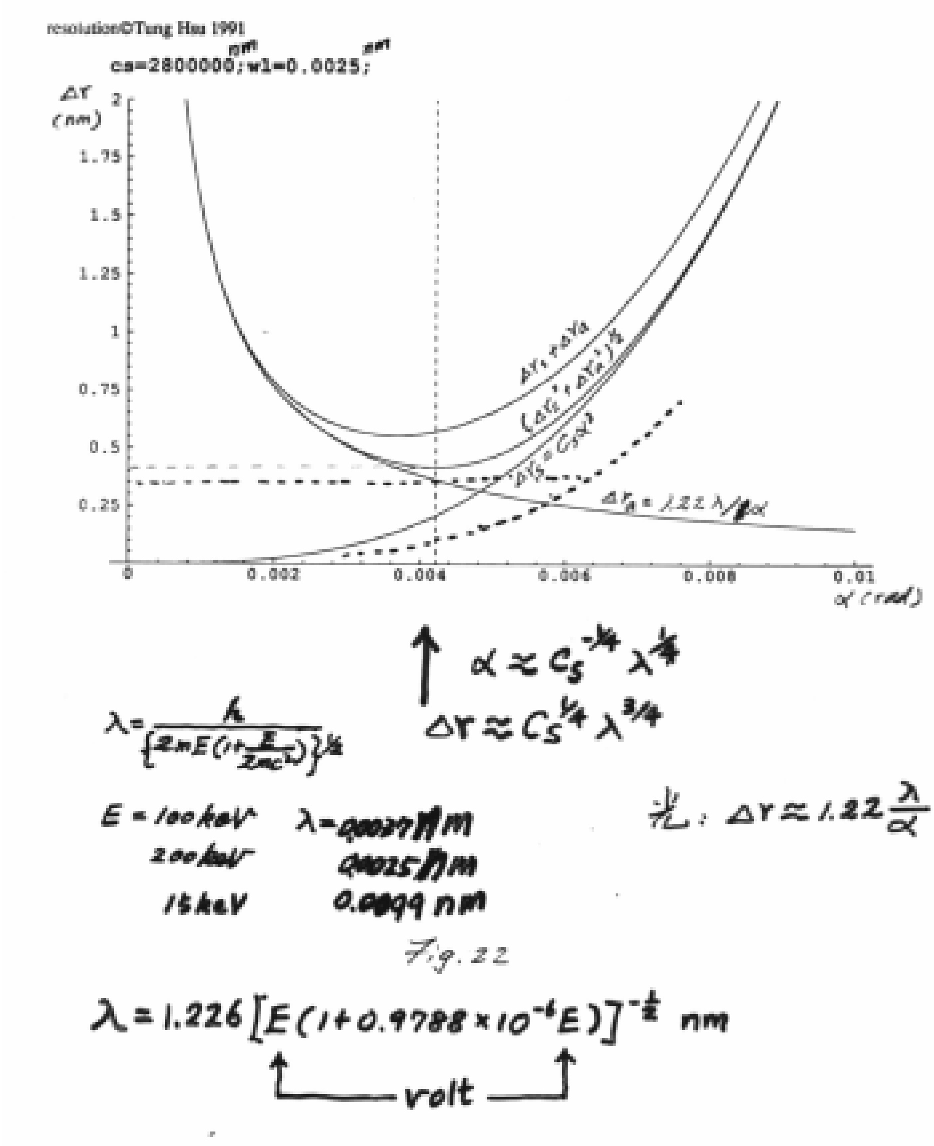
Beam/specimen interaction

- Amplitude and/or phase of the electron waves are altered by the specimen

Properties of lens

- Waves (rays) initiated from a point on the object cannot be converged by the lens to a point on the image.
  - Aperture limitation (“diffraction” related)
  - Spherical aberration
  - Chromatic aberration
  - Defocus (“diffraction” related)
  - Astigmatism

Detector: Fluorescence screen, Film, CCD, eyes



RESOLUTION:

Rayleigh's criterion

Balancing the spherical aberration effect and the diffraction effect:

Smaller aperture produces larger Airy disc (diffraction pattern of the aperture).

Larger aperture produces more diffused disc due to spherical aberration

Specimen preparation –

Specimen: What characterization is all about.

the ultimate limit of resolution and detectability

General requirements:

thin, small, conductive, firm, dry

Various methods

Ultramicrotomy

Mechanical

Chemical

Ion

(Lucky for nano-materials work: Minimal preparation)

Contrast enhancement:

Staining, evaporation, decoration

Specimen support and specimen holders

Specimen support

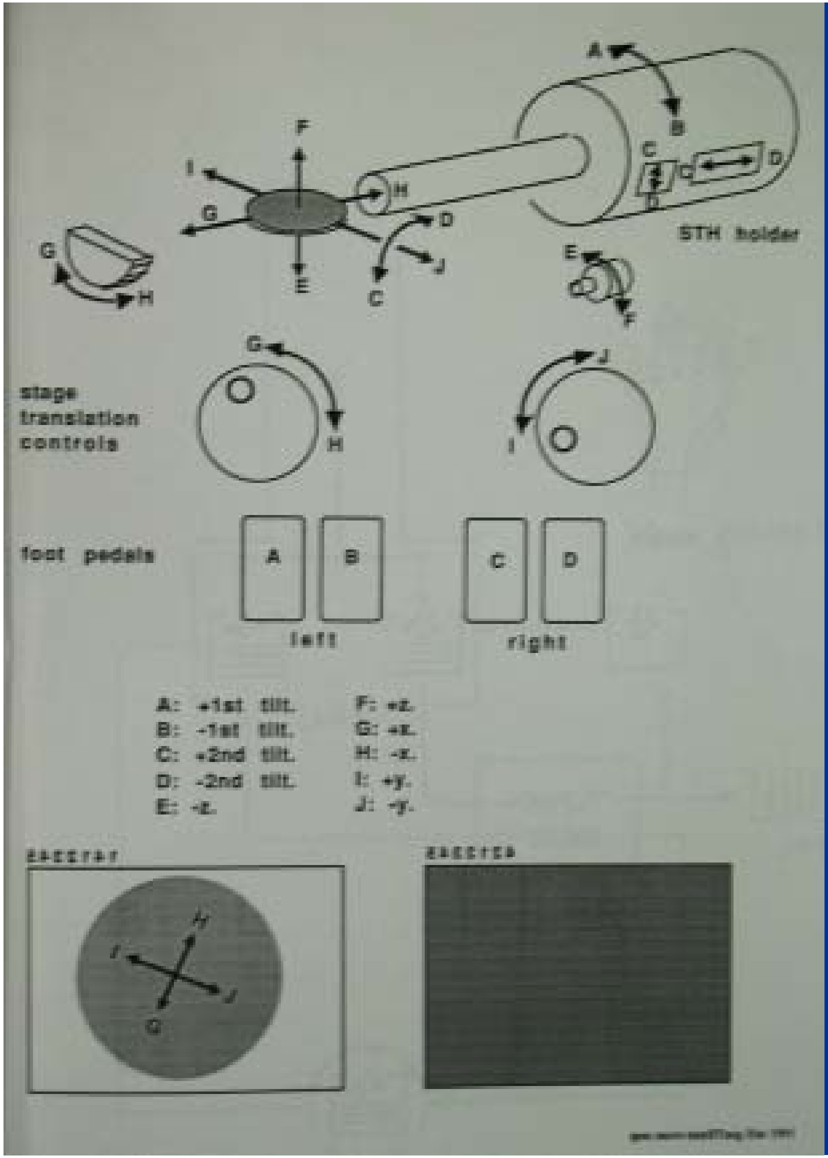
- Grid
- Holey carbon grid

Specimen holders:

- Top entry
- Side entry
- Single/double tilt
- Heating, cooling, tensile, environmental, etc.

Performance:

- Tilt angle, working distance,



Movements and controls of the specimen

Contrast mechanism (again!):
Conventional Transmission Electron Microscopy (CTEM)

Beam/specimen interaction - role of “diffraction”.

- In particular - crystalline specimens
- Atomic scattering factors
- Bending (plastic deformation)
- Thickness variation
- Line defects (dislocations)
- Planar defects (stacking faults)
- Crystal phases

Practice: small aperture, BF or DF.

- poor resolution.
- good contrast.
- good depth of focus.

High Resolution Electron Microscope (HREM):

Approaching atomic resolution.

Requirements:

(Ultra) high resolution pole piece

Electronic stability

Mechanical stability

Clean environment: (Ultra) high vacuum

Specimen preparation: very very thin

In general HREM is needed for studying nano-materials.

Scanning Electron Microscope (SEM)

O.L. Wells

Introduction 3

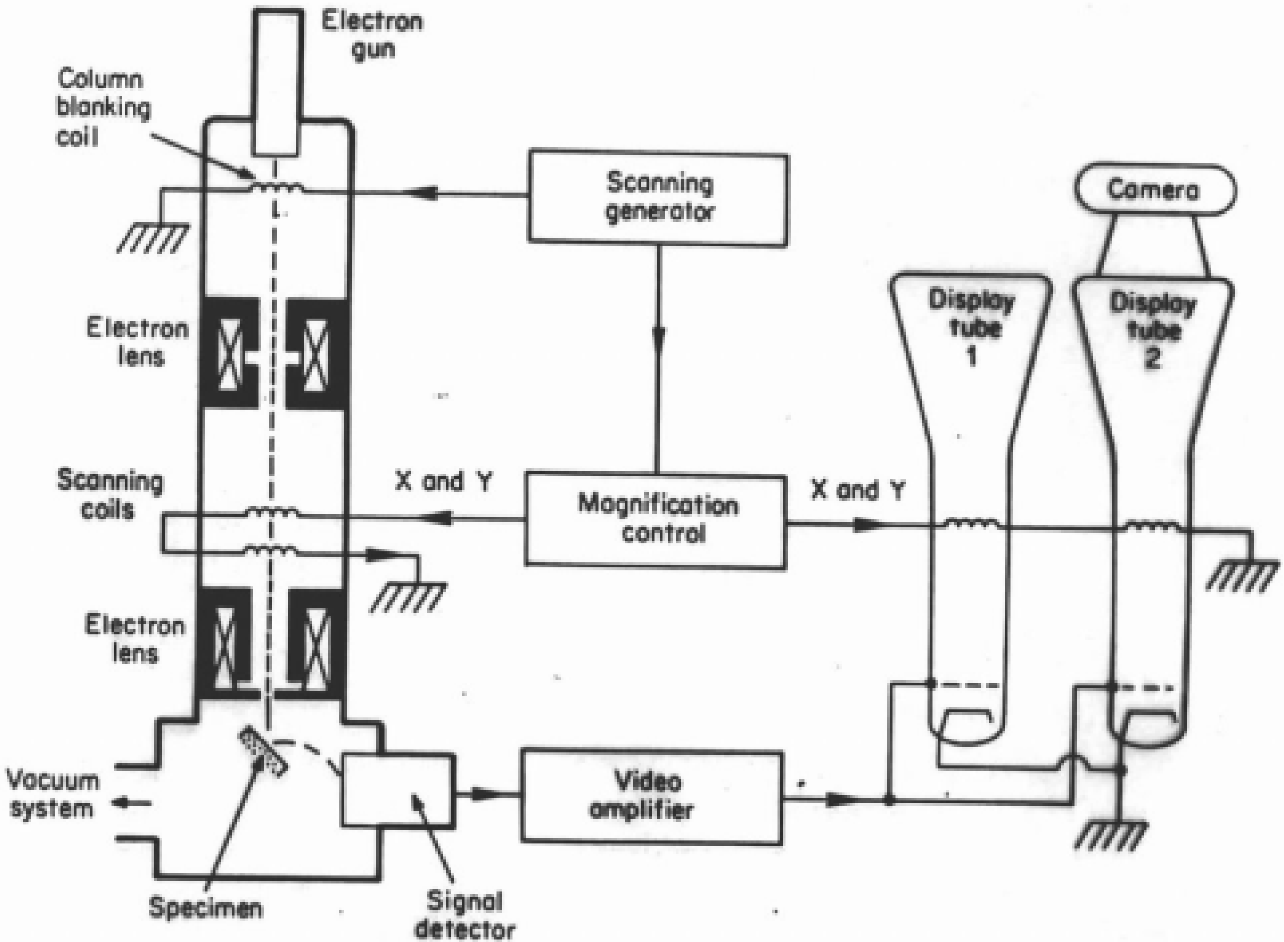


FIG. 1.2 Scanning electron microscope.

SEM:

Electron optics column (Probe forming system):

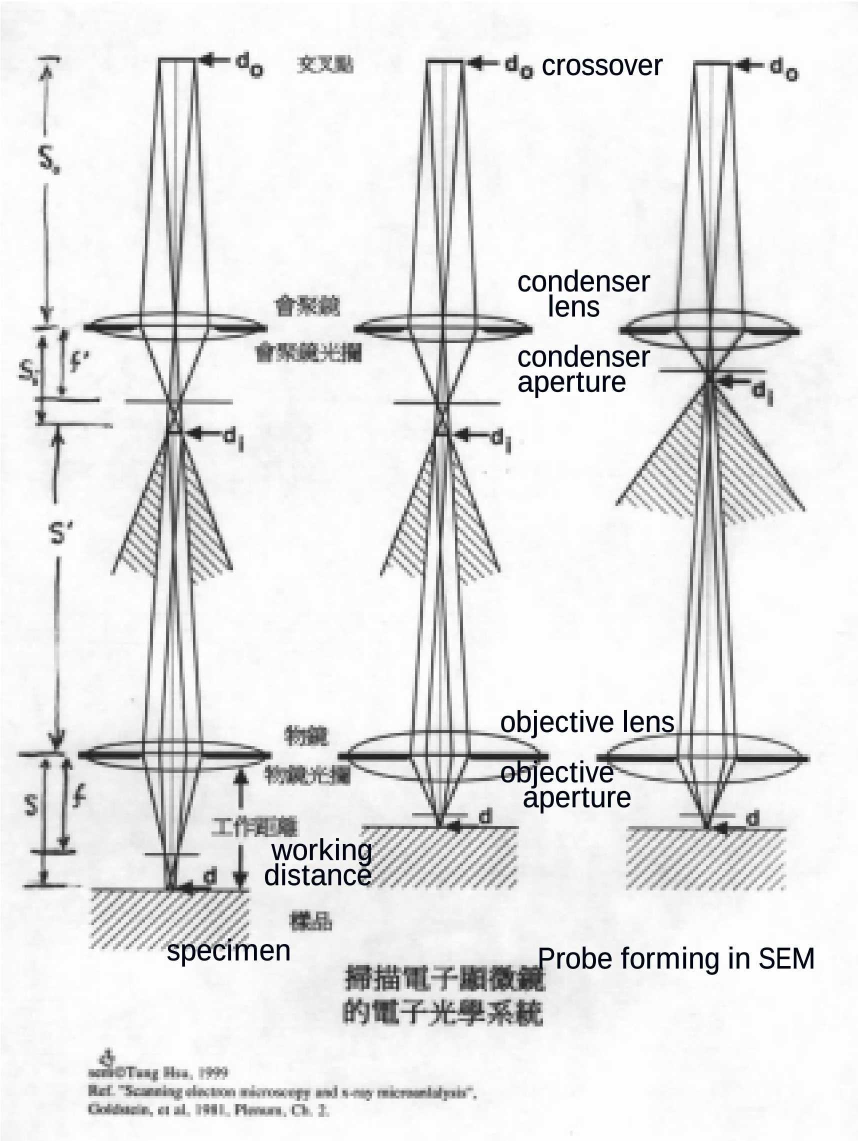
- Lens defects and probe size: same principles as in TEM.

Contrast mechanism:

- Scanning
- Beam/solid interaction (semi-infinite specimen)
- Detectors
- Resolution

Performance:

- Resolution much better than that of Optical microscope.
- “Surface” sensitive.
- Large depth of field.
- Easy to prepare specimens.
- Various modes of scanning and signal detection.



Scanning electron microscopy – microprobe

Beam/specimen interaction: When the specimen is thick, "semi-infinite".

Monte Carlo simulation

The probe forming system:

- Forming a small probe is the same as forming a small spot in the image
- The column

Contrast mechanism:

- Secondary electrons
- Back scattered electrons
- Other signals

Resolution:

- Low mag: limited by scan rate
- High mag: limited by lens defects – same as TEM

Detector

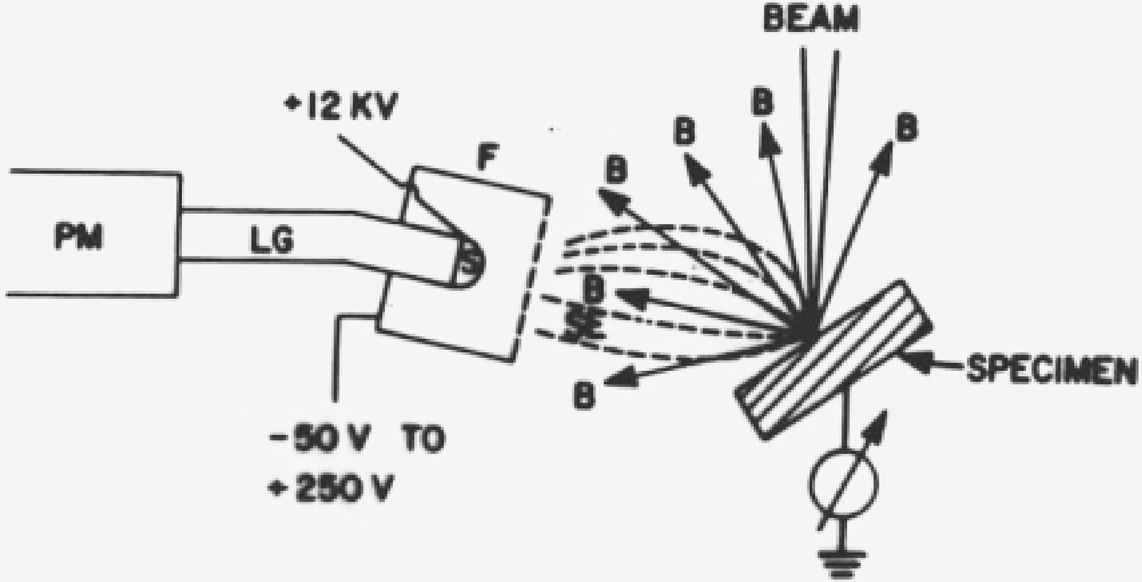


Figure 4.17. Schematic diagram of Everhart–Thornley scintillator–photomultiplier electron detector. B, backscattered electron; SE, secondary electron; F, Faraday cage; S, scintillator; LG, light guide; PM, photomultiplier.

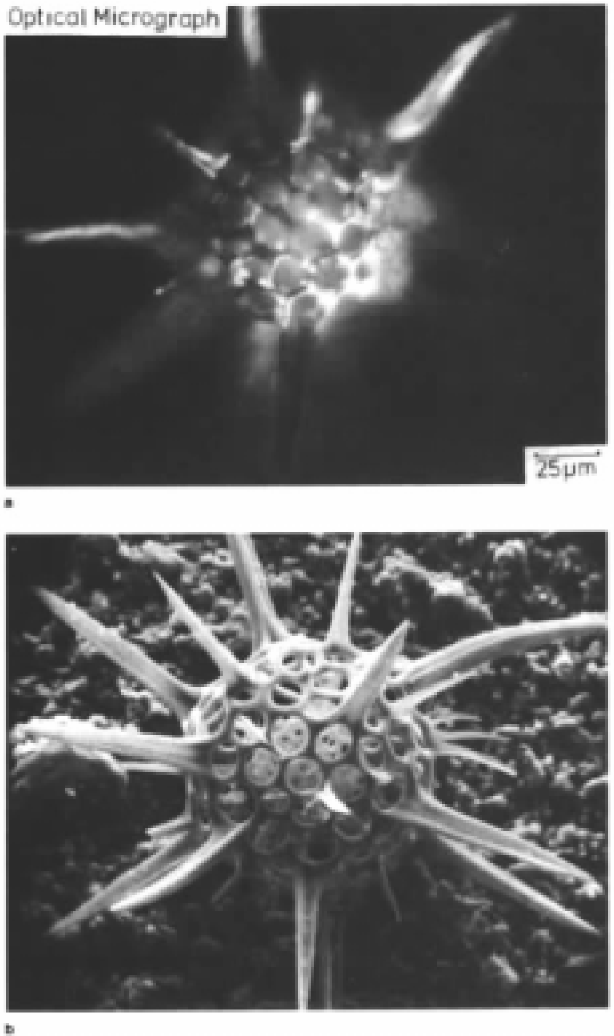
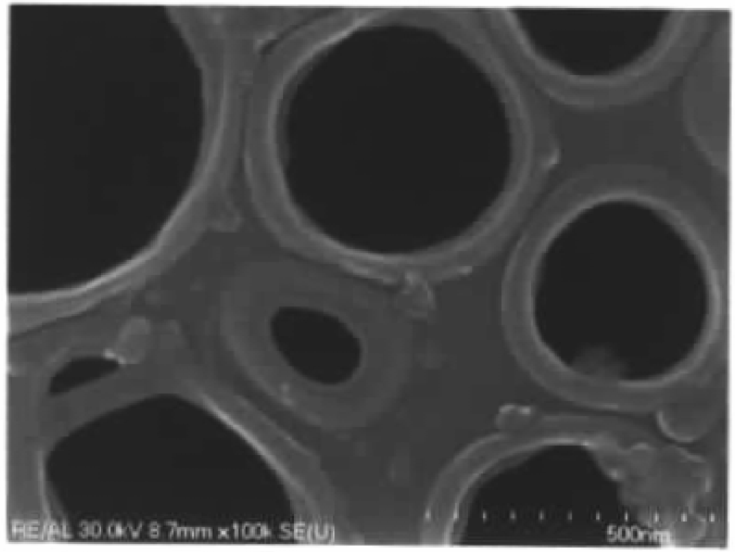
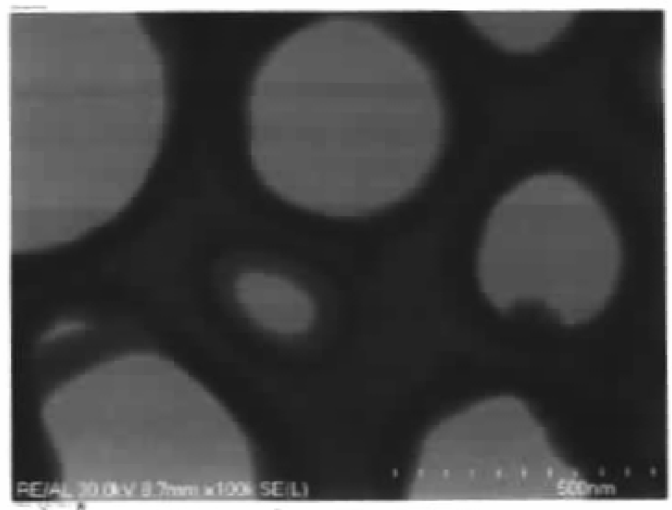


Figure 1.3. (a) Optical micrograph of the radiolarian *Trochodiscus longispinus*. (b) SEM micrograph of same radiolarian. The greater depth of focus and superior resolving capability of the SEM are apparent.





郑？, 2001 (Hitachi S-4700)

Examples of SEM images

	SEM			TEM		
E (kV)	10	20	30	100	200	400
λ (A)	0.122	0.0859	0.0698	0.037	0.025	0.0126
Cs (mm)		10-20			1-3	

Resolution: beam size — image point size

SEM: $r = \lambda^{3/4} Cs^{1/4}$

TEM: $r = \lambda^{3/4} Cs^{1/4}$

Electron microprobe / Analytical electron microscopy:

Energy dispersive (X-ray) spectrometer, EDS (EDX)

Wavelength dispersive (X-ray) spectrometer, WDS (WDX)

Electron energy loss spectroscopy, EELS

Quantitative analysis

etc.

	Co	Ni	Te	I
Mendeleev:	A = 58.9	58.6	127.7	126.9
Moseley:	Z = 27	28	52	53

Parameter	$n\lambda$	2d	$\sin\theta$
diffraction	known	calculated	measured
spectrometry(WDS)	calculated	known	measured
spectrometry(EDS)	E: measured		

$E = hc/\lambda$, $\lambda = C'(Z - \sigma)^{-2}$ (for the same spectral line, $K\alpha$, $K\beta$, ...)

Instrumentation: Electron probe/microscope

Other particle beam

x-ray fluorescence

radioactive sources

WDS: X-ray optics

regular crystals ⇒ O and up

"soap" film crystals ⇒ Be and up

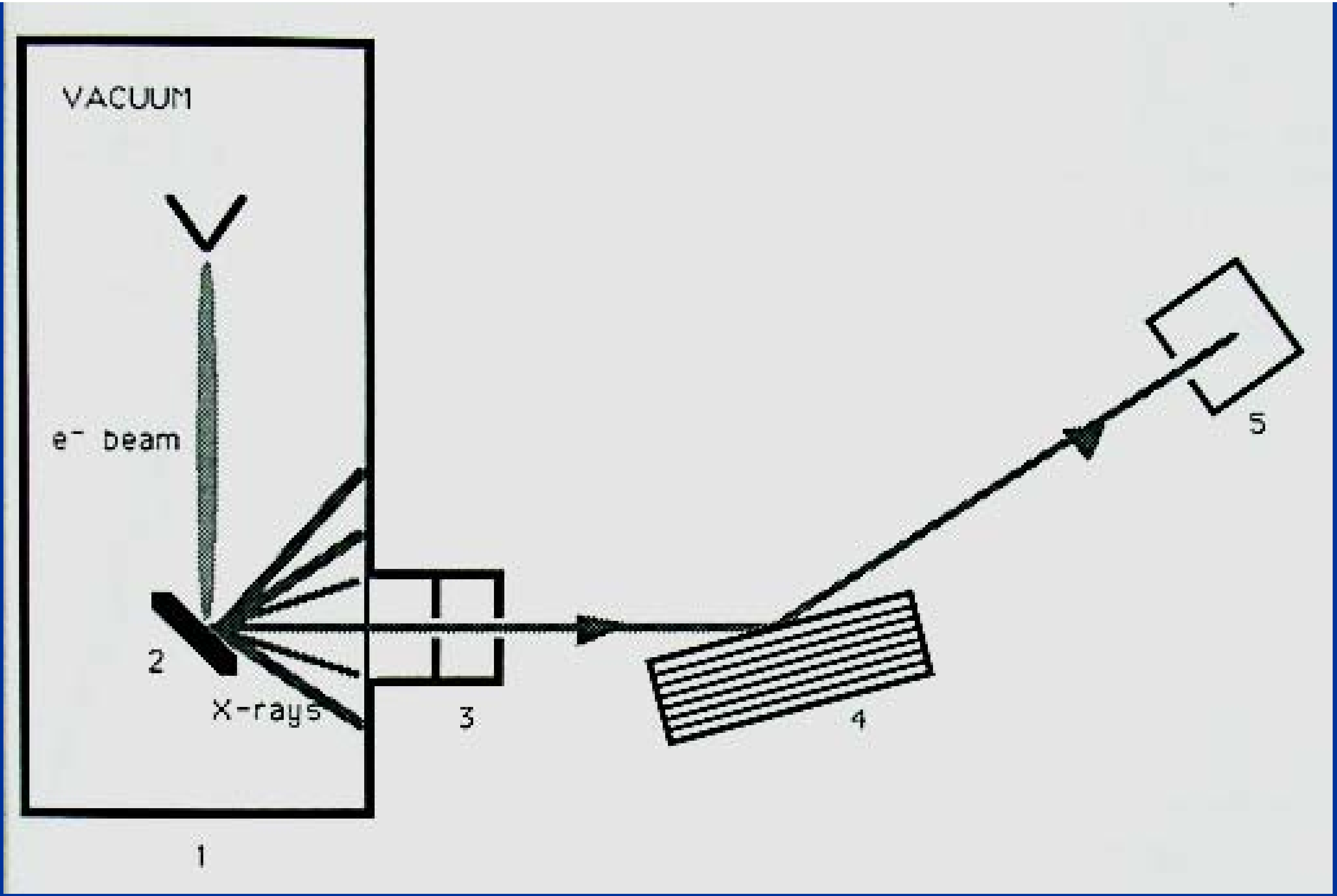
EDS: Si(Li) detector

Multi-channel analyzer (MCA)

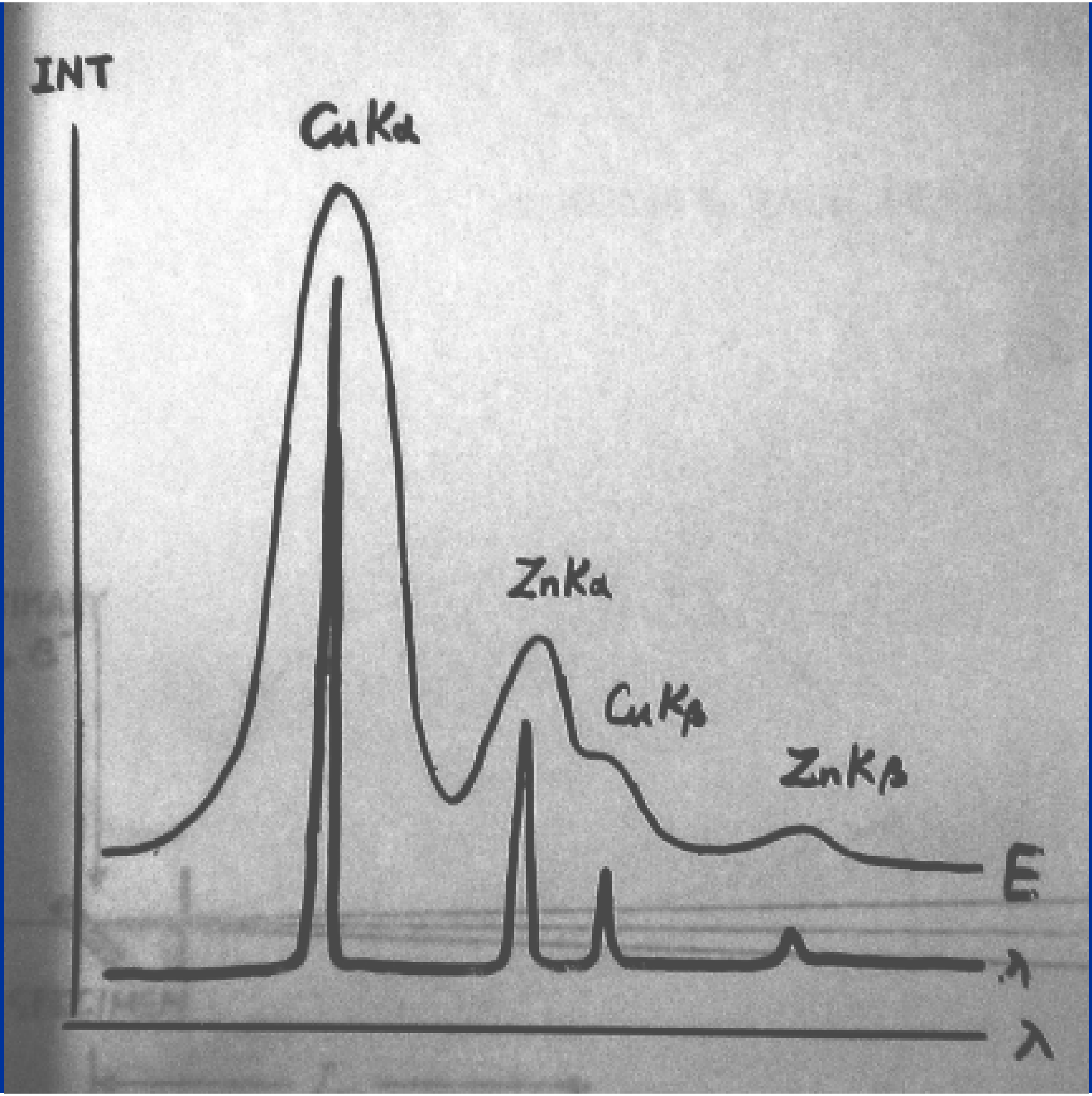
Be window ⇒ Na and up

Ultra-thin window or Windowless ⇒ B and up

Dead layer in Si(Li) detector is the limit



XRD and WDS



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Microscopy Society of America Position on Ethical Digital Imaging

RESOLUTION carried as follows: Be it resolved that the MSA position on digital image processing be approved as follows:

"Ethical digital imaging requires that the original uncompressed image file be stored on archival media (*e.g.*, CD-R) without any image manipulation or processing operation. All parameters of the production and acquisition of this file, as well as any subsequent processing steps, must be documented and reported to ensure reproducibility.

Generally, acceptable (non-reportable) imaging operations include gamma correction, histogram stretching, and brightness and contrast adjustments. All other operations (such as Unsharp-Masking, Gaussian Blur, *etc.*) must be directly identified by the author as part of the experimental methodology. However, for diffraction data or any other image data that is used for subsequent quantification, all imaging operations must be reported."

MSA 2003 Summer Council Meeting Minutes

Microscopy Today, 11:6(2003) 61.