ELECTRON MICROSCOPY

13:10 – 16:00, Nov. 5, 2007
Institute of Physics, Academia Sinica

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References:

"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, I976, 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

Scanning electron microscopy

Electron microprobe / Analytical electron microscopy

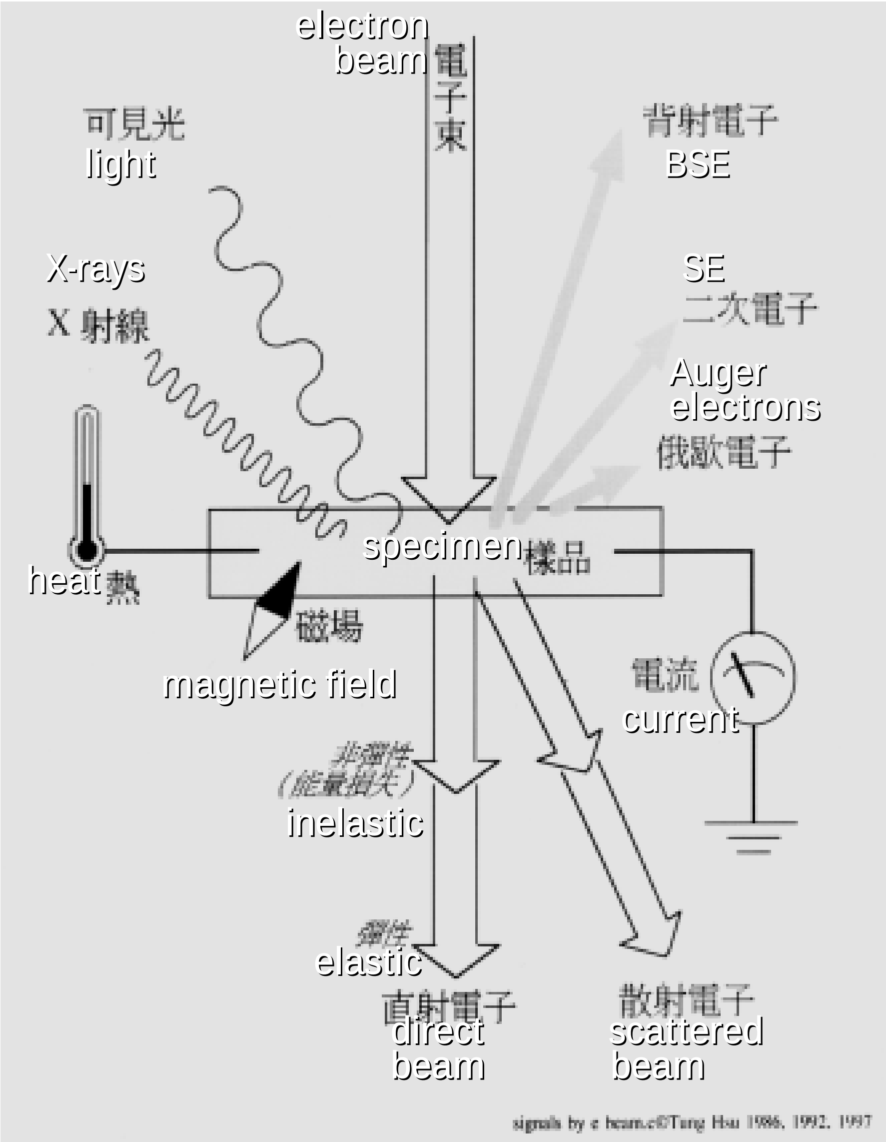
Introduction:

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

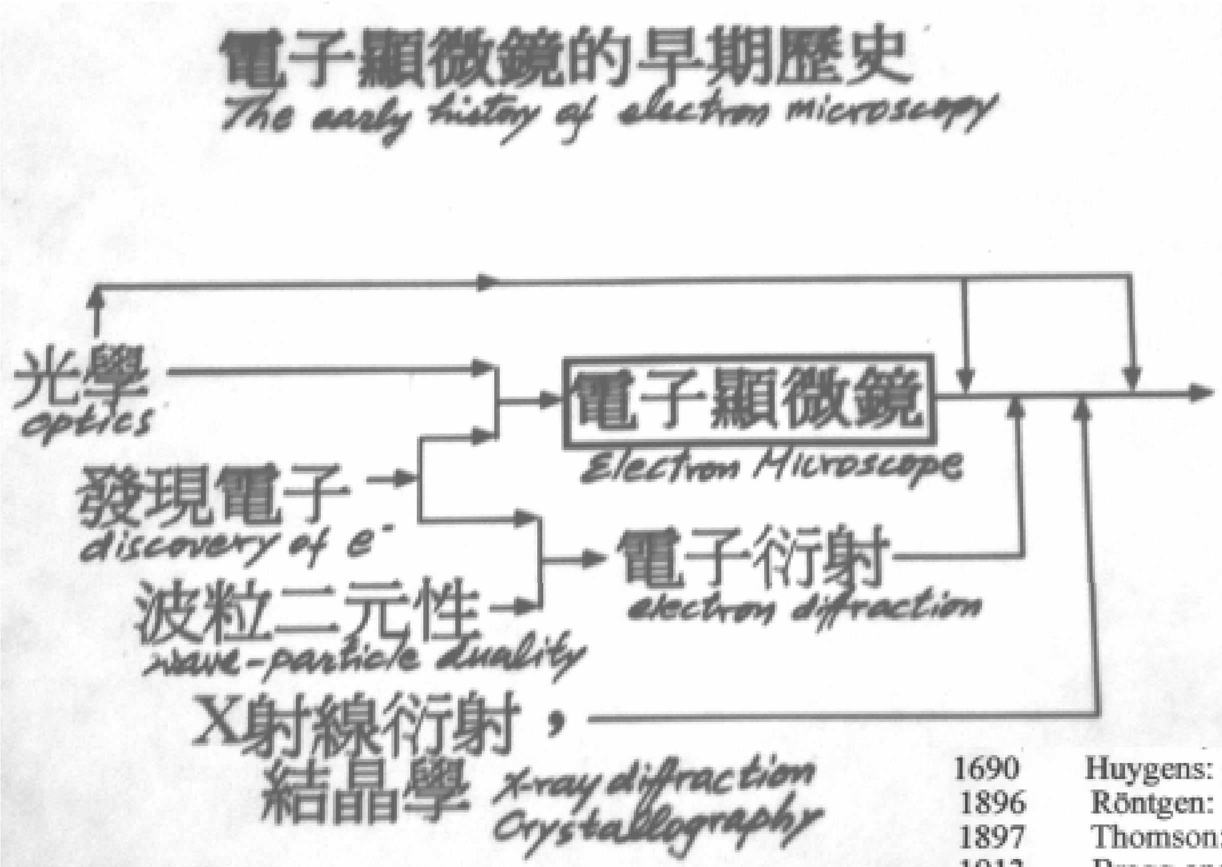
- image: morphology
  - scattering power
  - crystal structure
  - crystal defects
  - atomic structure
- other than the image:
  - (chemical) elemental composition
  - electronic structure

(Spatial) Resolution:

- Microscopy vs. microprobe
- Wavelength, properties of lens

A brief history of electron microscopy

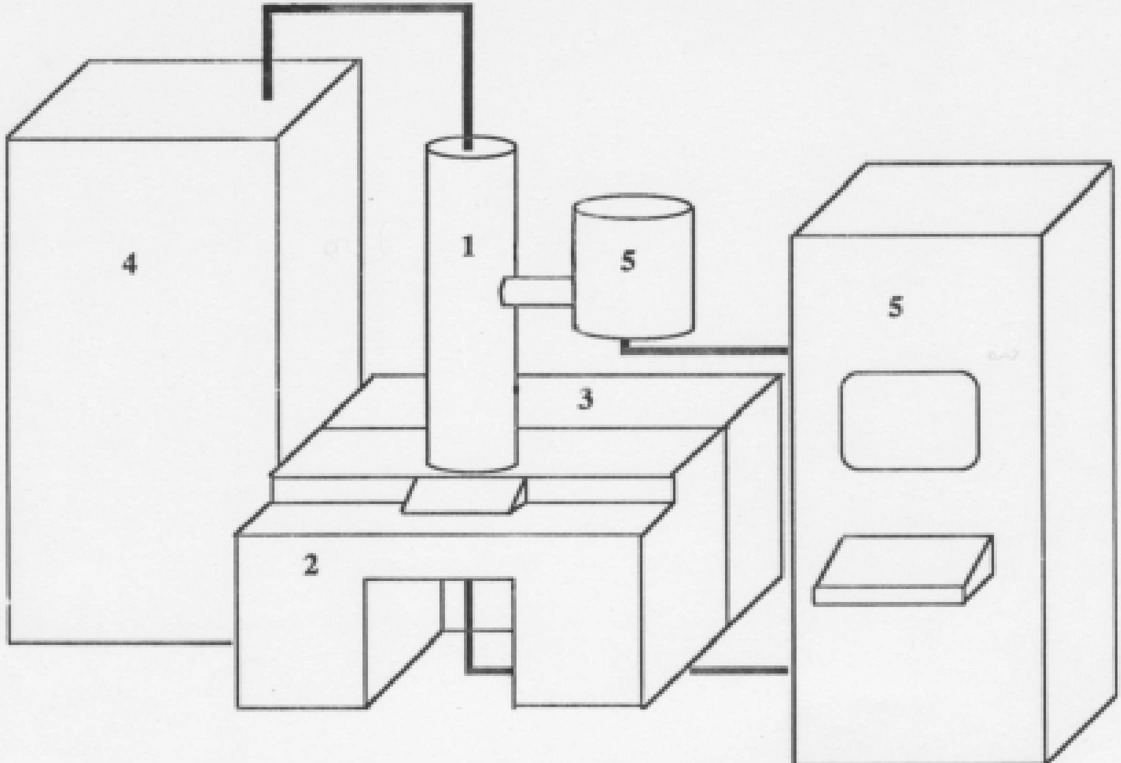
(vg)



Various Electron Microscopes

(vg)

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



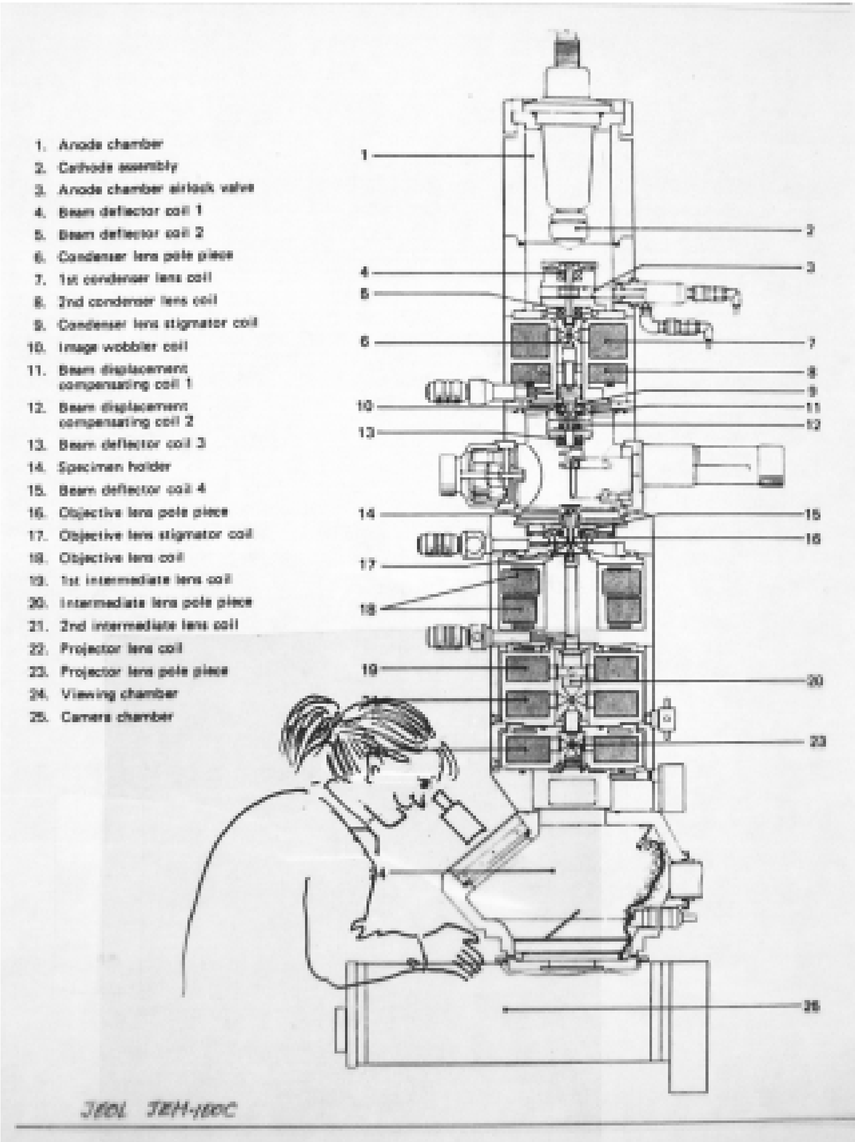
穿透式电子显微镜的主要部件

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

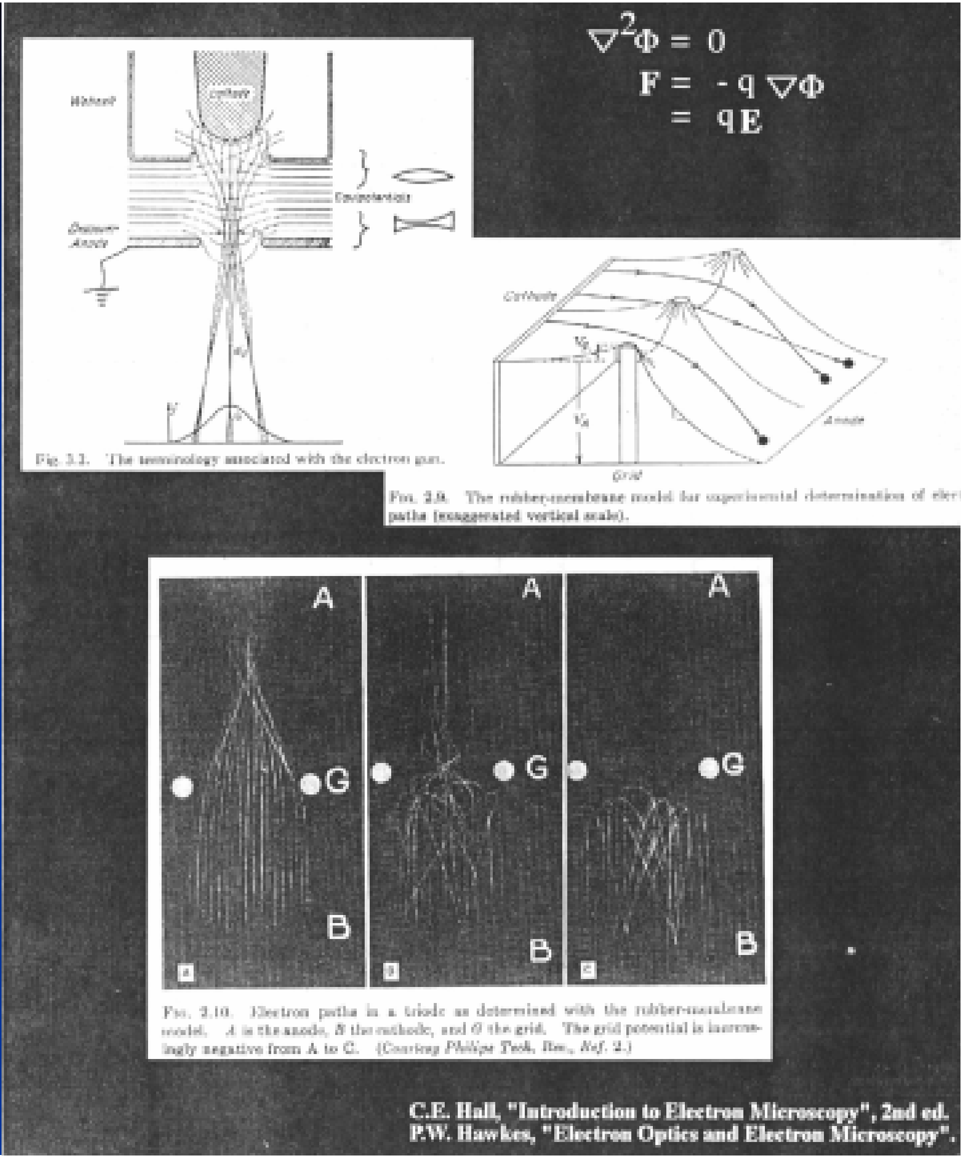
Camera

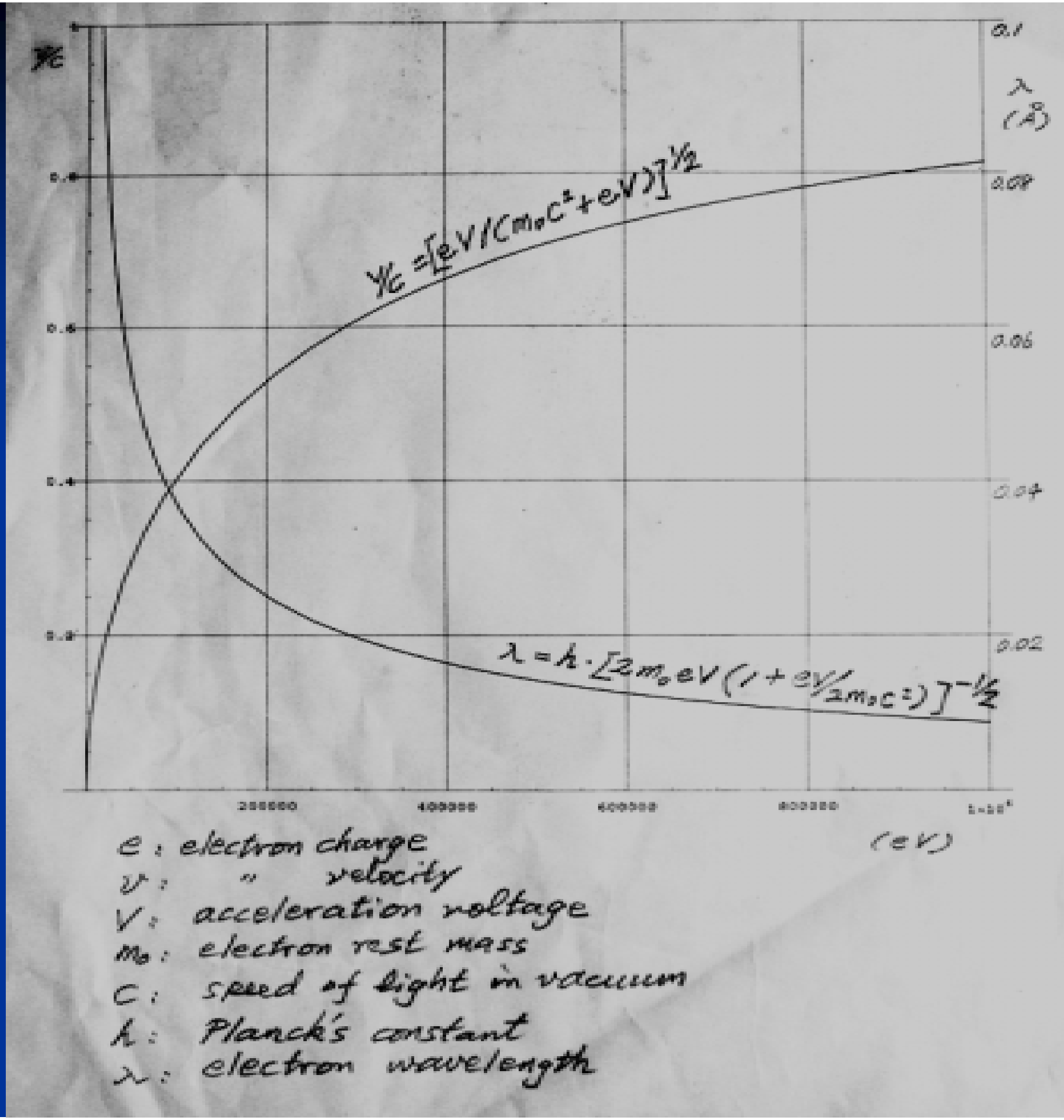
The electron gun:

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
200000
400000
600000
800000
(eV)
v/c = [eV/(m₀c² + eV)]^½
λ = h·[2m₀eV(1 + eV/2m₀c²)]^-½
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

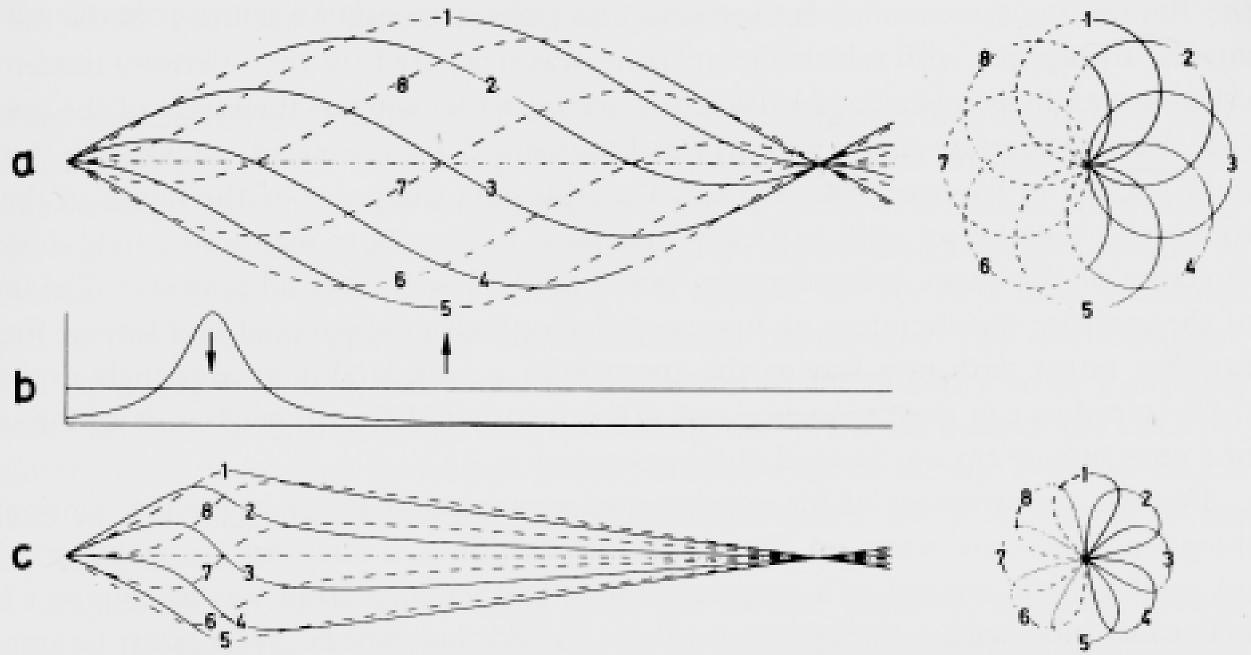
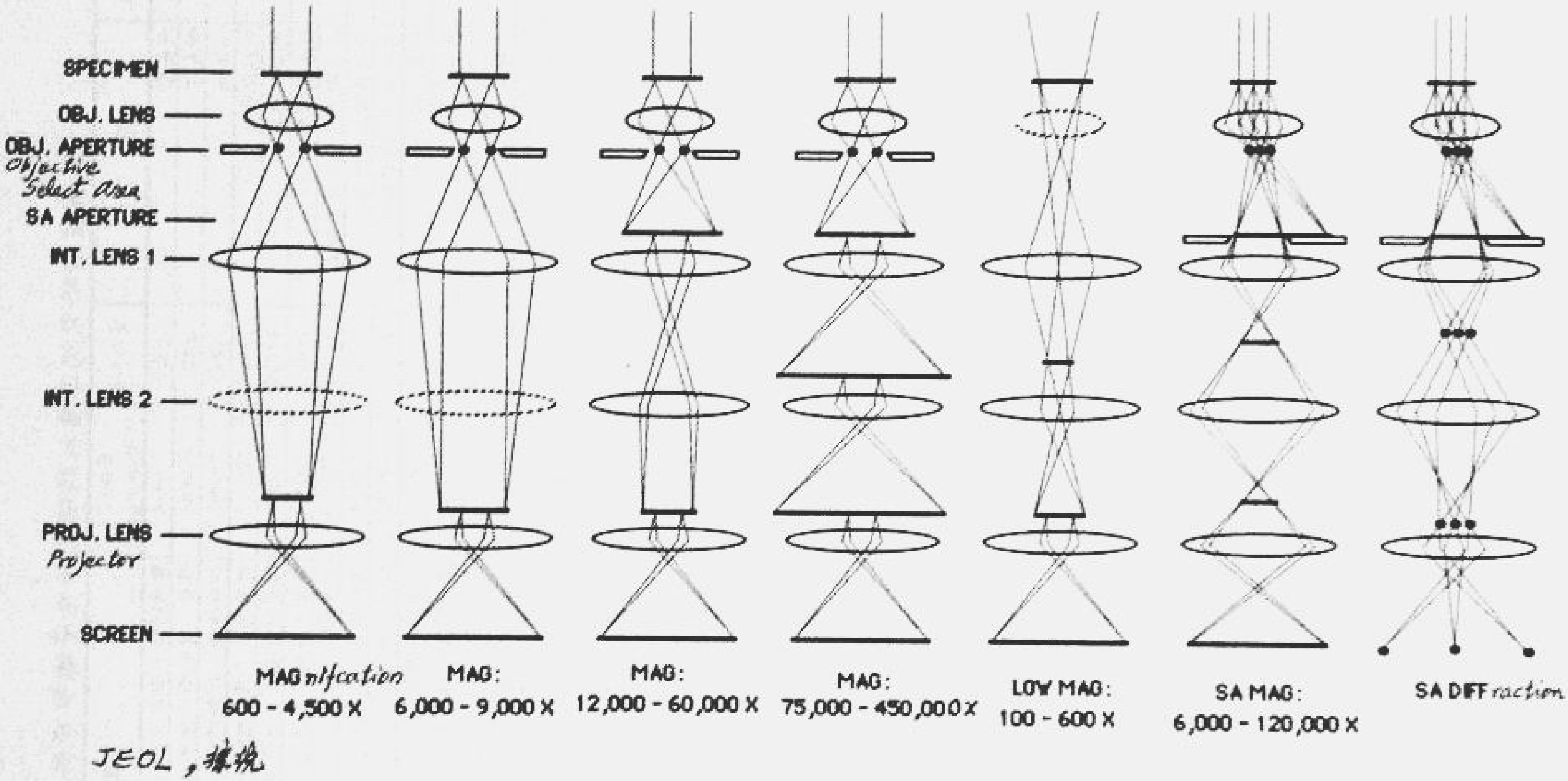


Fig. 1. Electron trajectories in a uniform (a) and in a non-uniform (c) magnetic field, issuing from an axial point of the specimen for different azimuth angles, but making the same angle with the lens axis. (b) Field distributions corresponding to (a) and (c).

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



The Electron microscope operation

diffraction pattern ↑

Principle of image formation

Fundamental geometrical and physical optics

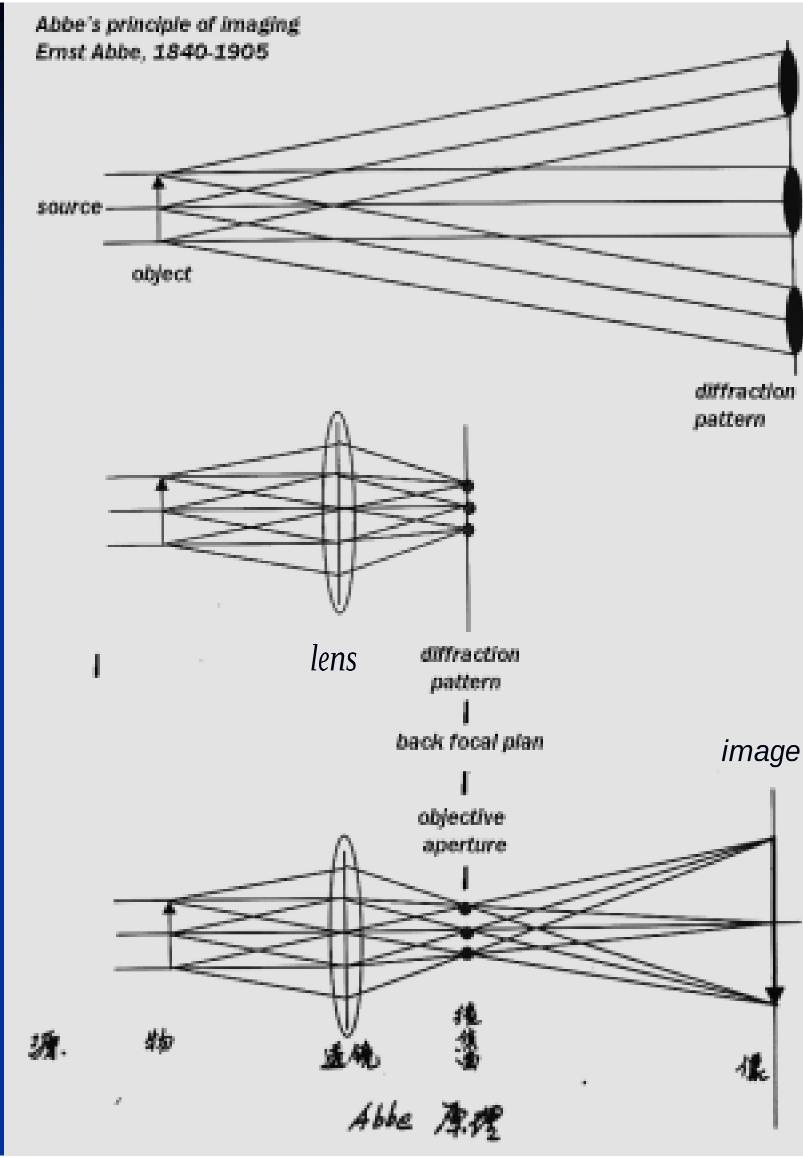
Abbe’s principle and the back focal plan (BFP)

Contrast: Beam/solid interaction

BFP and the objective aperture:

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

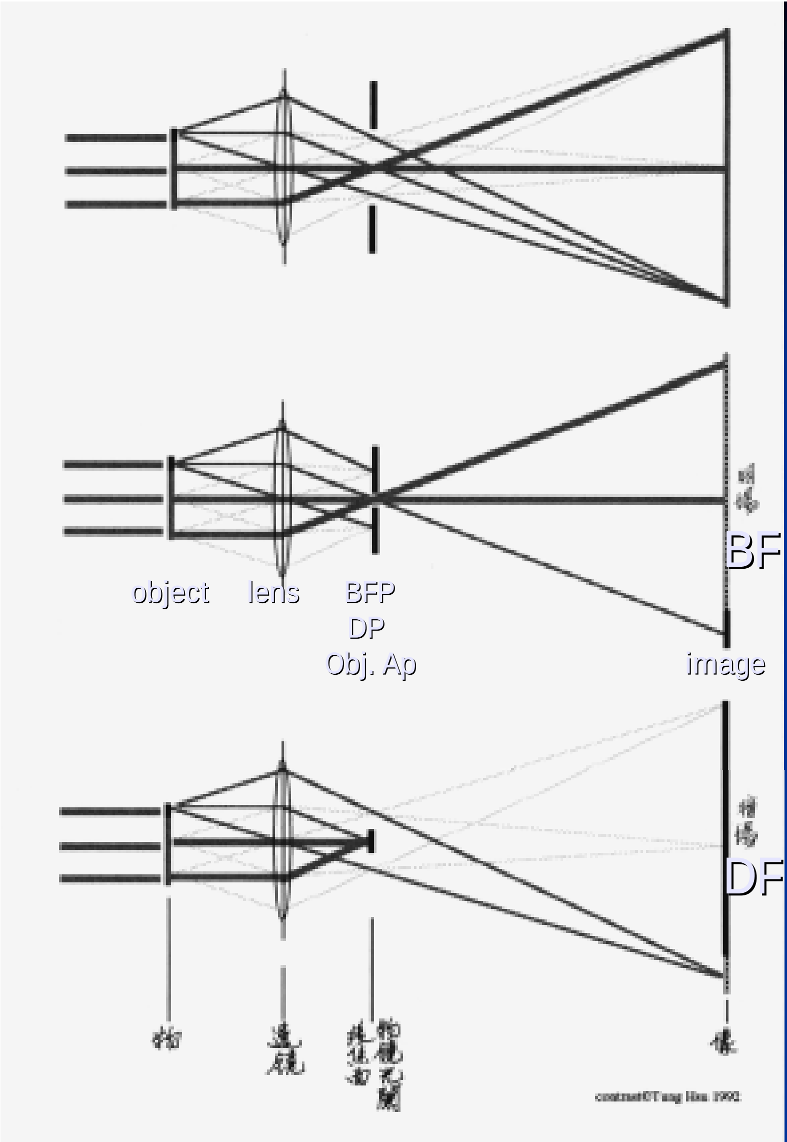
(vg)



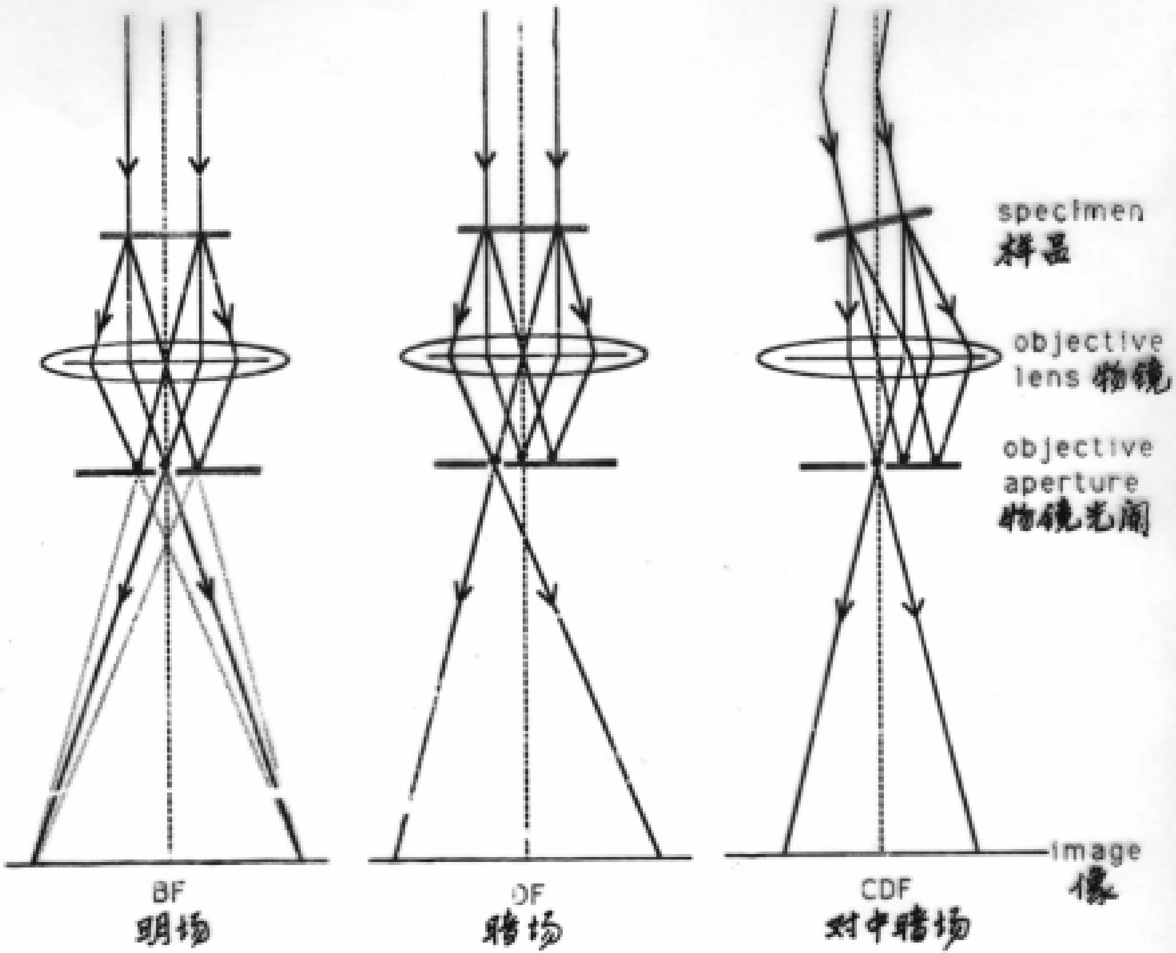
Abbe's Principle of image formation

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.

(vg)



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



DIFFRACTION CONTRAST

衍射衬度（衍衬）

Diffraction Pattern

Diffraction Contrast

What is Diffraction?

What is DIFFRACTION?

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 associated with the shape and nature of the object and the nature of the wave is formed. This can 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 (vg)

Amplitude-phase diagram (vector method) (vg)

Fourier transform

Optical bench (Atlas) (vg)

Computer (DigitalMicrograph)

Diffraction Patterns from 3D objects

Bragg's Law (vg)

$$n \lambda = 2d \sin \theta$$

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

(vg)

Contrast mechanism:

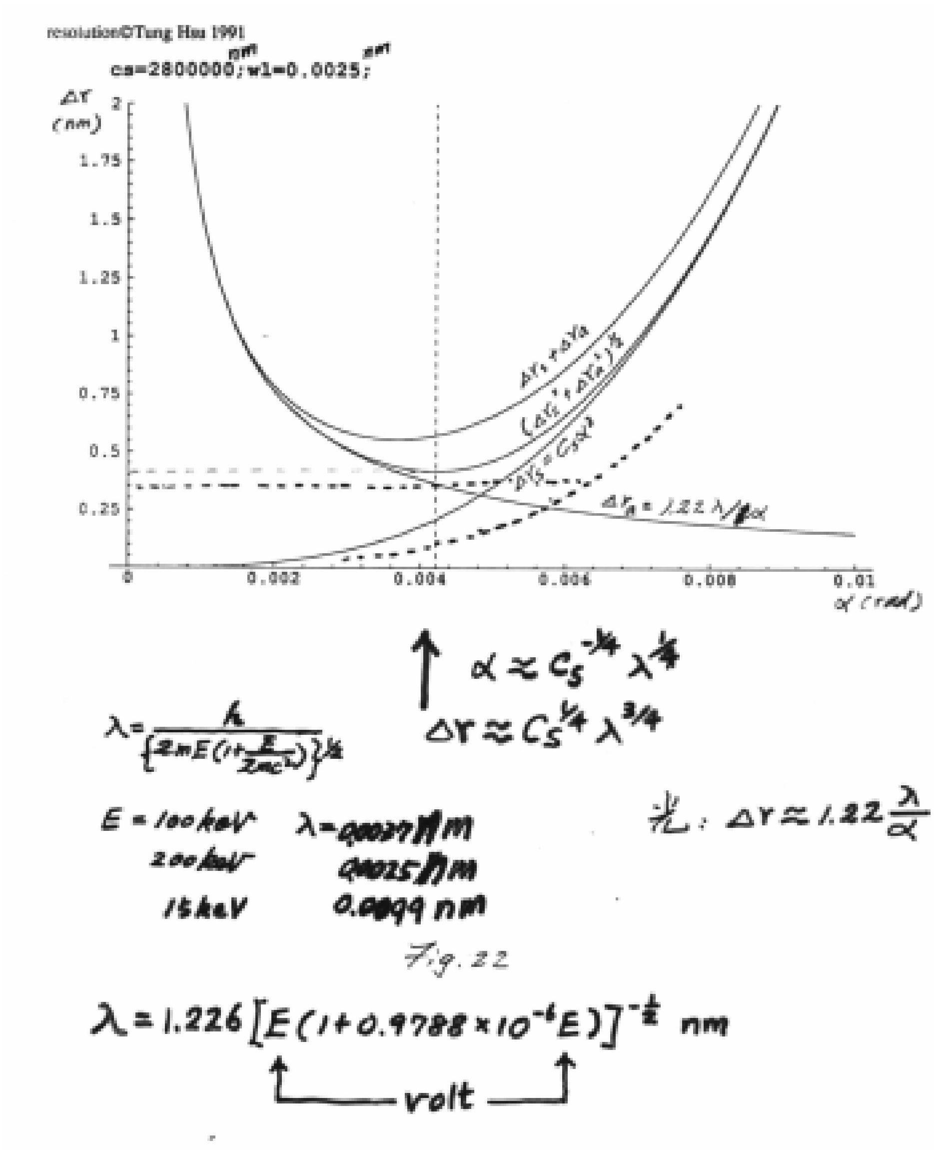
Beam/specimen interaction

- 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
  - Spherical aberration
  - Chromatic aberration
  - Defocus
  - Astigmatism

Detector: Film, CCD



RESOLUTION:

Rayleigh's criterion (vg)

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

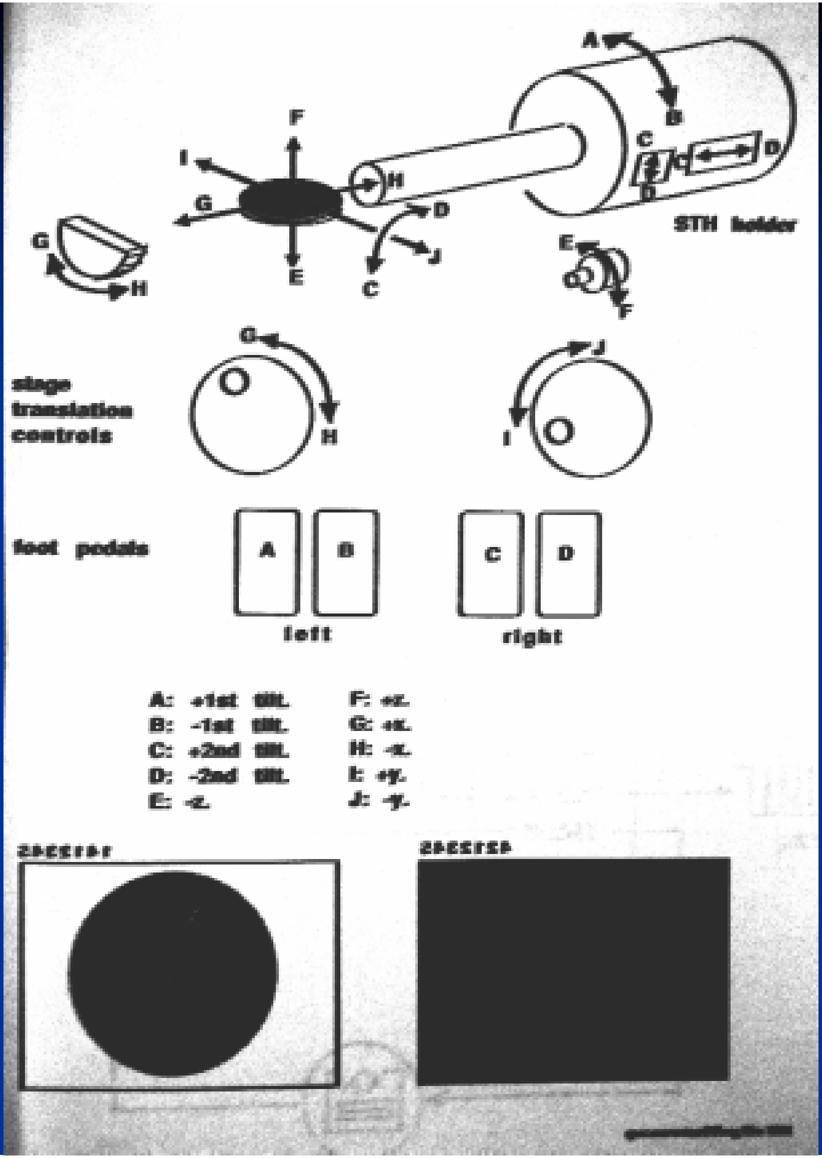
Specimen support and specimen holders

Specimen support: (vg)

Grid
Holey carbon grid

Specimen holders: (vg)
Top entry
Side entry
Single/double tilt
Heating, cooling, tensile, environmental, etc.

Performance:
Tilt angle, working distance,



Movements and controls of the specimen

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.

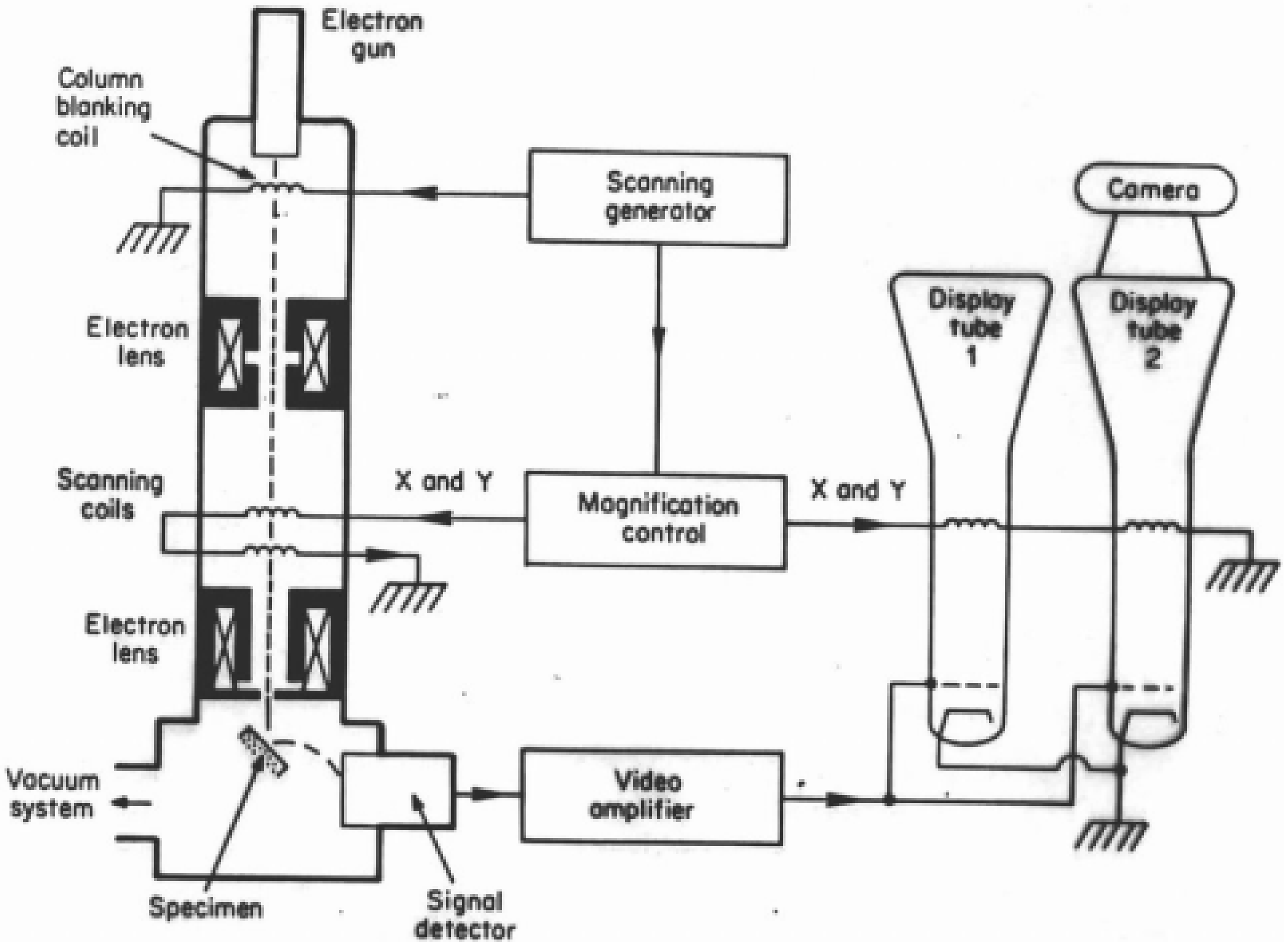
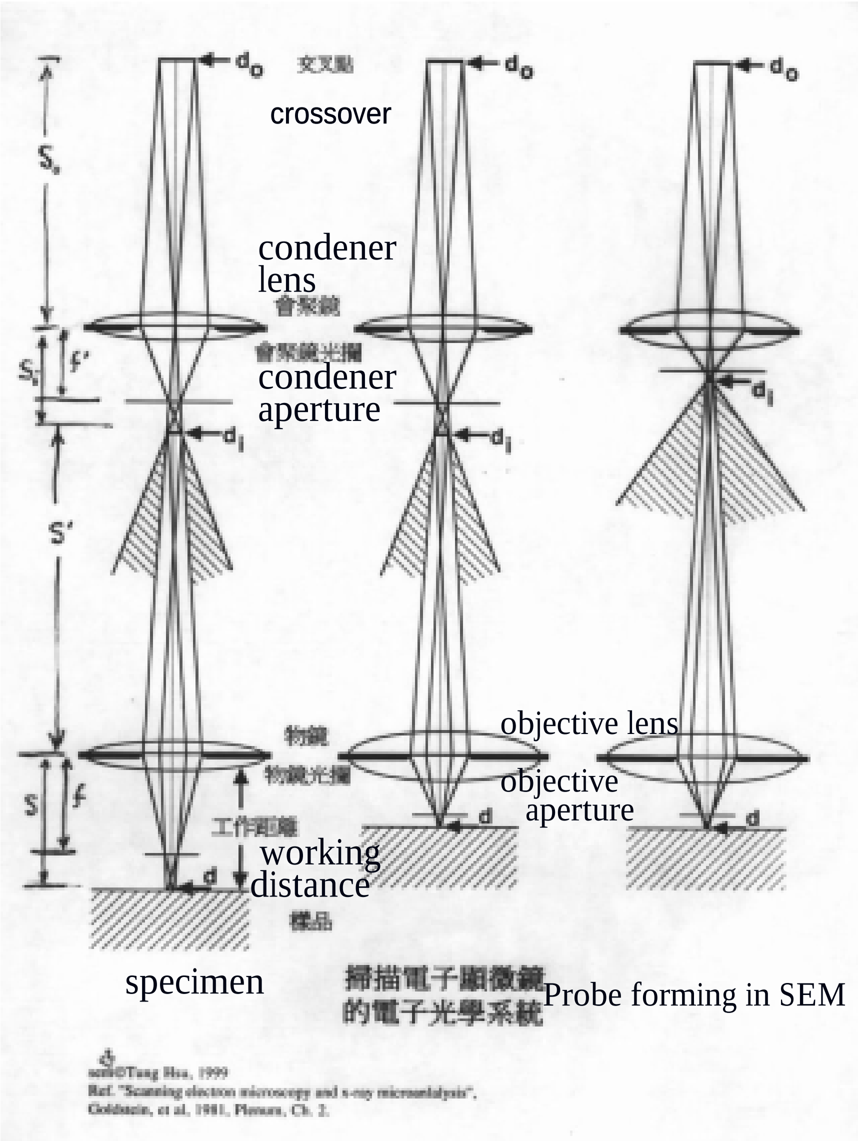


FIG. 1.2 Scanning electron microscope.



Scanning electron microscopy – microprobe

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

Monte Carlo simulation

The probe forming system: (vg)

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

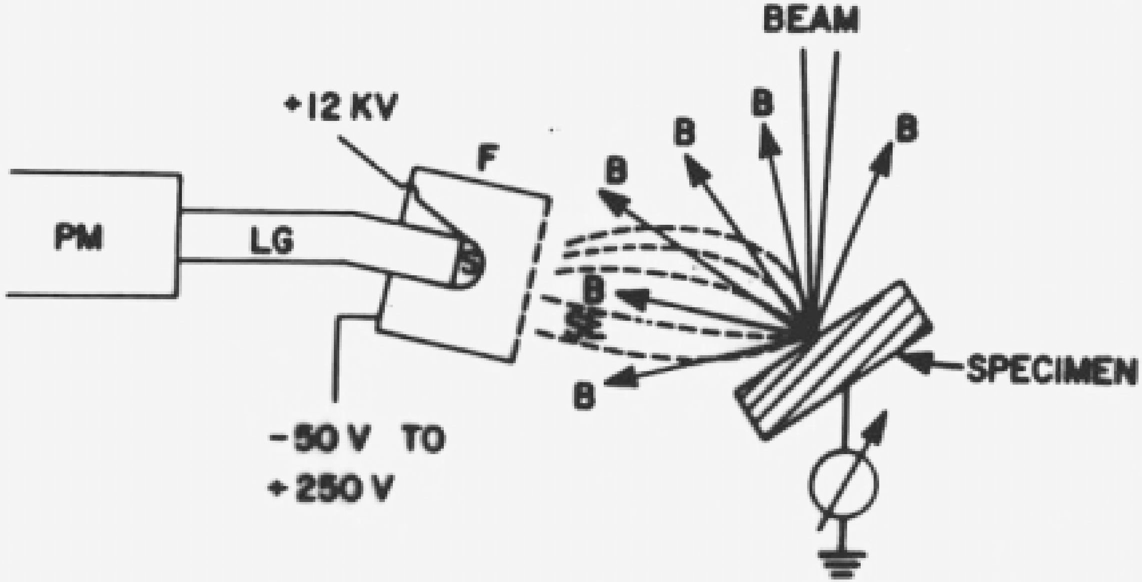


Figure 4.17. Schematic diagram of Everhart–Thornley scintillator–photomultiplier electron detector. B, backscattered electron; SE, secondary electron; F, Faraday cage; S, scintillator; LG, light guide; PM, photomultiplier.

Examples of SEM images (vg)

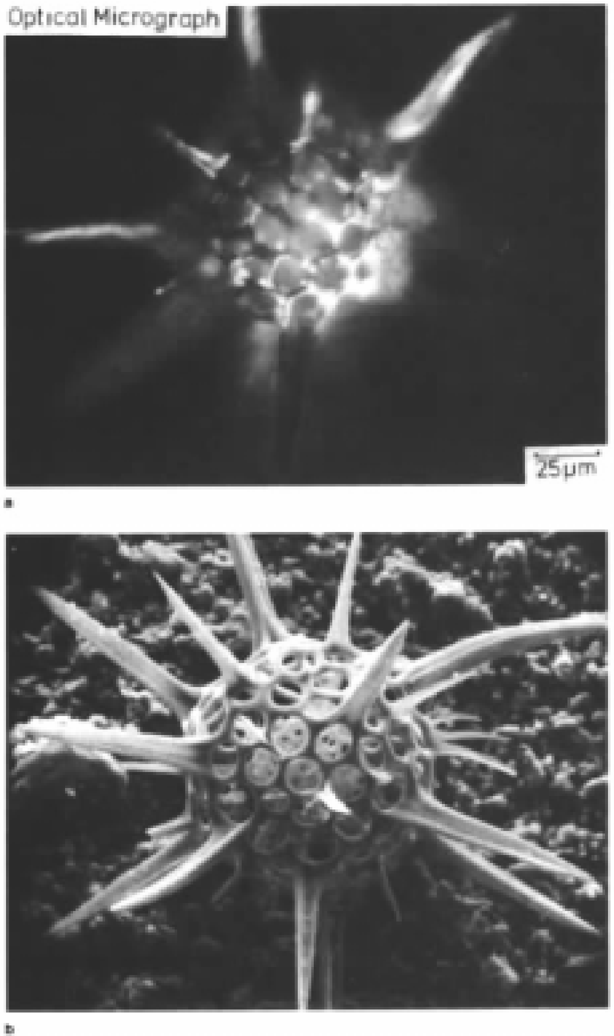
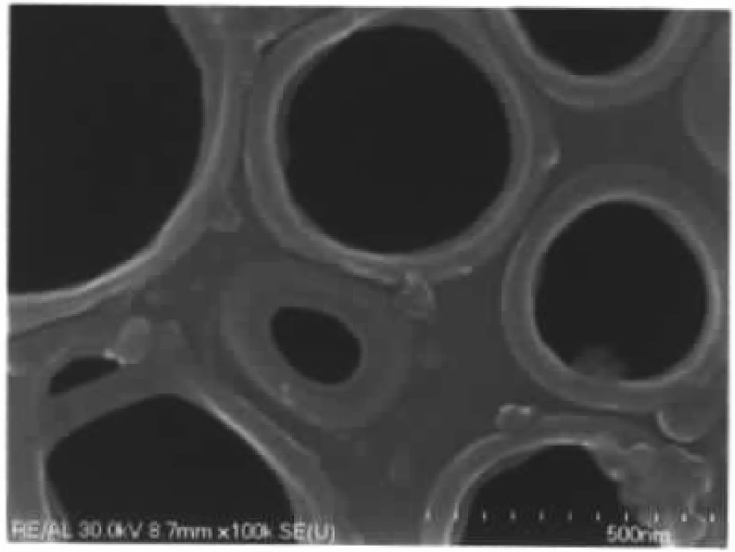
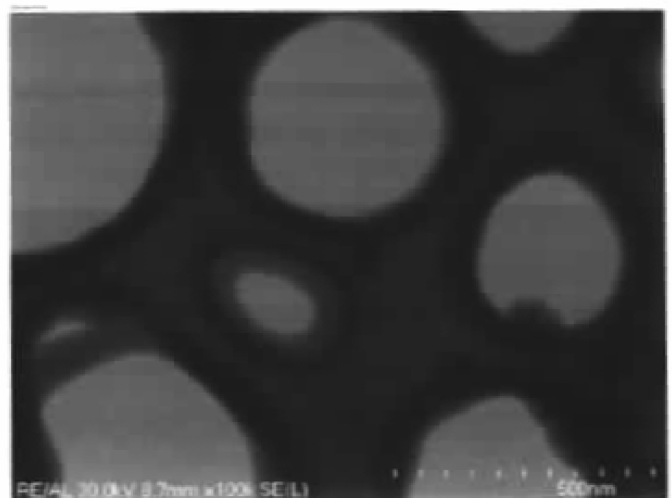


Figure 1.3. (a) Optical micrograph of the radiolarian *Trochodiscus longispinus*. (b) SEM micrograph of same radiolarian. The greater depth of focus and superior resolving capability of the SEM are apparent.





2001 (Hitachi S-4700)

	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

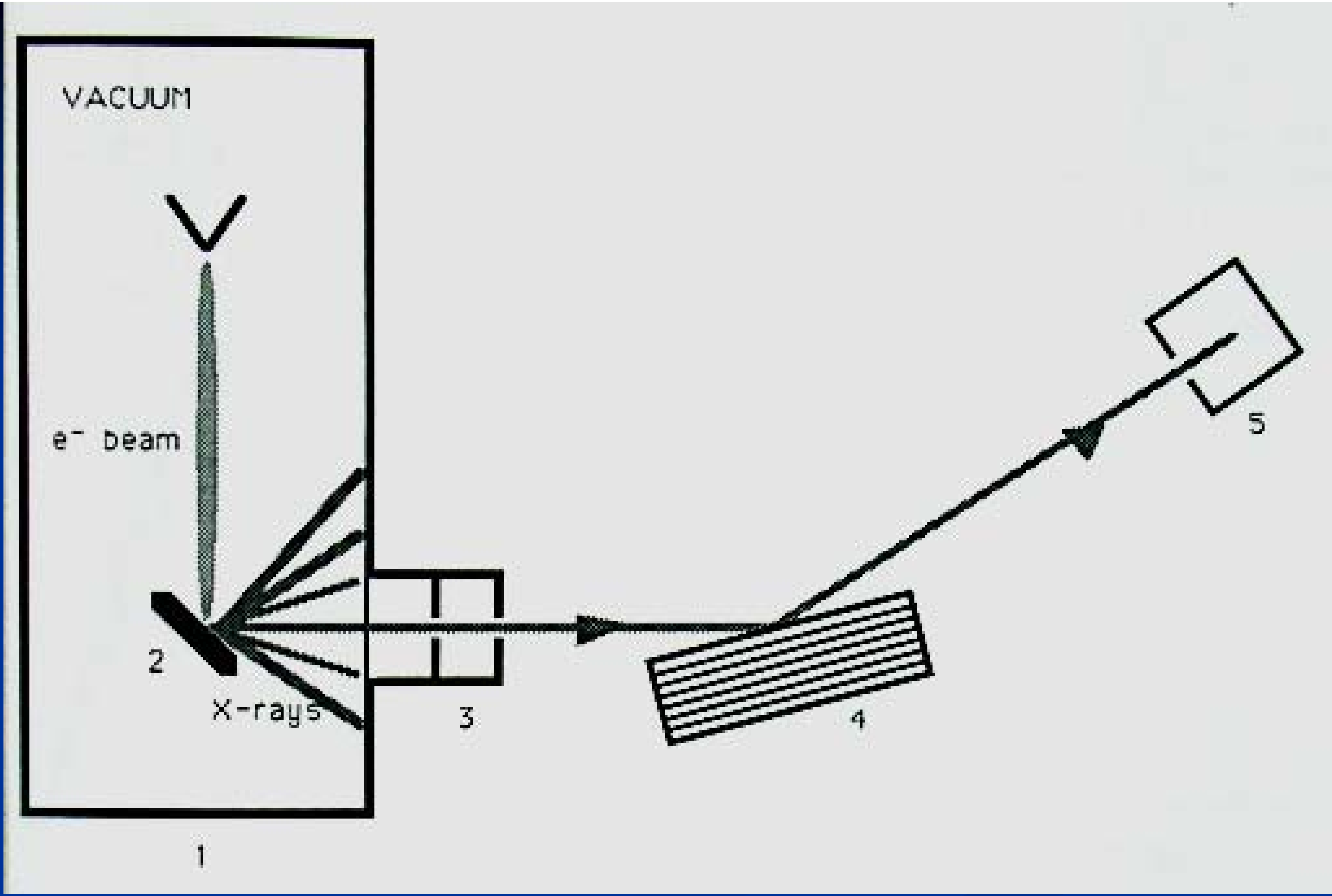
Parameter	$n\lambda$	2d	$\sin\theta$
diffraction	known	calculated	measured
spectrometry(WDS)	calculated	known	measured
spectrometry(EDS)	E: measured		

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

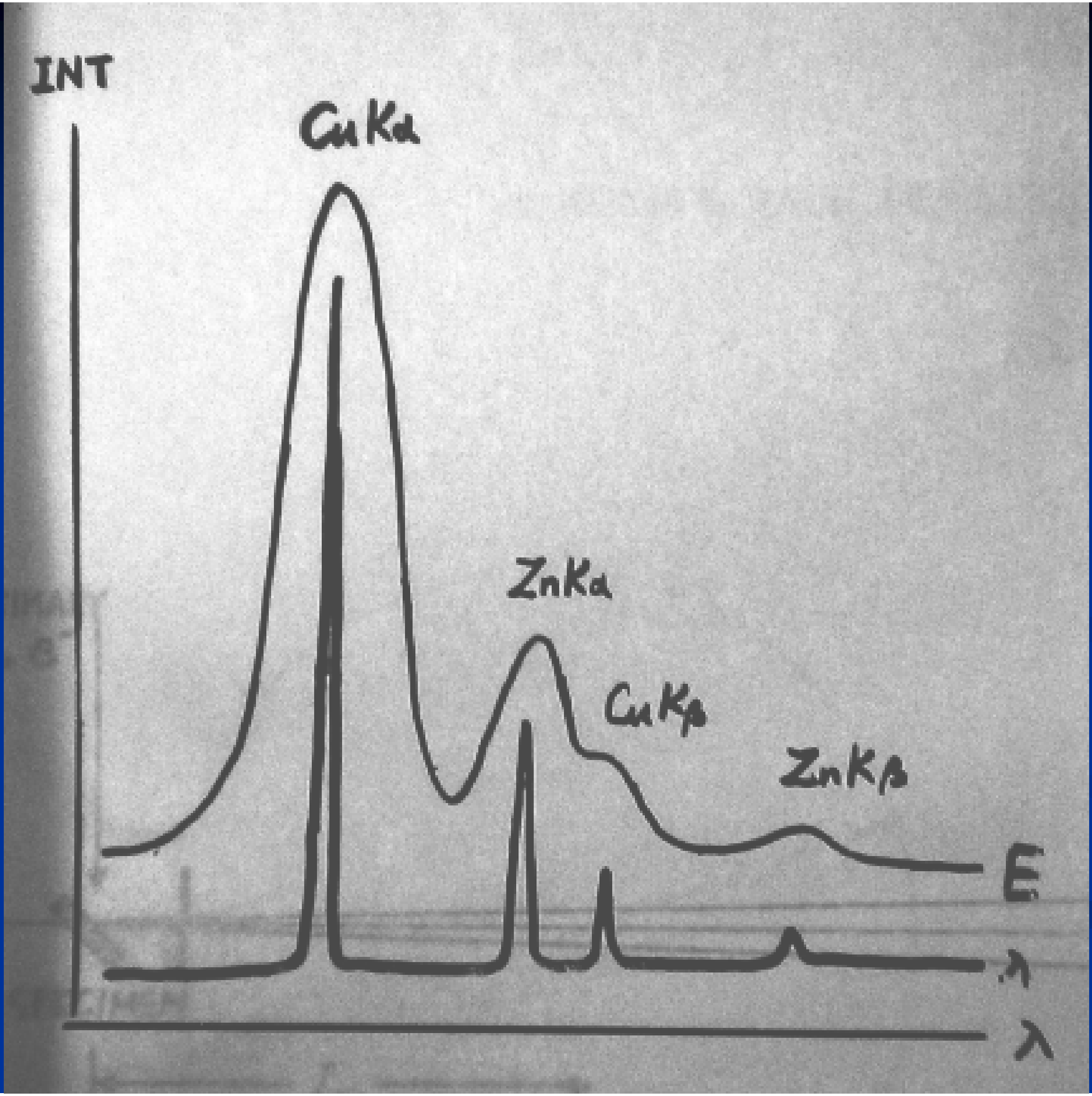
Instrumentation: Electron probe/microscope
Other particle beam
x-ray fluorescence
radioactive sources

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.