ELECTRON MICROSCOPY

14:20 – 17:20, Mar. 17, 2009
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智慧財產權聲明

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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

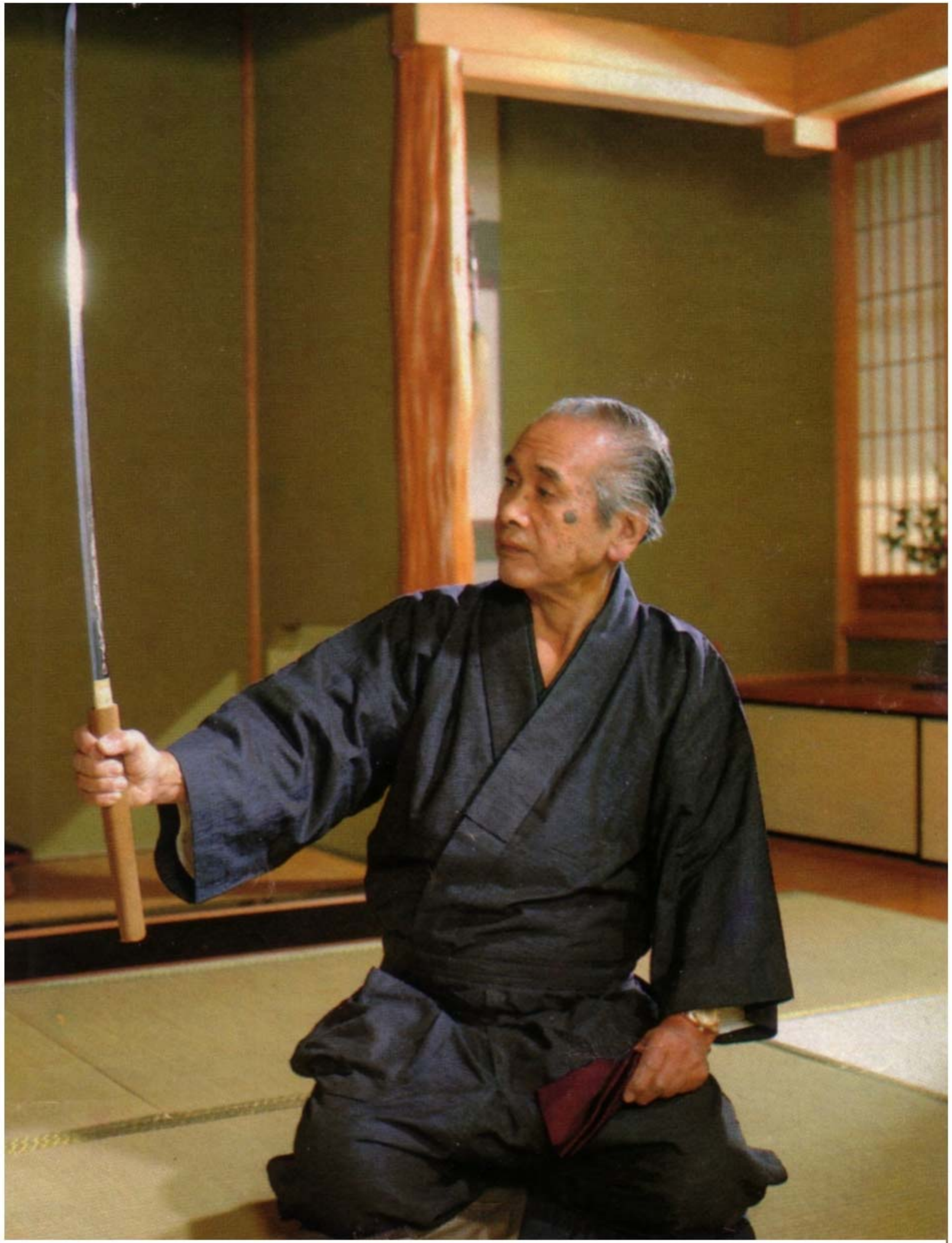
Electron microprobe / Analytical electron microscopy

Applications in nano-materials research

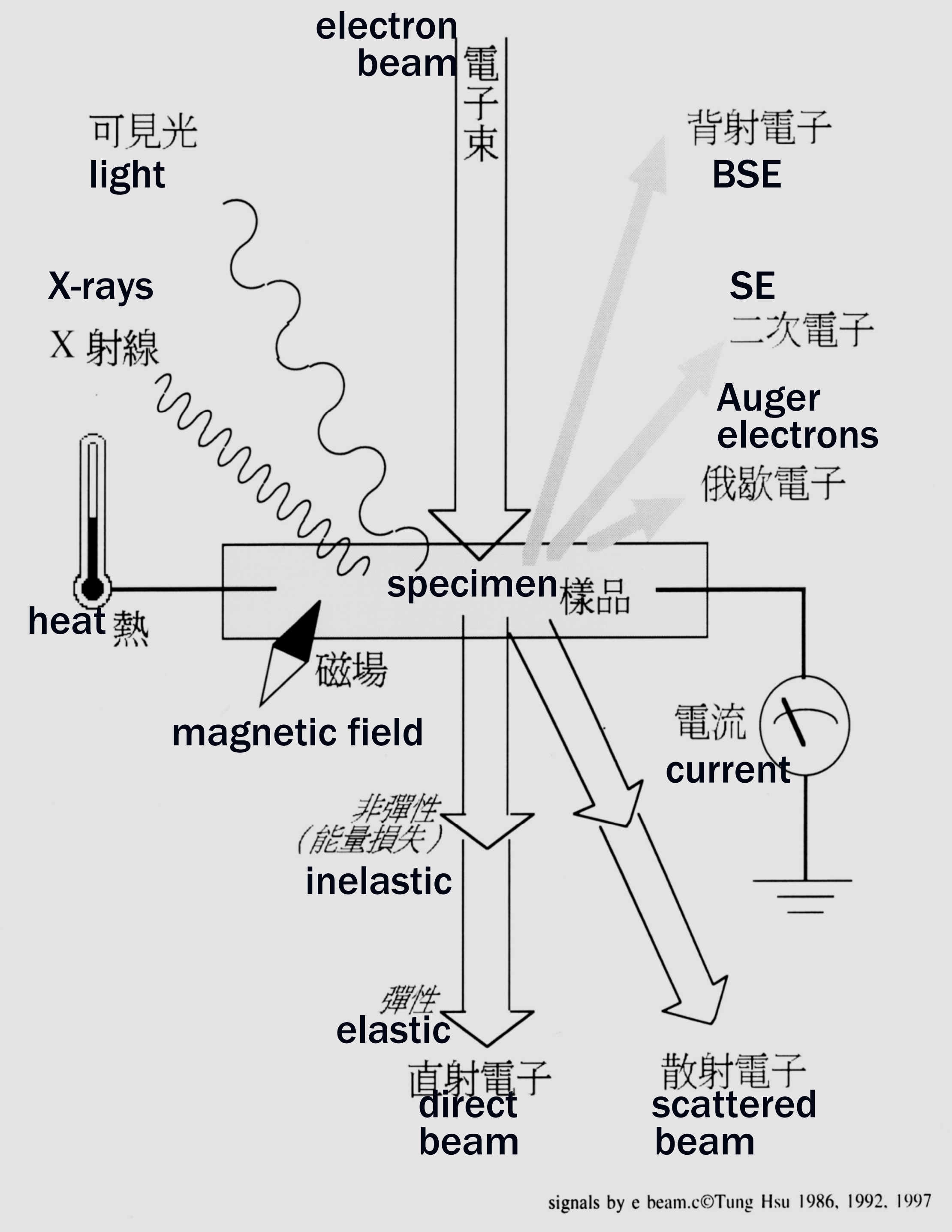
Introduction:

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



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

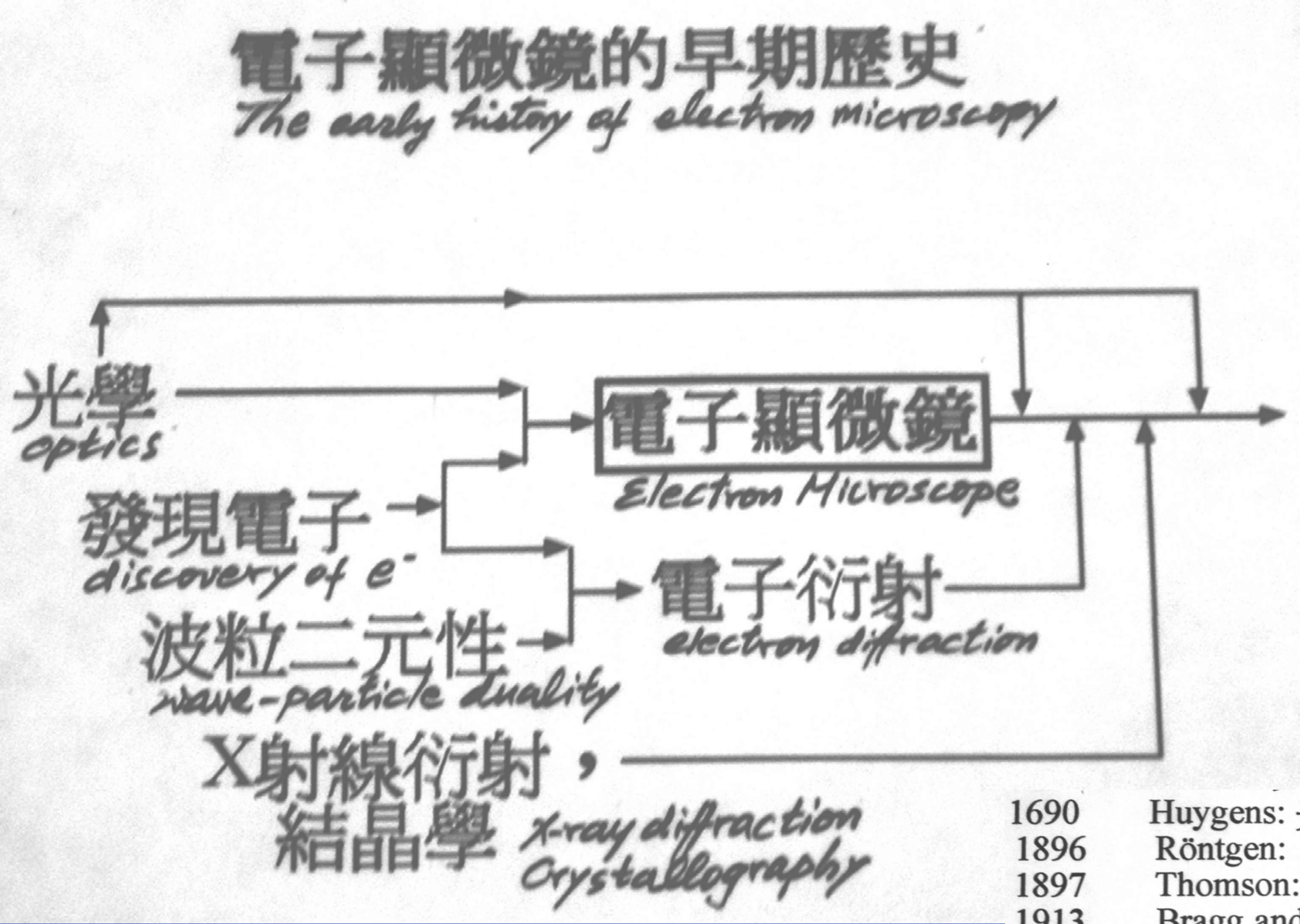
- image: morphology
  - scattering power
  - crystal structure
  - crystal defects
  - atomic structure
- other than the image:
  - (chemical) elemental composition
  - electronic structure

Microscope or microprobe

(Spatial) Resolution:

Wavelength, properties of lens

A brief history of electron microscopy

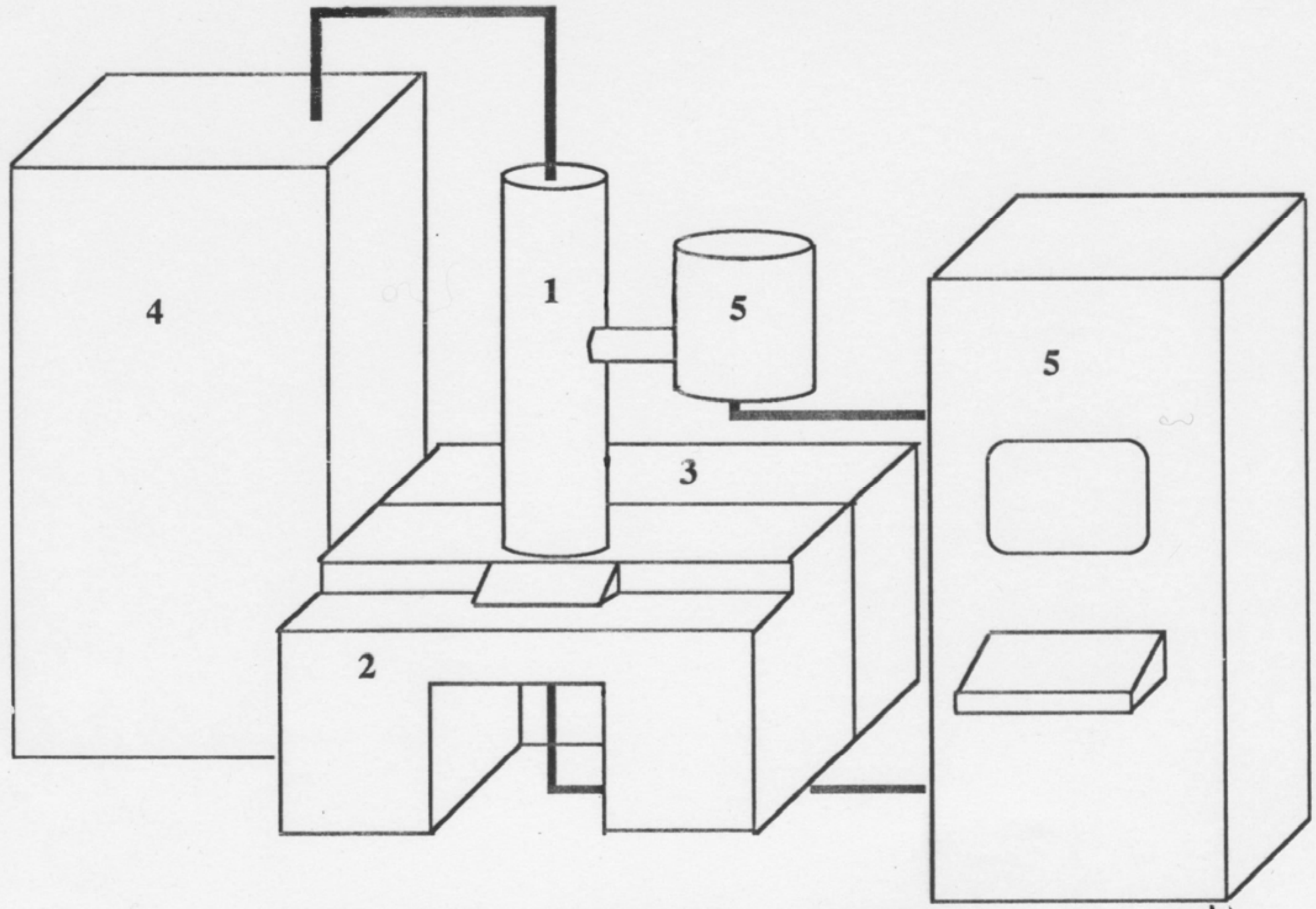


1690	Huygens: 光波，衍射
1896	Röntgen: 發現X射線
1897	Thomson: 發現電子
1913	Bragg and Bragg, von Laue: X射線衍射
1924	de Broglie 波
1926	Schödinger 方程式
	Busch: 電子束聚焦
1927	Davisson & Germer, Thomson: 電子衍射
1931	Ruska & Knoll: 鐵心磁鏡
1934	完成電子顯微鏡

Various Electron Microscopes (vg)

The Electron microscope

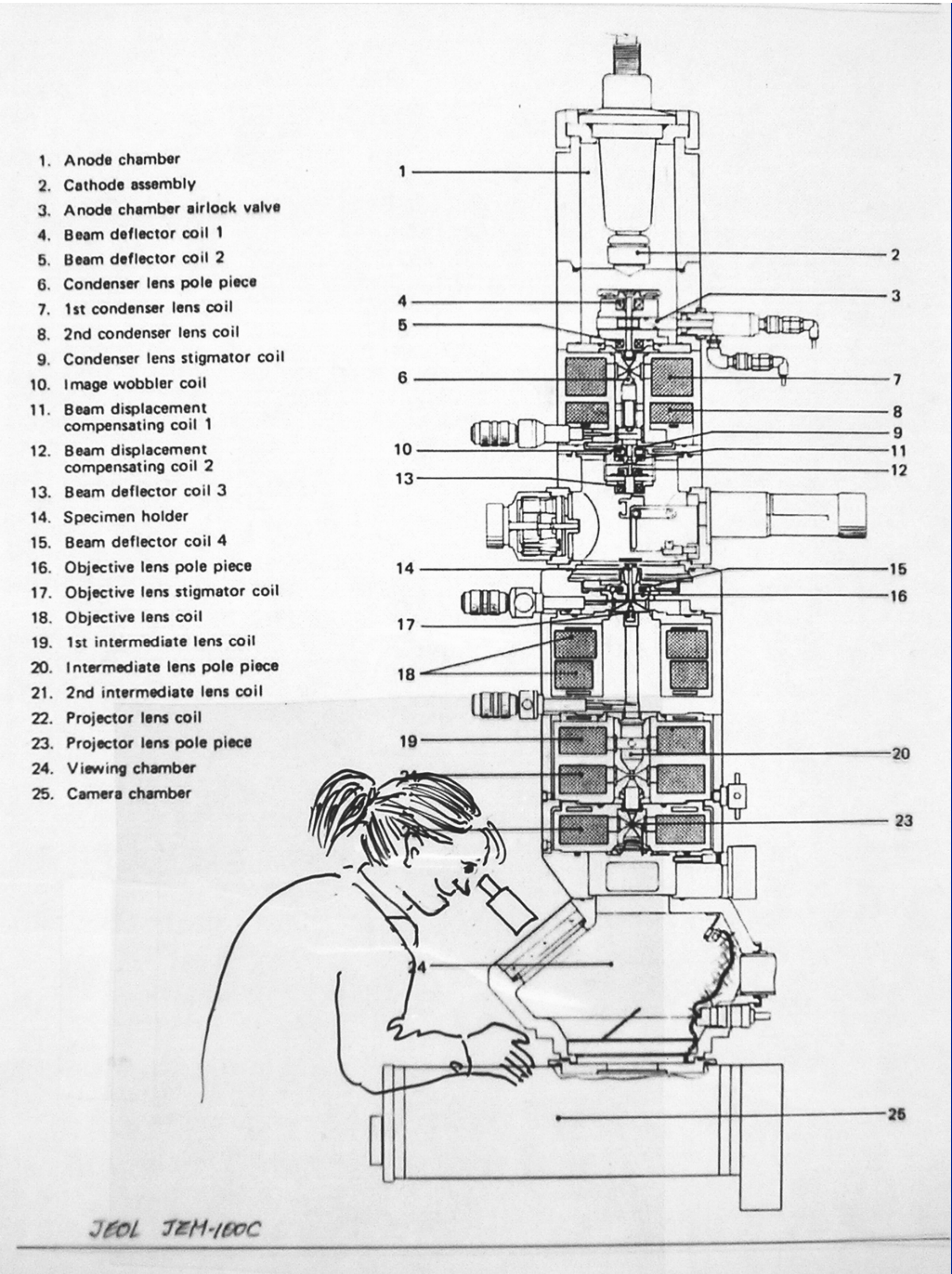
Structure and major components



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 Optics Column of JEOL JEM-100C (vg)

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

$$\nabla^2\Phi = 0$$
$$\mathbf{F} = -q\,\nabla\Phi = q\mathbf{E}$$

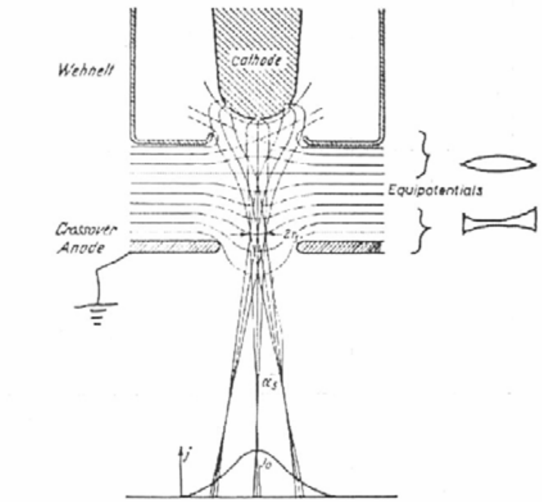


Fig. 3.2. The terminology associated with the electron gun.

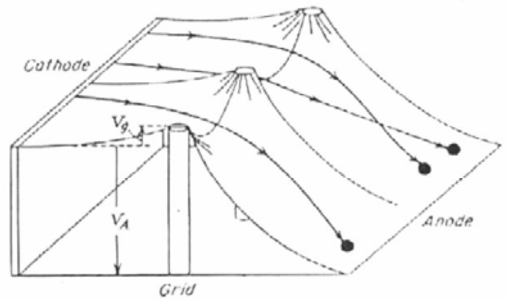


Fig. 2.9. The rubber-membrane model for experimental determination of elect paths (exaggerated vertical scale).

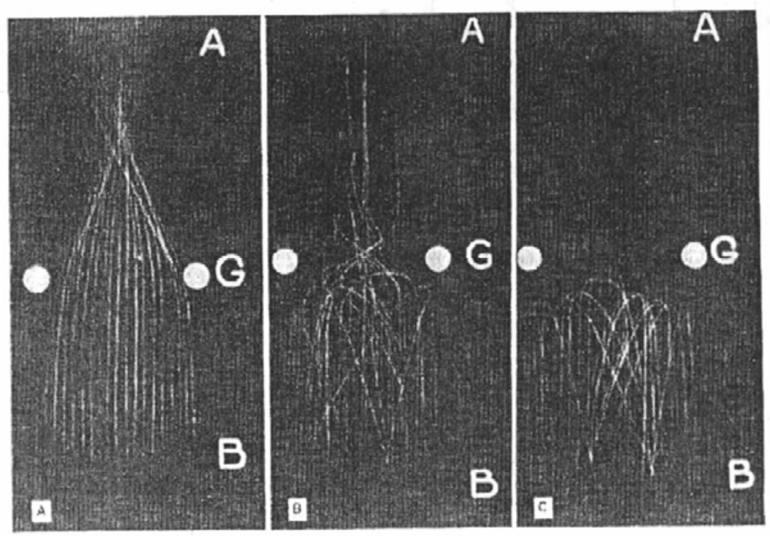
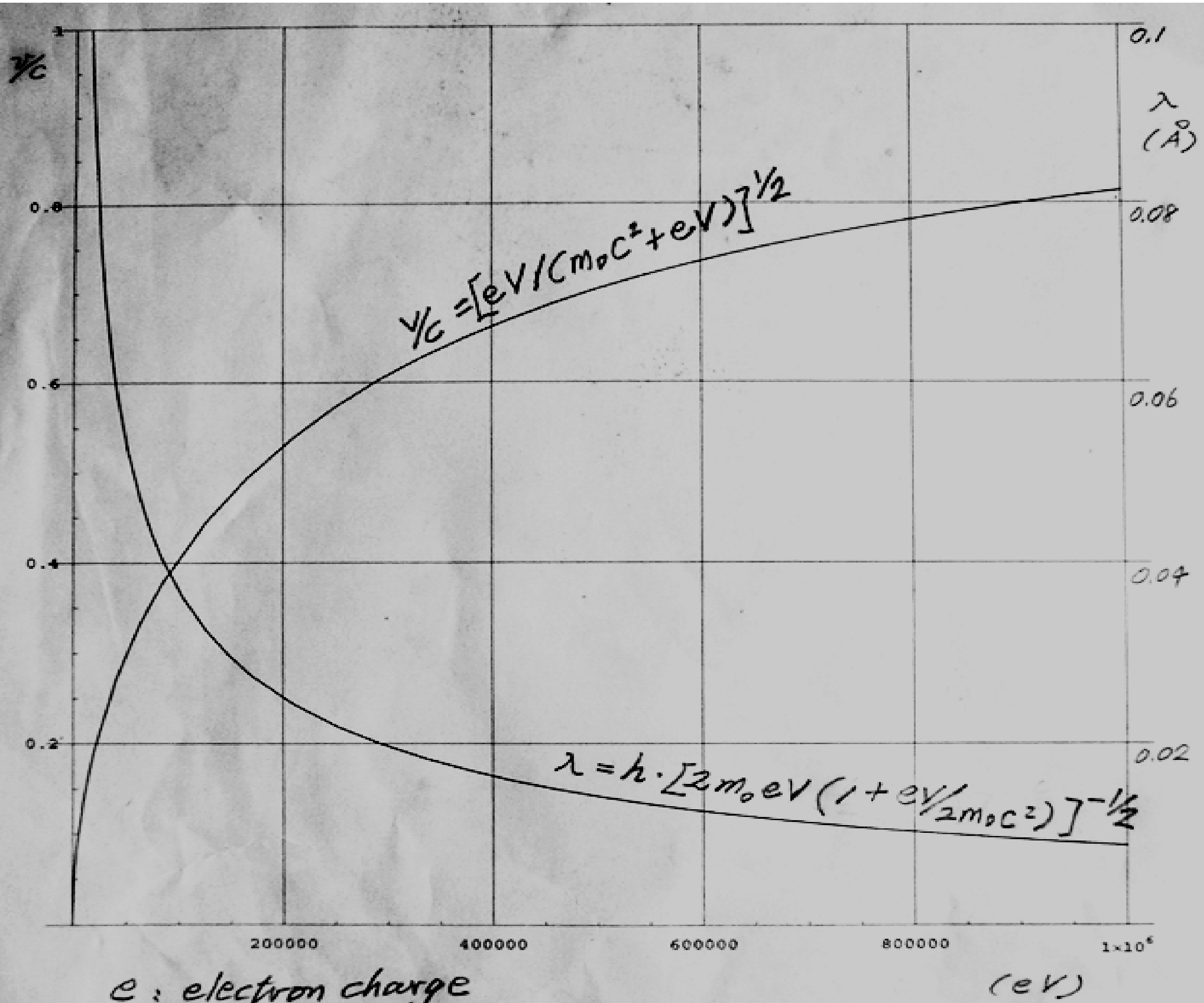


Fig. 2.10. Electron paths in a triode as determined with the rubber-membrane model. *A* is the anode, *B* the cathode, and *G* the grid. The grid potential is increasingly negative from A to C. (*Courtesy Philips Tech. Rev., Ref.* 2.)

C.E. Hall, "Introduction to Electron Microscopy", 2nd ed.
P.W. Hawkes, "Electron Optics and Electron Microscopy".



e : electron charge

v : " velocity

V : acceleration voltage

m_0 : electron rest mass

c : speed of light in vacuum

h : Planck's constant

λ : electron wavelength

The electromagnetic lens

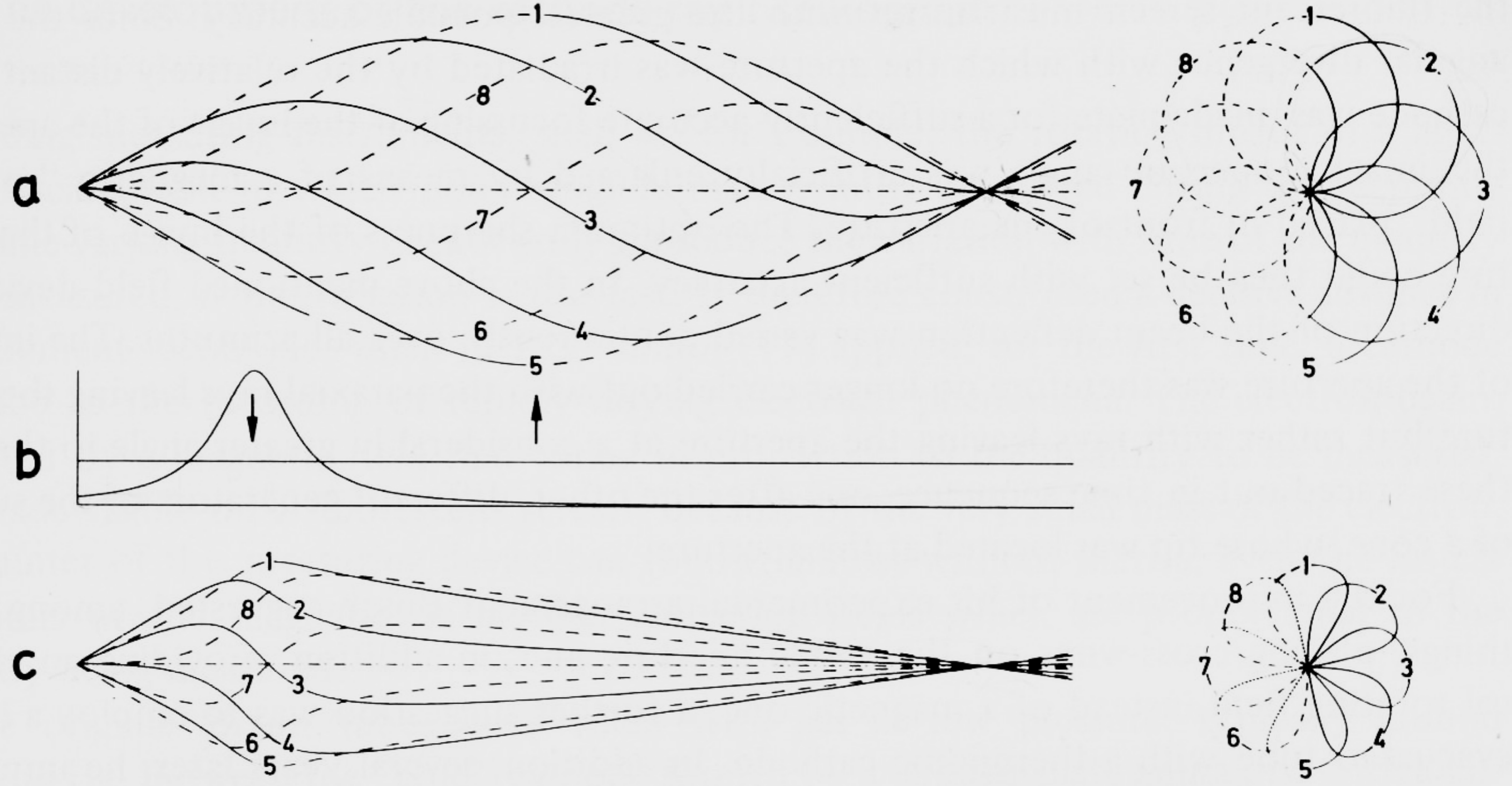
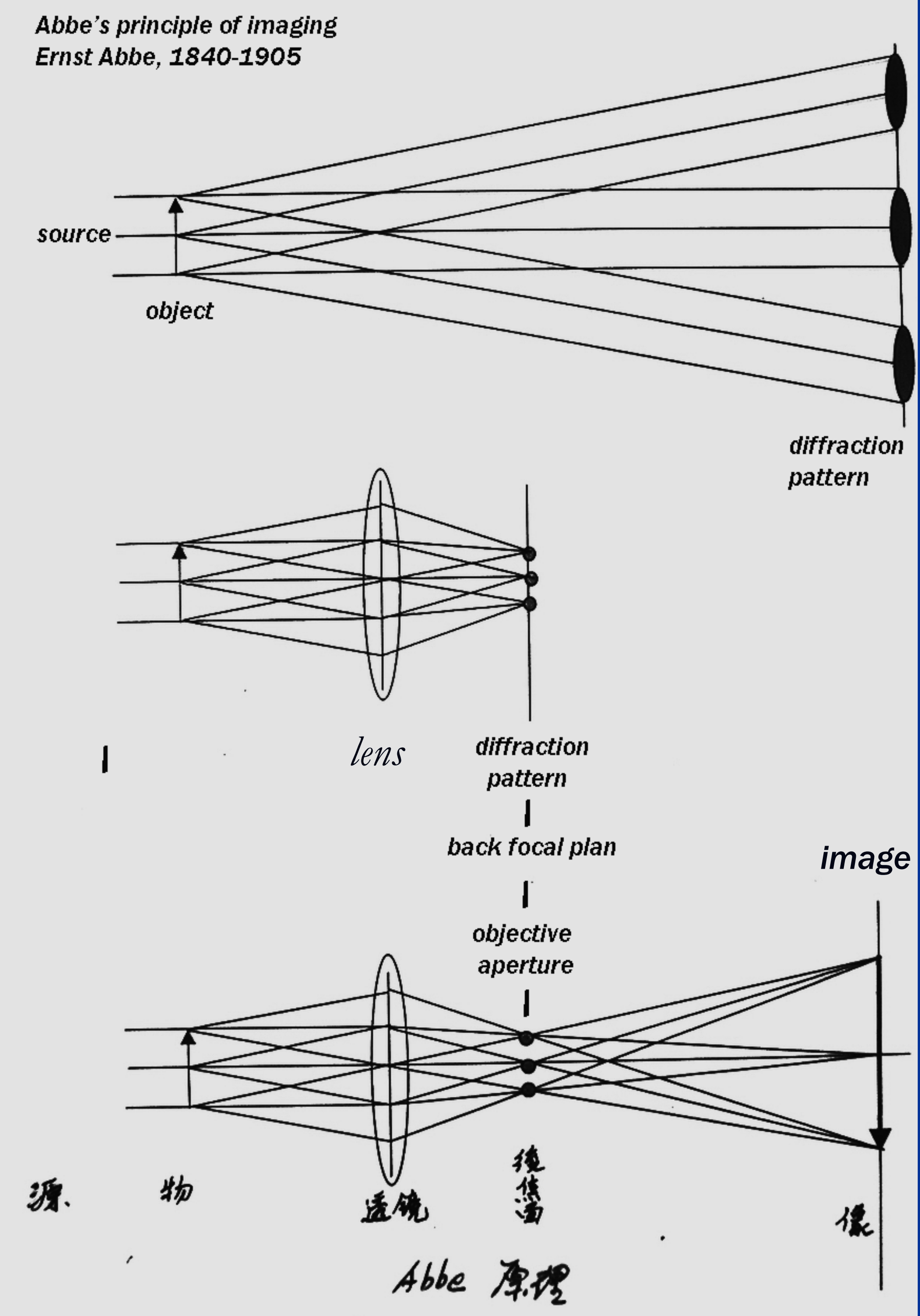


Fig. 1. Electron trajectories in a uniform (a) and in a non-uniform (c) magnetic field, issuing from an axial point of the specimen for different azimuth angles, but making the same angle with the lens axis. (b) Field distributions corresponding to (a) and (c).

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

ABBE’S PRINCIPLE

Ernst Abbe (1840-1905),
Carl Zeiss Lab



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

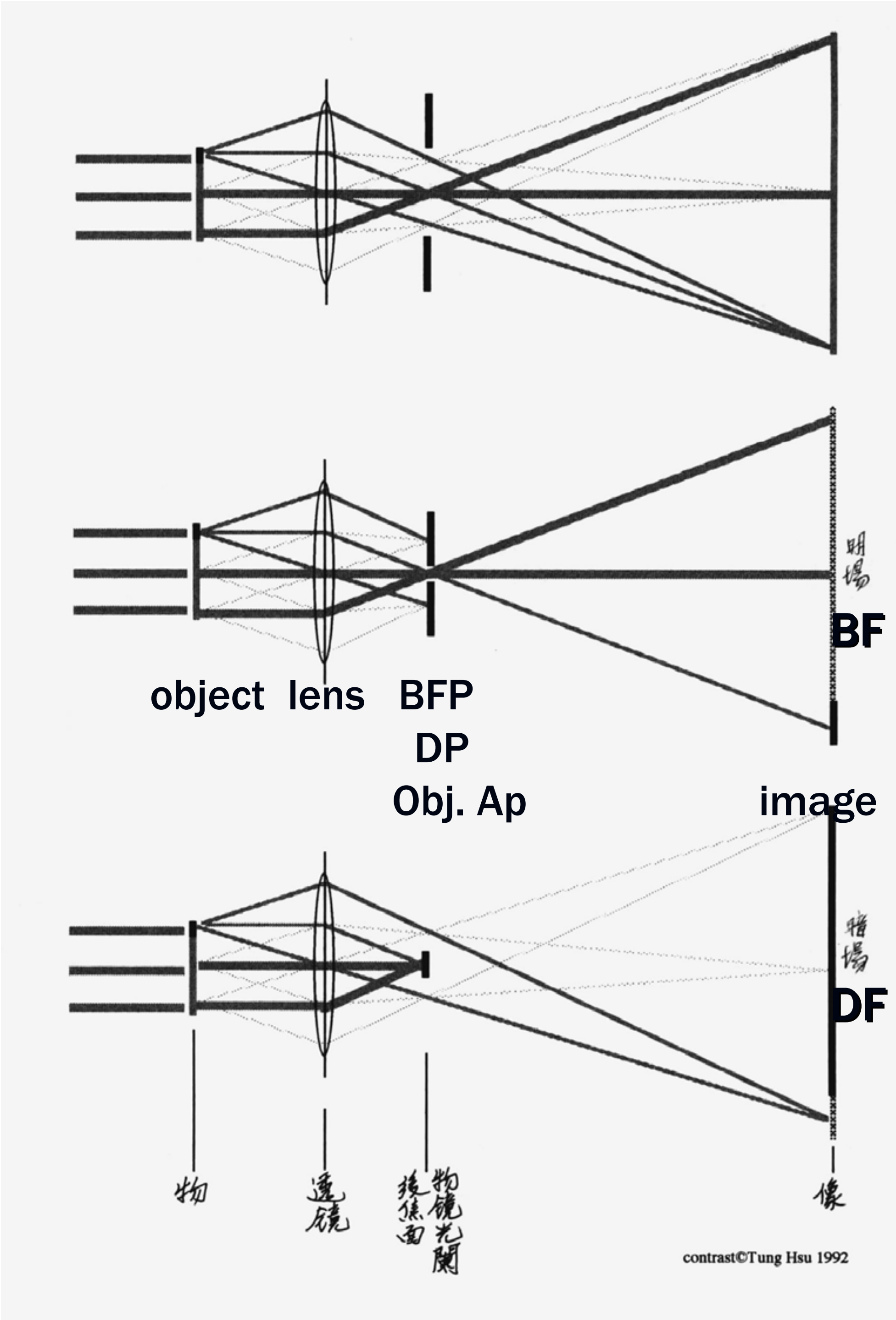
Fundamental geometrical and physical optics

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

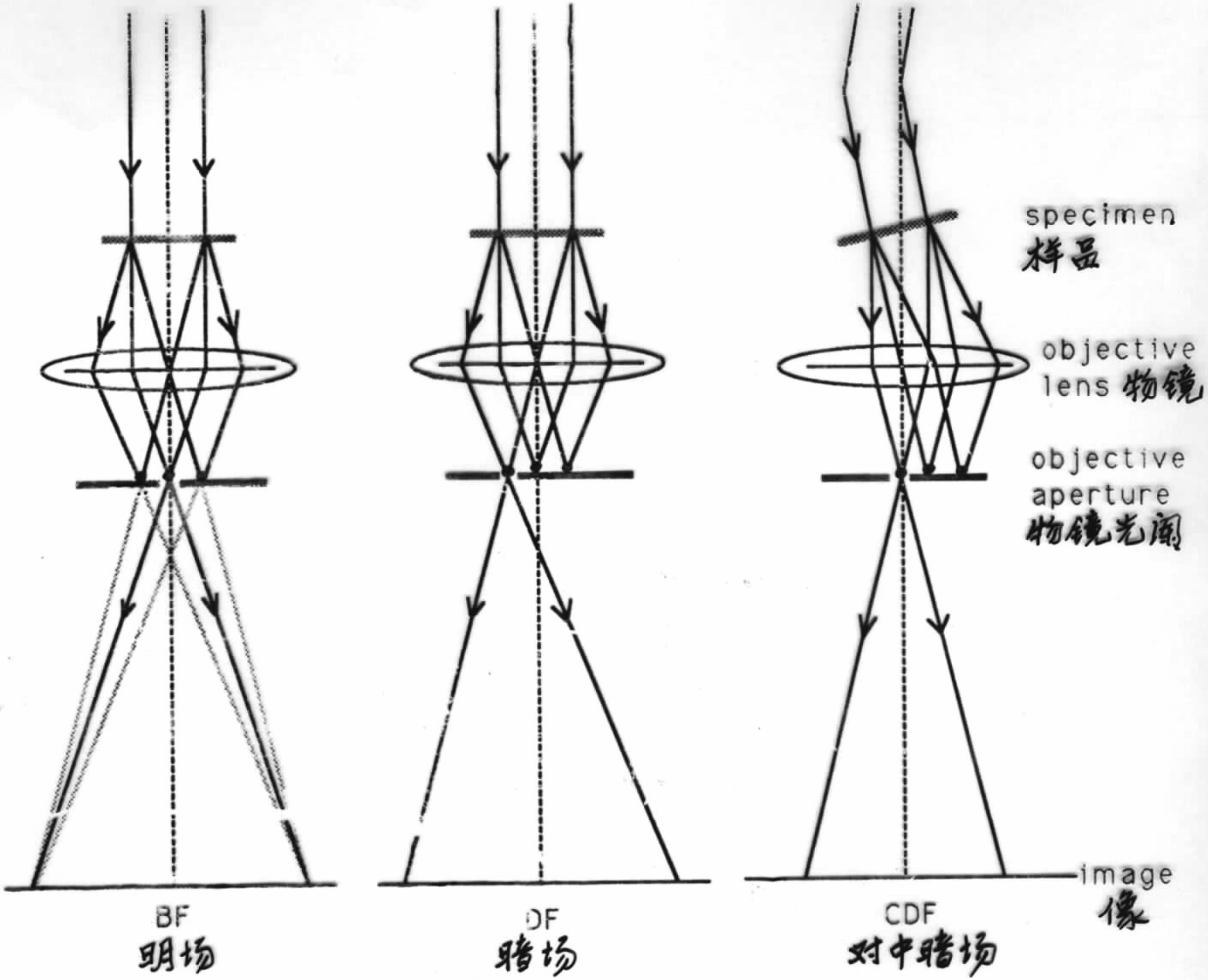
Contrast: Beam/solid interaction

BFP and the objective aperture:

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

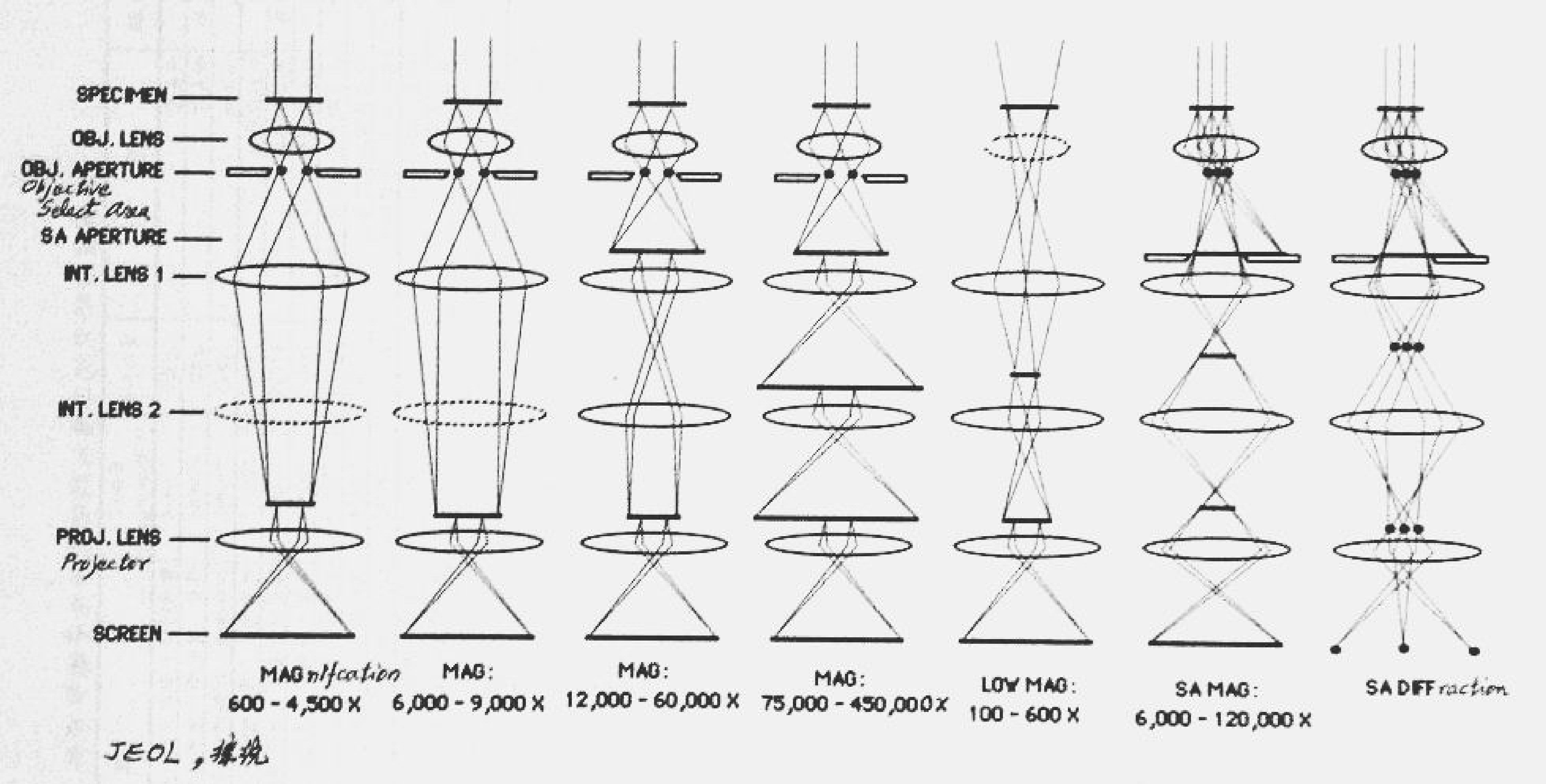


Contrast: Beam/solid interaction BFP and the objective aperture: Bright field (BF) and dark field (DF) images.



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) (vg)

Diffraction Pattern

Diffraction Contrast

What is Diffraction?

What is DIFFRACTION?

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*. *No one has ever been able to define the difference between interference and diffraction satisfactorily*. 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.

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 (superposition).

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

Contrast mechanism:

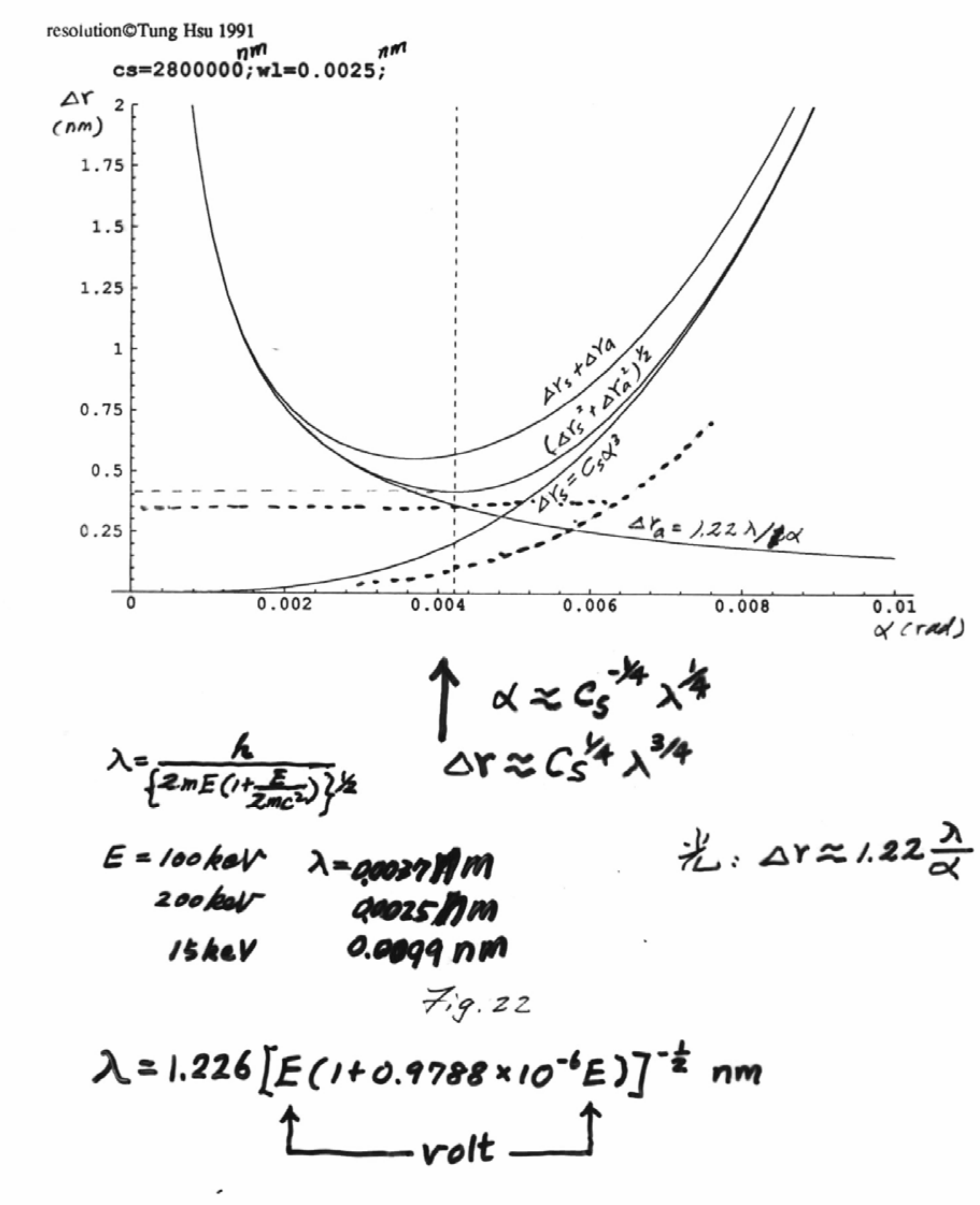
Beam/specimen interaction

- 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

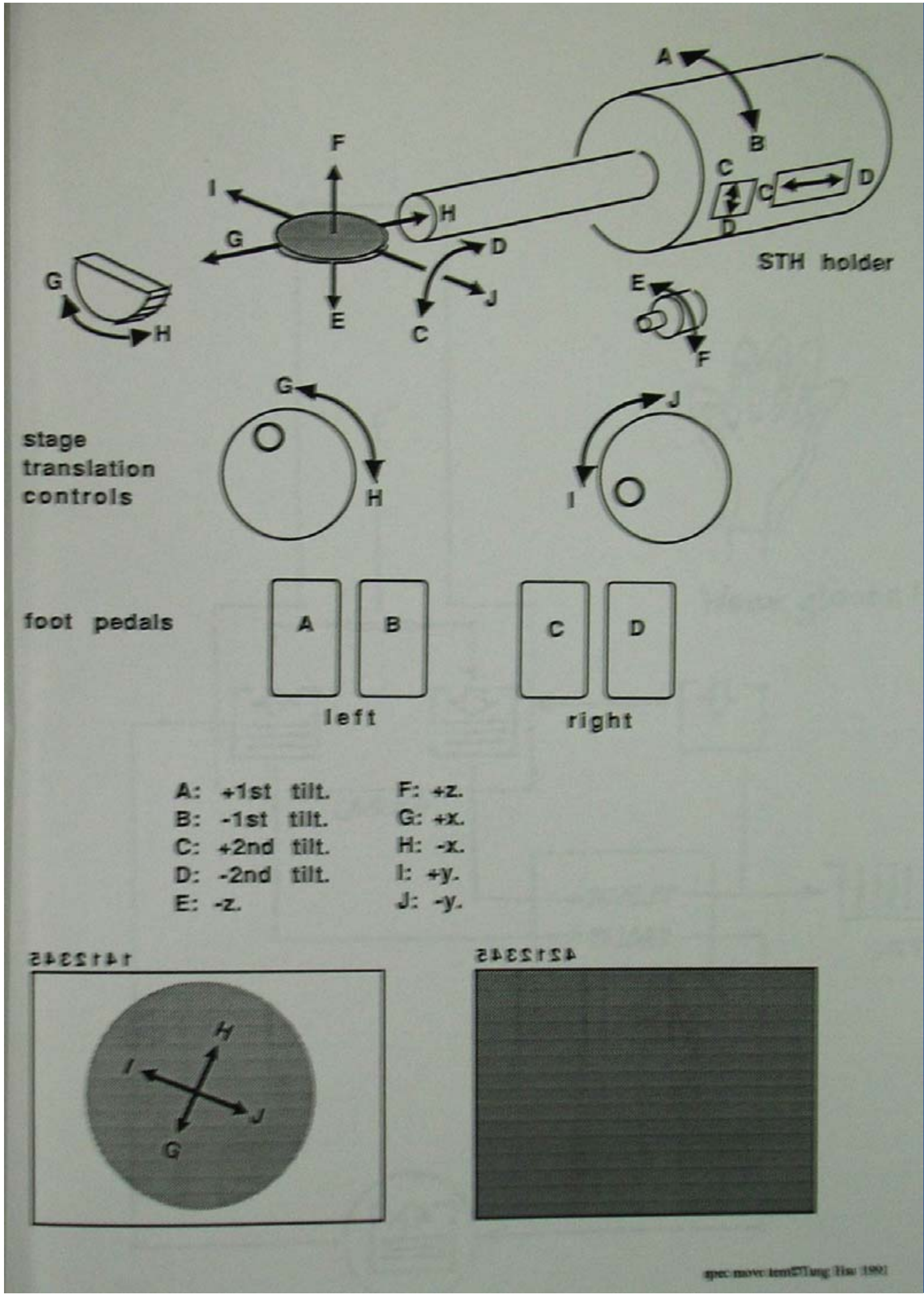
- Grid
- Holey carbon grid

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

WHY VACUUM (LOW PRESSURE)?

Specimen
Filament
Mean free path
of electrons

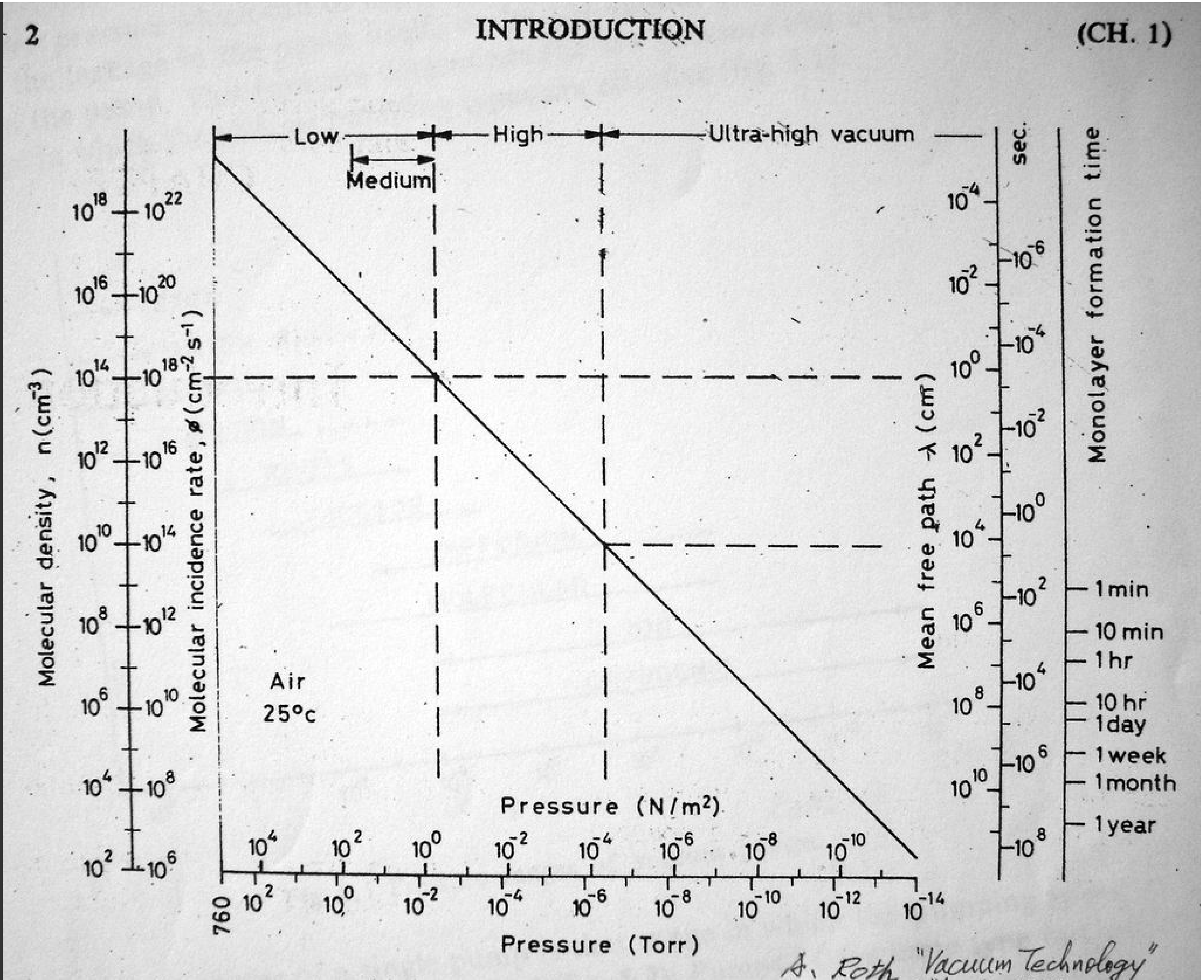


Fig. 1.1 Relationship of several concepts defining the degree of vacuum.

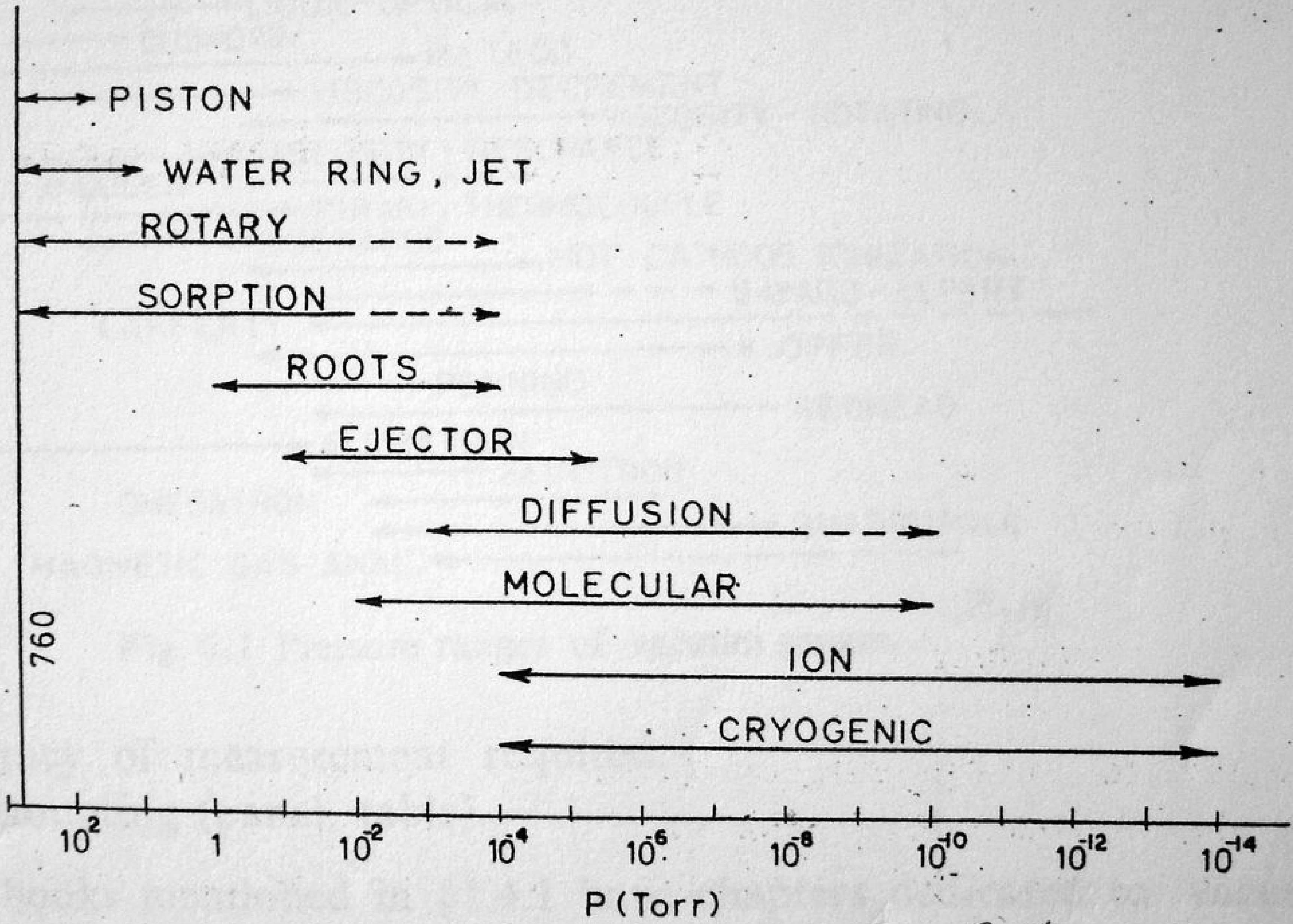


Fig. 5.1 Pressure ranges of vacuum pumps.

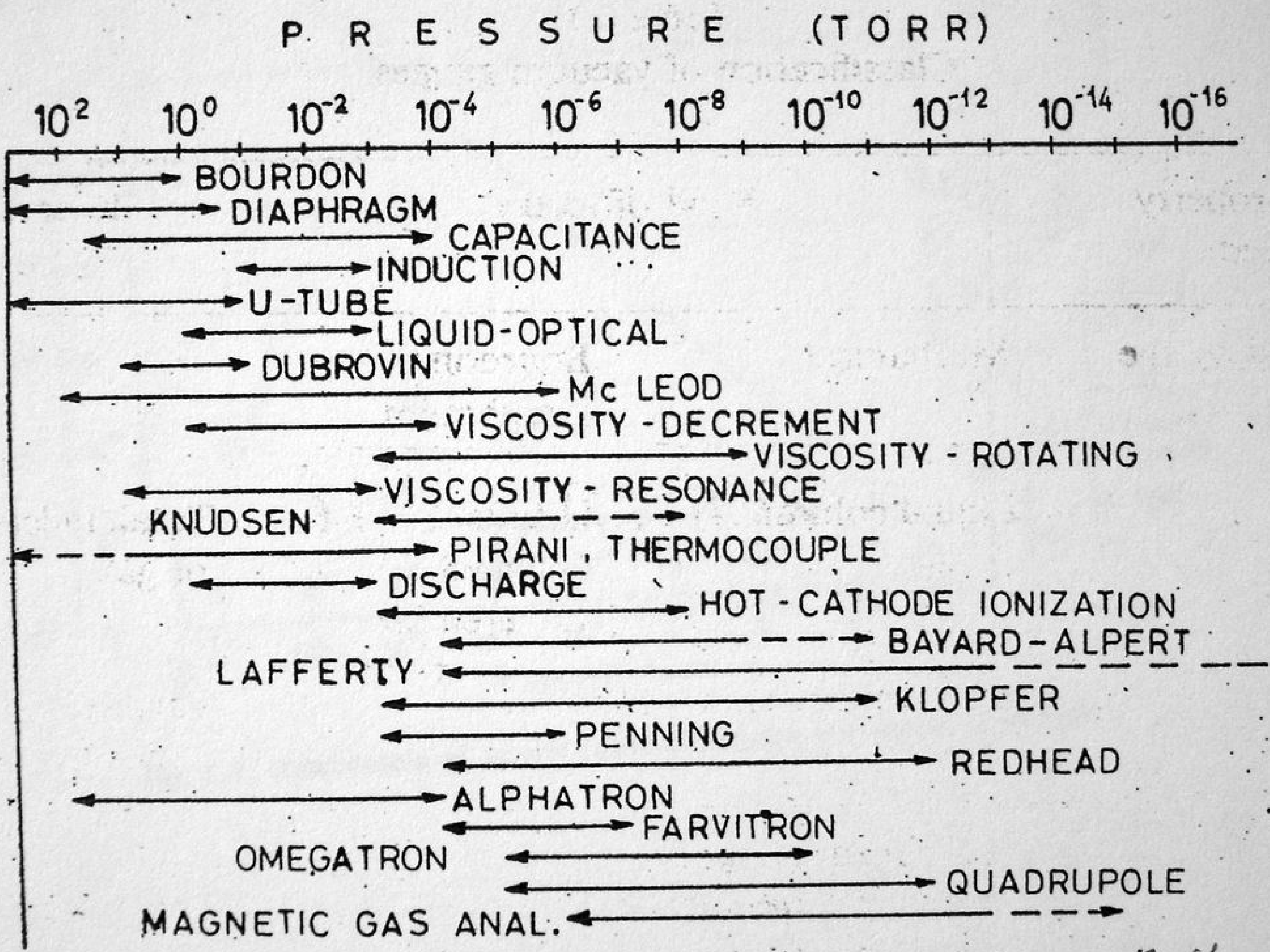
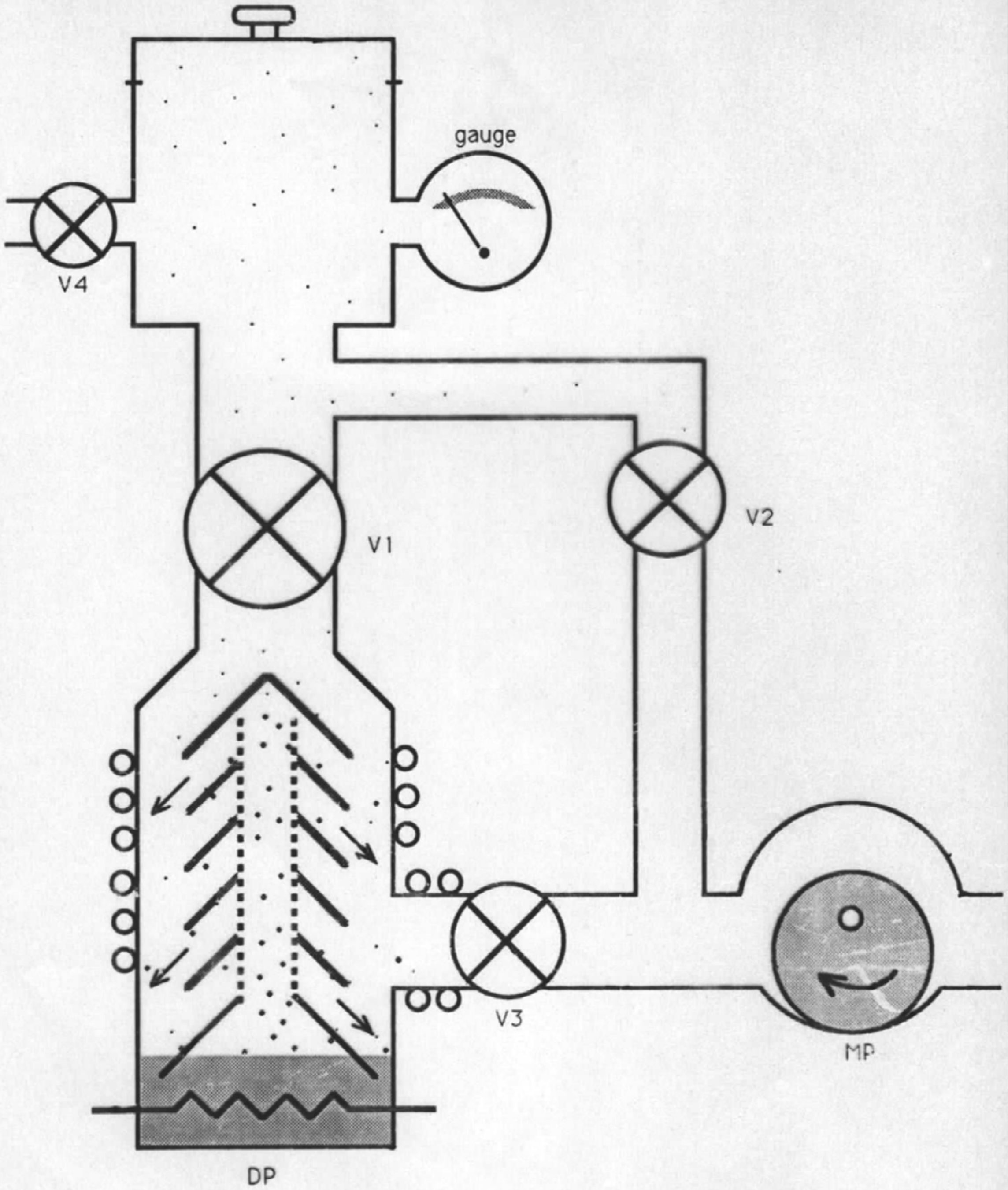


Fig. 6.1 Pressure ranges of vacuum gauges.


gauge
V4
V1
V2
V3
MP
DP

High Resolution Electron Microscopy (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.

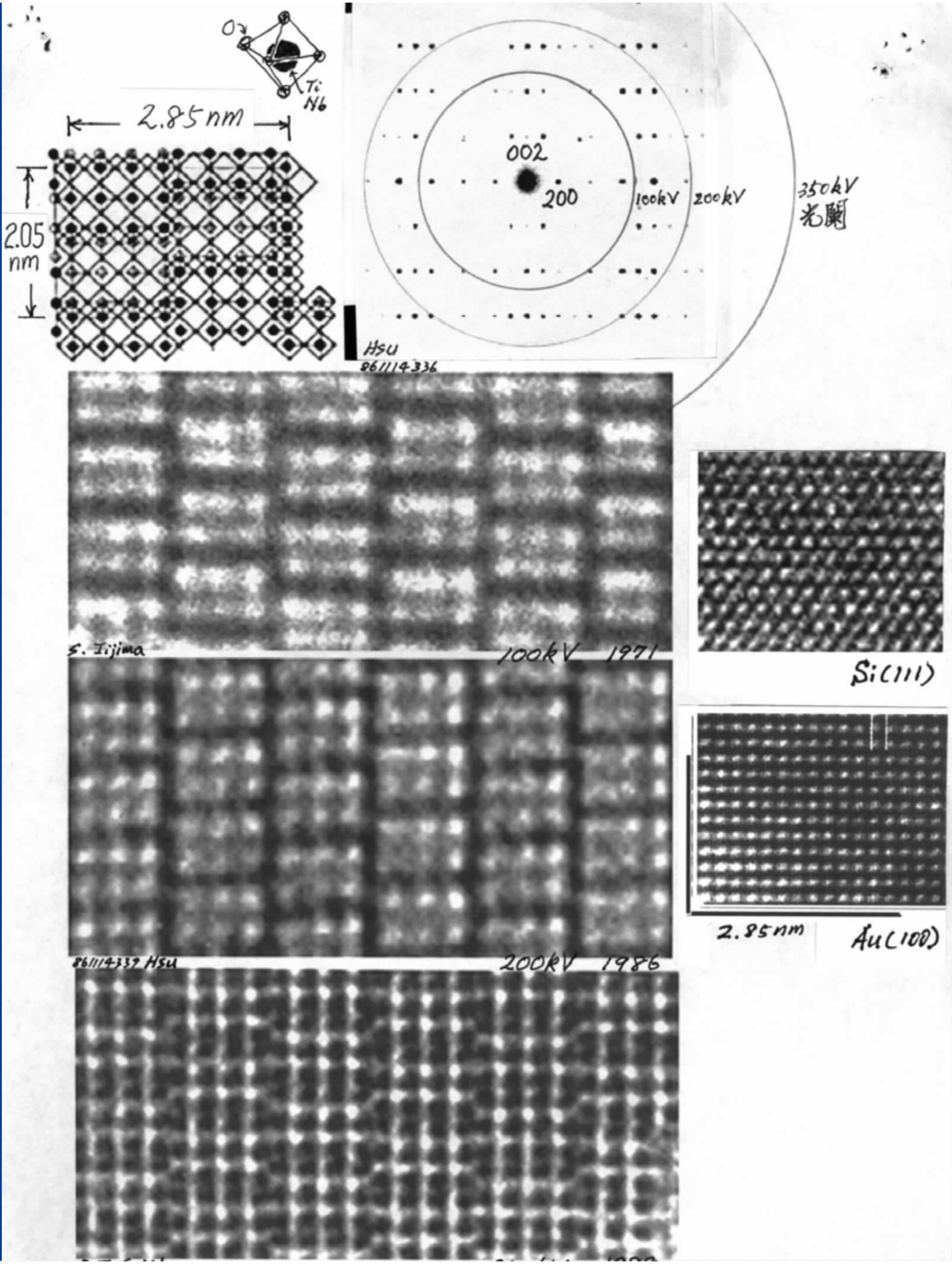
HREM examples:

Specimens:

- **$Ti_2Ni_{10}O_{19}$**
- **Si**
- **Au**

Microscopes:

- **JEOL JEM-100B**
- **JEOL JEM-200CX**
- **JEOL JEM-4000EX**



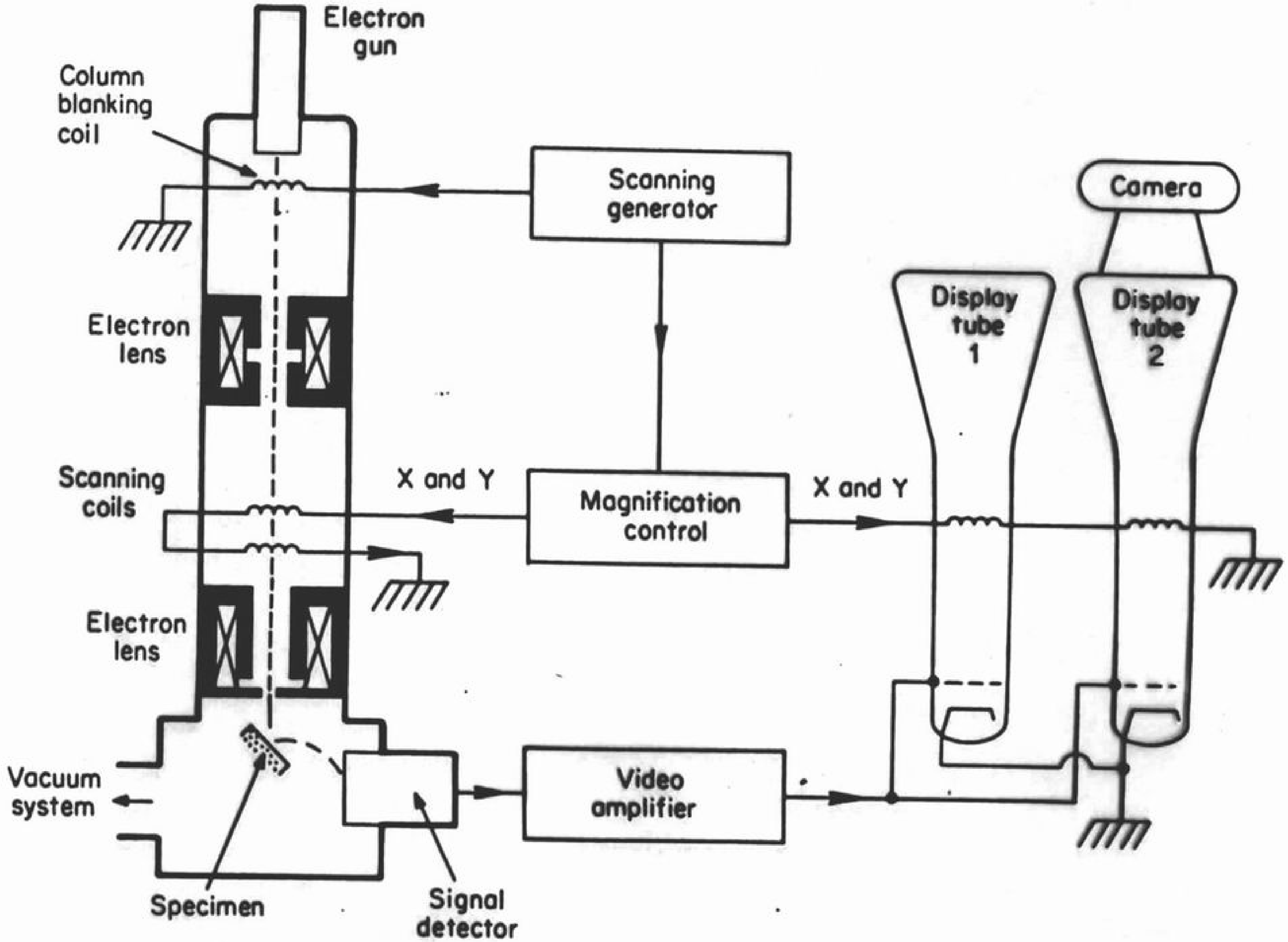
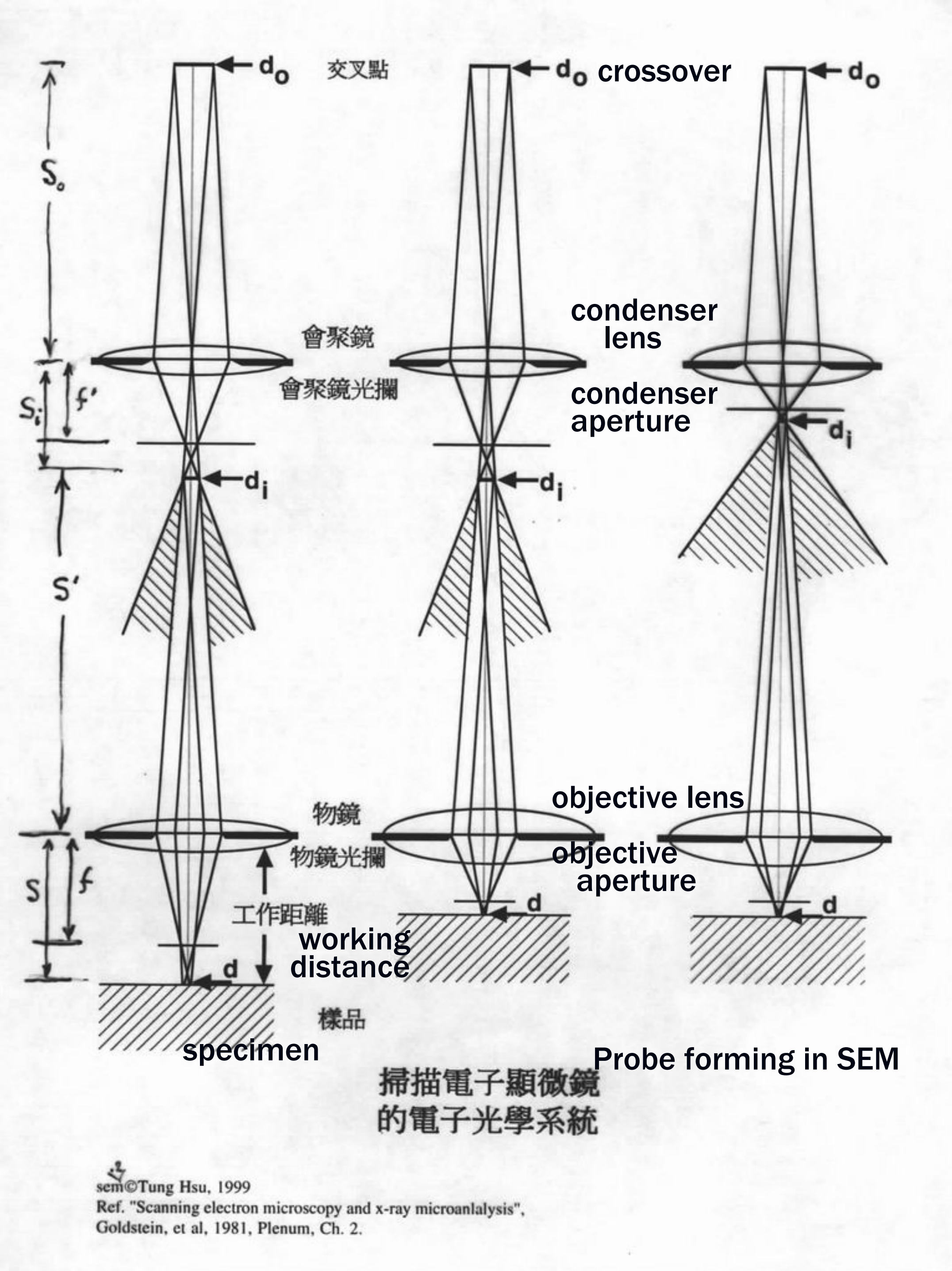


FIG. 1.2 Scanning electron microscope.



Scanning electron microscopy – microprobe

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

Monte Carlo simulation

The probe forming system:

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

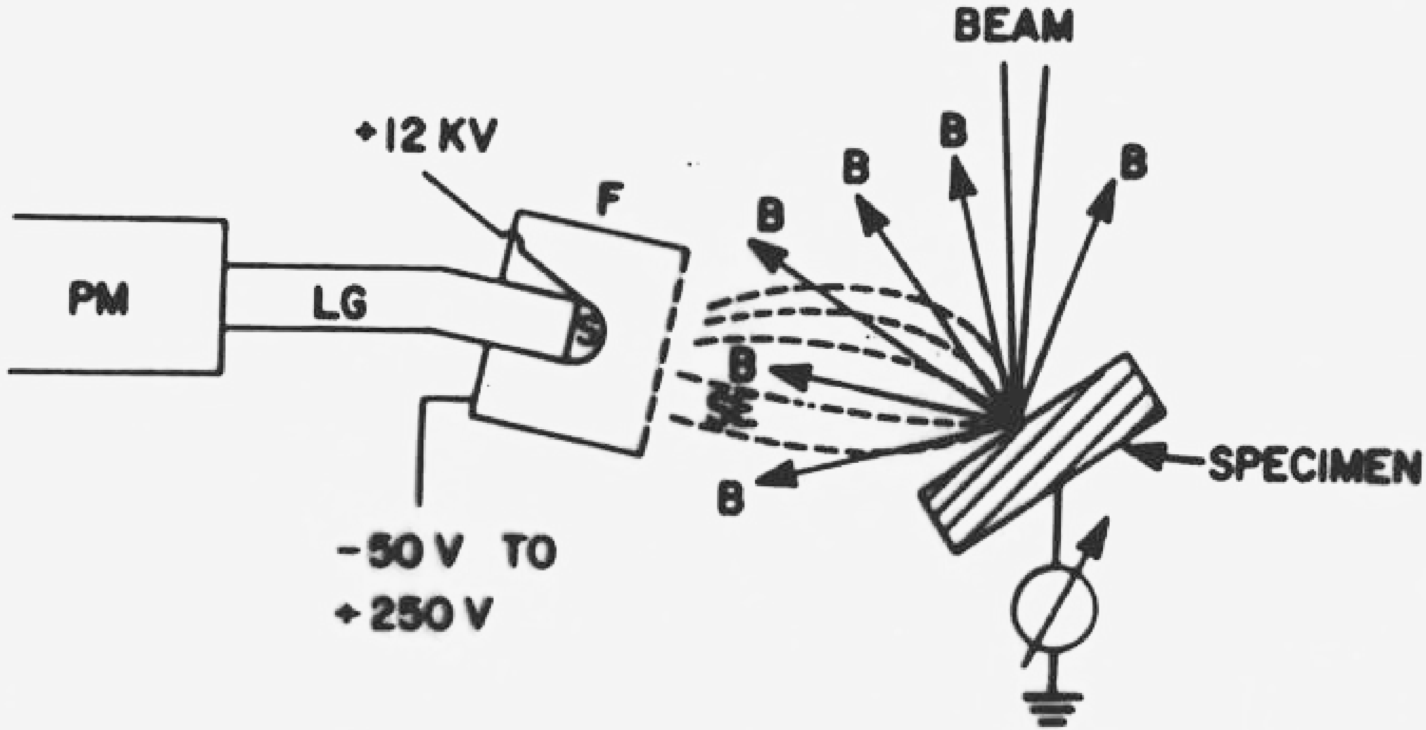


Figure 4.17. Schematic diagram of Everhart–Thornley scintillator–photomultiplier electron detector. B, backscattered electron; SE, secondary electron; F, Faraday cage; S, scintillator; LG, light guide; PM, photomultiplier.

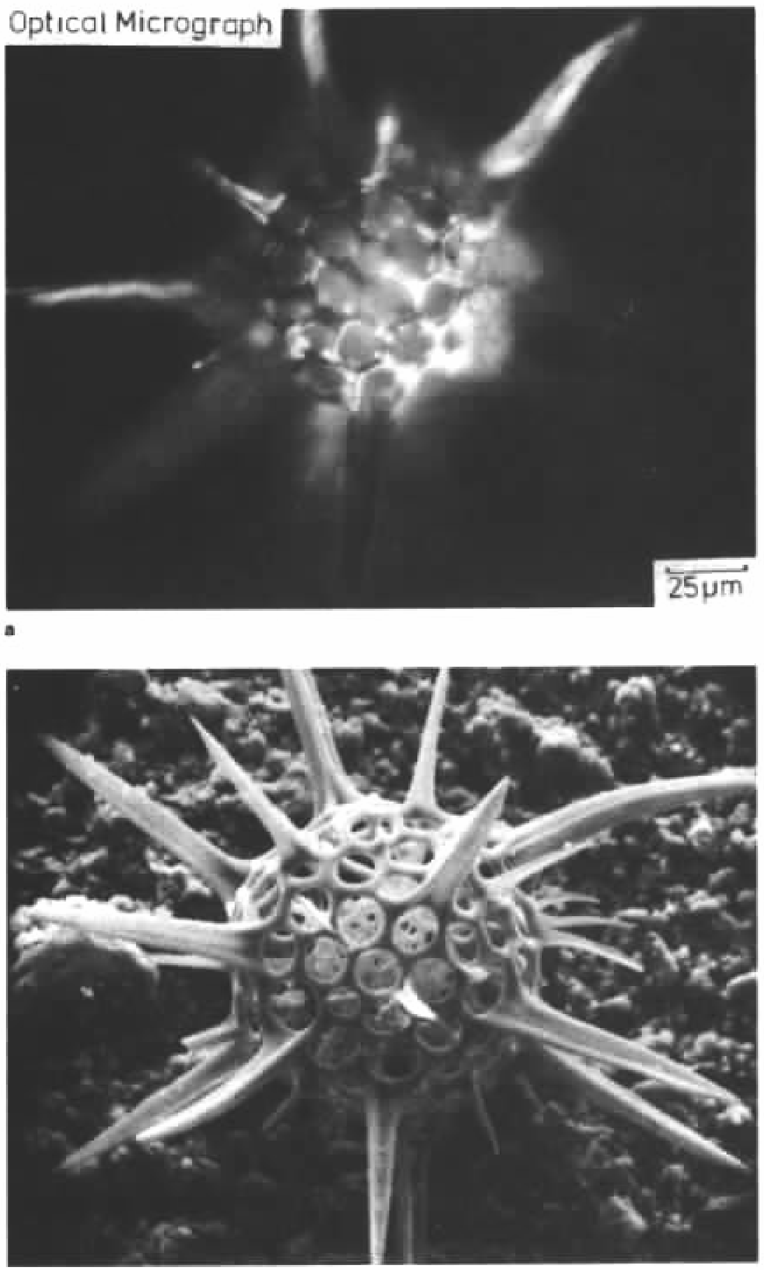
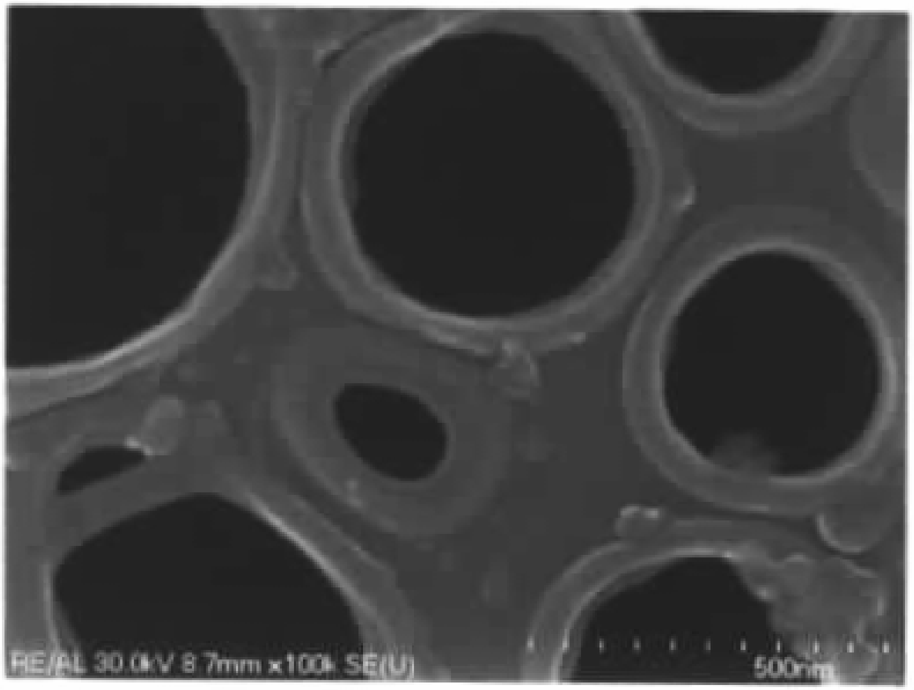
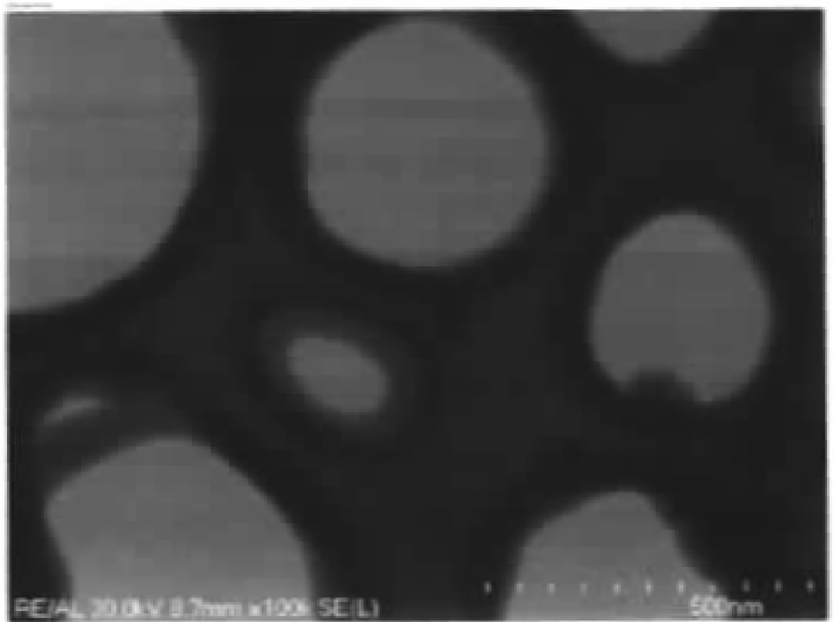


Figure 1.3. (a) Optical micrograph of the radiolarian *Trochodiscus longispinus*. (b) SEM micrograph of same radiolarian. The greater depth of focus and superior resolving capability of the SEM are apparent.

Examples of SEM images





[illegible], 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 $r = \lambda^{3/4} Cs^{1/4}$			image point size $r = \lambda^{3/4} Cs^{1/4}$		

Electron microprobe / Analytical electron microscopy:

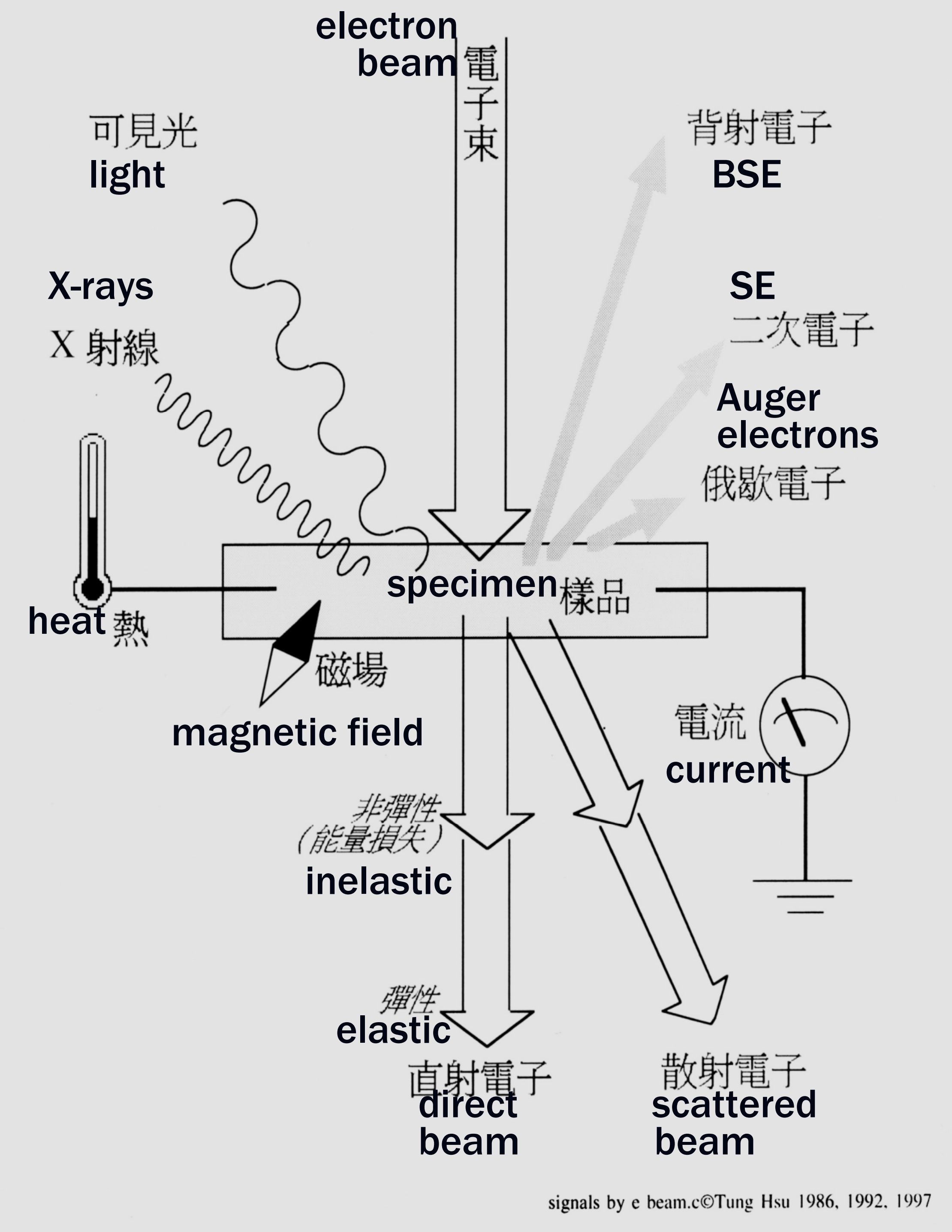
Energy dispersive (X-ray) spectrometer, EDS (EDX)

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

Electron energy loss spectroscopy, EELS

Quantitative analysis

etc.



ANALYSIS

Analytical Electron Microscopy

Electron Probe Micro Analysis (EPMA)

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

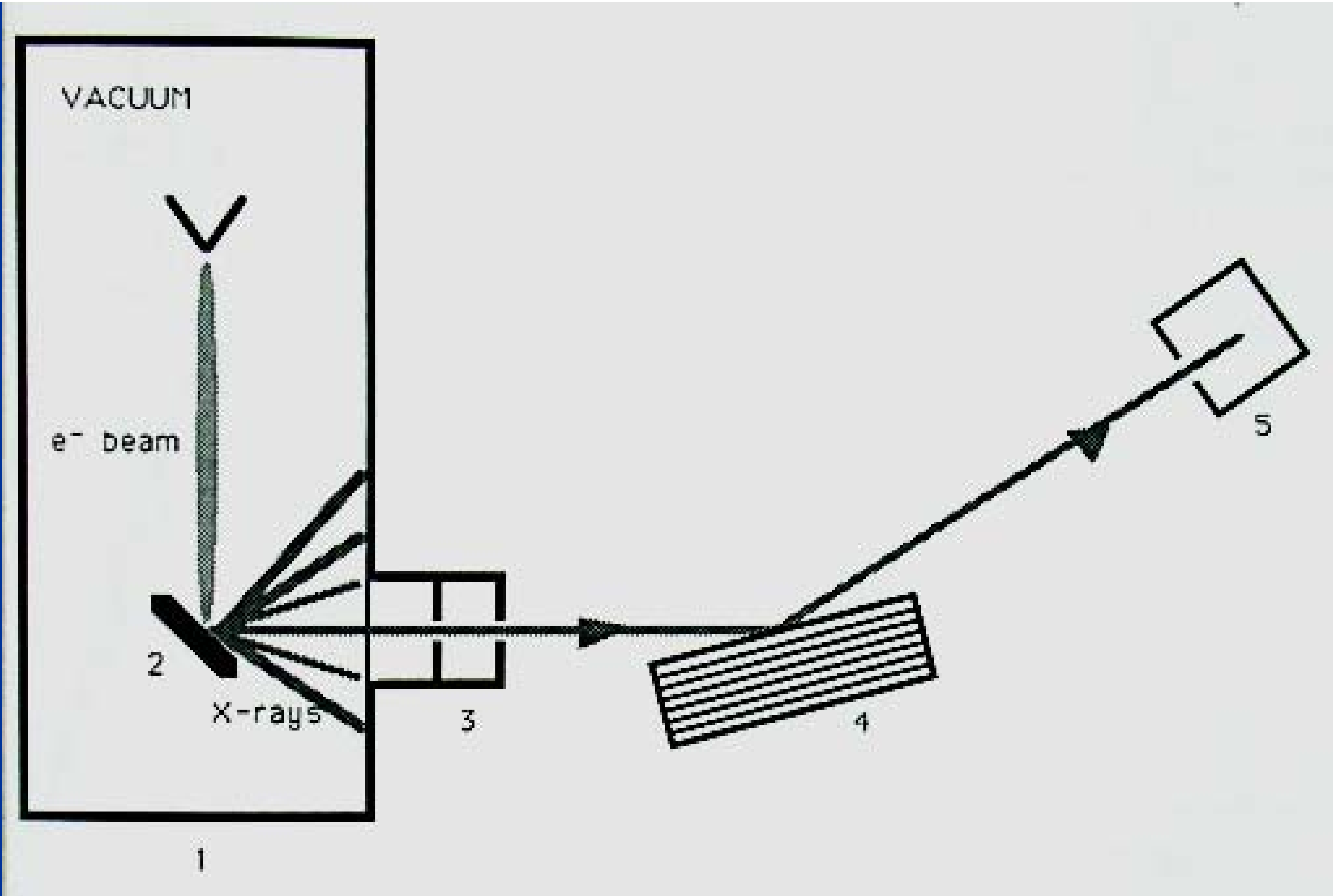
Parameter	$n\lambda$	$2d$	$\sin\theta$
diffraction	known	calculated	measured
spectrometry(WDS)	calculated	known	measured
spectrometry(EDS)	E: measured		

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

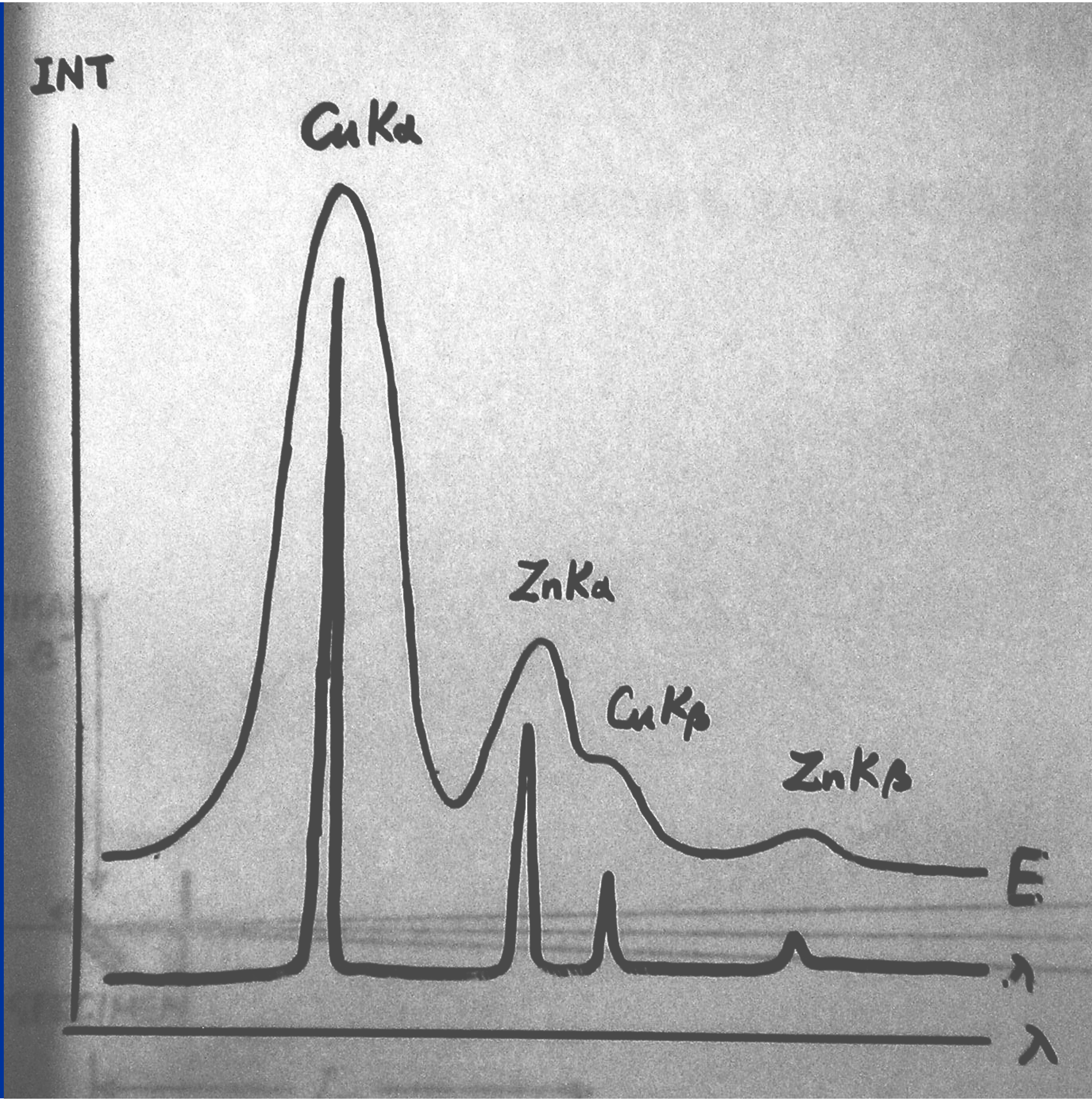
Instrumentation: Electron probe/microscope
Other particle beam
x-ray fluorescence
radioactive sources

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



E

Resolution

Quantitative analysis

Monte Carlo simulation

ZAF

Quantitative analysis: ZAF correction

MonteCarlo.demo

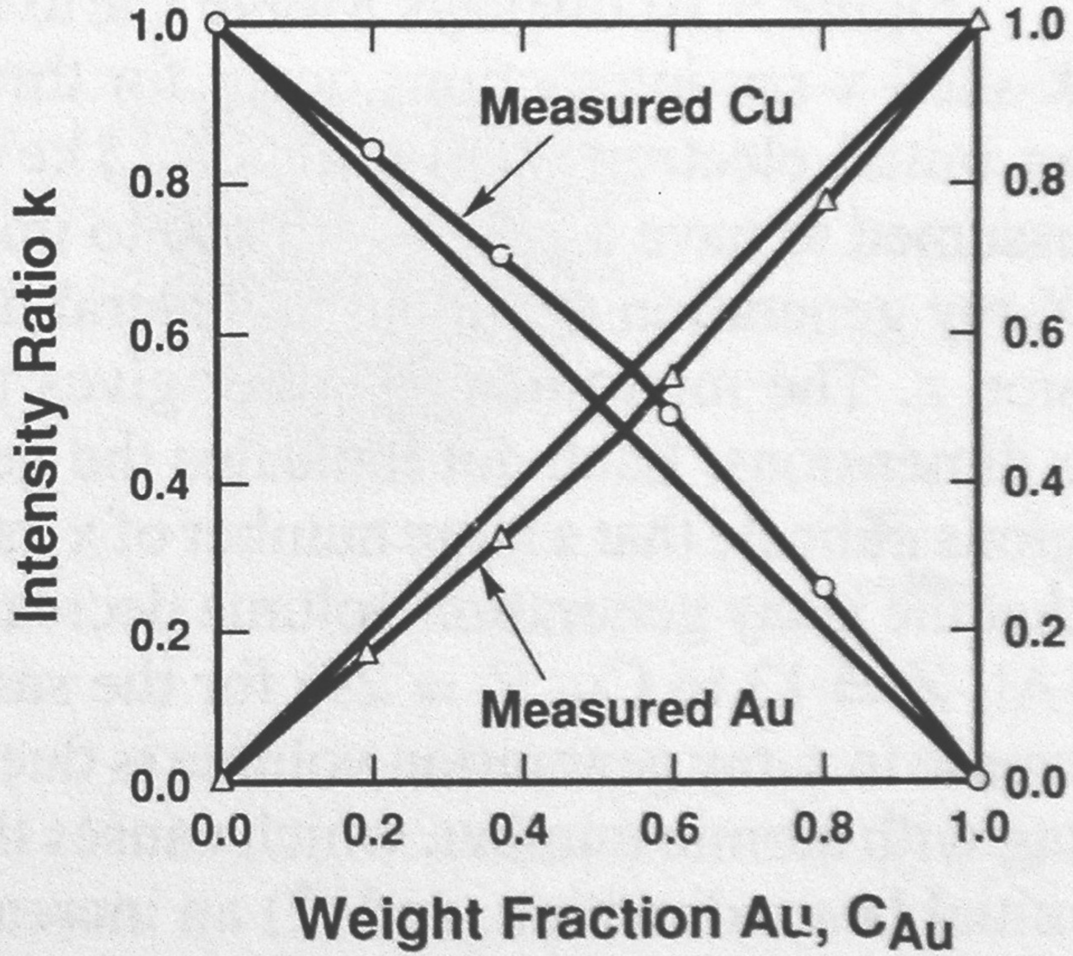


Figure 9.9. Measured Au–Cu intensity ratios k_{Au} and k_{Cu} versus the weight fraction of Au at 25 keV. Curves are measured k ratios, straight lines represent ideal behavior.

Goldstein, et al

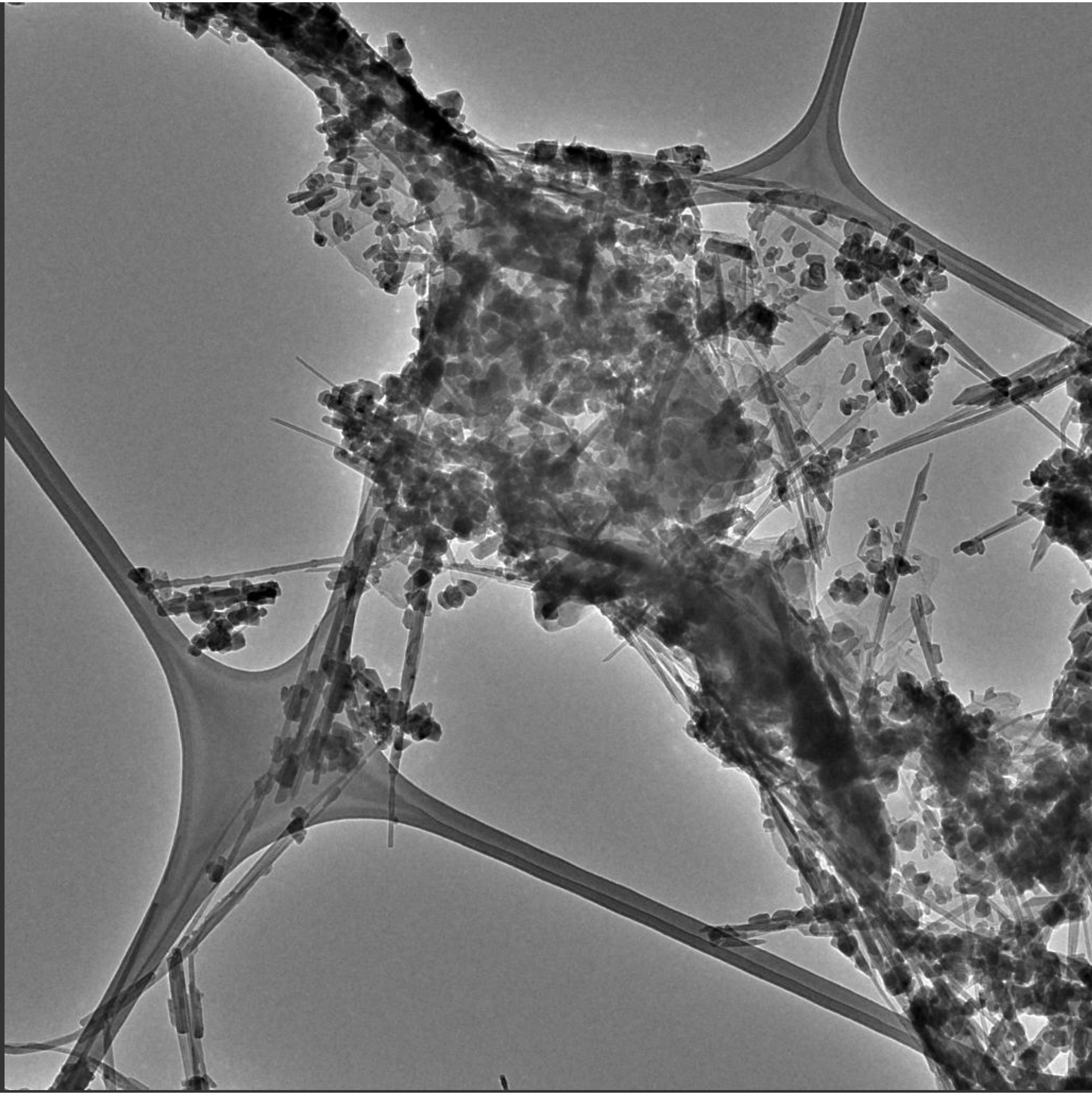
Quantitative analysis:

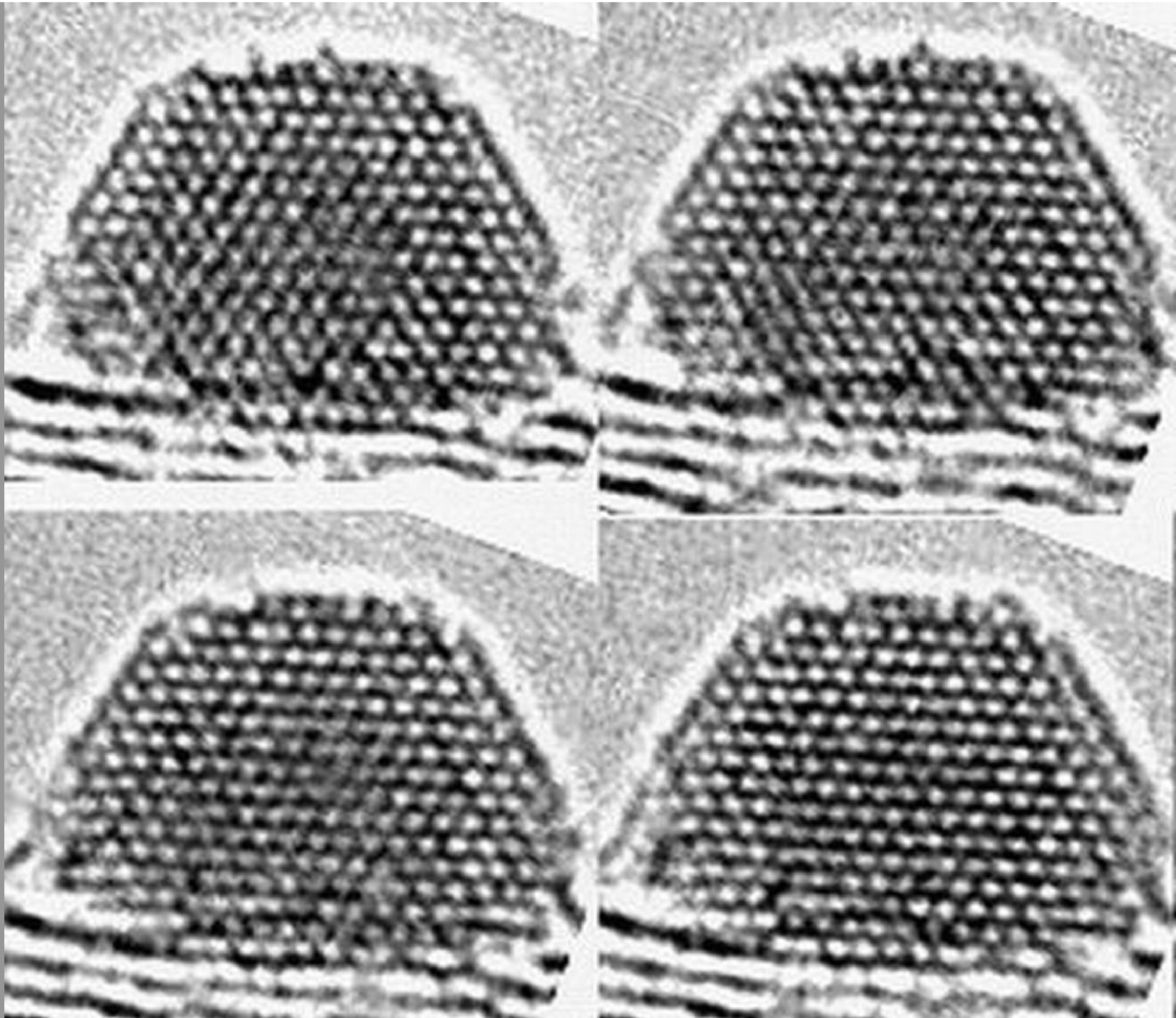
ZAF correction

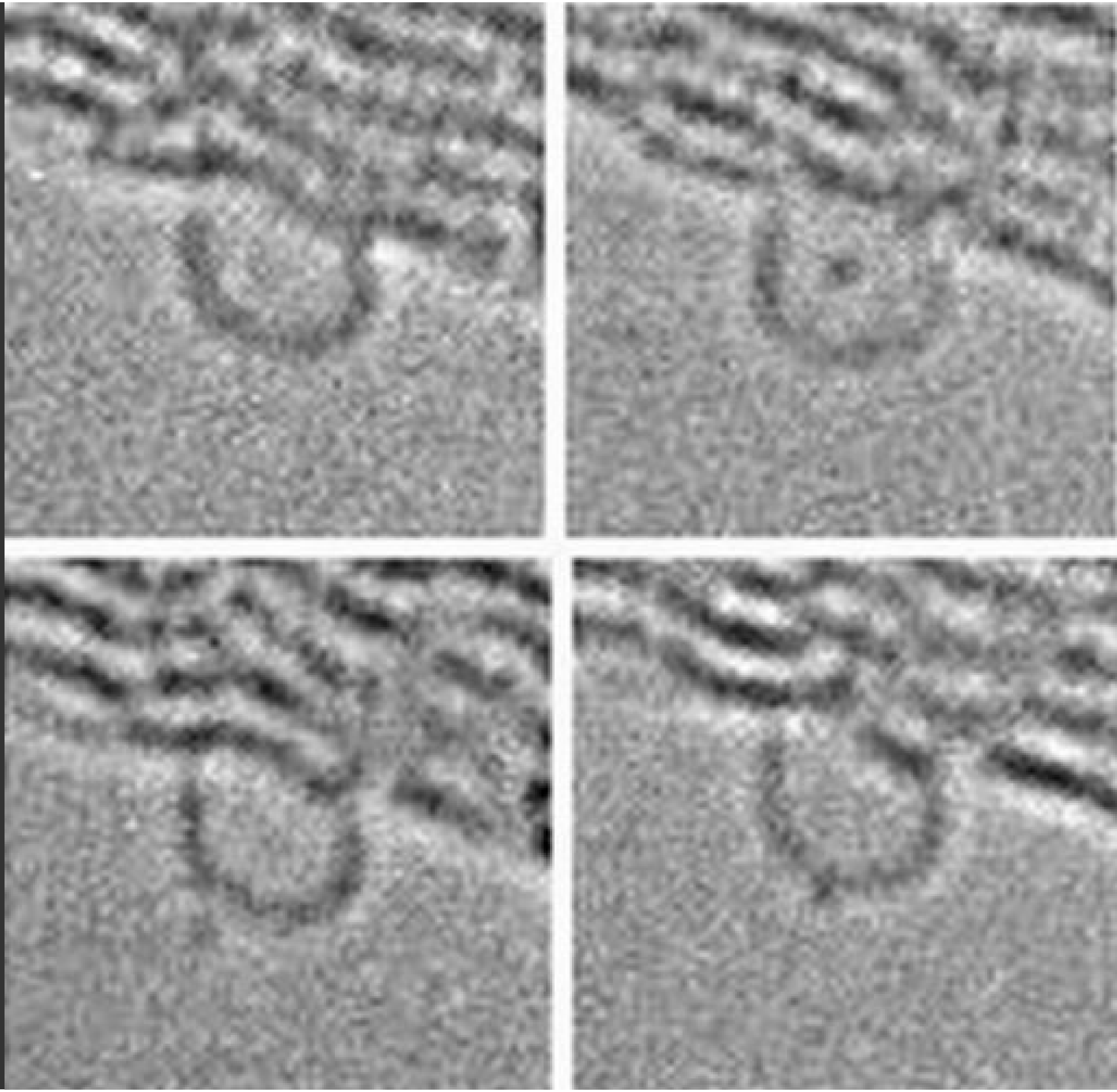
MonteCarlo.demo

Applications of TEM in characterization and manipulation of nano-materials

Specimen preparation: often straight forward
Observation: straight forward
beware of e-beam damage
Diffraction: weak signal
orientation
X-ray analysis: weak signal
EELS: to be investigated
In situ observation of dynamical phenomena
Manipulation: mechanical
electrical
temperature
deposition, evaporation







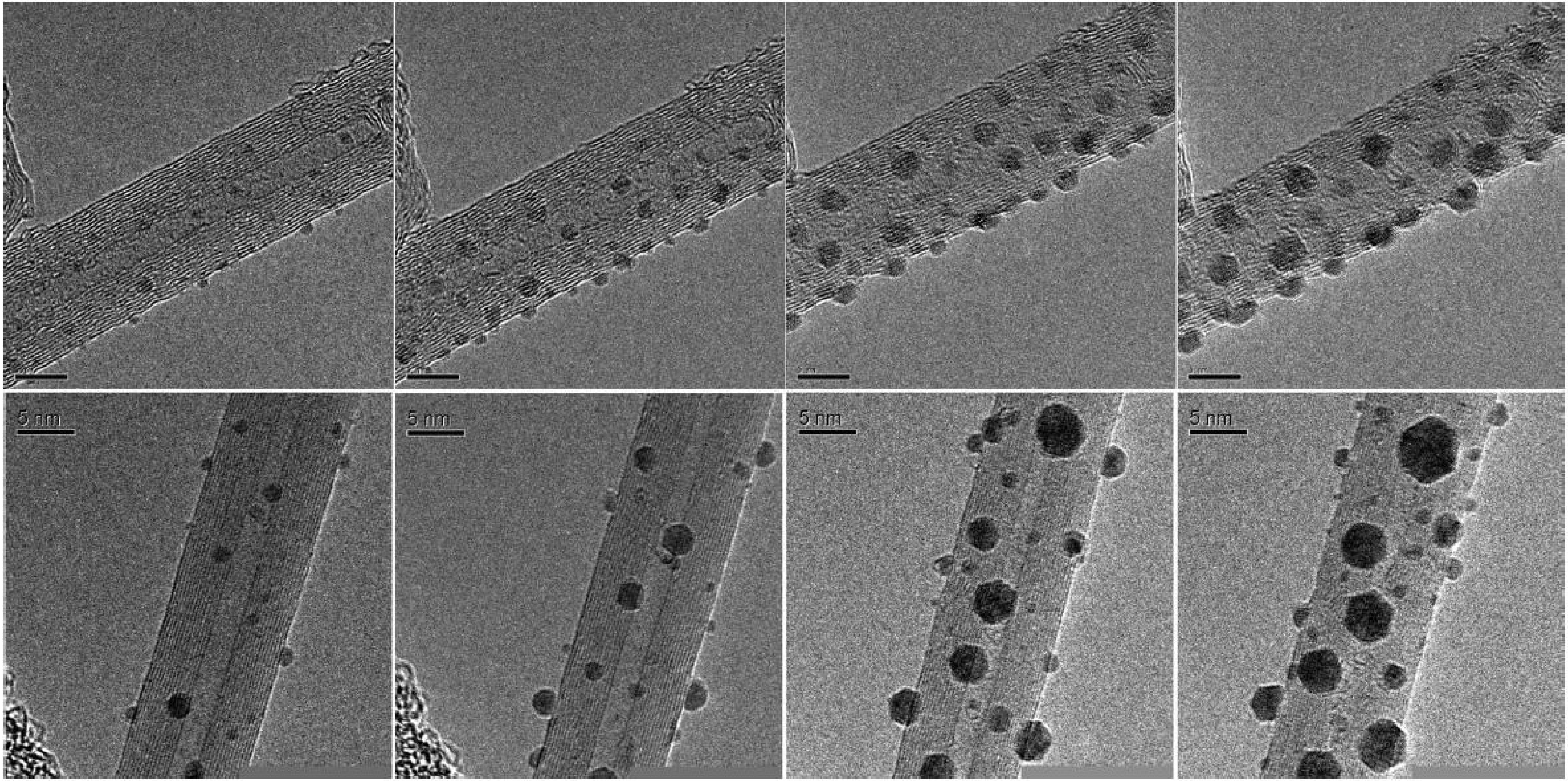


FIG. 1. Sequential TEM images of the formation of Ag clusters on the CNTs at two different temperatures. Images in the top row were taken at room temperature 296 K (heating current $I_h=0$) and those in the bottom at 503 K ($I_h=0.17$ A). From the left frame to right, the images were taken with time at about 5, 15, 35, and 55 min after Ag deposition. The scale bars are 5 nm in length.

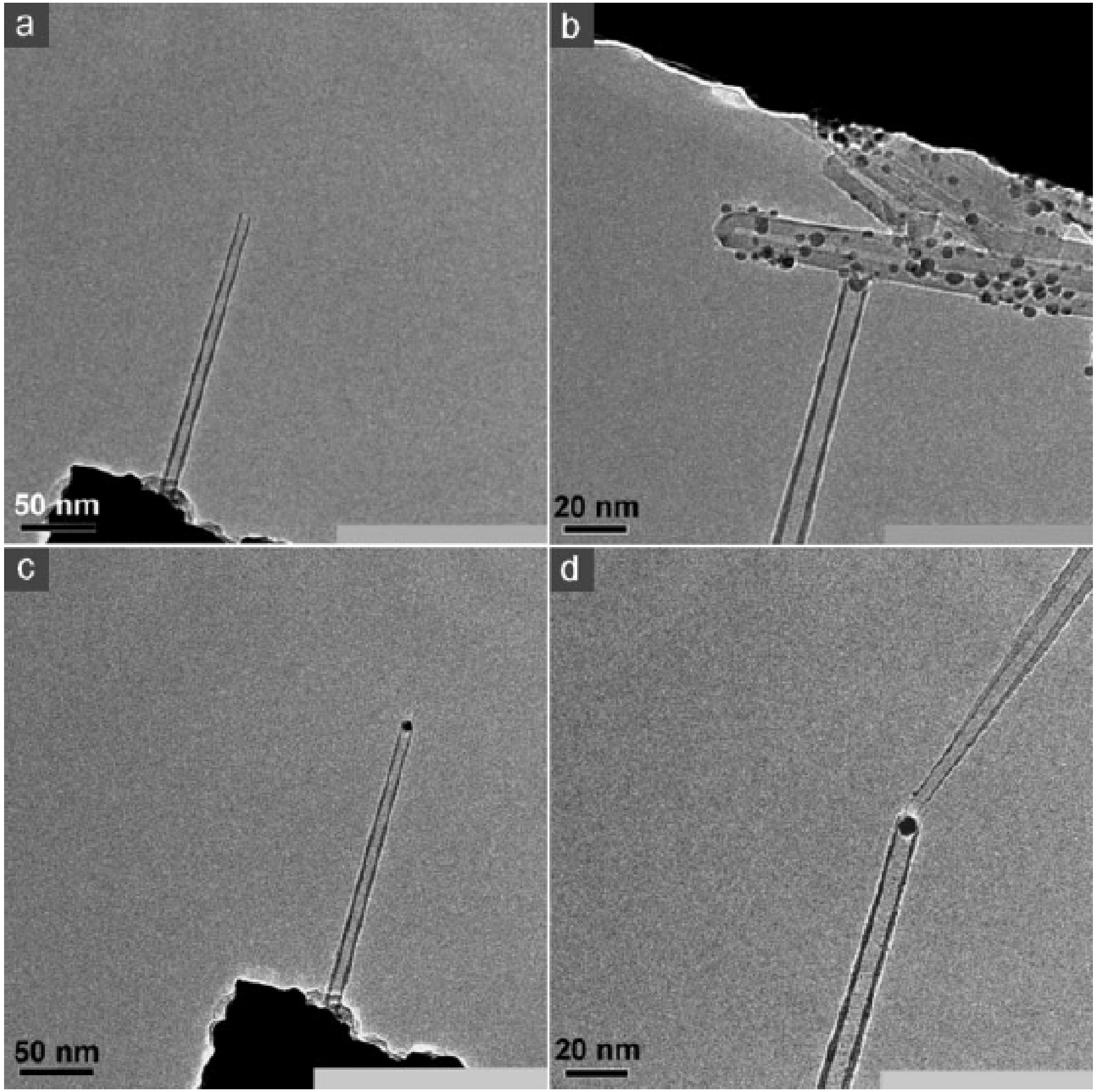


Figure 4. Transmission electron micrographs showing the insertion of Ag clusters into a peeled MWNT. a) A MWNT with a defined inner diameter (≈6nm) after the peeling process. b) This tube making contact with a chosen Ag cluster. Ag clusters of various sizes deposited on the nanotubes were formed by electron-beam evaporation under UHV. c) The Ag cluster transferred to the tube by pure mechanical force. d) The transferred Ag cluster pushed slightly into the tube by the piston rod.

DOI: 10.1002/smll.200800563

“In Situ Tailoring and Manipulation of Carbon Nanotubes”

Yuan-Chih Chang,
Yuan-Hong Liaw,
Yang-Shan Huang,
Tung Hsu,
Chia-Seng Chang,
and Tien-Tzou Tsong

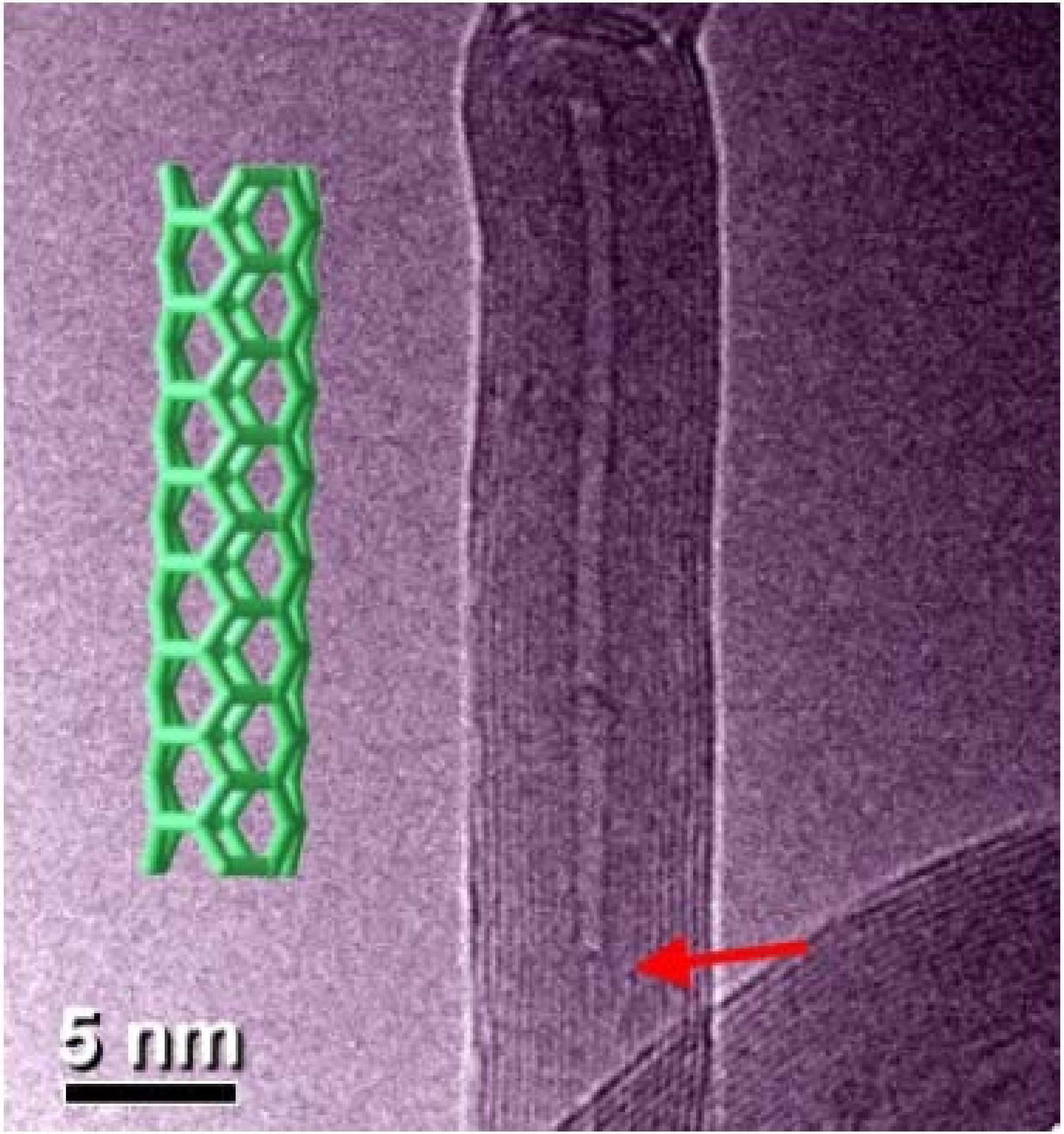


Fig. 1. Transmission electron micrograph shows the smallest SWNT formation. The smallest SWNT is arrowed. Schematic drawing is accompanied to illustrate the smallest SWNT.

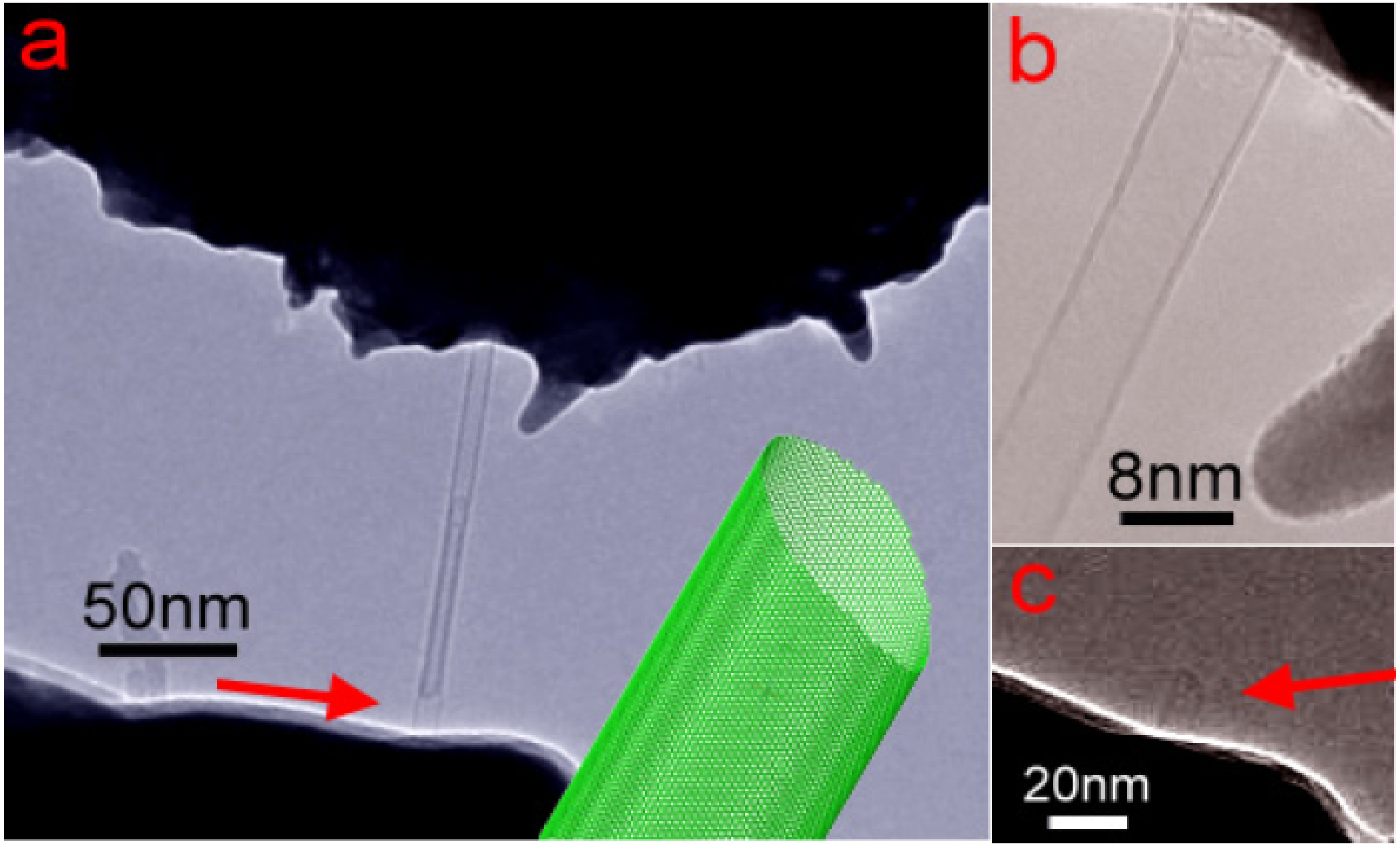
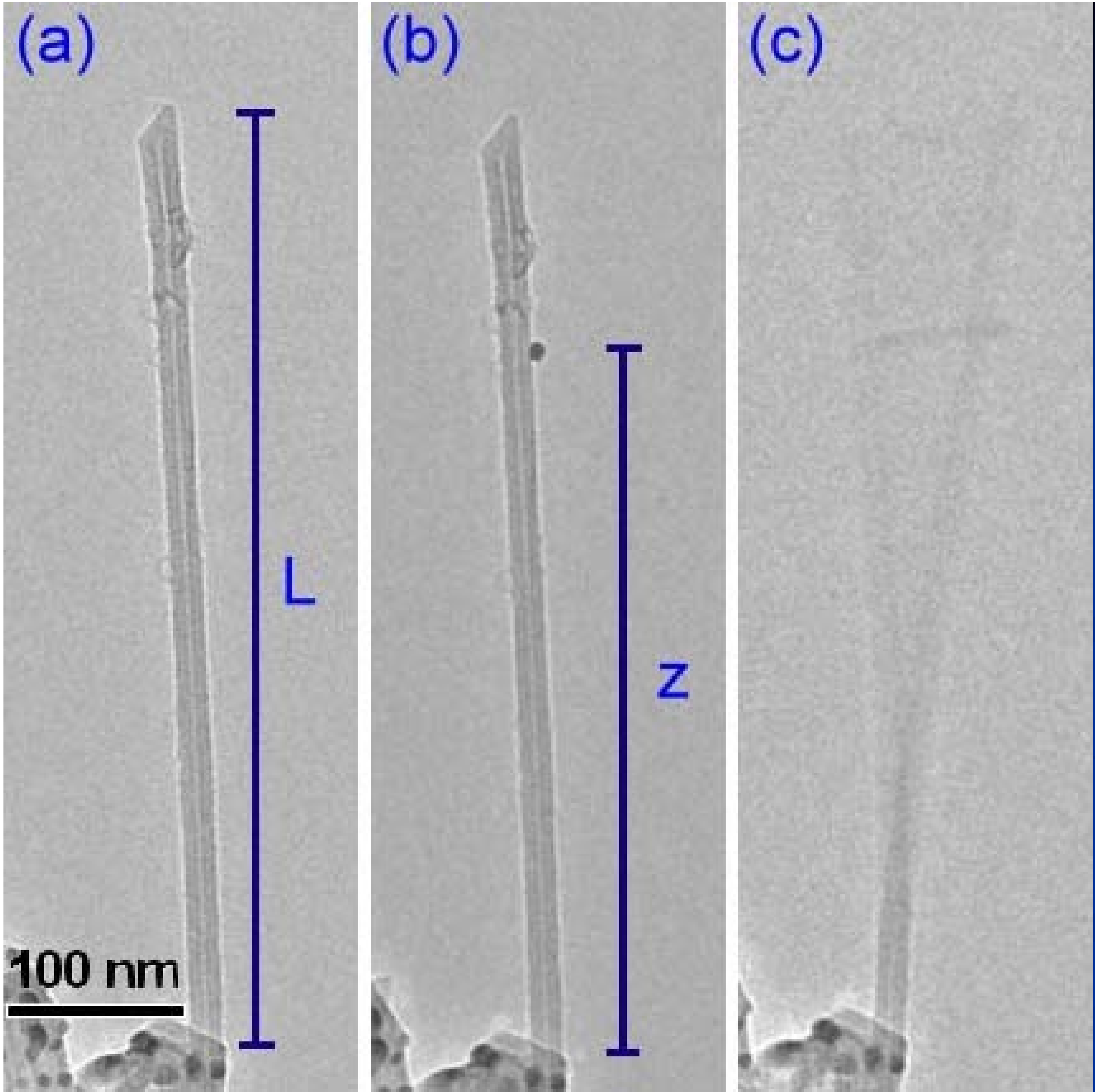


Fig. 2. Transmission electron micrographs show the largest MWNT fabrication at last stage of the extraction process. Schematic drawing is accompanied to illustrate the largest SWNT. (a) The SWNT formation (see arrow) during second internal peeling process. (b) A two-wall tube pulled out after the second peeling process. (c) The remaining SWNT on the base (see arrow) obtained by two internal peeling processes.



**Shau-Chieh Wang,
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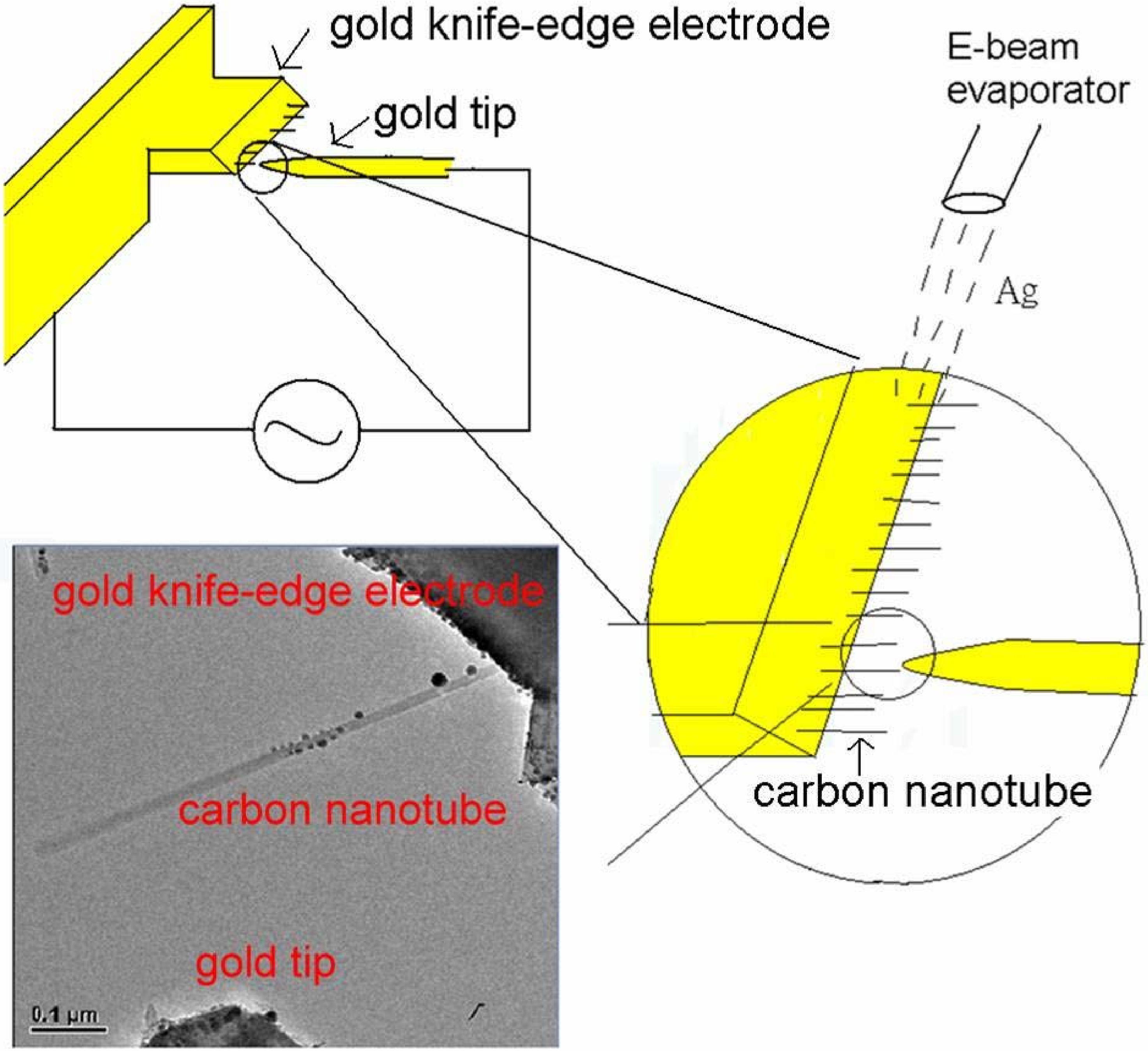
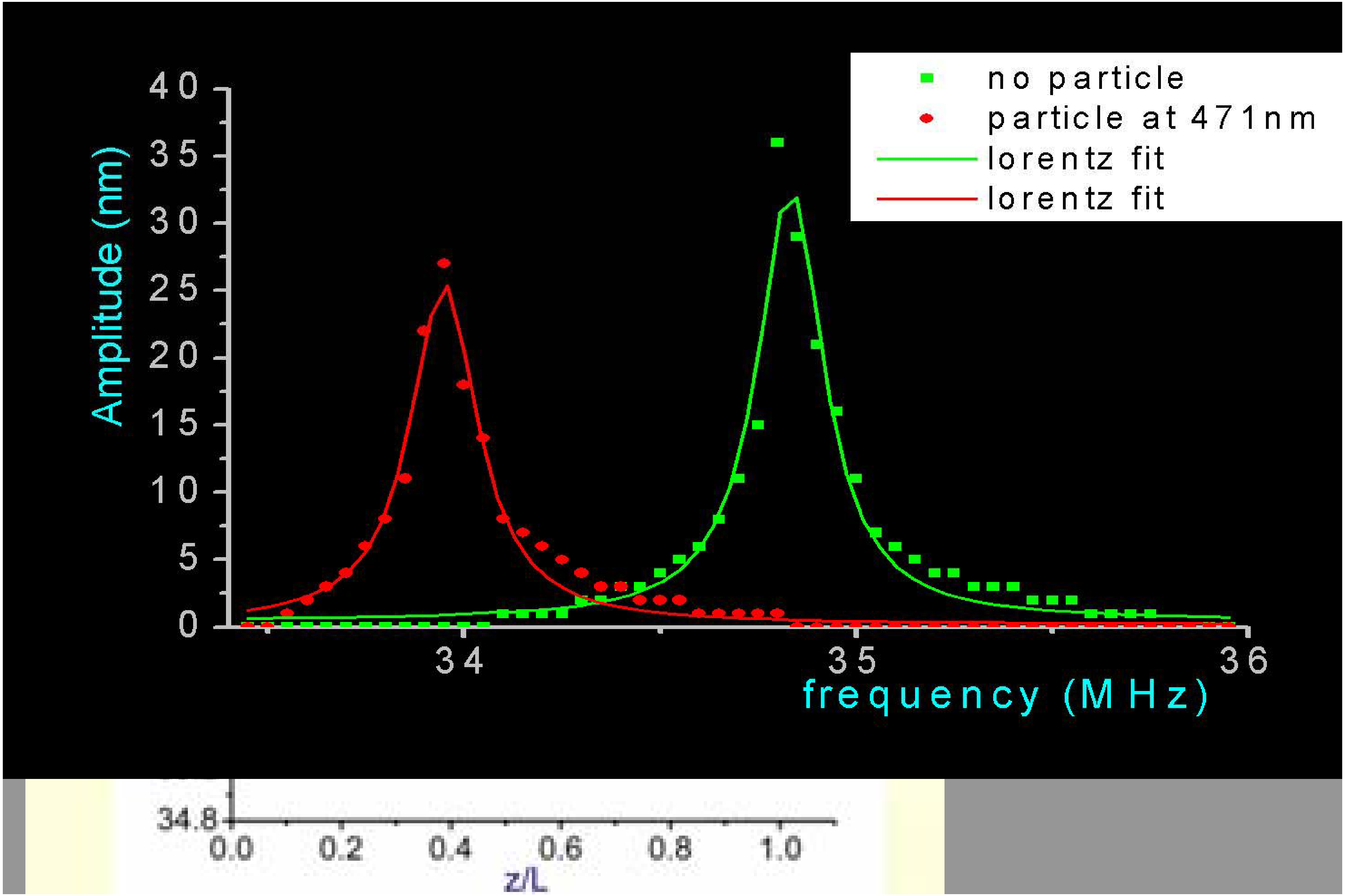
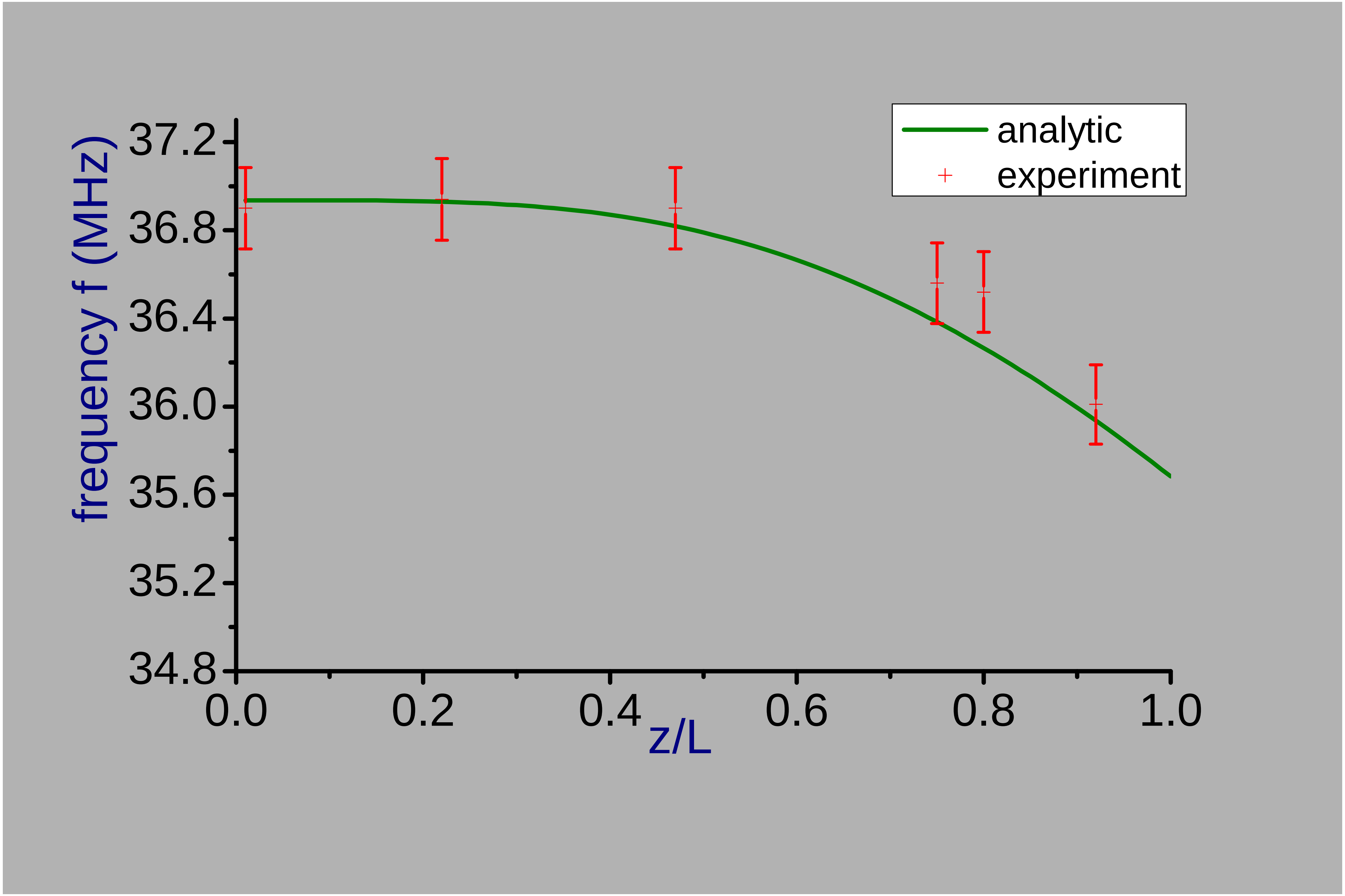
Der-Hsien Lien,
Tung Hsu,
Chia-Seng Chang,
and Tien-Tzou Tsong**


gold knife-edge electrode
gold tip
E-beam
evaporator
Ag
carbon nanotube
gold knife-edge electrode
carbon nanotube
gold tip
0.1 µm



no particle
particle at 471nm
lorentz fit
lorentz fit
Amplitude (nm)
0
5
10
15
20
25
30
35
40
34
35
36
frequency (MHz)
34.8
0.0
0.2
0.4
0.6
0.8
1.0
z/L


analytic
experiment
frequency f (MHz)
37.2
36.8
36.4
36.0
35.6
35.2
34.8
0.0
0.2
0.4
0.6
0.8
1.0
z/L

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.