ELECTRON MICROSCOPY

13:10 – 16:00, Oct. 6, 2008
Institute of Physics, Academia Sinica

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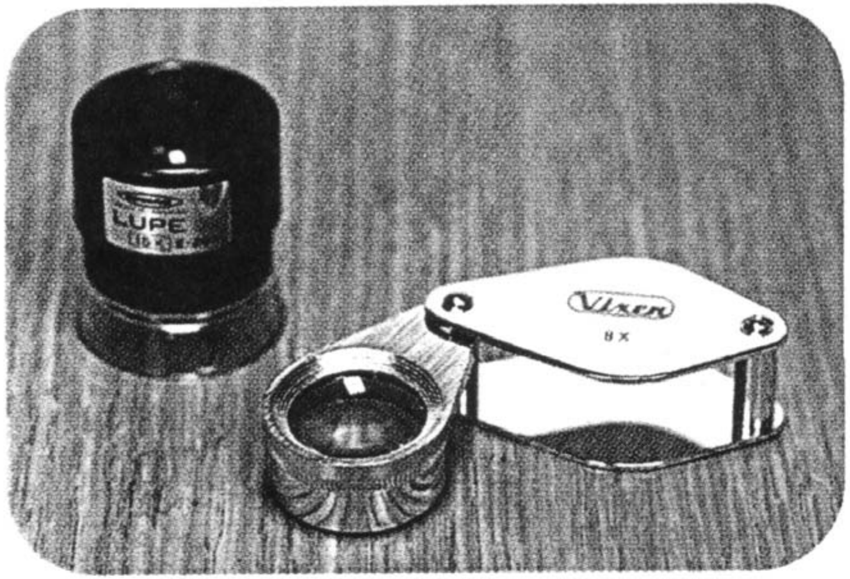
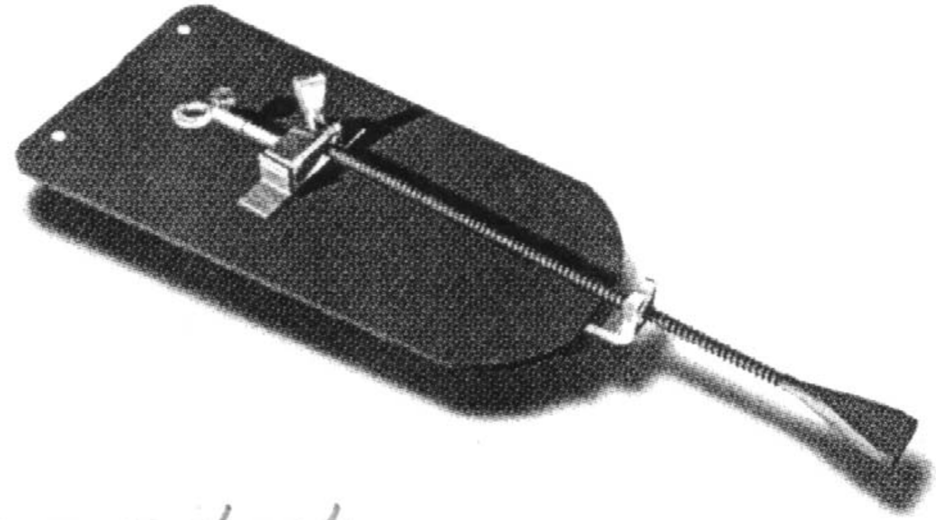
[redacted]

《顕微鏡入門》沼澤茂美, 2001, 誠文堂新光社, 20頁.



10〜20倍に拡大できる理科観察用ルーペ。野外での理科観察や、岩石採集などに気軽に持ってゆくことができます。ほとんどのものは「繰り出し式」と呼ばれる保護枠がついています。



Leeuwenhoek

レーウェンフックが作った初期の顕微鏡。対物レンズと接眼レンズの組合せではなく、1枚のレンズしか用いない単レンズ式でした。これで200倍以上の高倍率を作り出しました。

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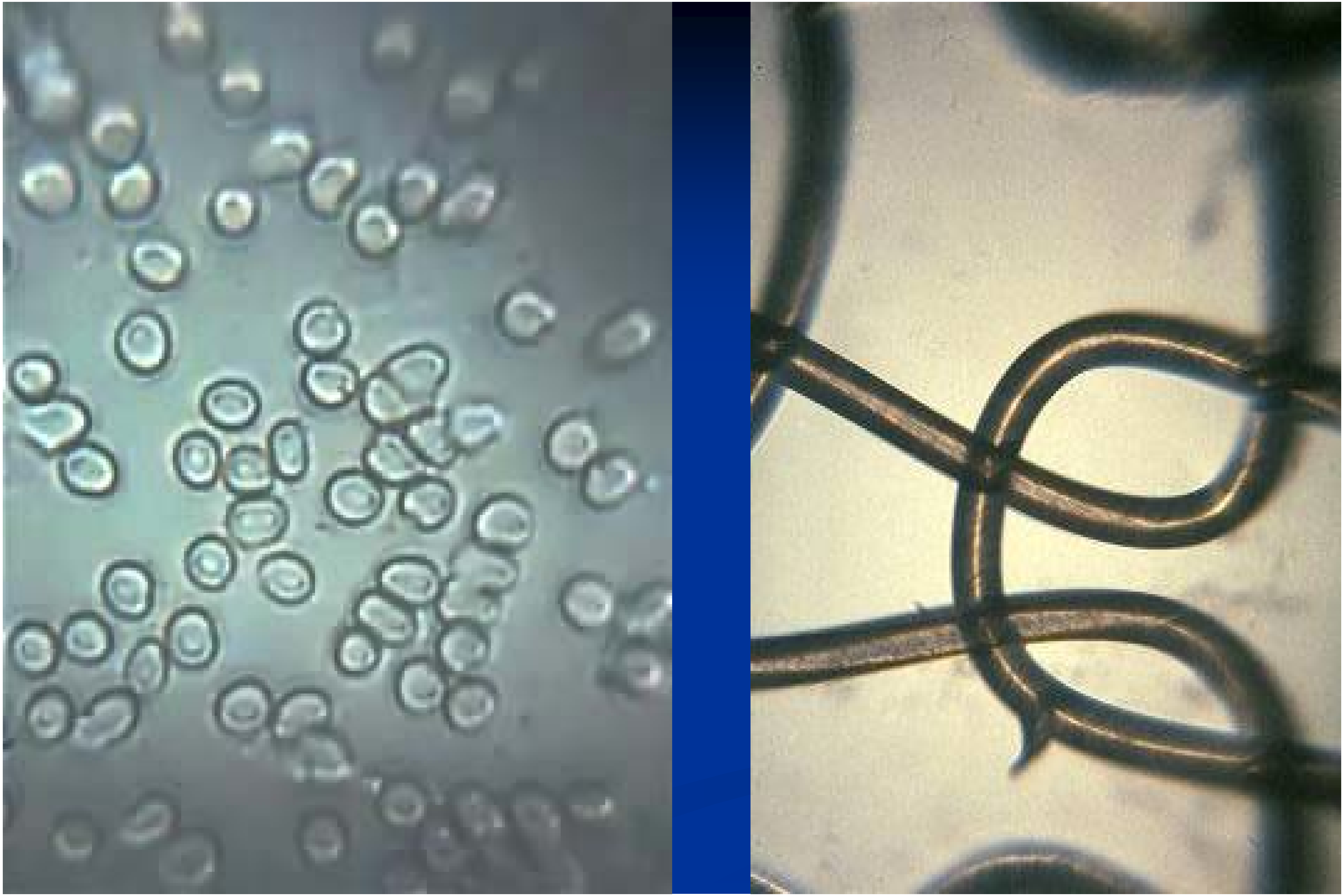
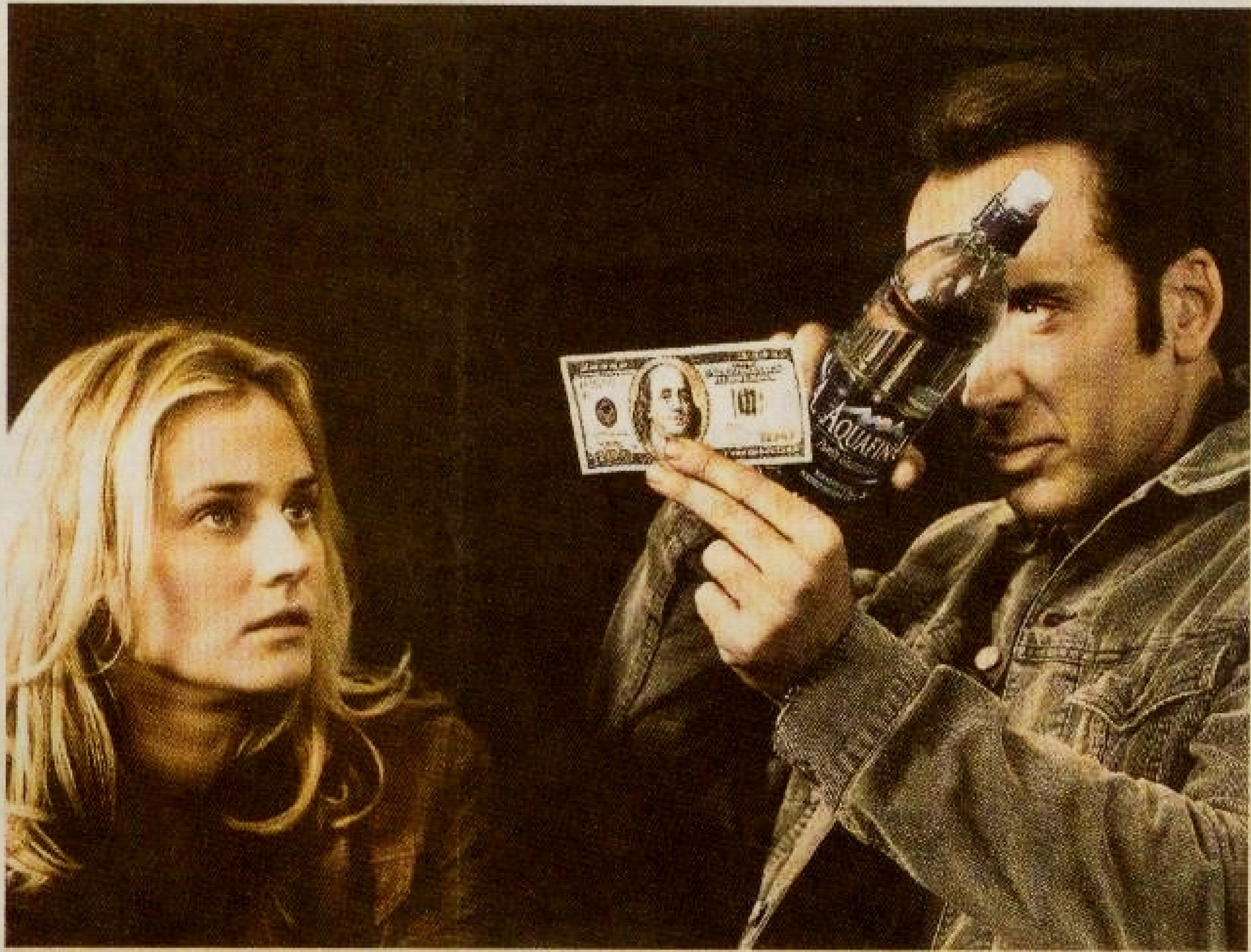




巨星登寶島

聯合.04.12.14,D1

▲「國家寶藏」男女主角尼可拉斯凱吉（右）和黛安克魯格等一行人，今天浩浩蕩蕩從南韓飛來台灣宣傳。

本報資料照片

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, 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/Applications

Scanning electron microscopy

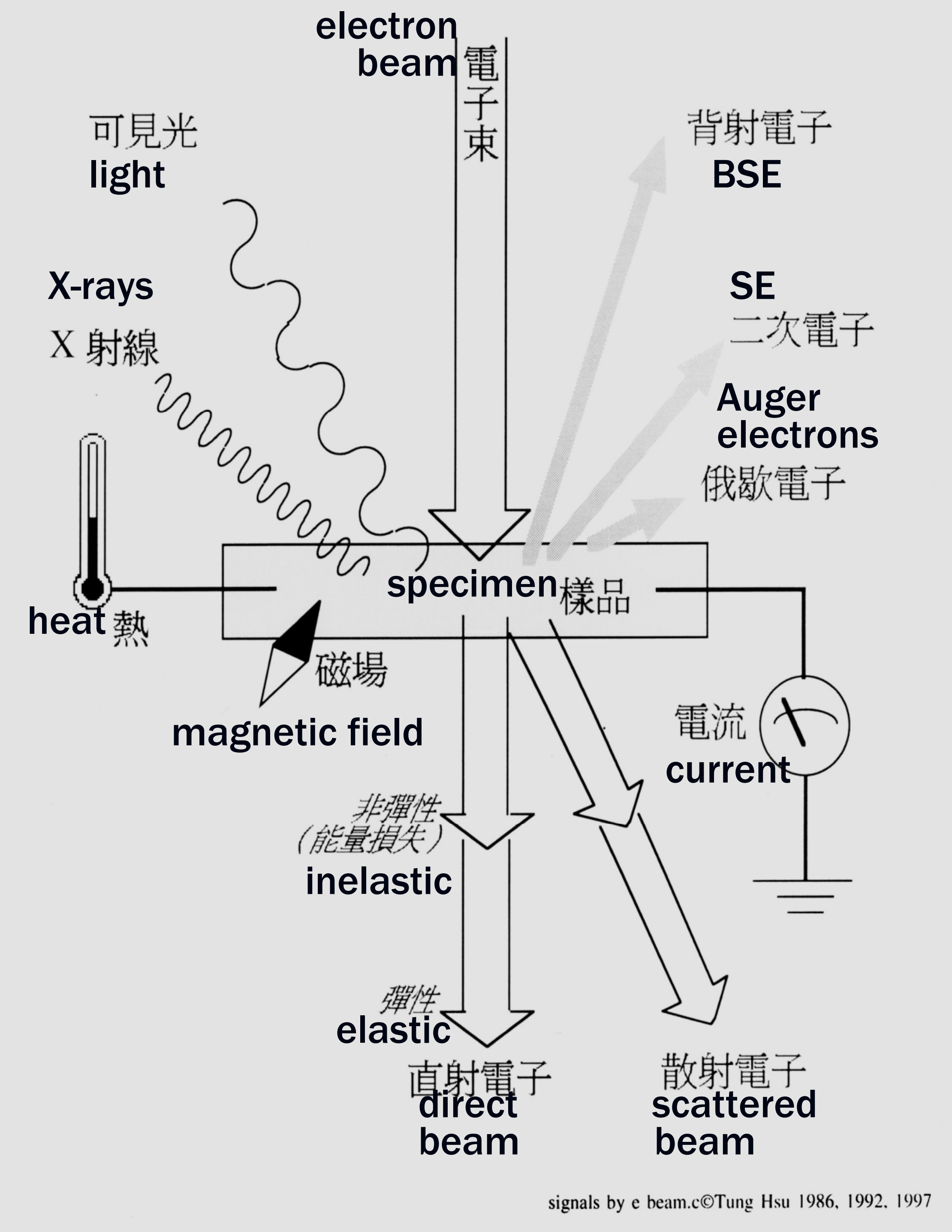
Electron microprobe / Analytical electron microscopy

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



Why electron microscopy (EM)?

Information obtainable from EM

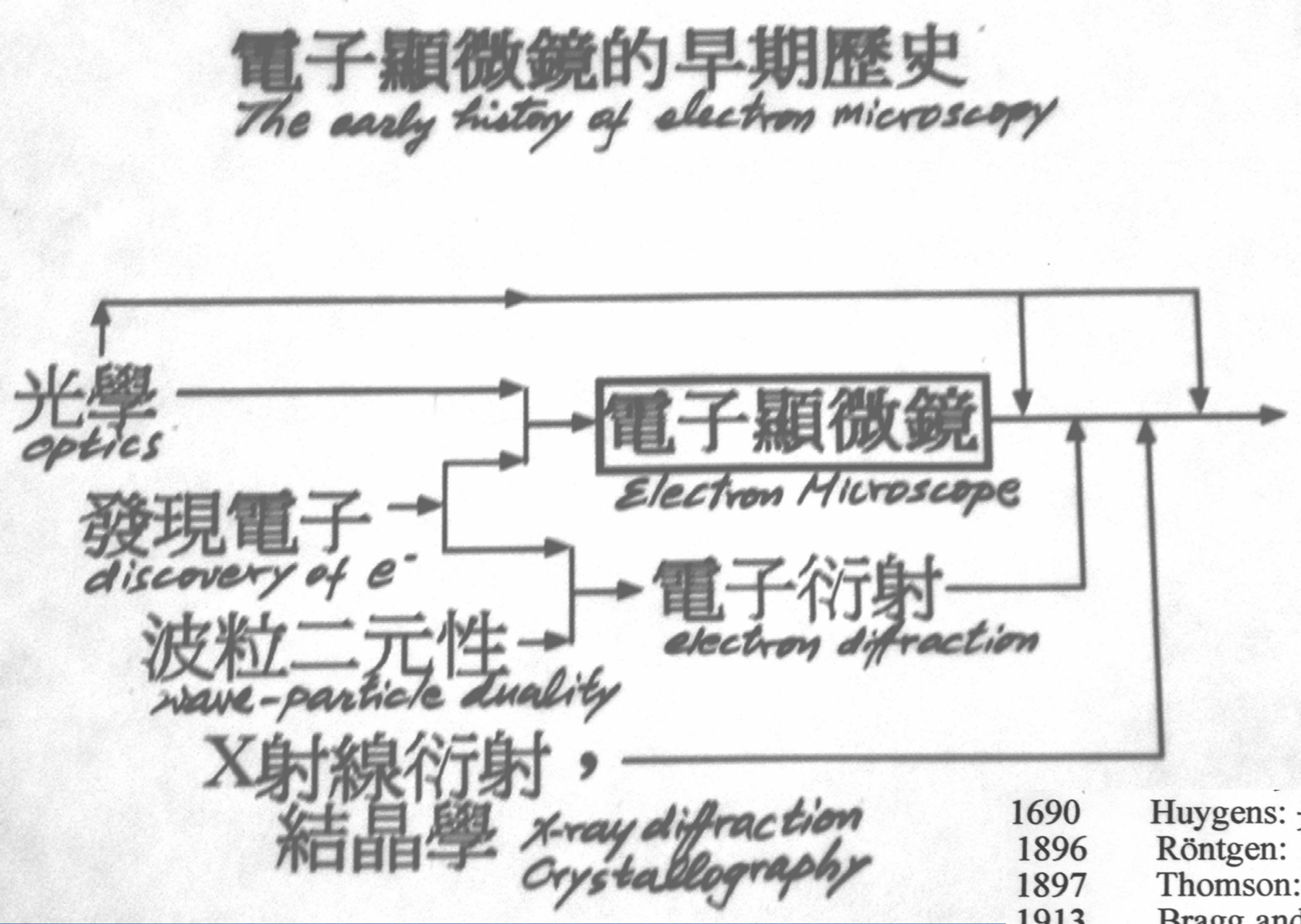
Beam/solid interaction

- 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

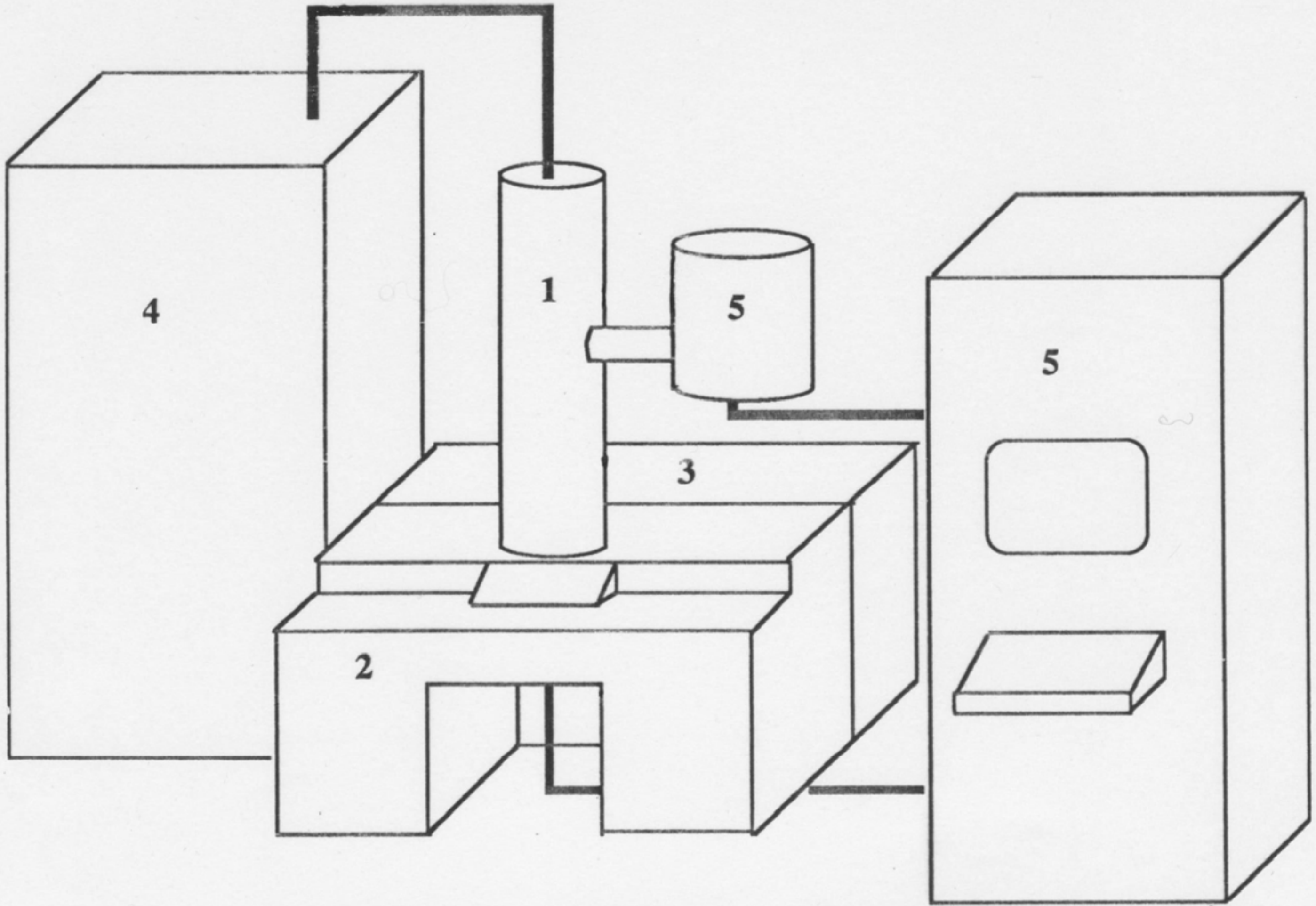
A brief history of electron microscopy



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

The Electron microscope

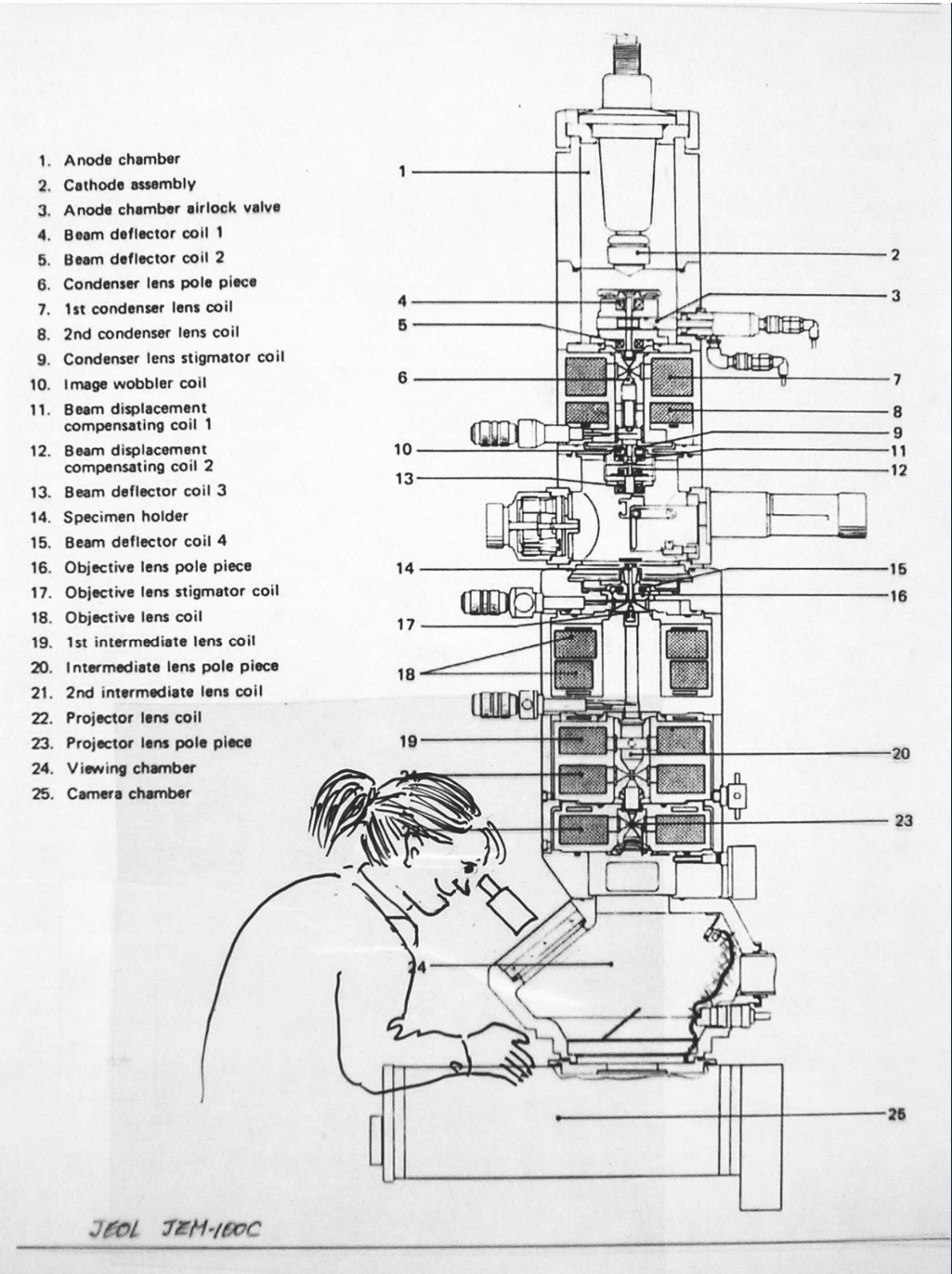
Structure and major components



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

The Lens System:

Condenser Lens:

Controls beam intensity, density, convergence, coherence.

Objective Lens:

Magnification, introducing contrast.

Intermediate Lens:

Further magnification, imaging or diffraction.

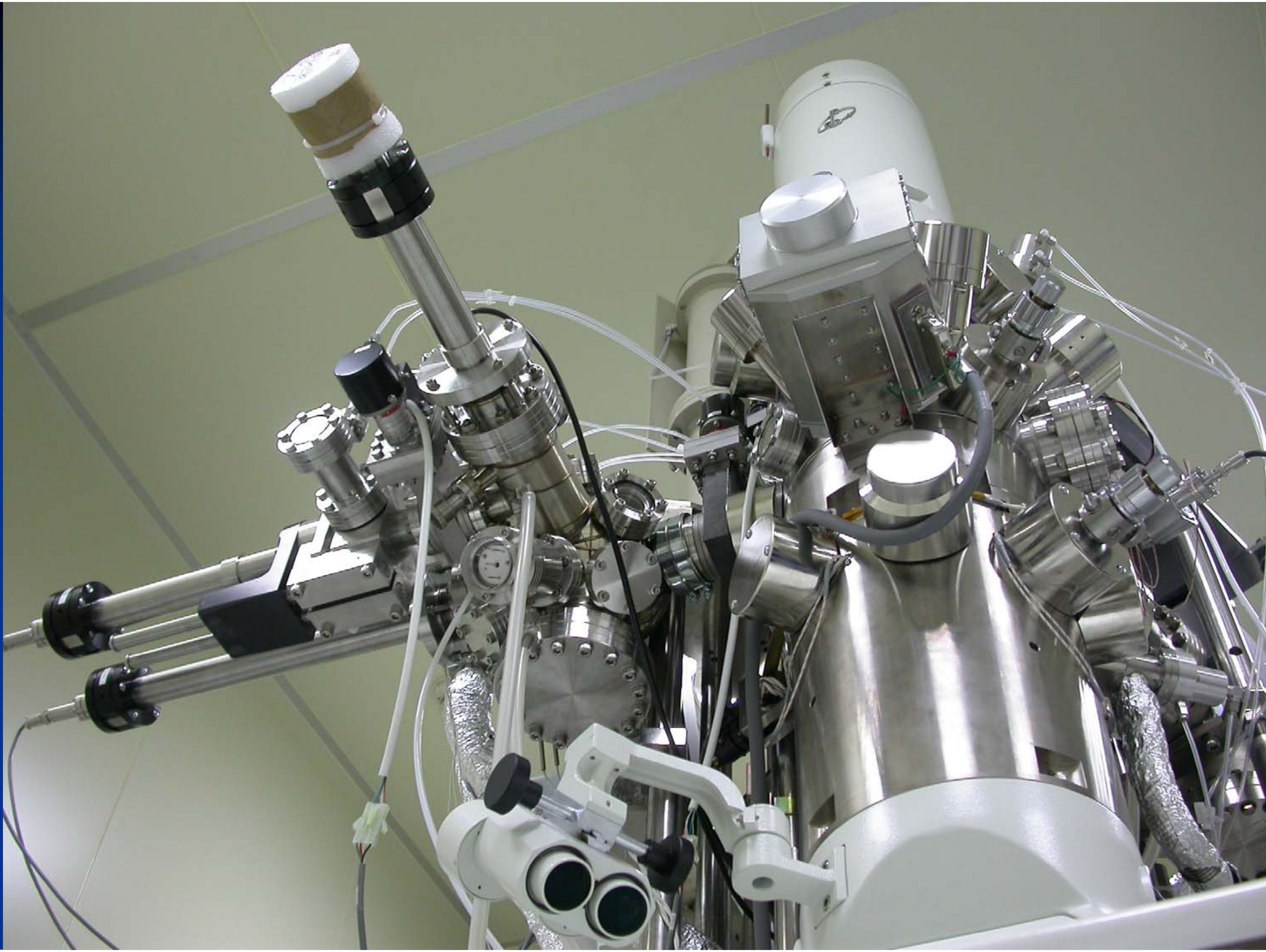
Projector Lens:

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

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

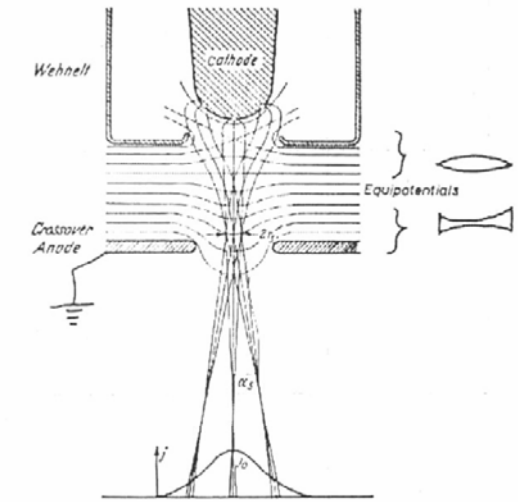


Fig. 3.2. The terminology associated with the electron gun.

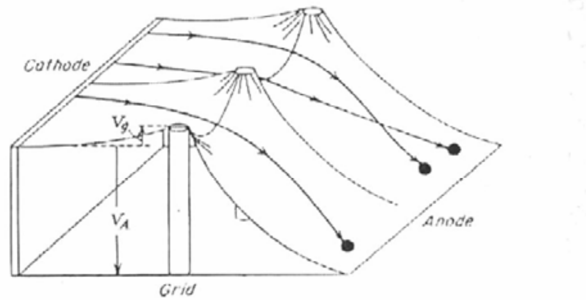


FIG. 2.9. The rubber-membrane model for experimental determination of elect[ron]
paths (exaggerated vertical scale).

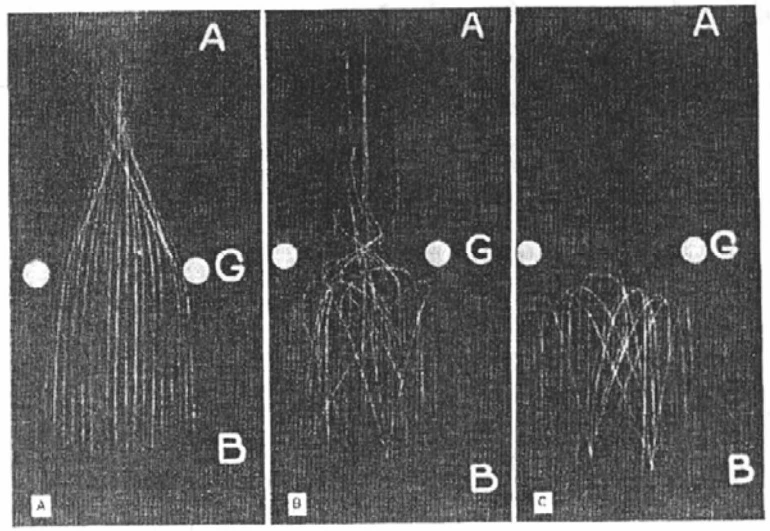
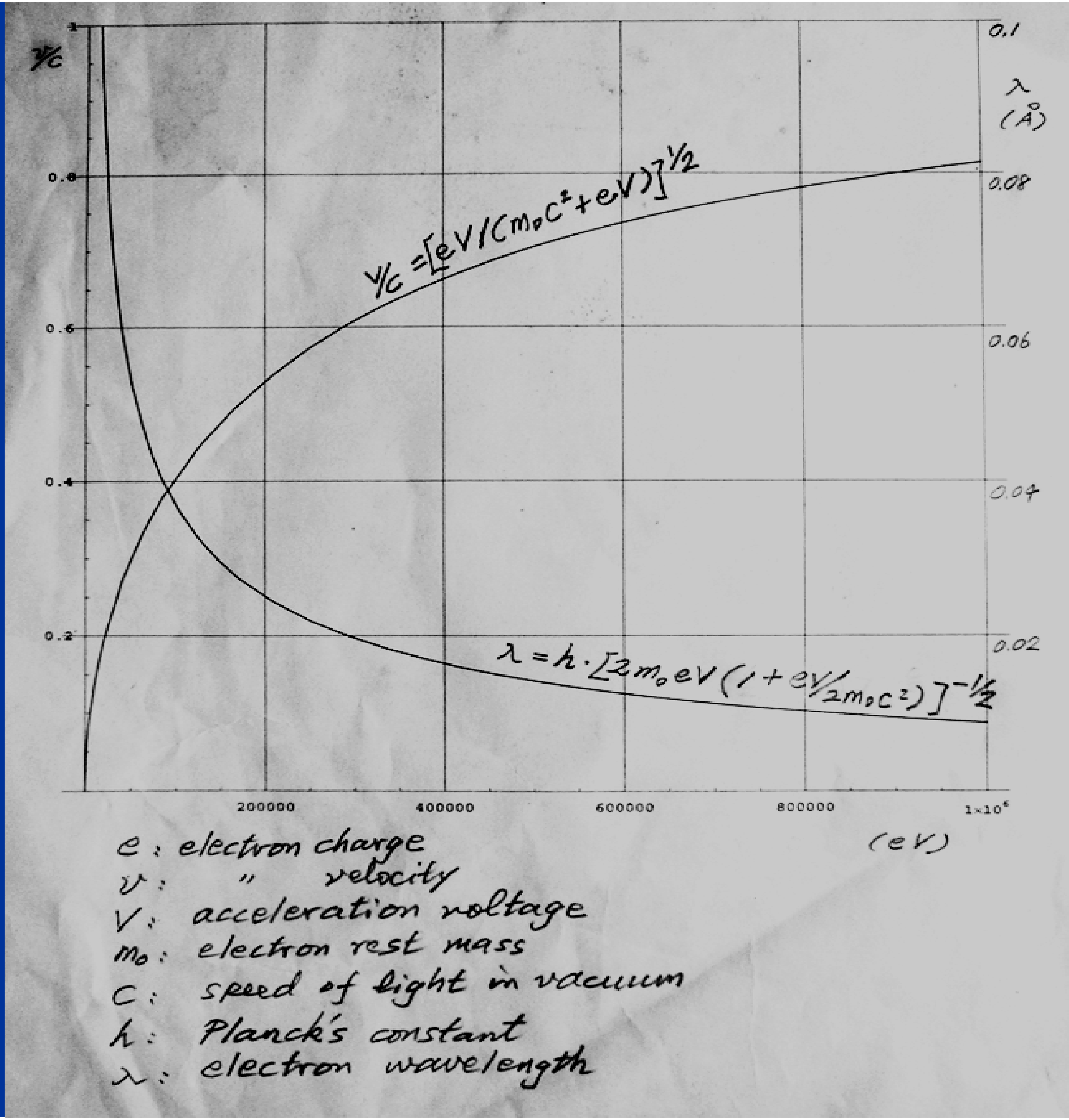


FIG. 2.10. Electron paths in a triode as determined with the rubber-membrane model. *A* is the anode, *B* the cathode, and *G* the grid. The grid potential is increasingly negative from A to C. (*Courtesy Philips Tech. Rev., Ref.* 2.)

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



v/c
0.8
0.6
0.4
0.2
0.1
λ (Å)
0.08
0.06
0.04
0.02
200000
400000
600000
800000
1×10^6
(eV)
$v/c = [eV/(m_0c^2+eV)]^{1/2}$
$\lambda = h\cdot[2m_0eV(1+eV/2m_0c^2)]^{-1/2}$
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

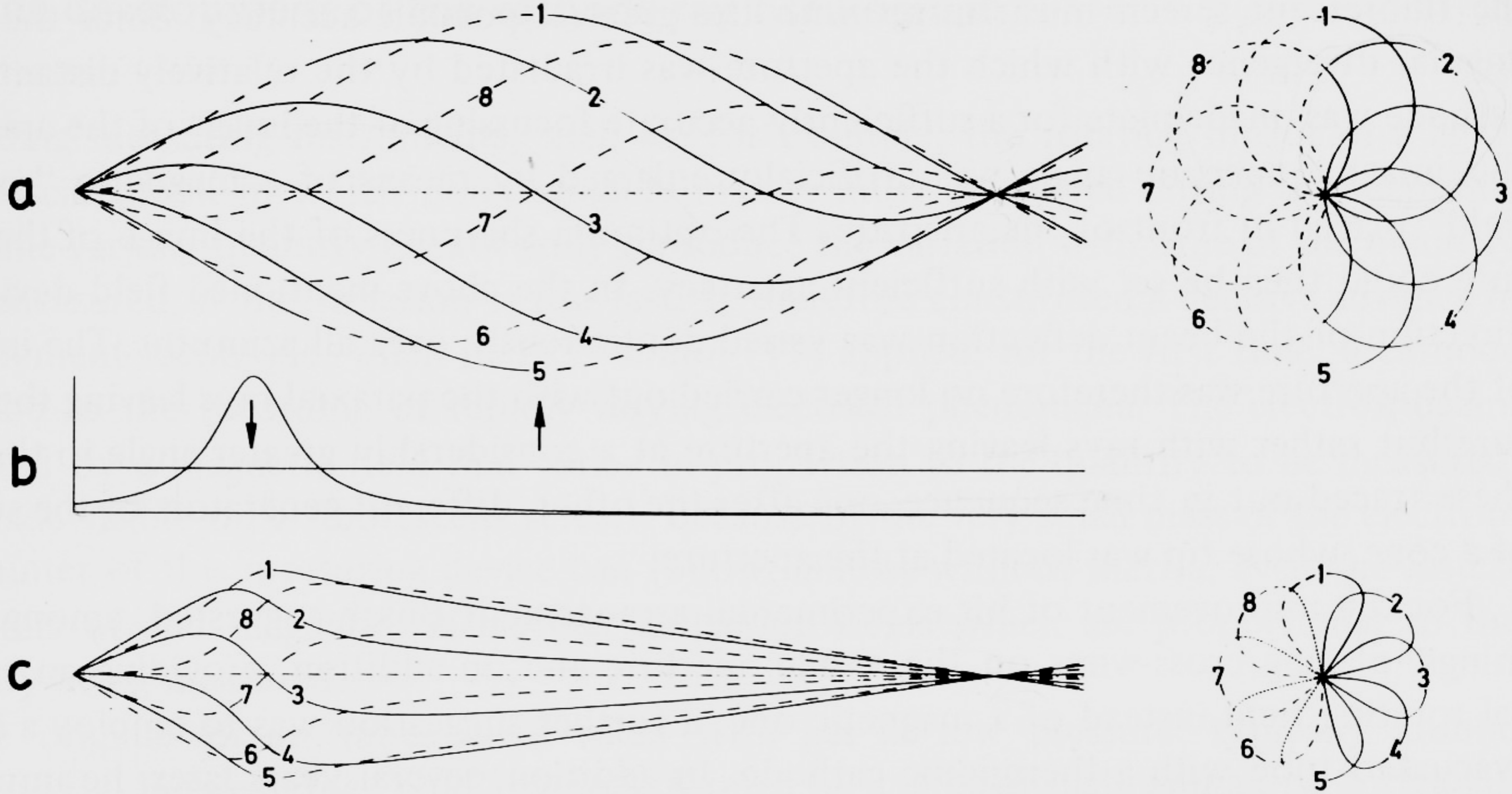
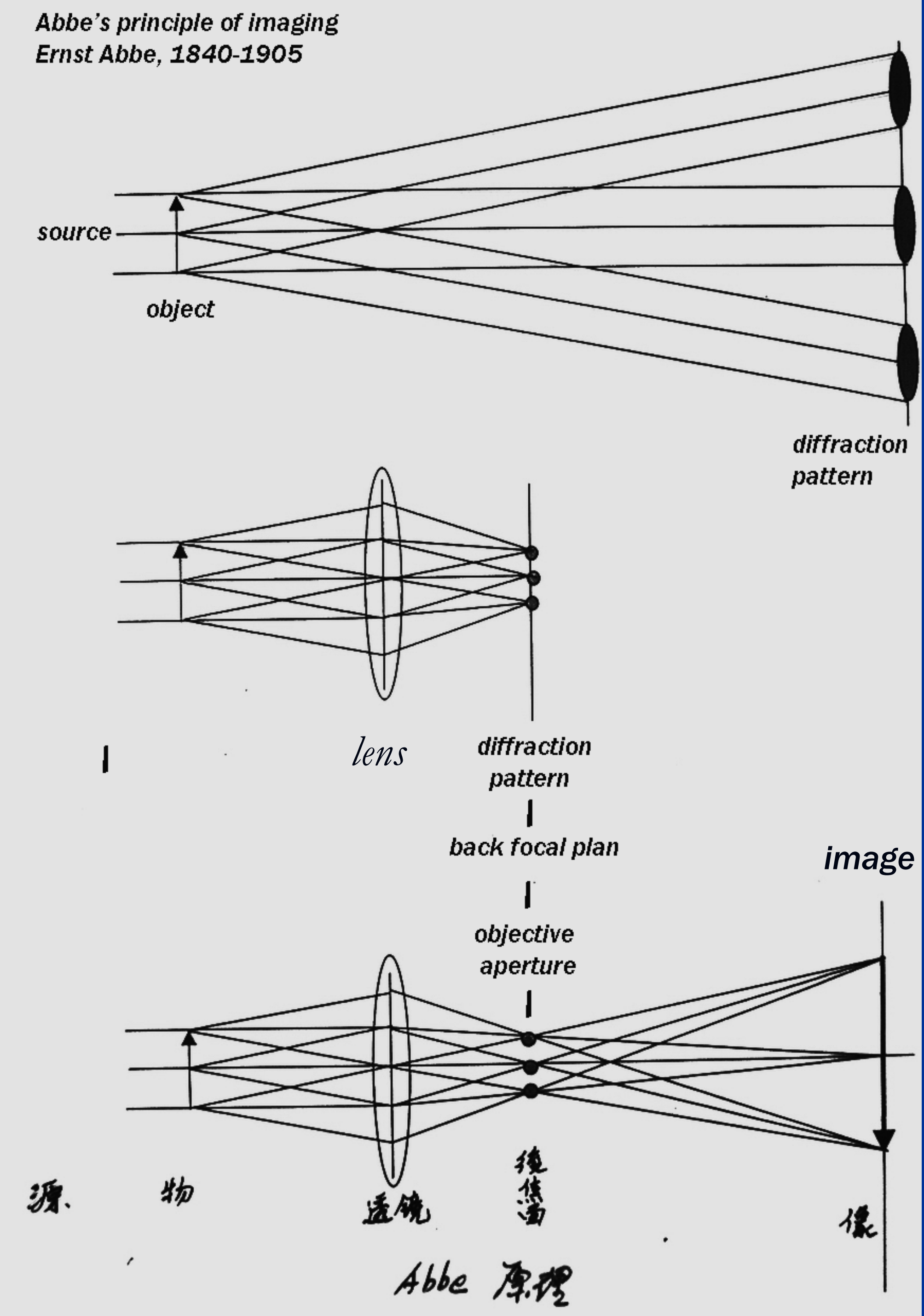


Fig. 1. Electron trajectories in a uniform (a) and in a non-uniform (c) magnetic field, issuing from an axial point of the specimen for different azimuth angles, but making the same angle with the lens axis. (b) Field distributions corresponding to (a) and (c).

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

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

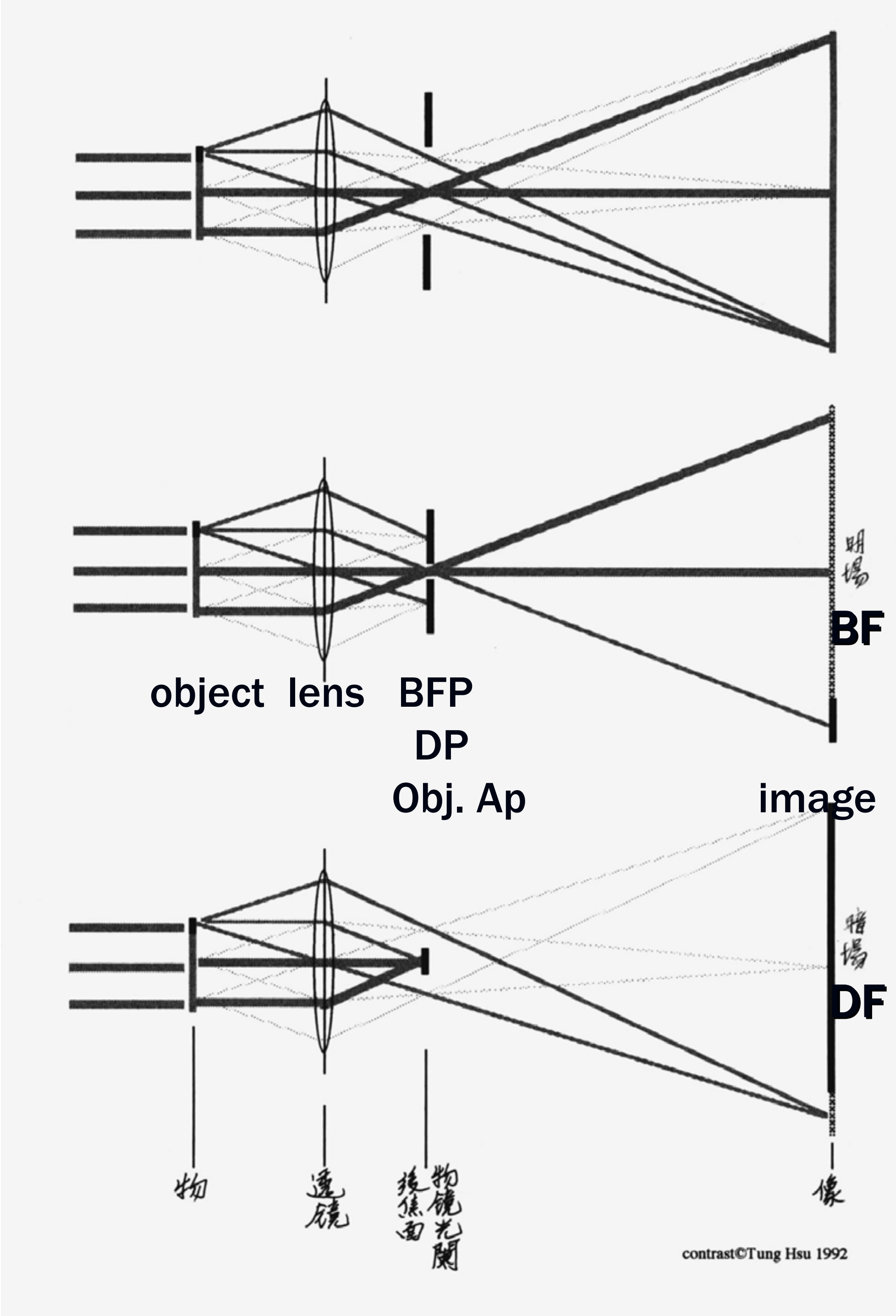
Fundamental geometrical and physical optics

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

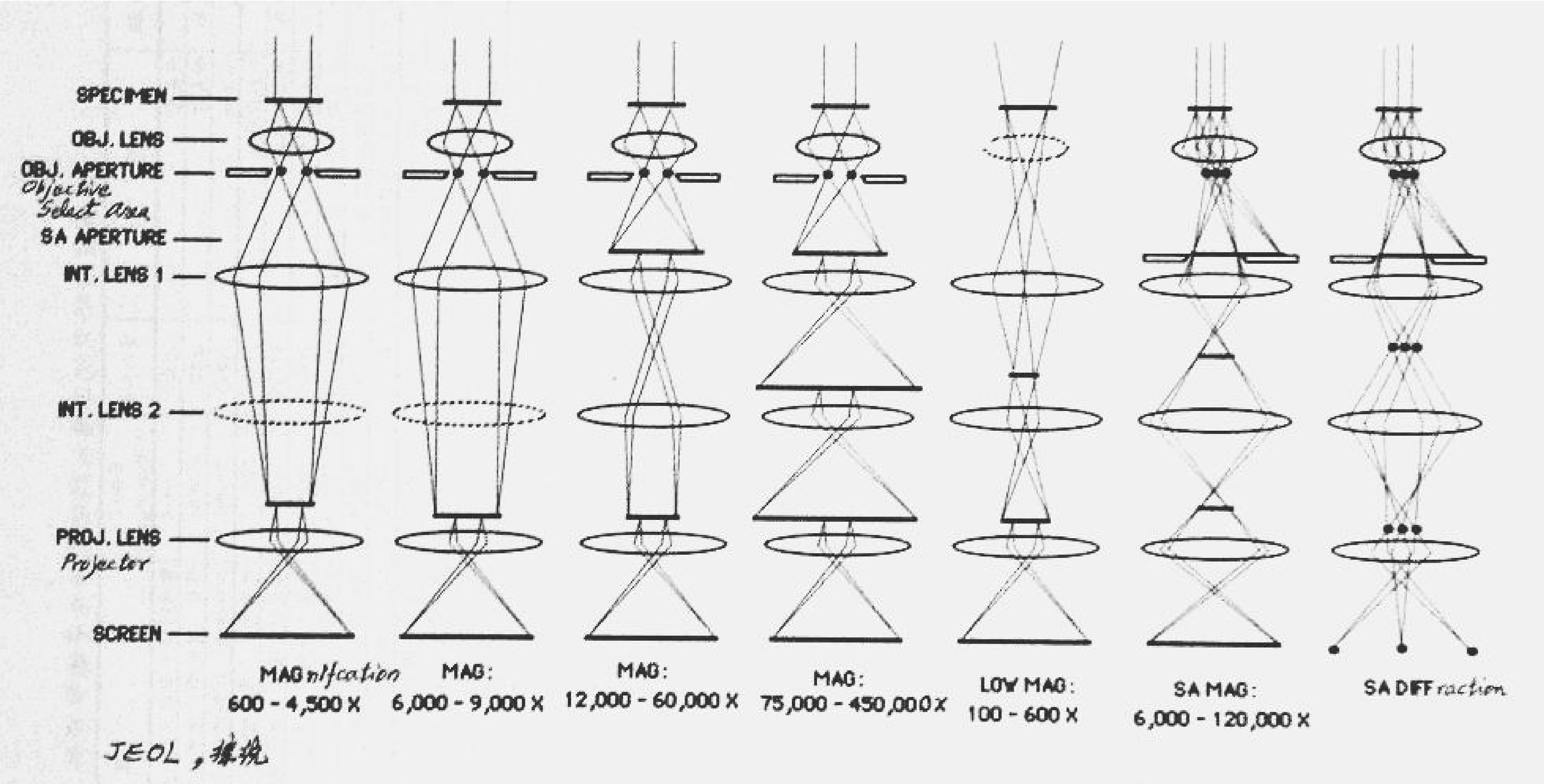
Contrast: Beam/solid interaction

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.



The Electron microscope operation

diffraction pattern ↑

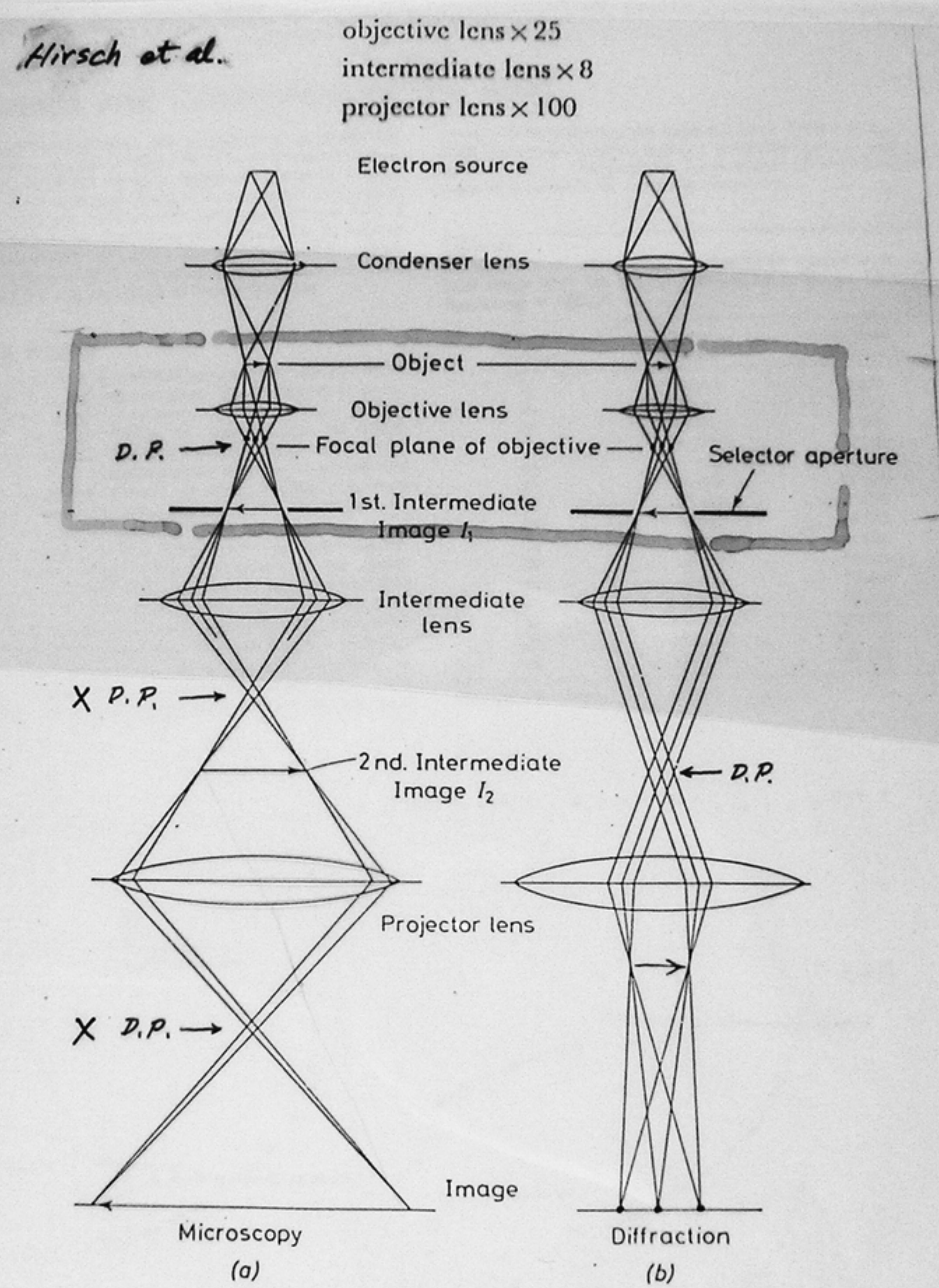


Figure 1.1. *Ray paths in the electron microscope (a) under microscopy conditions and (b) diffraction conditions*

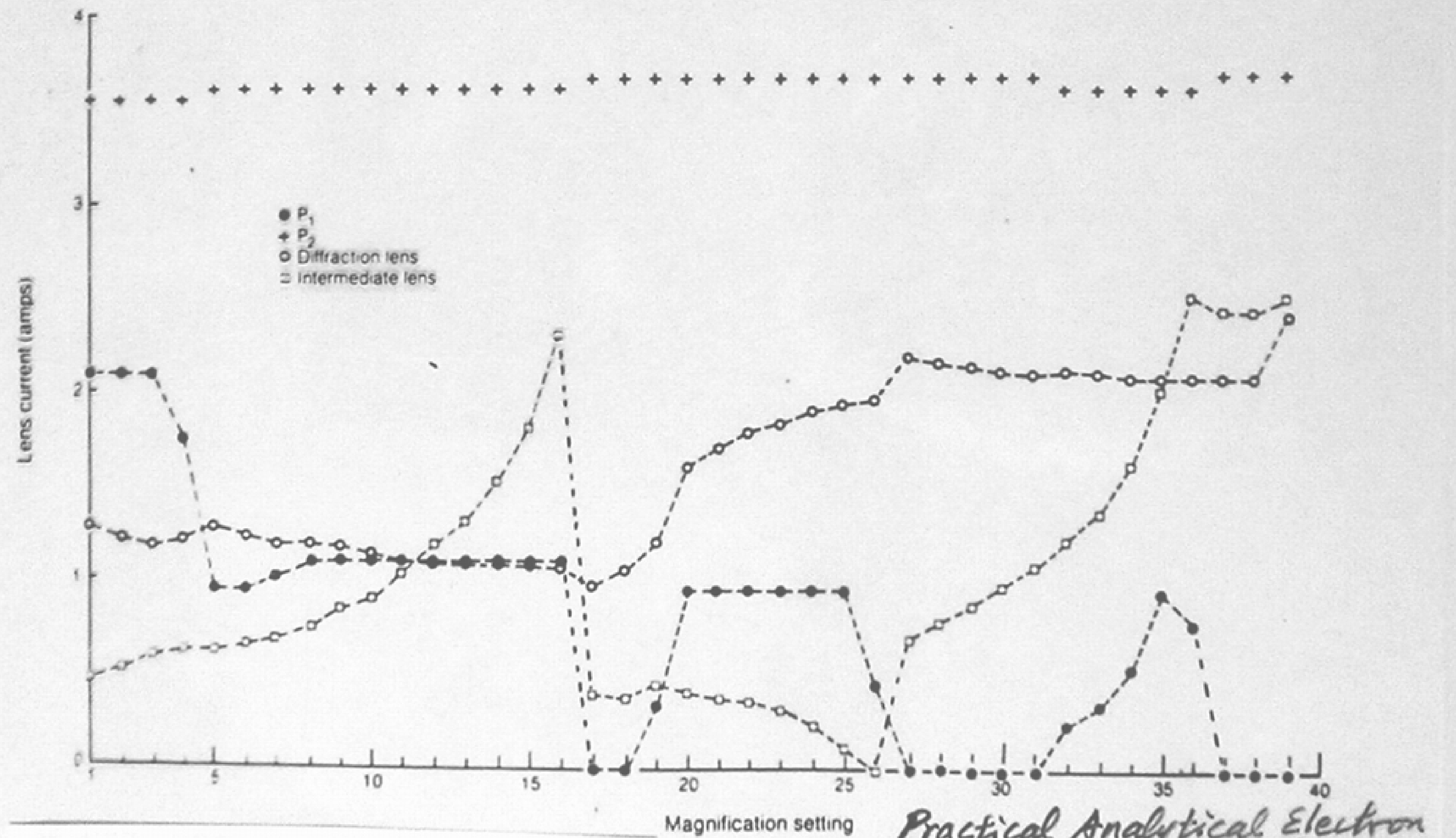


Figure 21. The variation of the current in the imaging lenses as a function of magnification setting in a Philips EM400T operated at 100kV. (Courtesy of J.R. Michael)

Electron micrographs
(EM, TEM images)

And

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

Diffraction Pattern

Diffraction Contrast

What is Diffraction?

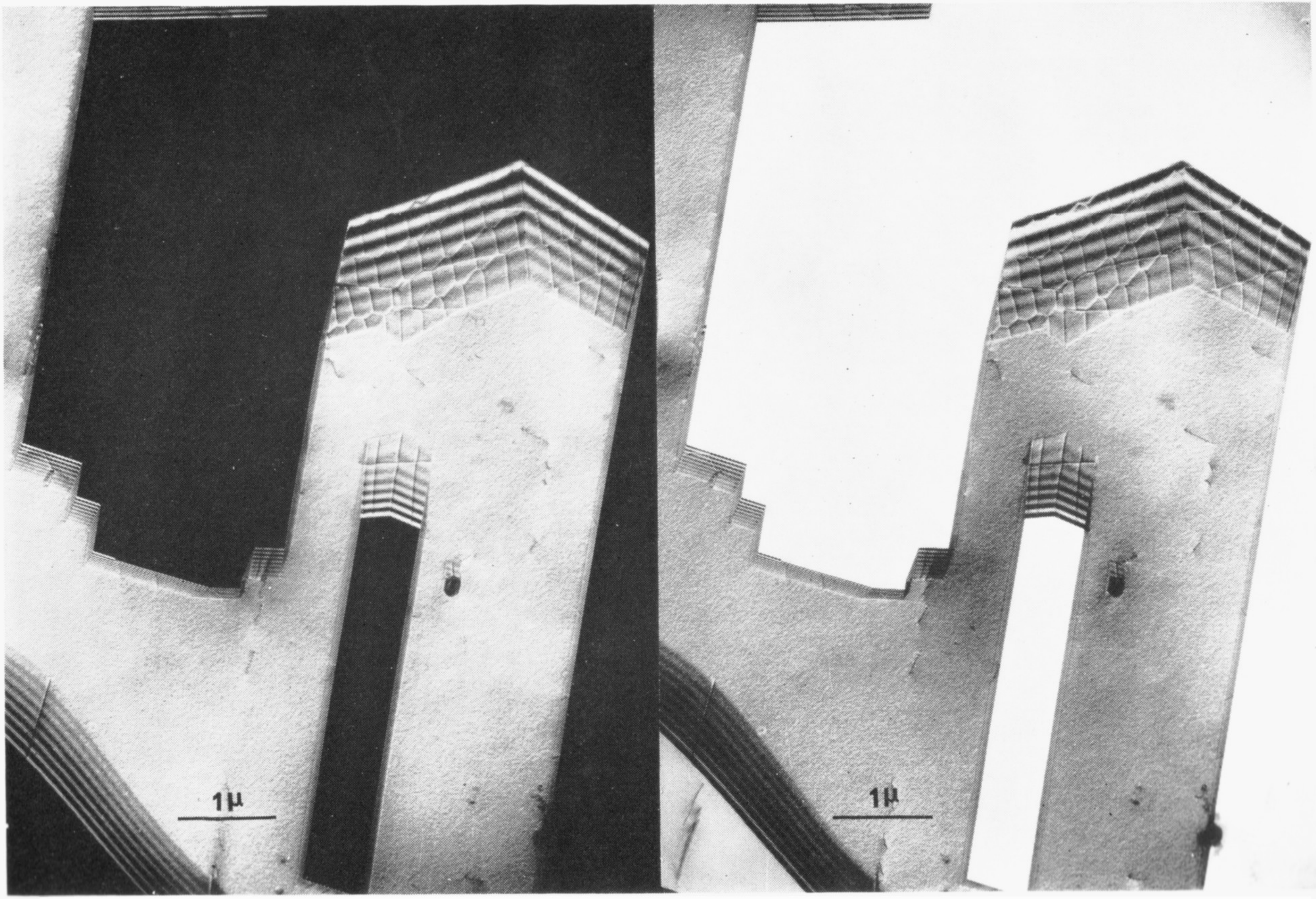


Plate X. Bright and dark-field micrographs of stainless steel, obtained with the Toulouse 1·5 MV microscope. 12 000 × ; Φ = 1 MV.

(Courtesy of Professor G. Dupouy, Laboratoire d'Optique Electronique du C.N.R.S., Toulouse)

Ref: Hawkes, P.W. (1974), Electron Optics and Electron Microscopy, Taylor & Francis, London.

Third of the series of articles by Dvorak and Monahan on the use of electron microscopy in pathology.

Weight Loss, Fatigue, Diarrhea, Fever and Lymphadenopathy in a Forty-Eight Year Old Male Industrial Engineer.

by Ann M. Dvorak and Rita A. Monahan, Electron Microscopy Unit, Department of Pathology, Beth Israel Hospital, Boston, Massachusetts

Clinical Summary

The patient, a 48-year-old male industrial engineer, was ill for 10 months before a definitive diagnosis by electron microscopy allowed specific antibiotic therapy to be given. The illness was characterized by severe weight loss (70 lbs.), diarrhea, night sweats, fevers, lymphadenopathy, and fatigue, all of which necessitated that he discontinue working. At the time of diagnosis he could barely walk or sit and exertional dyspnea had become severe. The medical workup at two institutions was extensive. Diagnoses entertained were in excess of twenty and he was treated unsuccessfully for one of these — sarcoidosis. Diagnoses and problems that were considered included the following: lymphoma, sarcoidosis, industrial exposures to plastics, polyvinylchloride and asbestos; duodenal ulcer, interstitial lung disease, sprue, pancreatic insufficiency, Crohn's

(Figures 7–9). Intercommunication of individual lipid bodies and the appearance of irregular losses of lipid from lipid bodies was also characteristic of these lipid-laden, lysosome-poor macrophages.

Other host inflammatory cells were either closely involved with, or surrounded by viable bacteria. Plasma cells (Figure 10) were surrounded by organisms. Mast cells showed focal losses of granule content and surface attachment of unaltered bacteria (Figure 11). Neutrophils (Figure 12) characteristically revealed phagocytosis and alteration of bacteria, loss of their cytoplasmic granules, and a marked increase in the number of lipid bodies — all changes we have regularly observed in reactive, activated neutrophils in tissues involved with inflammatory processes.

Electron Microscopic Diagnosis: Whipple's Disease

Comment

Whipple first described the disease which bears his name in 1907 in a 36-year-old male physician who presented with symp-

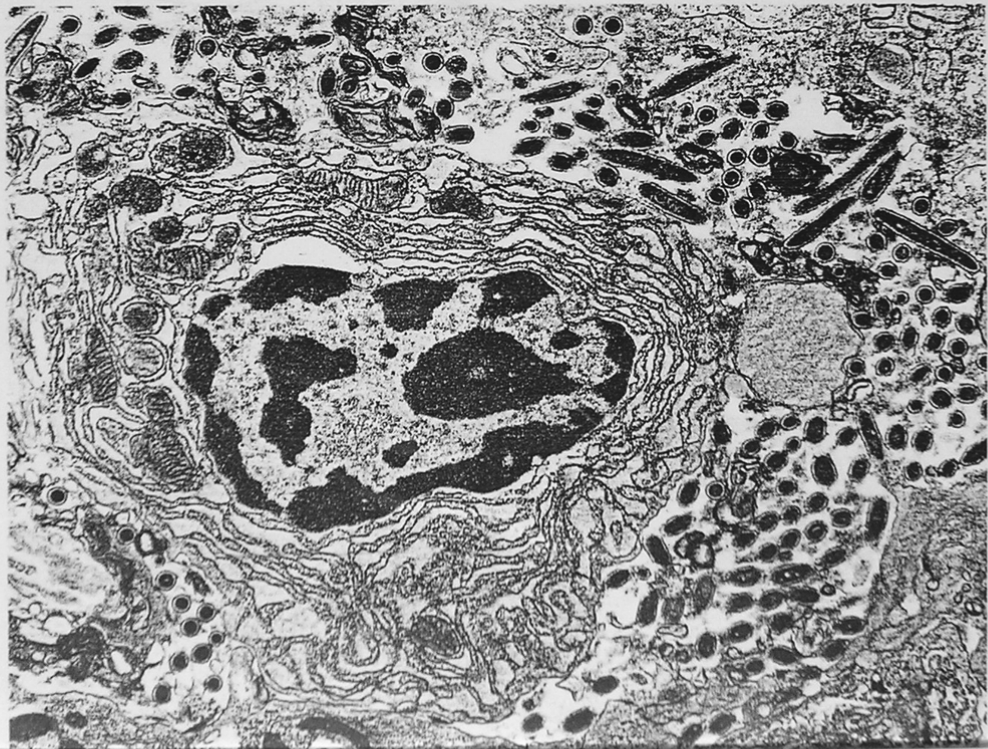


Figure 10. A plasma cell in the lamina propria is surrounded by unaltered bacilli. The plasma cell shows a typical nuclear pattern of dense block-shaped chromatin aggregates, and extensive cytoplasmic cisternae of rough endoplasmic reticulum. This cell does not internalize bacteria. 16,000×.

NORELCO REPORTER
31: 1EM (Apr. 1984) 10

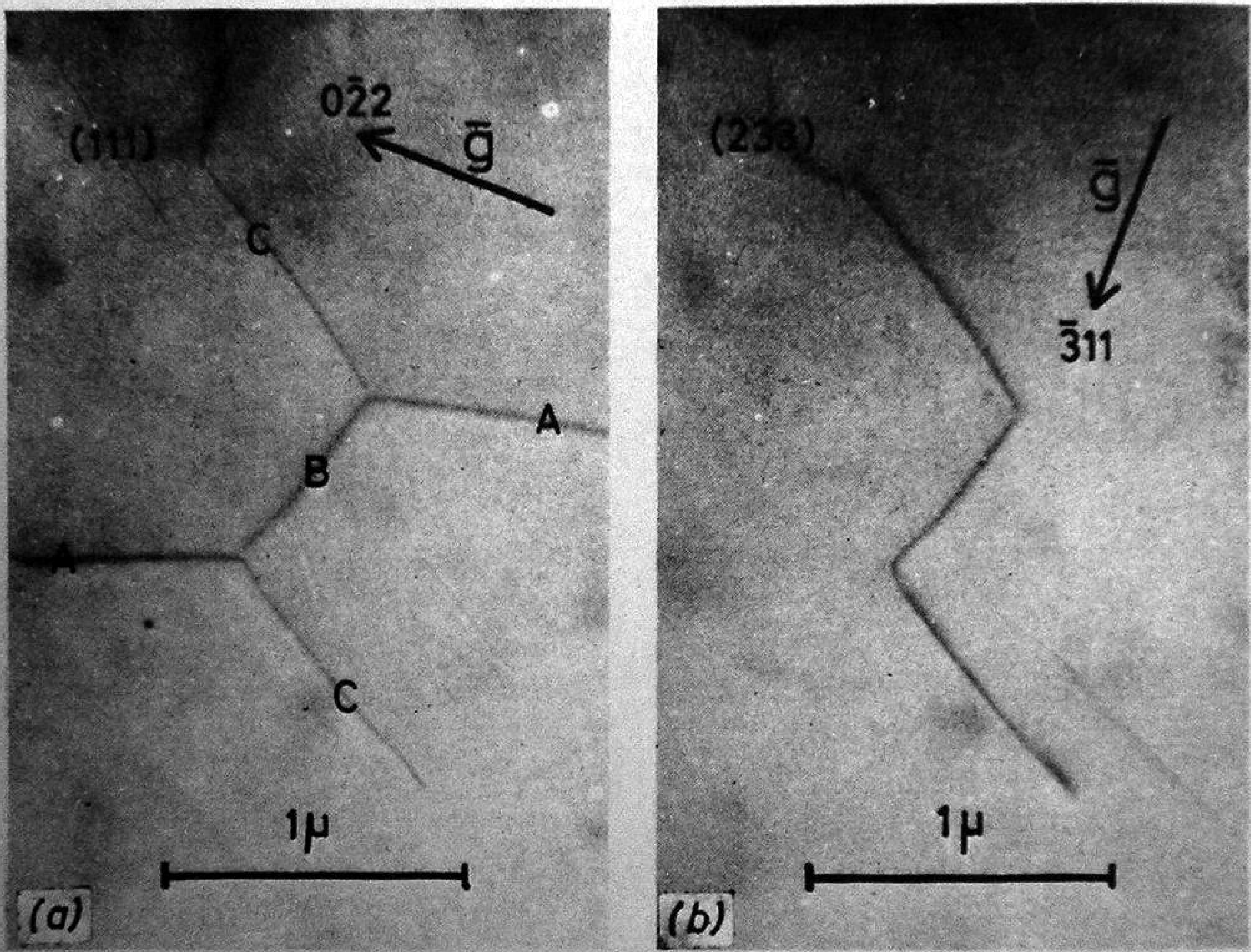


Figure 7.14. Network of dislocations with nearly pure screw character, in Si, *lying approximately in a* (111) *plane. In* (*a*), *taken in* 0$\bar{2}$2, *the dislocations A, B, C are all visible; in* (*b*), *taken in* $\bar{3}$11, *the image of dislocations A vanishes, which is consistent with a screw dislocation with Burgers vector* $\frac{1}{2}[0\bar{1}1]$. *Using other reflections dislocations B and C were also found to be nearly screws with Burgers vectors* $\frac{1}{2}[\bar{1}01]$ *and* $\frac{1}{2}[\bar{1}10]$

(From Booker, 1965,
by courtesy of The Faraday Society)

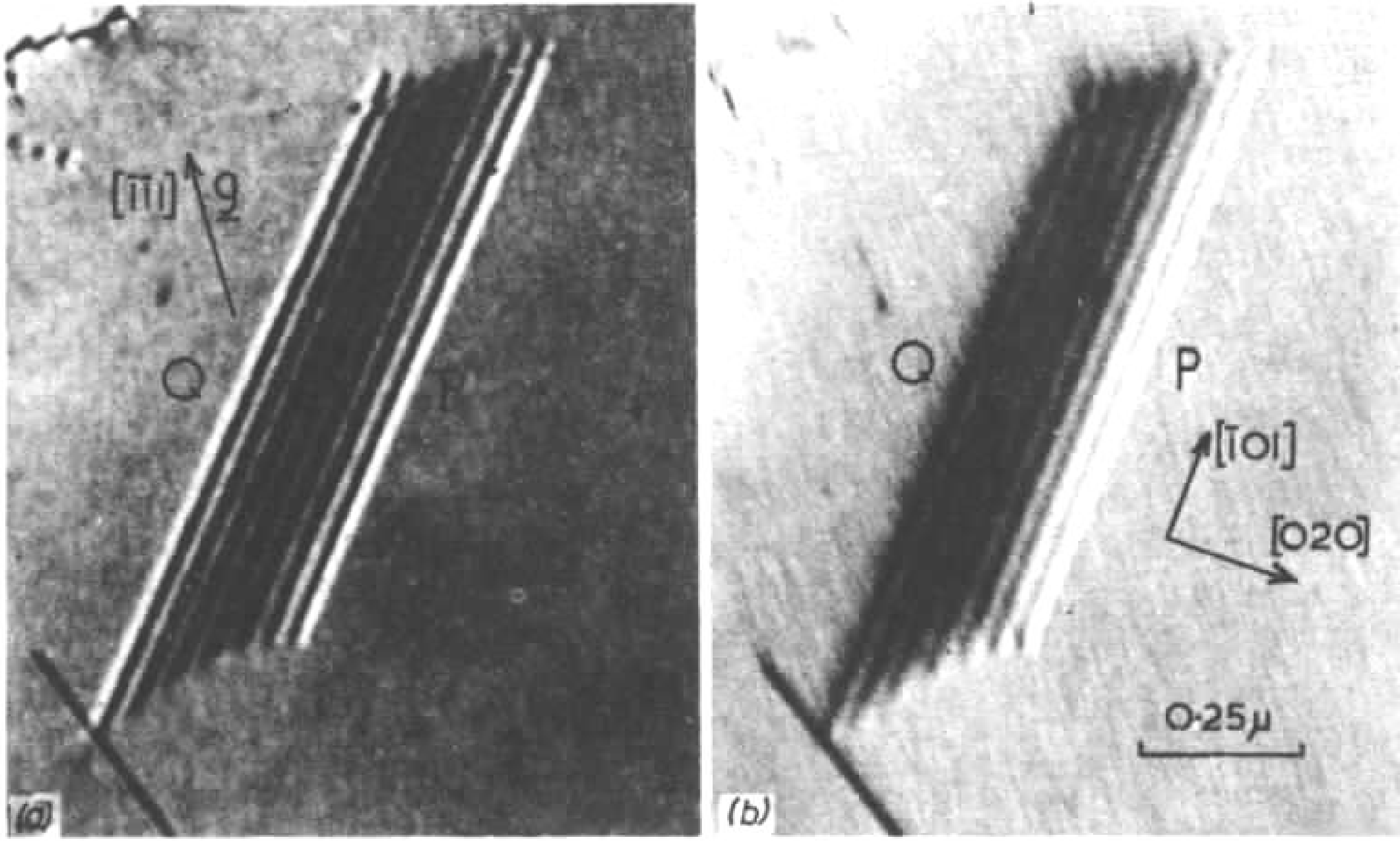
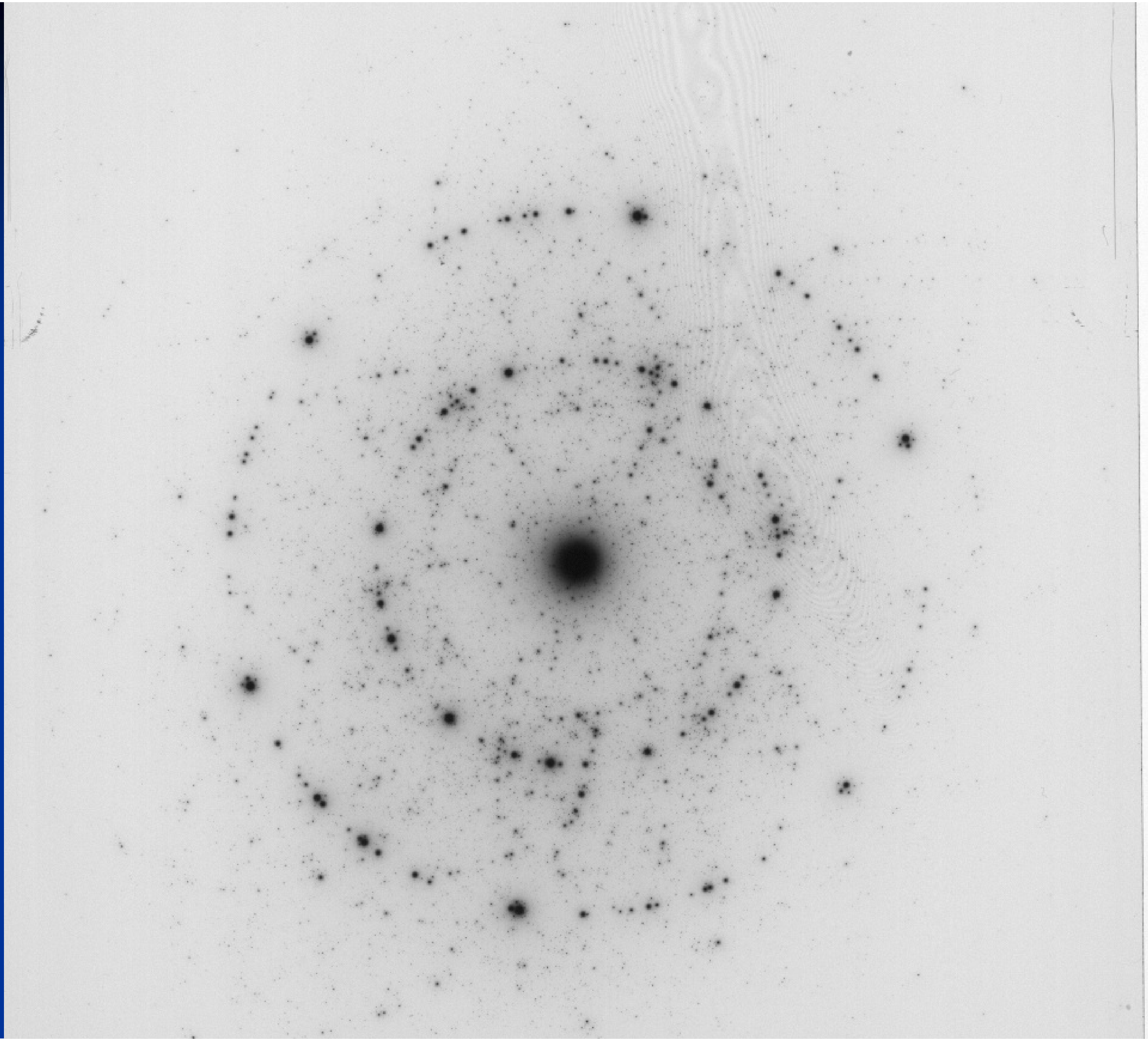


Figure 10.10. Bright-field (*a*) *and dark-field* (*b*) *images of a stacking fault with* $\alpha = +2\pi/3$ *in* Cu + 7% Al *alloy,* [101] *orientation. The reflection* $\mathbf{g} = [\bar{1}\bar{1}1]$ *is shown. Compare with Figure 10.8*

(From Hashimoto, Howie and Whelan, 1962, by courtesy of The Royal Society)



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.

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

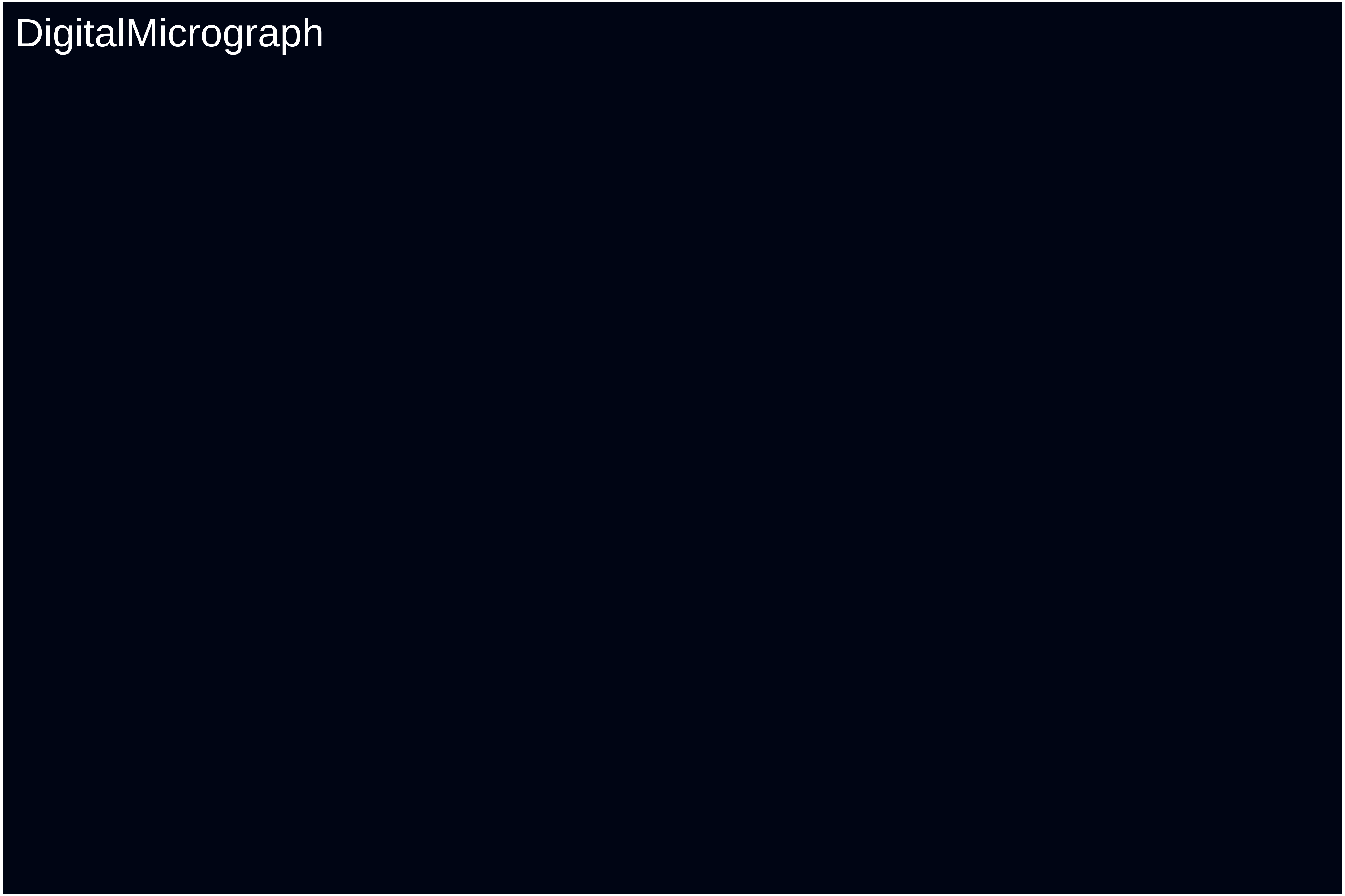
Amplitude-phase diagram (vector method)

Fourier transform

- Optical bench (Atlas)
- Computer

Diffraction Patterns from 3D objects

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


DigitalMicrograph

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

Contrast mechanism:

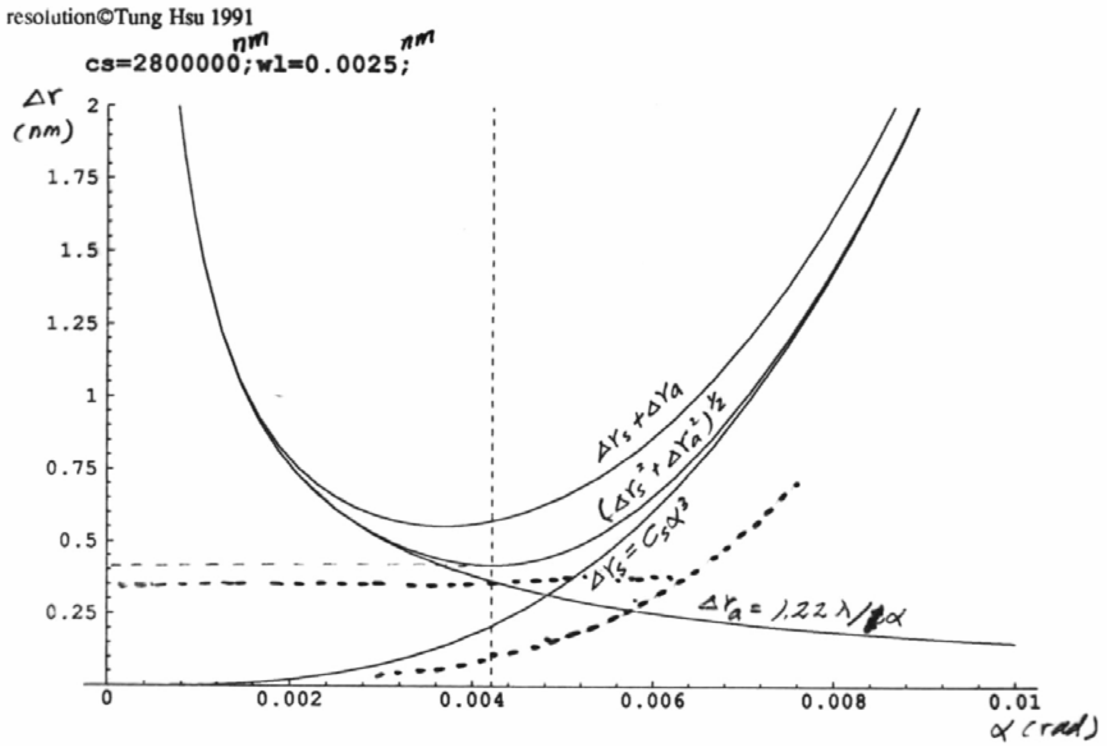
Beam/specimen interaction

- 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



$$\alpha \approx C_s^{-1/4}\lambda^{1/4}$$

$$\Delta r \approx C_s^{1/4}\lambda^{3/4}$$

$$\lambda = \frac{h}{\{2mE(1+\frac{E}{2mc^2})\}^{1/2}}$$

E = 100 keV	λ = 0.0037 nm
200 keV	0.0025 nm
15 keV	0.0099 nm

光: $\Delta r \approx 1.22\frac{\lambda}{\alpha}$

Fig. 22

$$\lambda = 1.226[E(1+0.9788\times10^{-6}E)]^{-\frac{1}{2}}\ \text{nm}$$

↑—— volt ——↑

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,

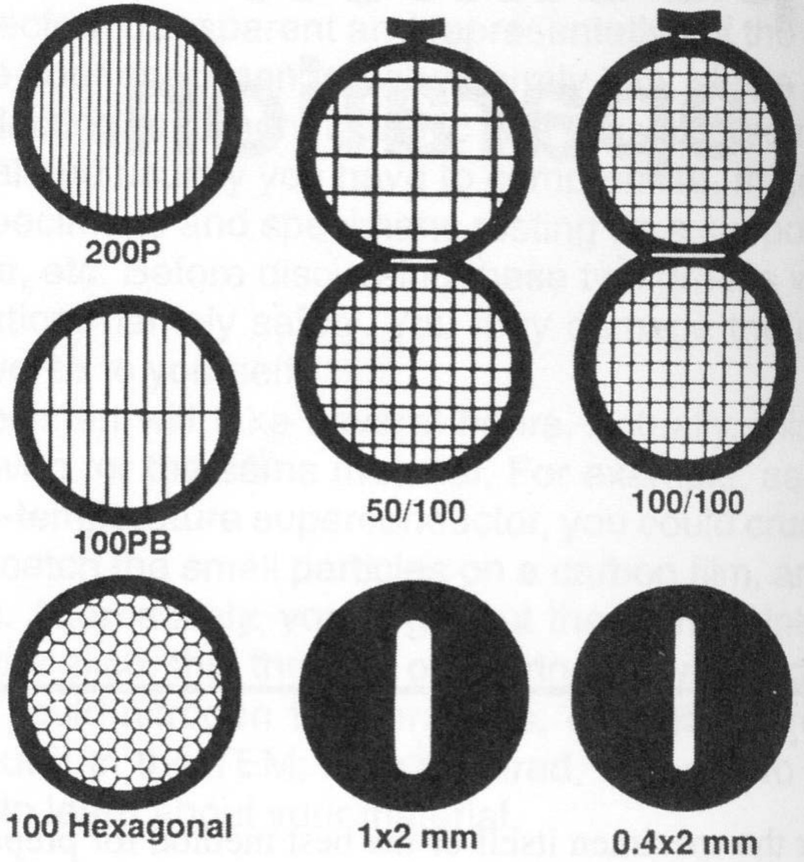
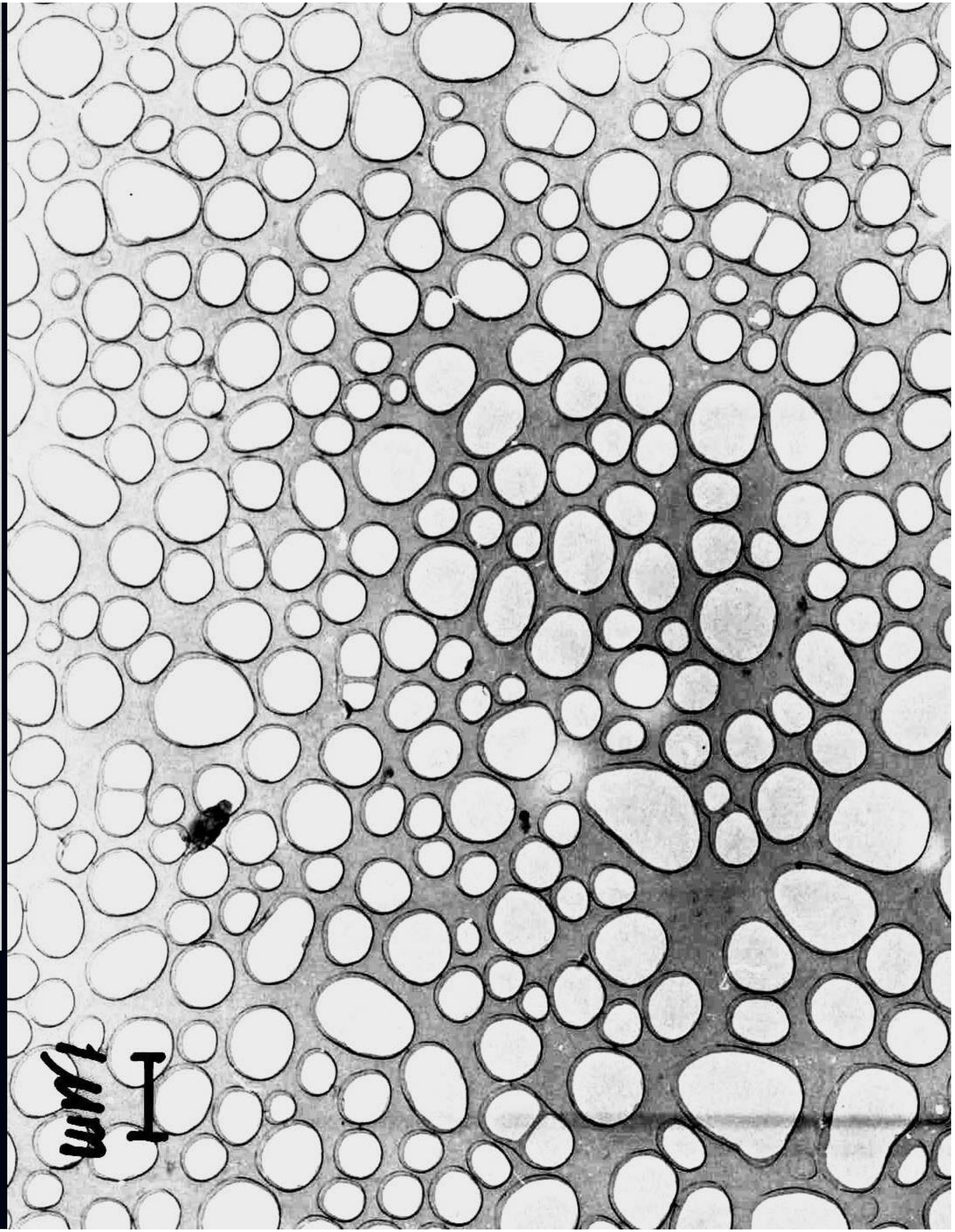
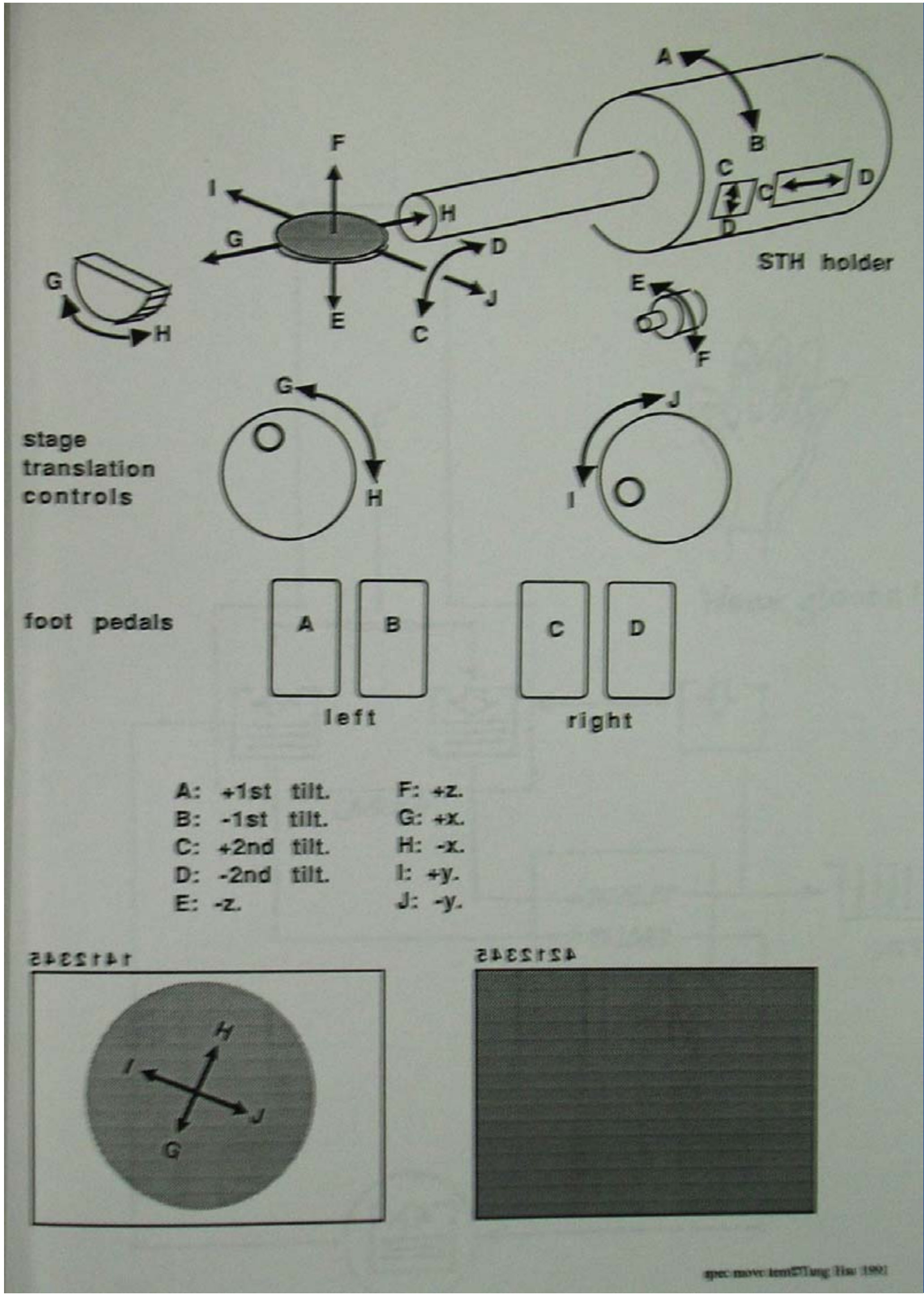


Figure 10.2. A variety of specimen support grids of different mesh size and shape. At the top right is the oyster grid, useful for sandwiching small slivers of thin material.





Movements and controls of the specimen

High Resolution Electron Microscope (HREM):

Approaching atomic resolution.

Requirements:

(Ultra) high resolution pole piece

Electronic stability

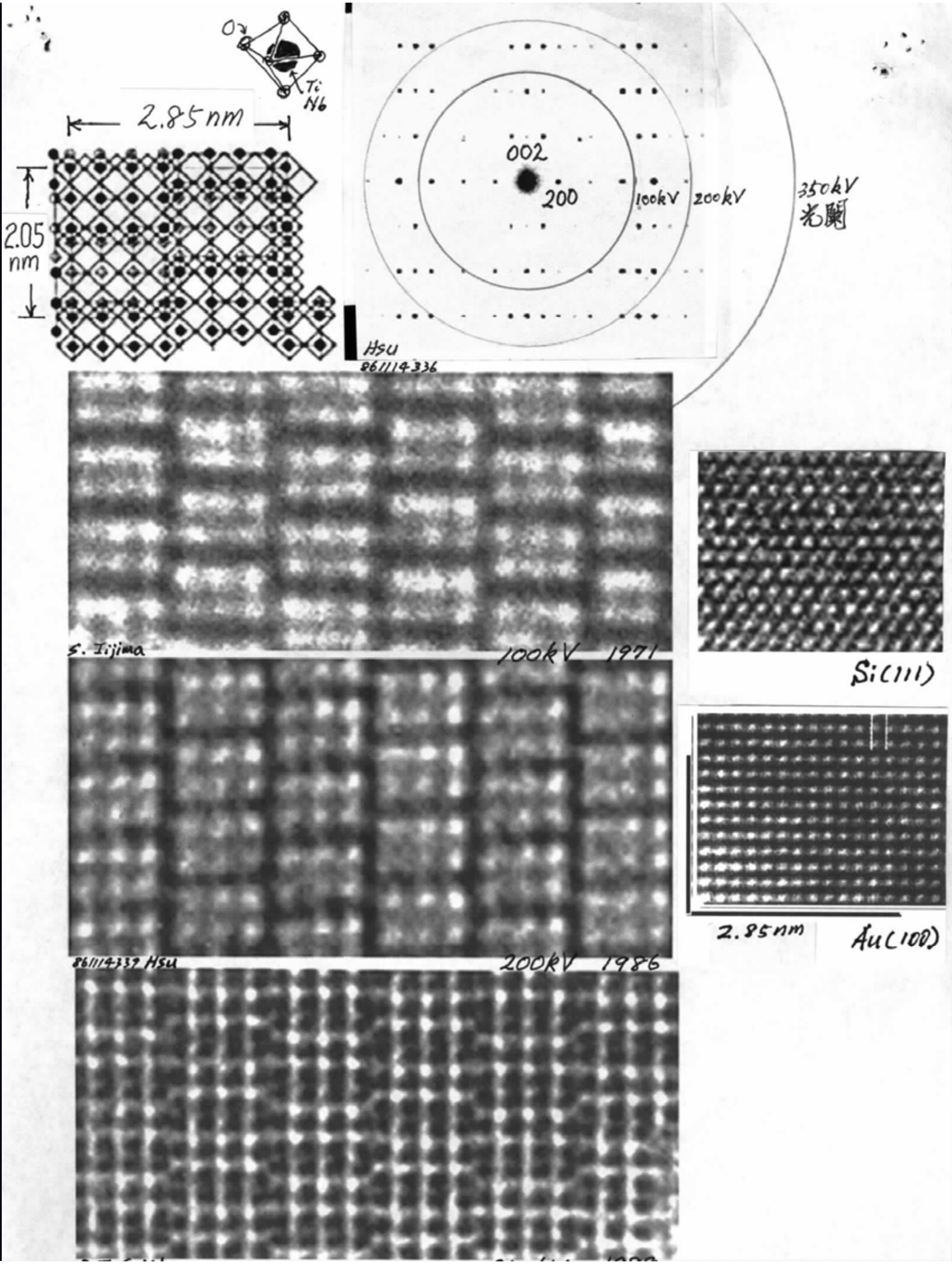
Mechanical stability

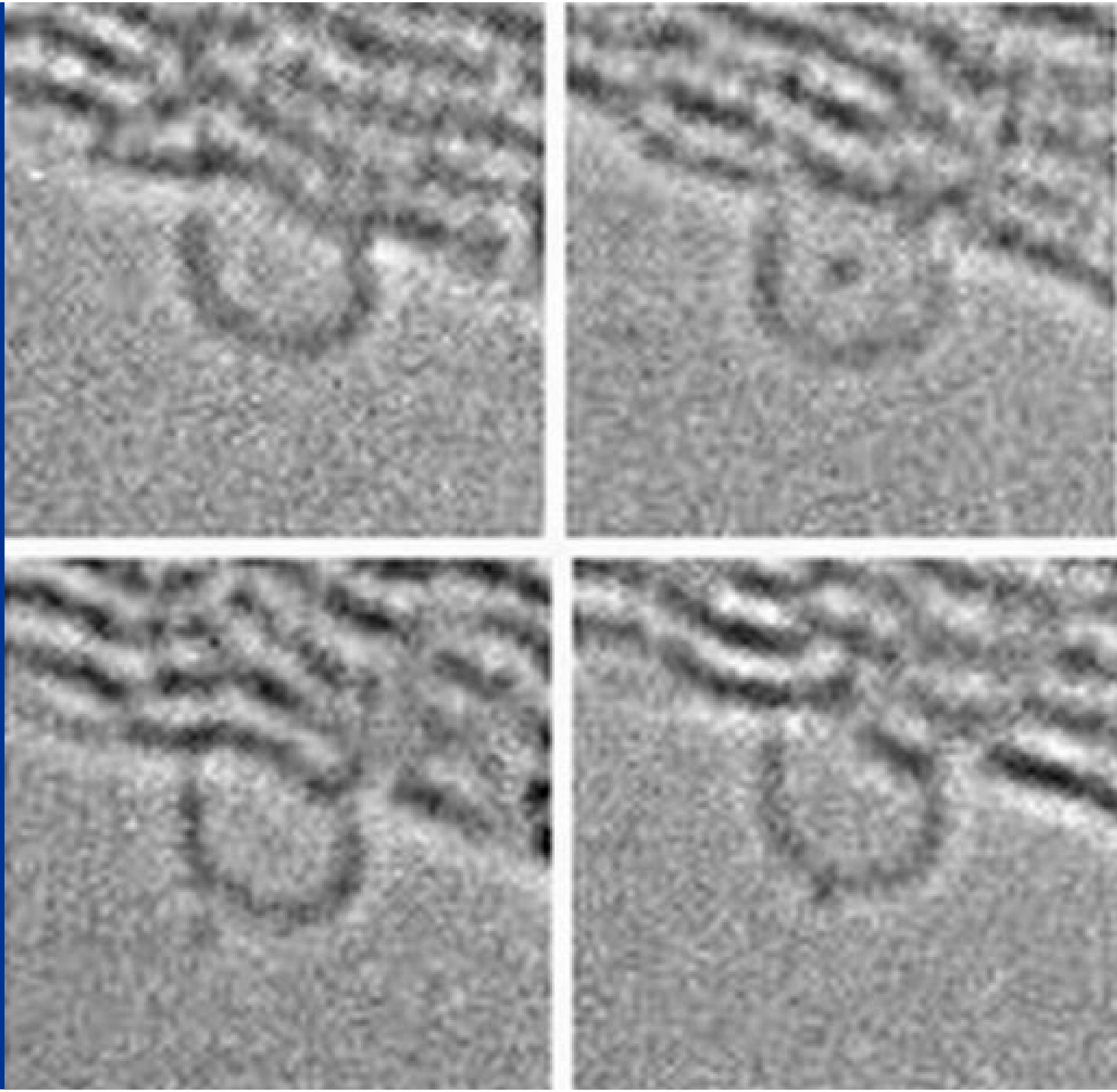
Clean environment: (Ultra) high vacuum

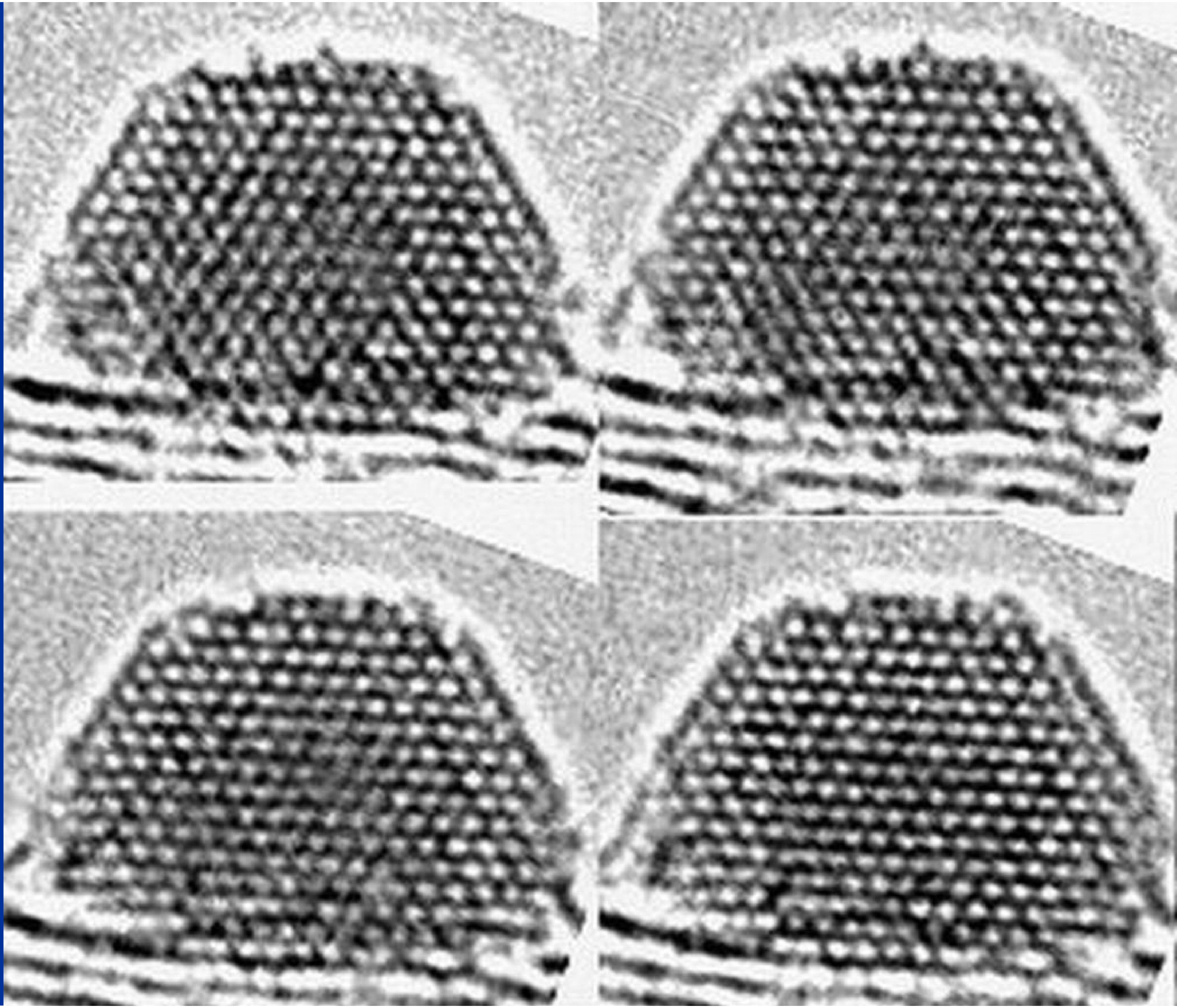
Specimen preparation: very very thin

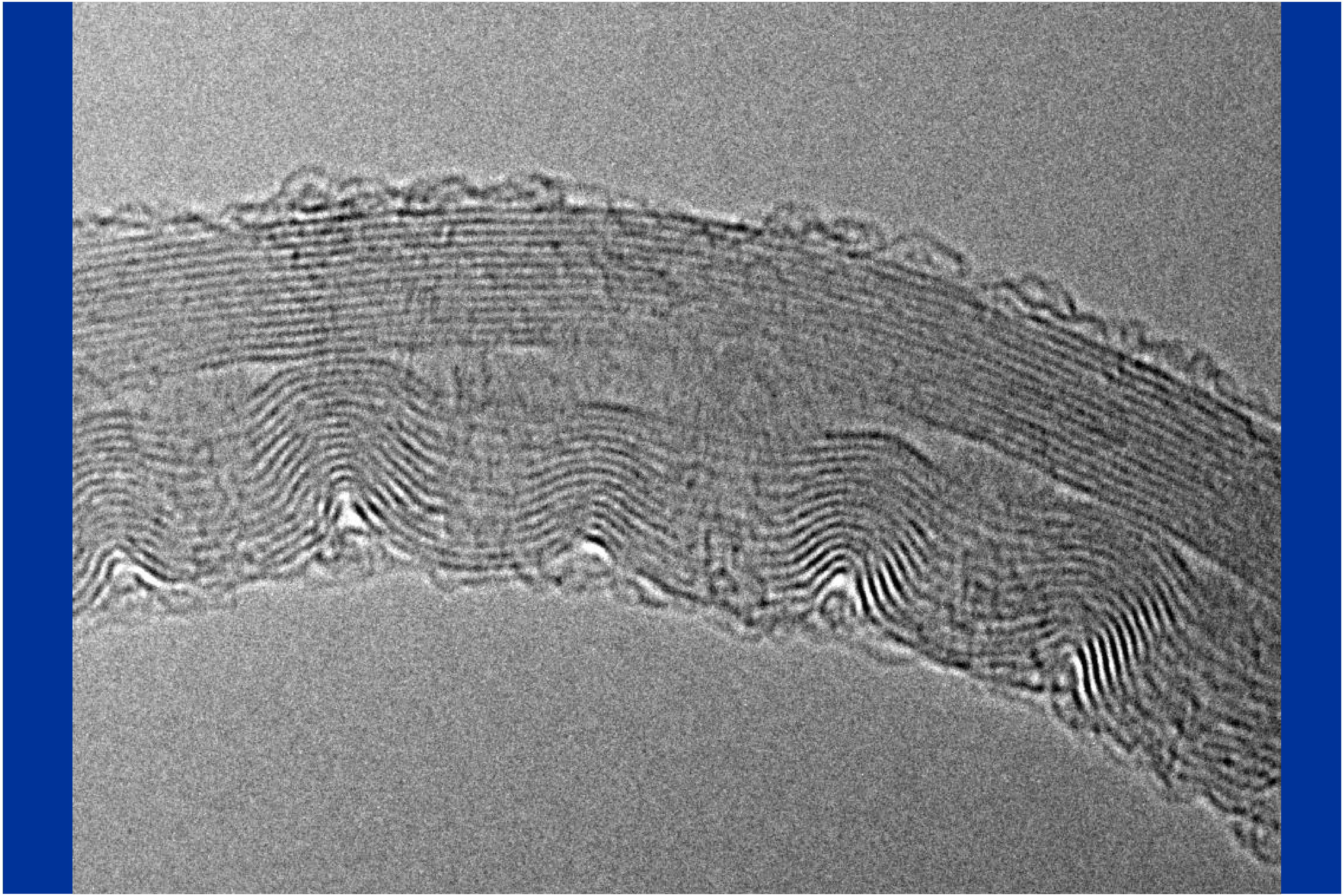
In general HREM is needed for studying nano-materials.

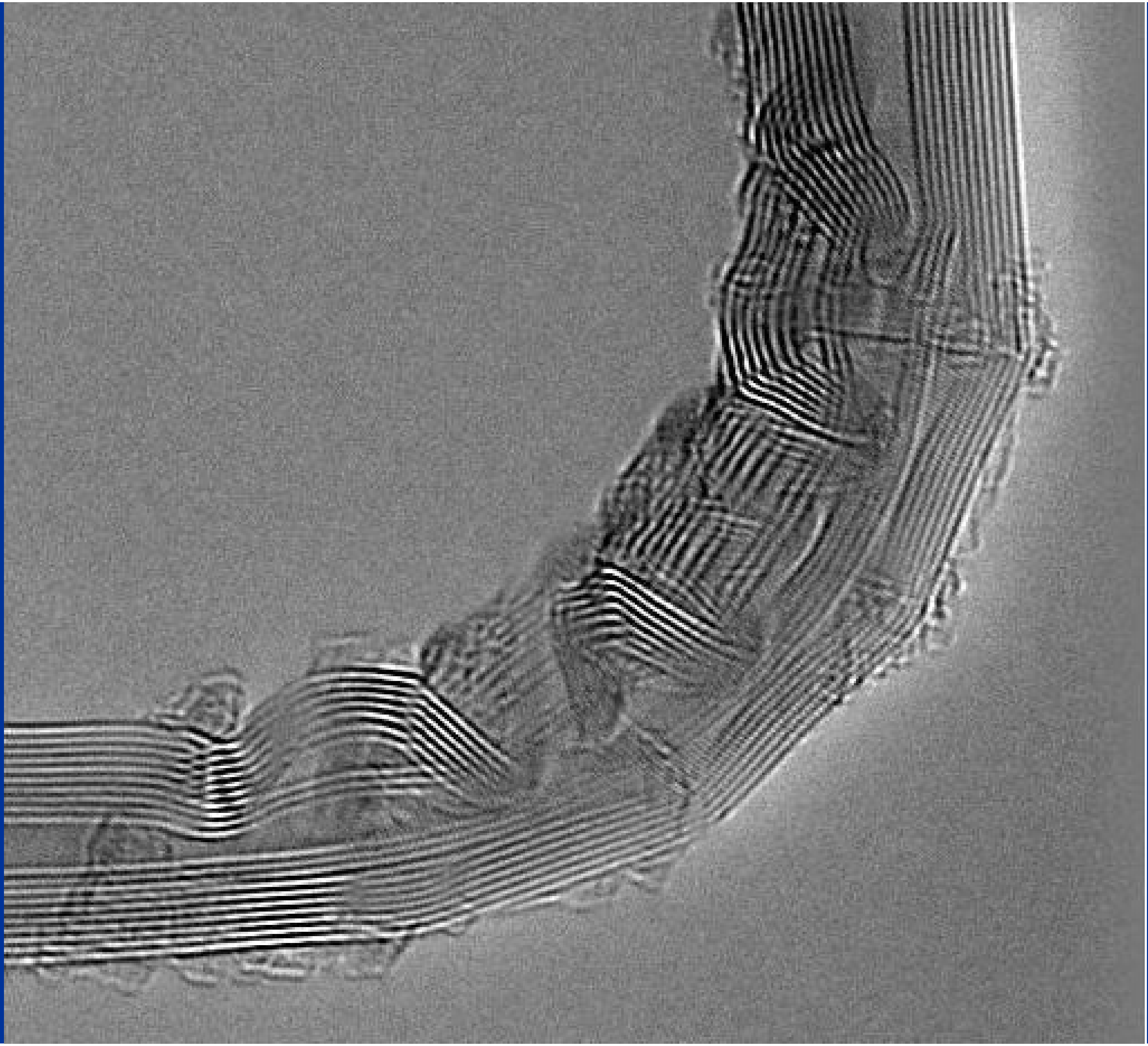
HREM examples

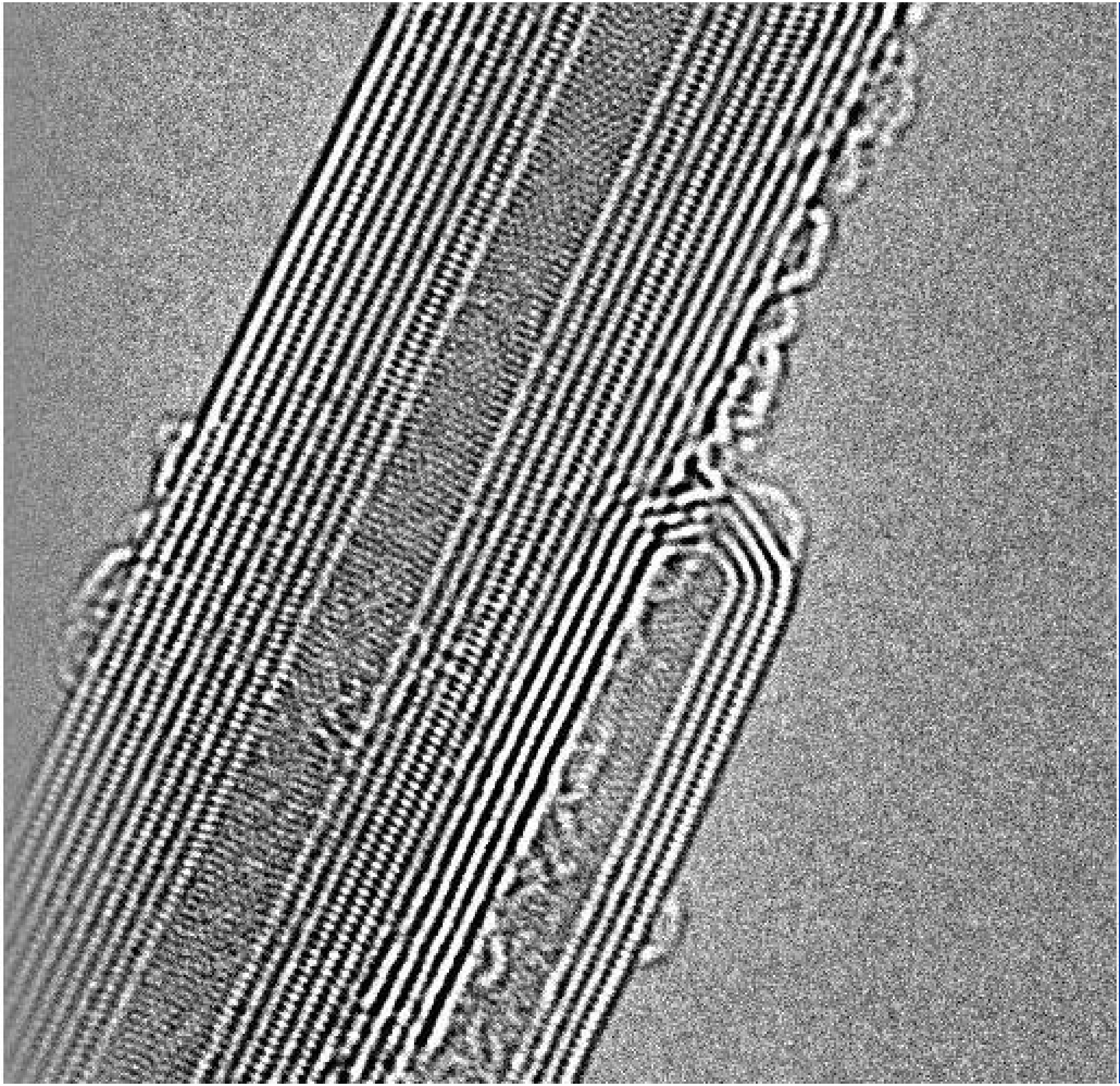


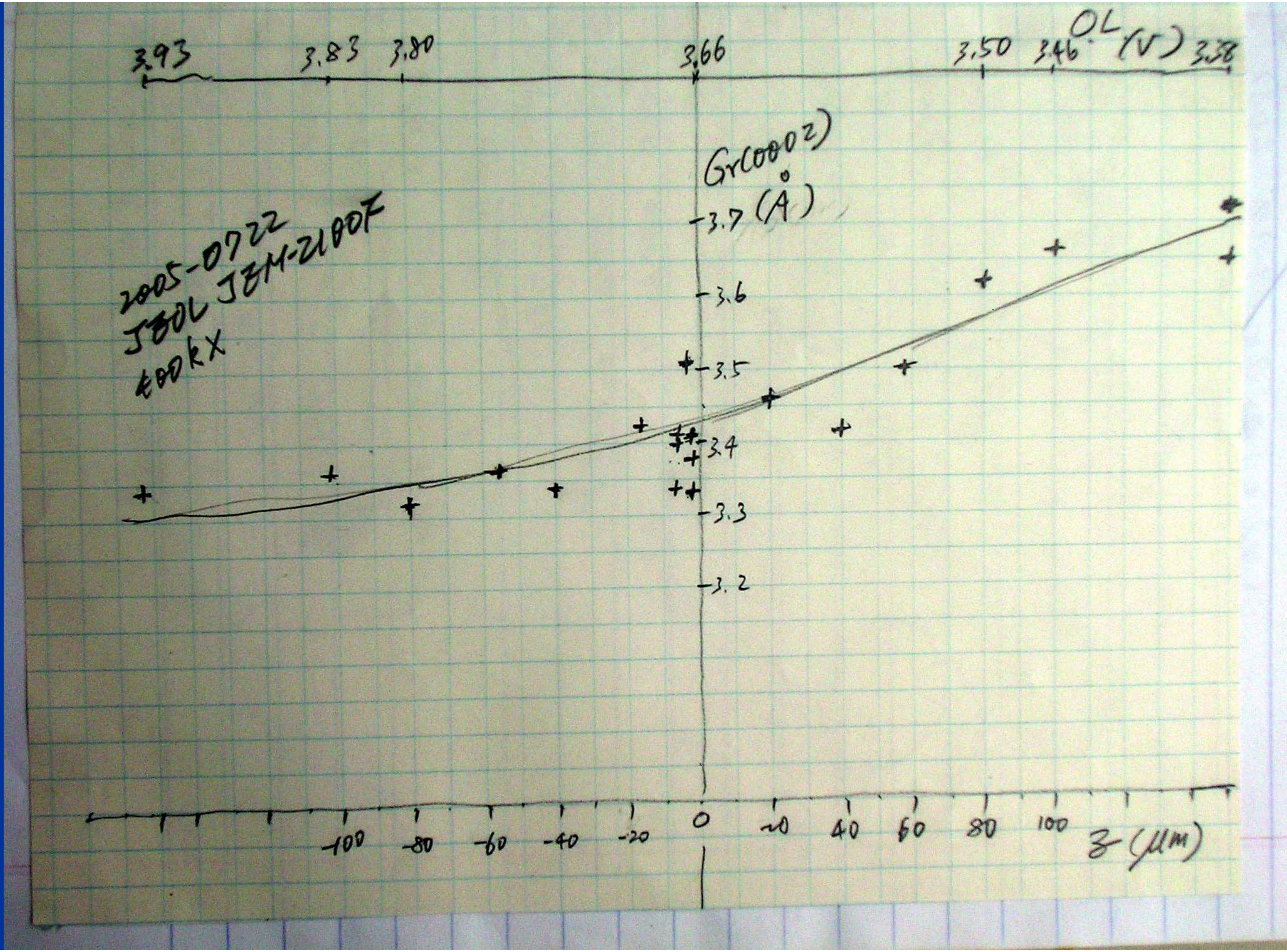












3.93
3.83
3.80
3.66
3.50
3.46
OL (V)
3.38
Gr(0002)
(Å)
3.7
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3.4
3.3
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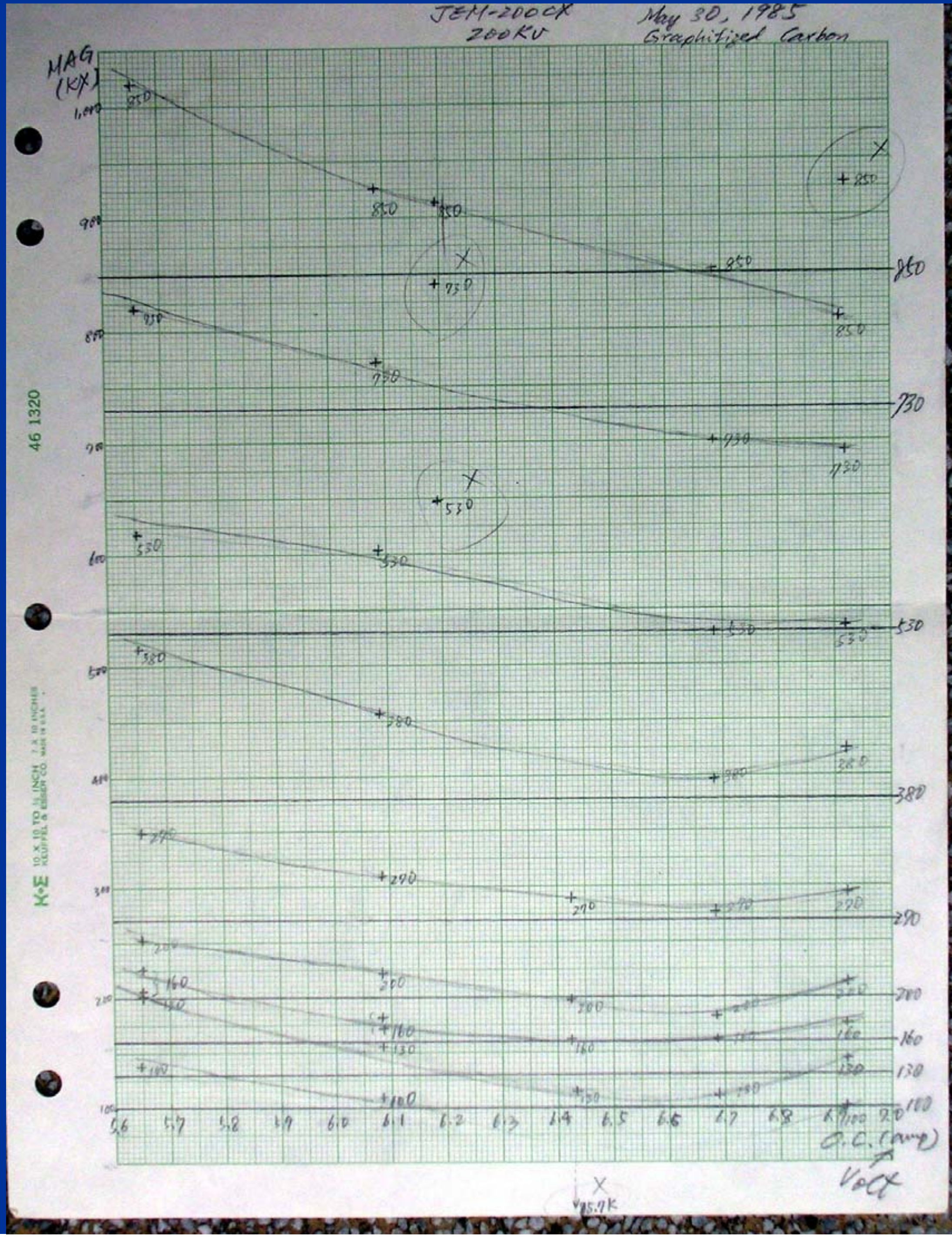
2005-0722
JEOL JEM-2100F
400kX
-100
-80
-60
-40
-20
0
20
40
60
80
100
z (μm)


JEM-200CX
200KV
May 30, 1985
Graphitized Carbon
MAG (KX)
850
730
530
380
270
200
160
130
100
5.6
5.7
5.8
5.9
6.0
6.1
6.2
6.3
6.4
6.5
6.6
6.7
6.8
6.9
7.0
O.C. (amp)
Volt
46 1320
K+E