ELECTRON MICROSCOPY

14:10 - 17:00, Mar. 8, 2018

14:10 - 17:00, Mar. 15, 2018

P101, Institute of Physics, Academia Sinica

Tung Hsu

Mail: Department of Materials Science and Engineering
National Tsing Hua University
Hsinchu 300, TAIWAN

Tel: 0983230927

E-mail: tunghsu@hotmail.com

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, 1976, 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

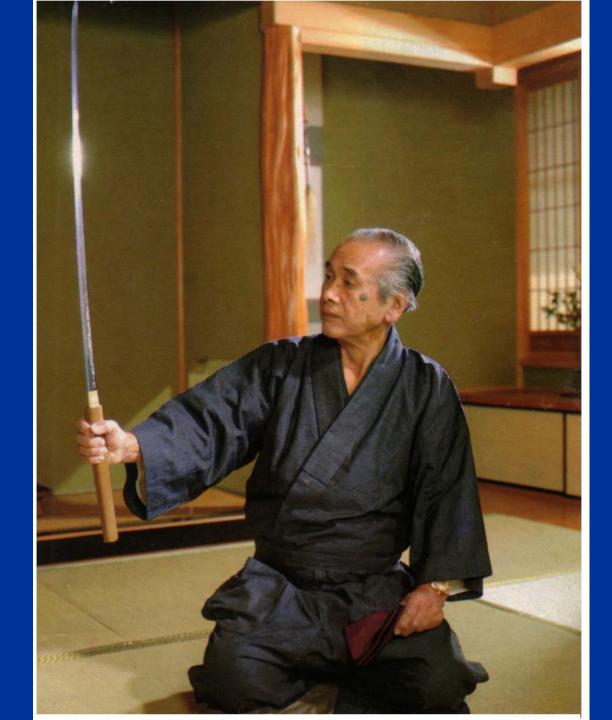
(Spatial) Resolution:

Microscopy vs. microprobe

Wavelength, properties of lens

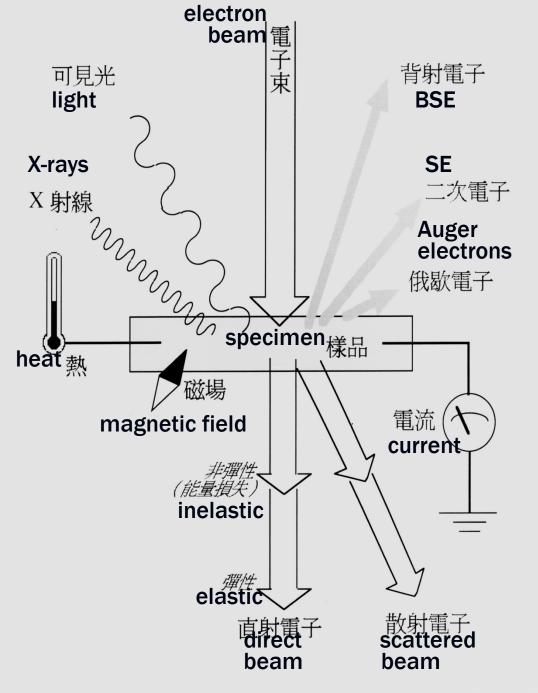
Information other than the image

A brief history of electron microscopy



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

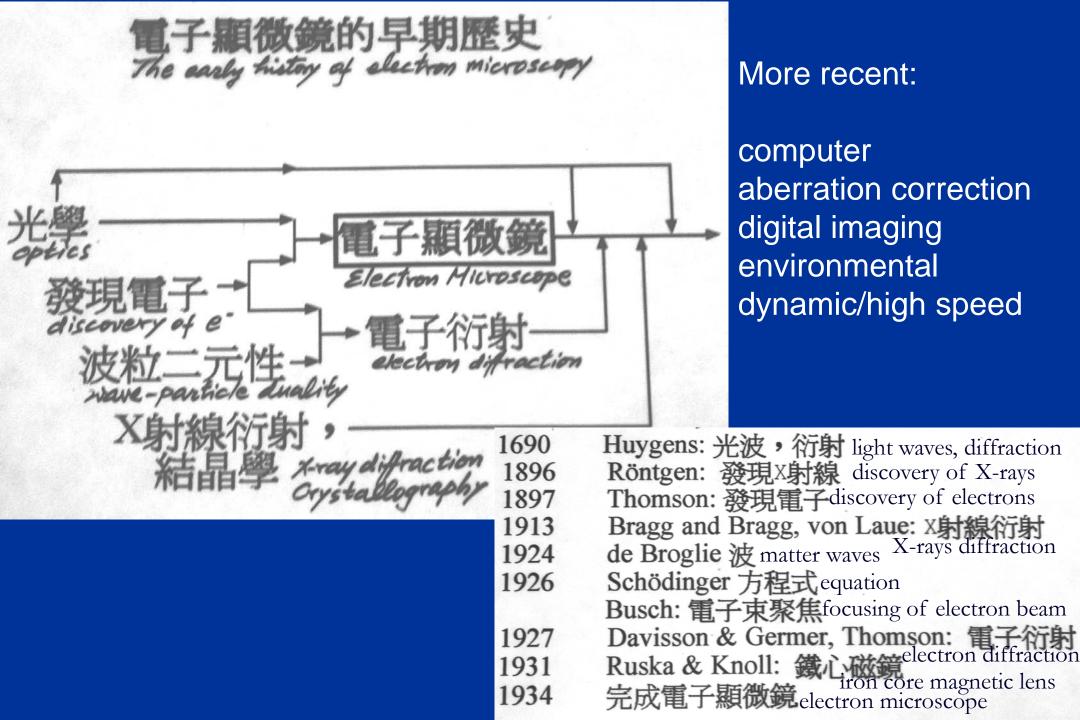
(ref. Taiyo)



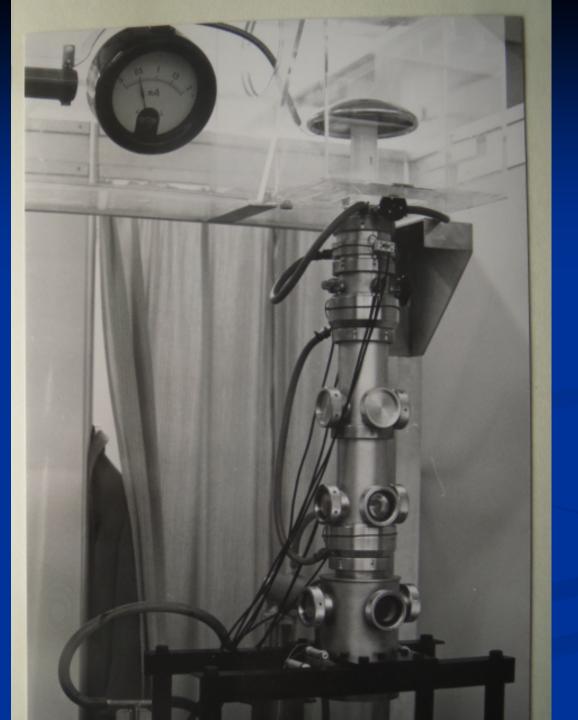
Why electron microscopy (EM)?

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



Various Electron Microscopes

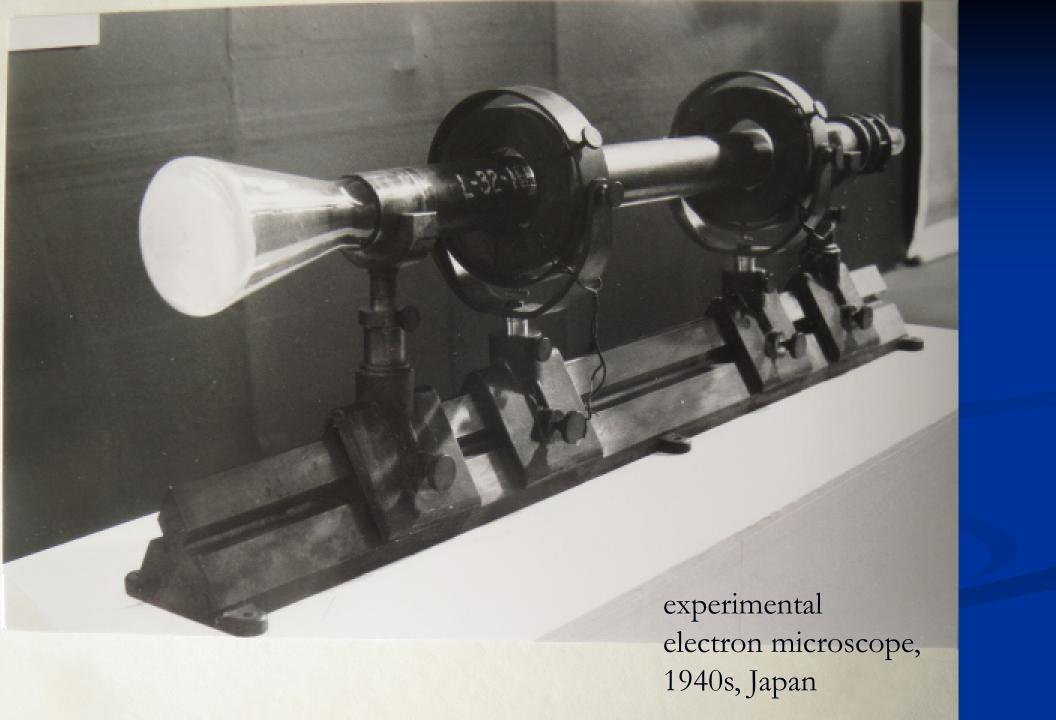


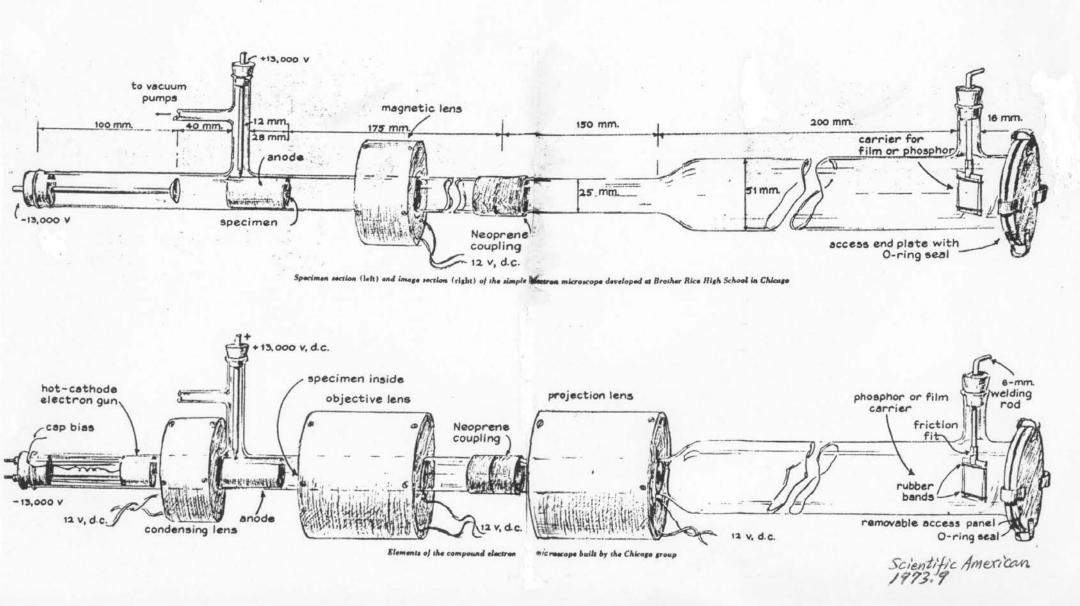
first electron microscope, (replica)



first commercial electron microscope, 1934

Ruska's Commercial Electron Microscope (1934); Museum f. Verkehr u. Technik, Berlin; 1987.





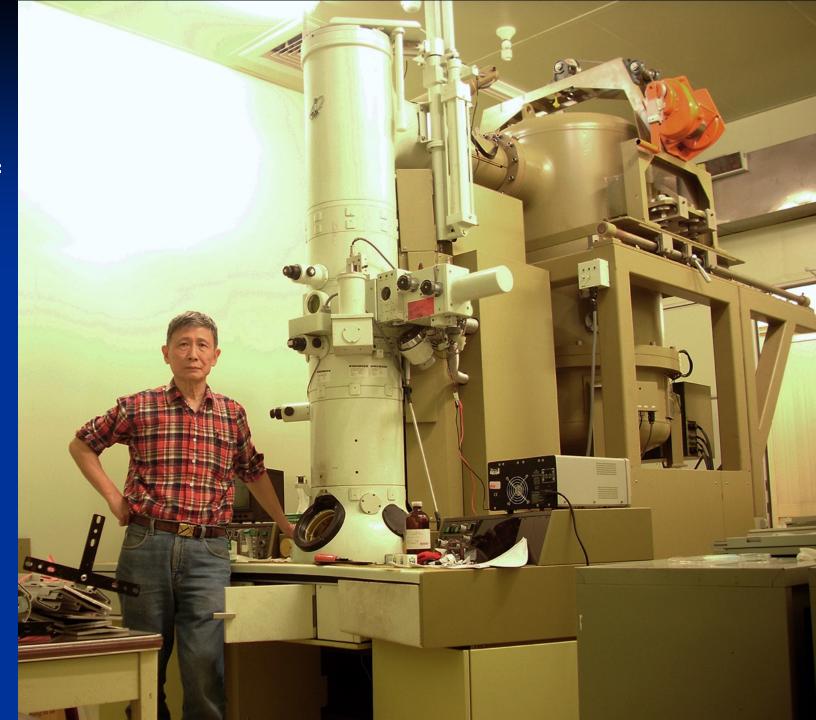
Experimental electron microscope, Loyola High School, Chicago

Hitachi H-600 NTHU, 2008



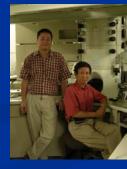
JEOL JEM-4000EX

NTHU, 2016
After retirement of machine and man



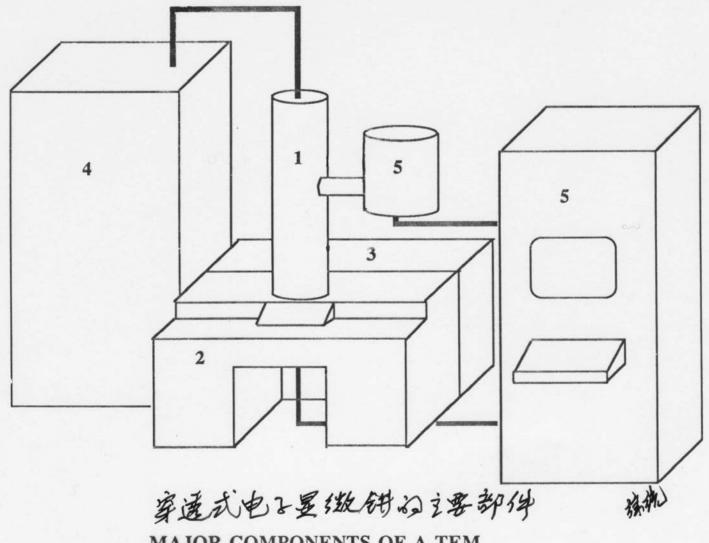


JEOL JEM-ARM1250/1000









MAJOR COMPONENTS OF A TEM

1. electron optics column

2. electronics and controls

3. vacuum system

4. high voltage power supply

5. accessories

研斷

电子系统

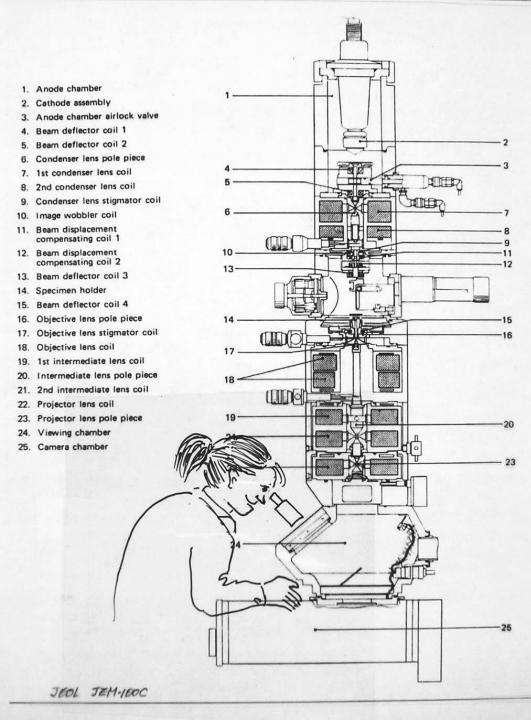
真空系统

马压电路

1344.

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

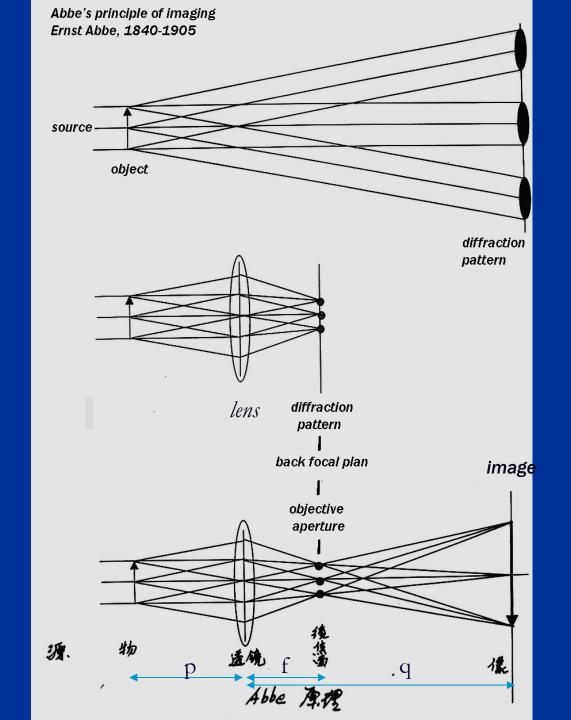
Apertures
Specimen chamber
Camera





OPTICAL MICROSCOPY

ABBE'S PRINCIPLE



Abbe's Principle of lectron electron electron microscope

Principle of Fundamental geometrical and physical optics
Abbe's principle and the

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.

$$1/p + 1/q = 1/f$$

Principle of image formation

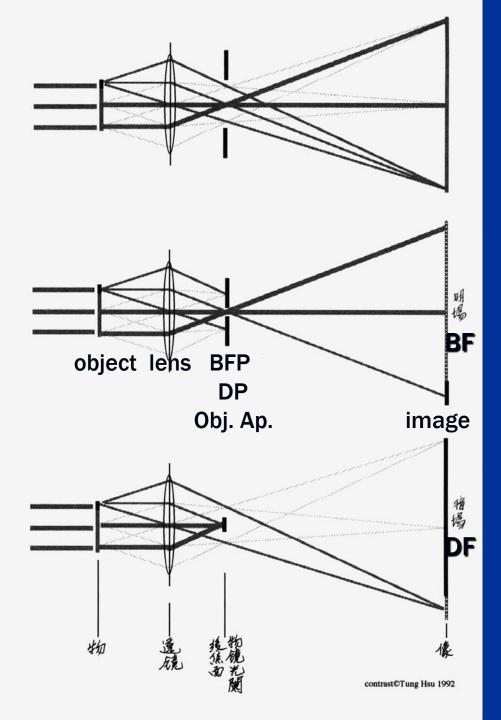
Fundamental geometrical and physical optics

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

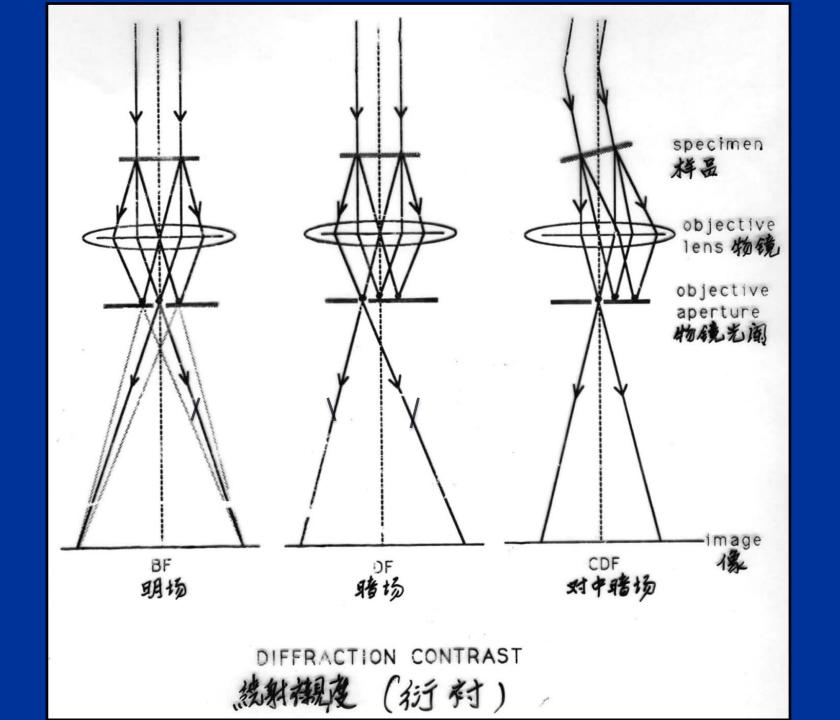
Contrast: Beam/solid interaction

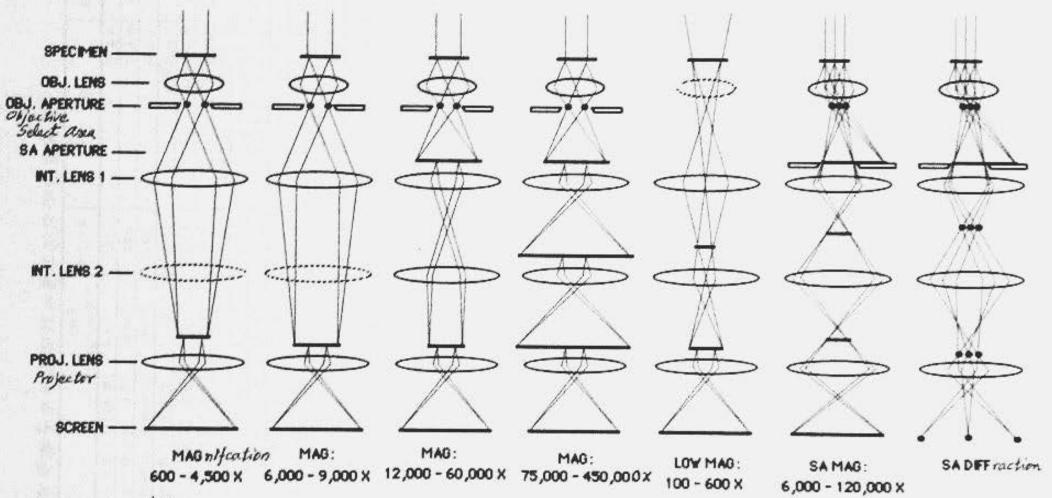
BFP and the objective aperture:

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



Contrast: Beam/solid interaction
Back focal plane,
objective aperture,
diffraction pattern
Bright field (BF) and dark field (DF)
images.





JEOL ,接税

The electron gun:

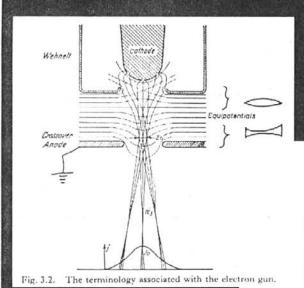
An electrostatic lens + an electron accelerator

Filament: Tungsten

LaB₆

Field emission

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



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

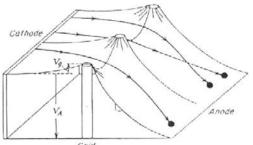


Fig. 2.9. The rubber-membrane model for experimental determination of elecpaths (exaggerated vertical scale).

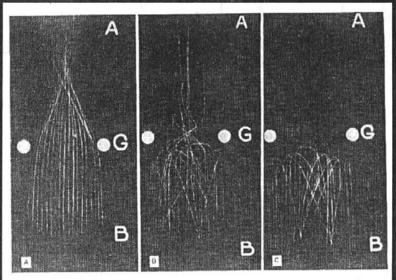
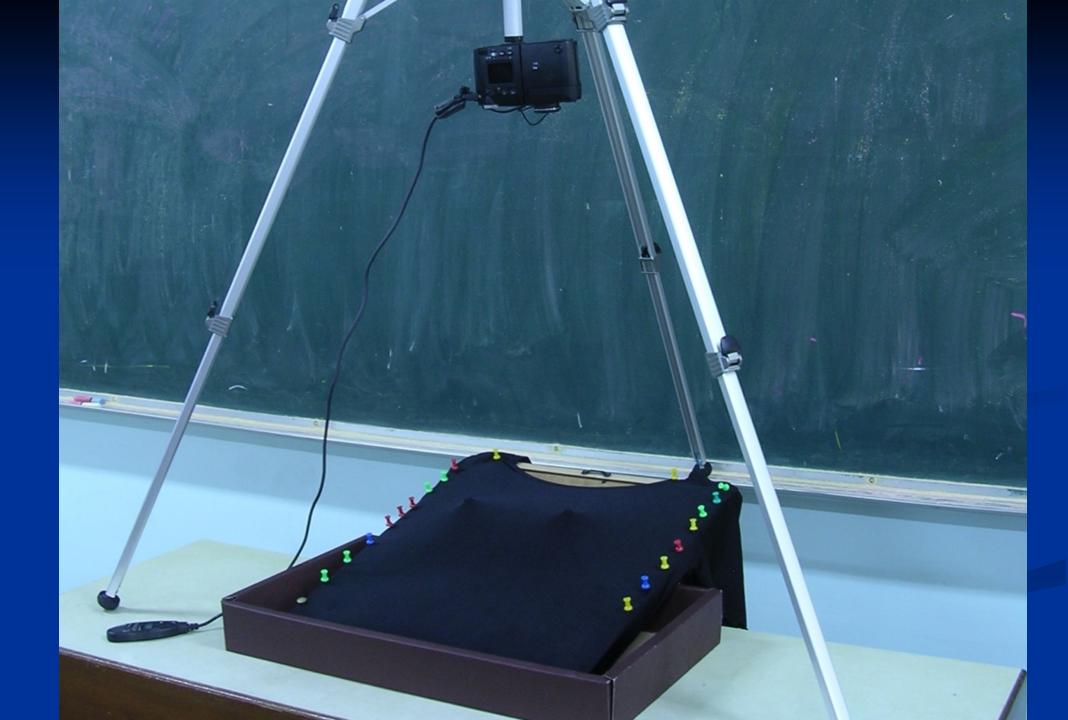
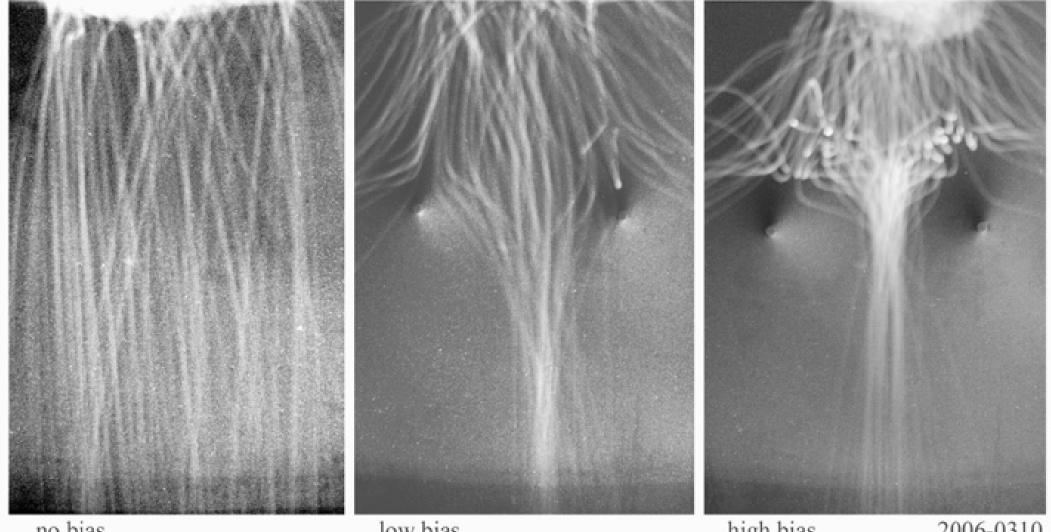


Fig. 2.10. Electron paths in a triode as determined with the rubber-membrane model. A is the anode, B the cathode, and G the grid. The grid potential is increasingly negative from A to C. (Courtesy Philips Tech. Rev., Ref. 2.)

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





low bias high bias no bias 2006-0310

The electromagnetic lens

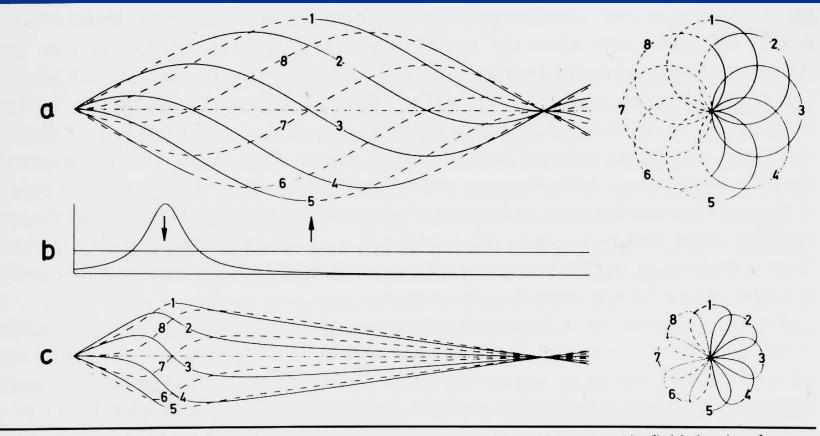


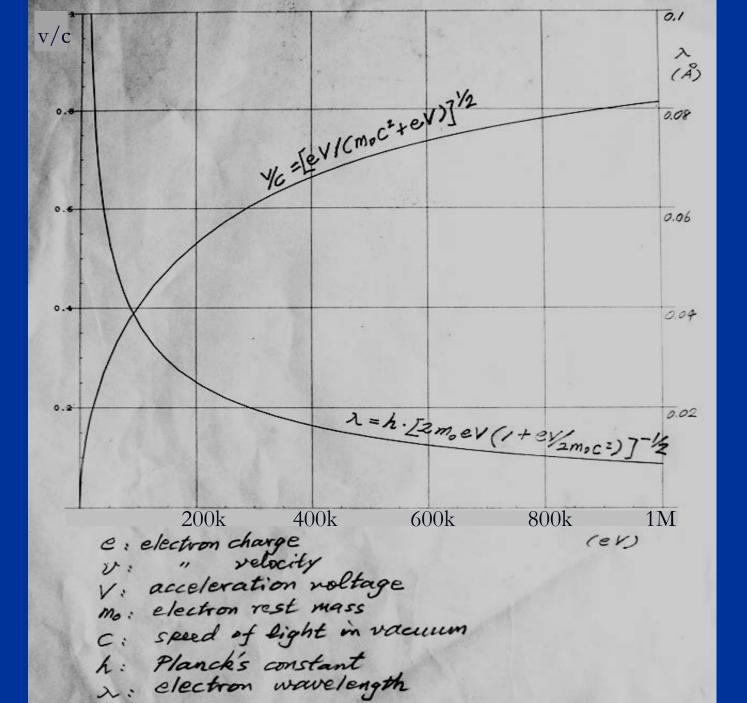
Fig. 1. Electron trajectories in a uniform (a) and in a non-uniform (c) magnetic field, issuing from an axial point of the specimen for different azimuth angles, but making the same angle with the lens axis. (b) Field distributions corresponding to (a) and (c).

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

Electron micrographs (EM, TEM images)

And

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



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 <u>interference</u>, 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. 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.

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 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

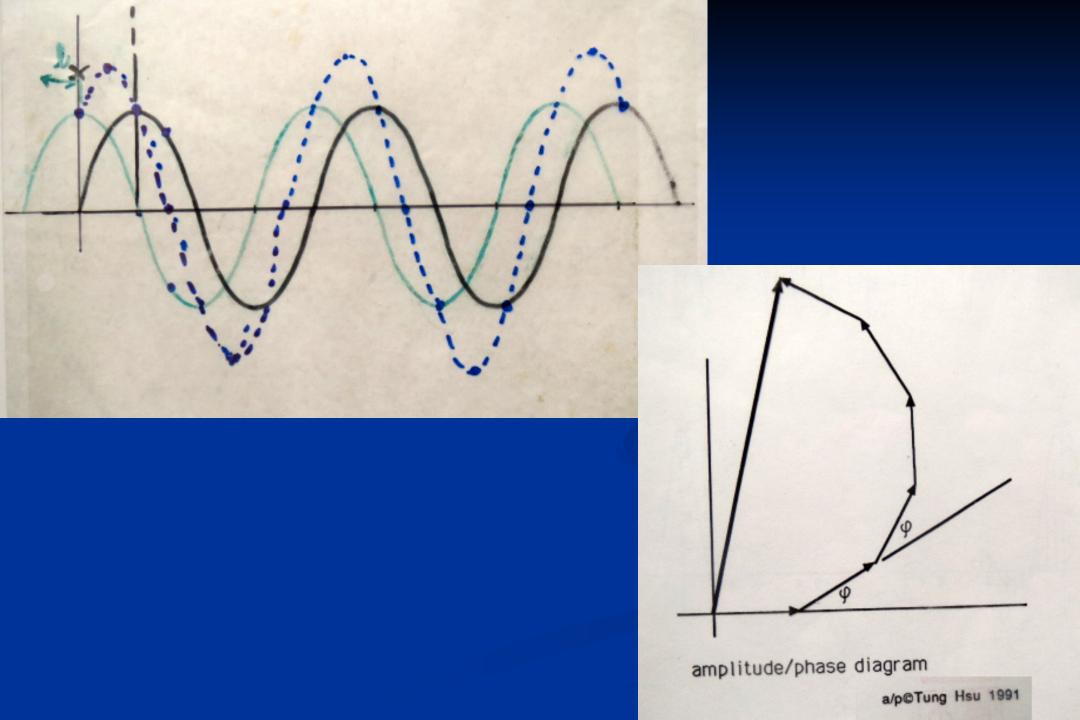
Optical bench (Atlas)

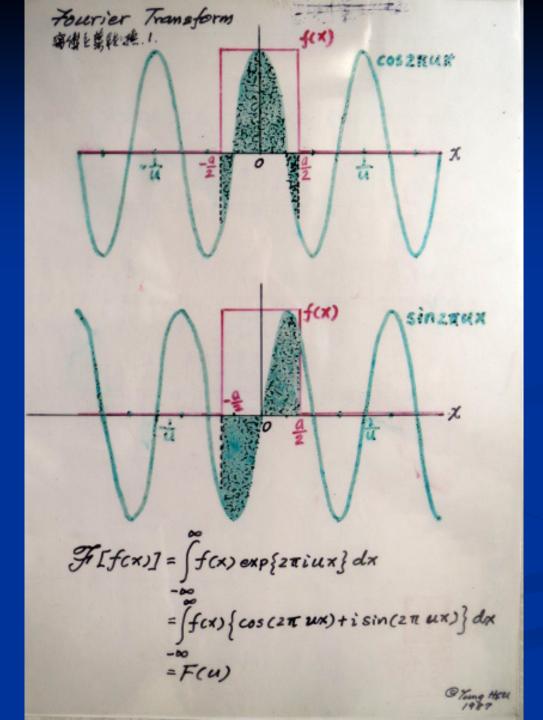
Computer

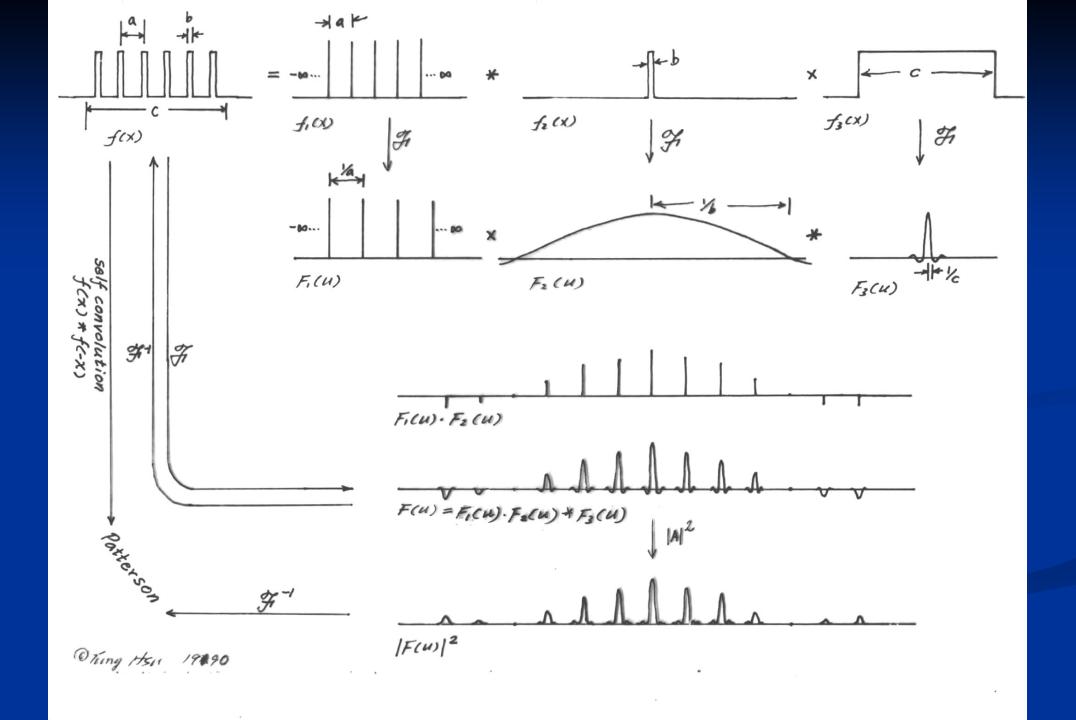
Diffraction Patterns from 3D objects

Bragg's Law

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







For finding diffraction patterns:

ImageJ®:

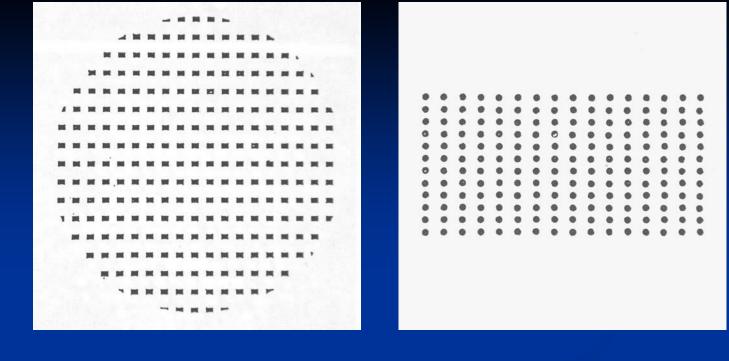
basic image processing. Ask Ms. Chen (2789-8394) about down loading.

DigitalMicrograph®:

professional image processing. Ask Ms. Chen about free demo copy.

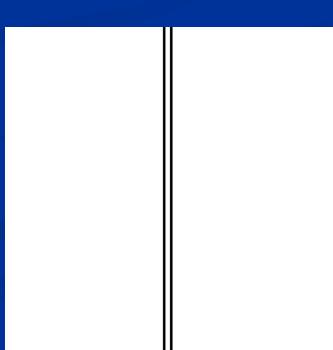
Reciprocal lattice and Ewald construction:

These are so cool and important. Sorry we did not have time to cover them. If you are interested, I can give a special lecture and demo on this subject. It will be free except that you have to arrange a time outside your scheduled classes.



Try these on ImageJ® or DigitalMicrograph®







Try this on ImageJ® or DigitalMicrograph®

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

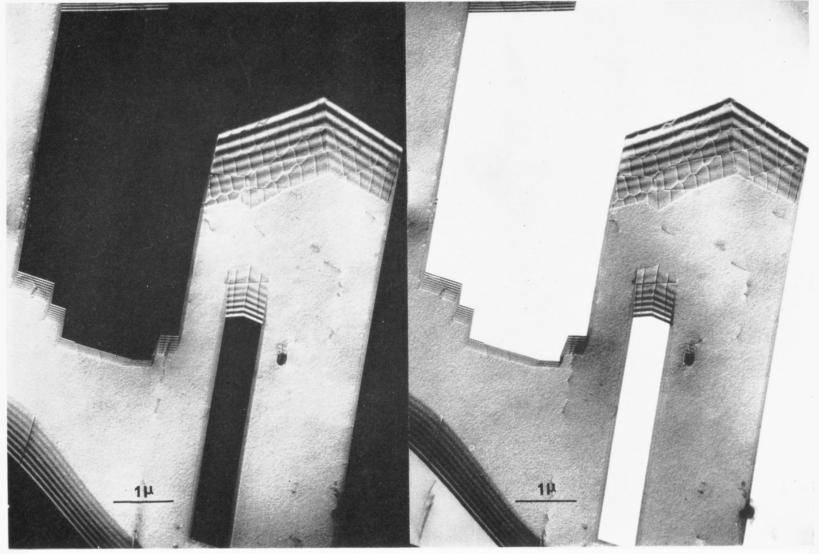
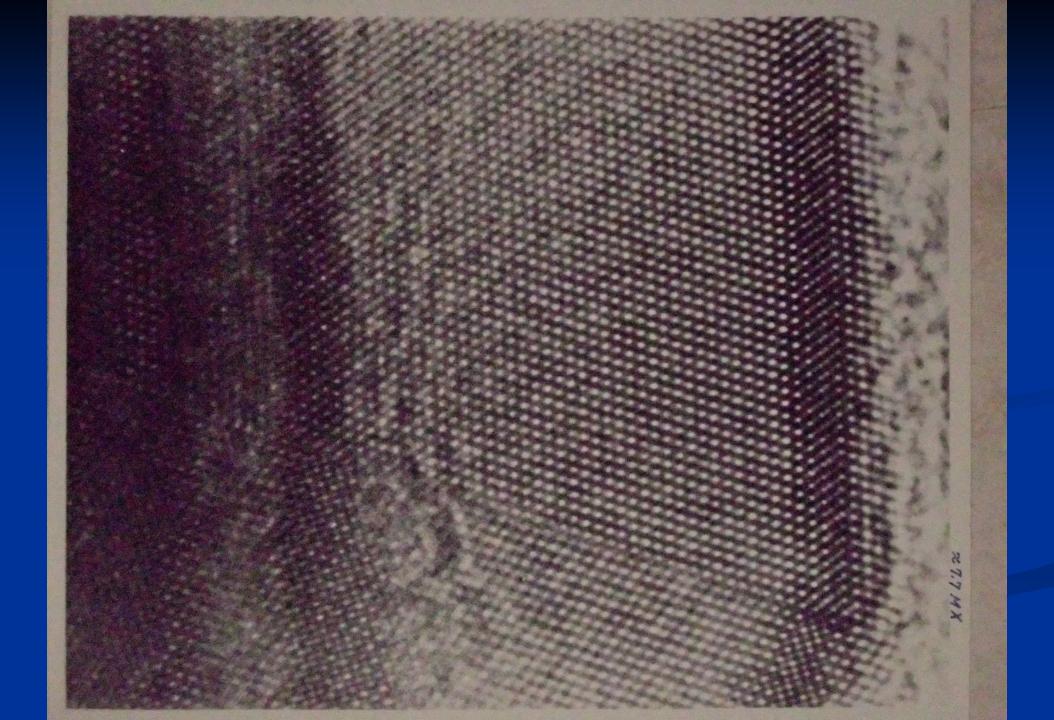
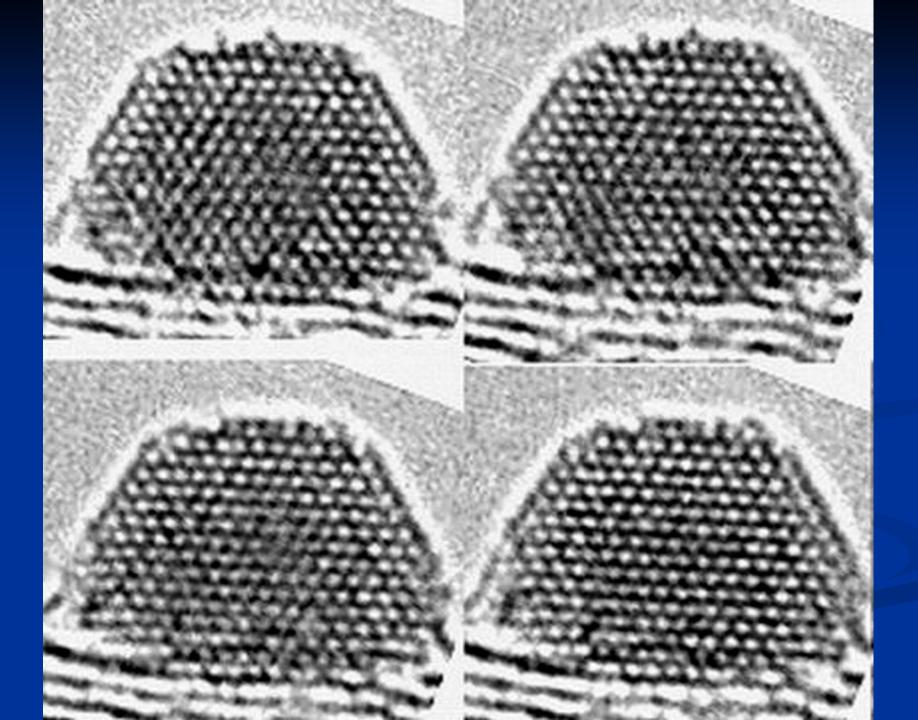


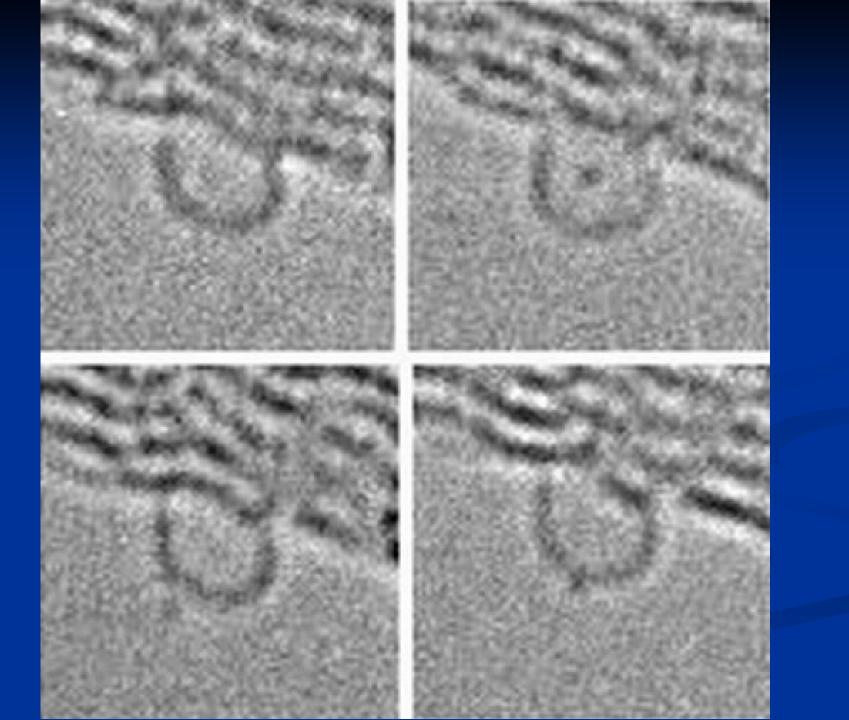
Plate X. Bright and dark-field micrographs of stainless steel, obtained with the Toulouse 1·5 MV microscope. $12\,000 \times$; $\Phi = 1$ MV.

(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.







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)

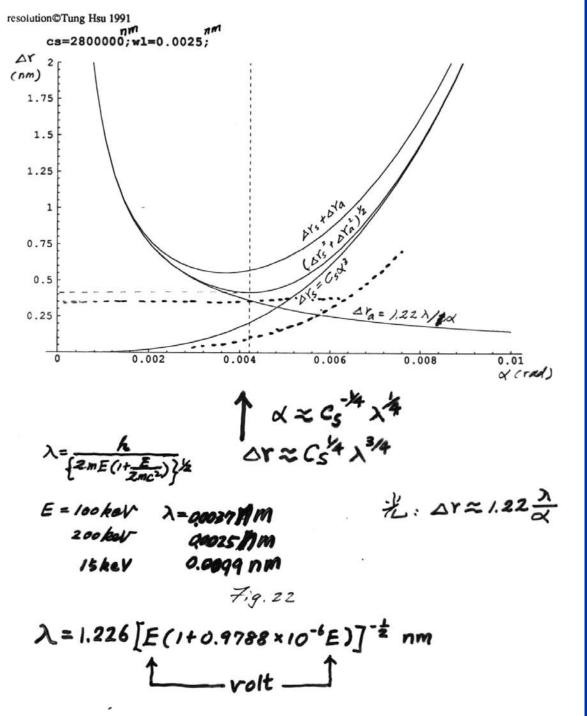
Spherical aberration

Chromatic aberration

Defocus ("diffraction" related)

Astigmatism

Detector: Fluorescence screen, Film, CCD, eyes



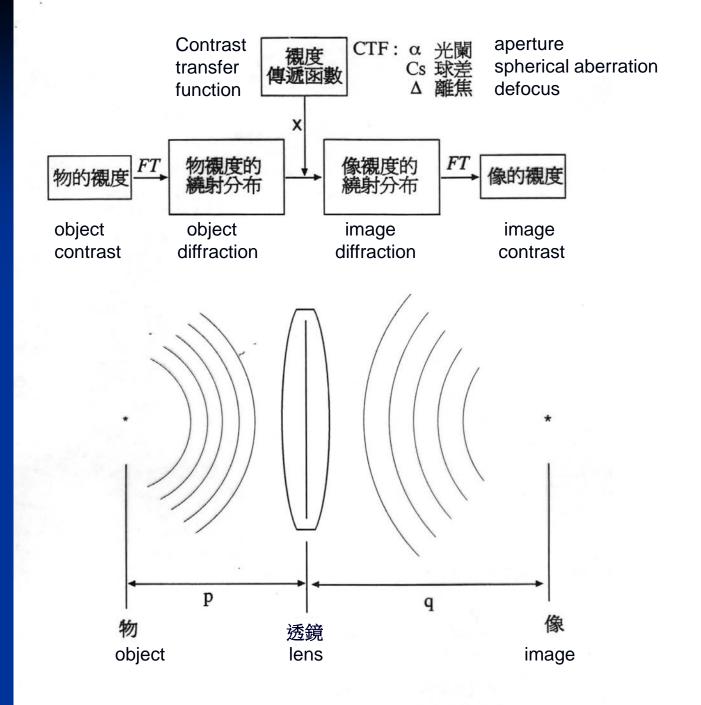
RESOLUTION:

Rayleigh's criterion

Balancing the spherical aberration effect and the diffraction effect:

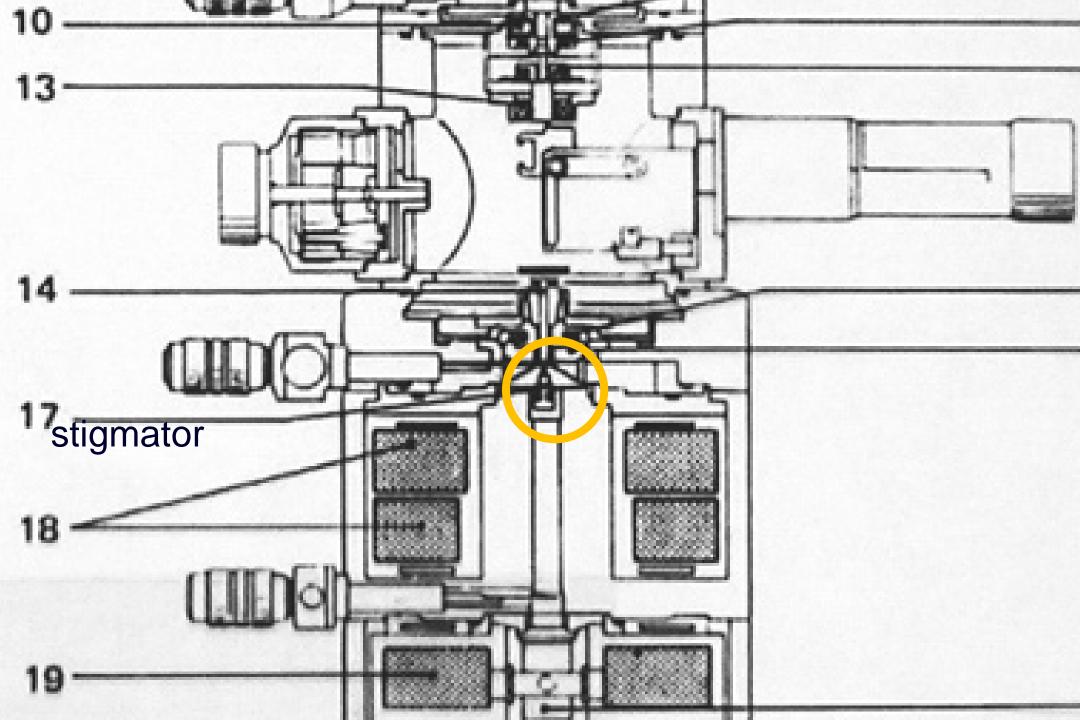
Smaller aperture produces larger Airy disc (diffraction pattern of the aperture).

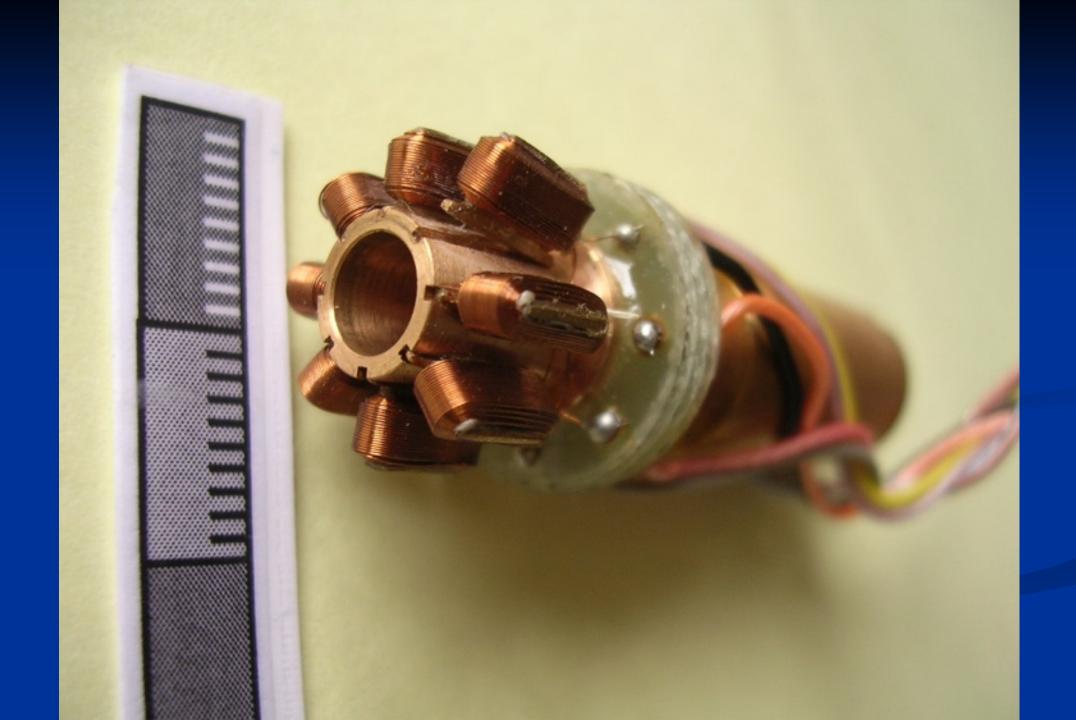
Larger aperture produces more diffused disc due to spherical aberration



ASTIGMATISM

Stigma
Stigmatize
Astigmatism
Anastigmatic
Stigmator





```
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
  lon
  (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

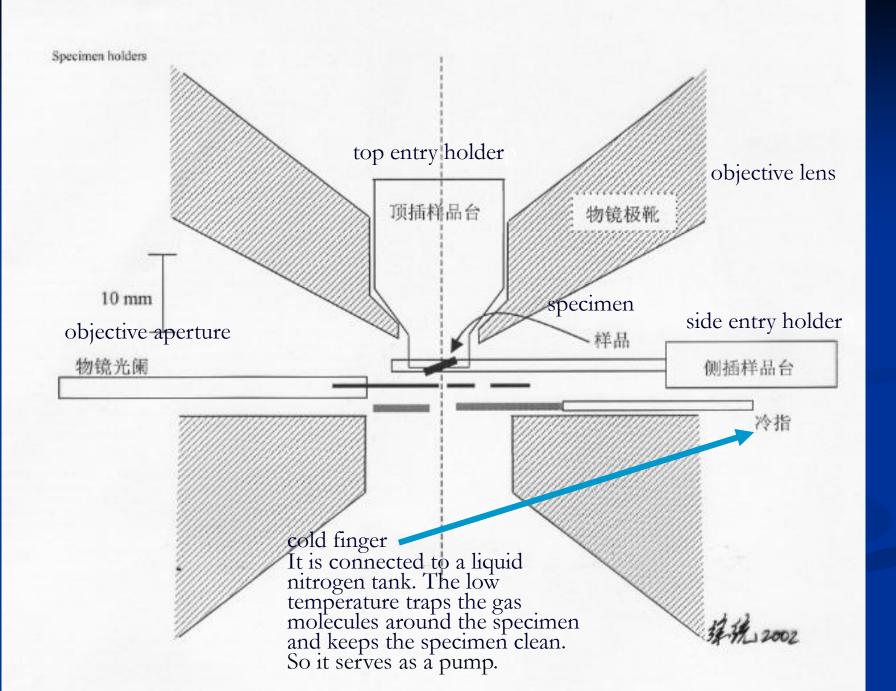
Side entry

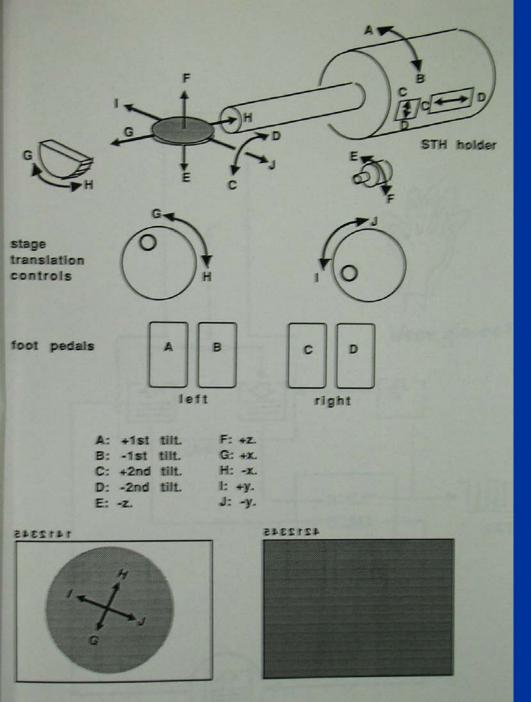
Single/double tilt

Heating, cooling, tensile, environmental, etc.

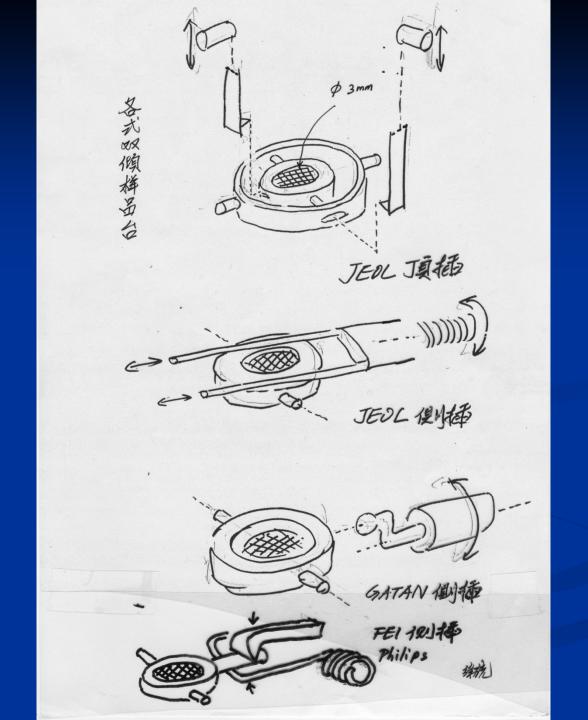
Performance:

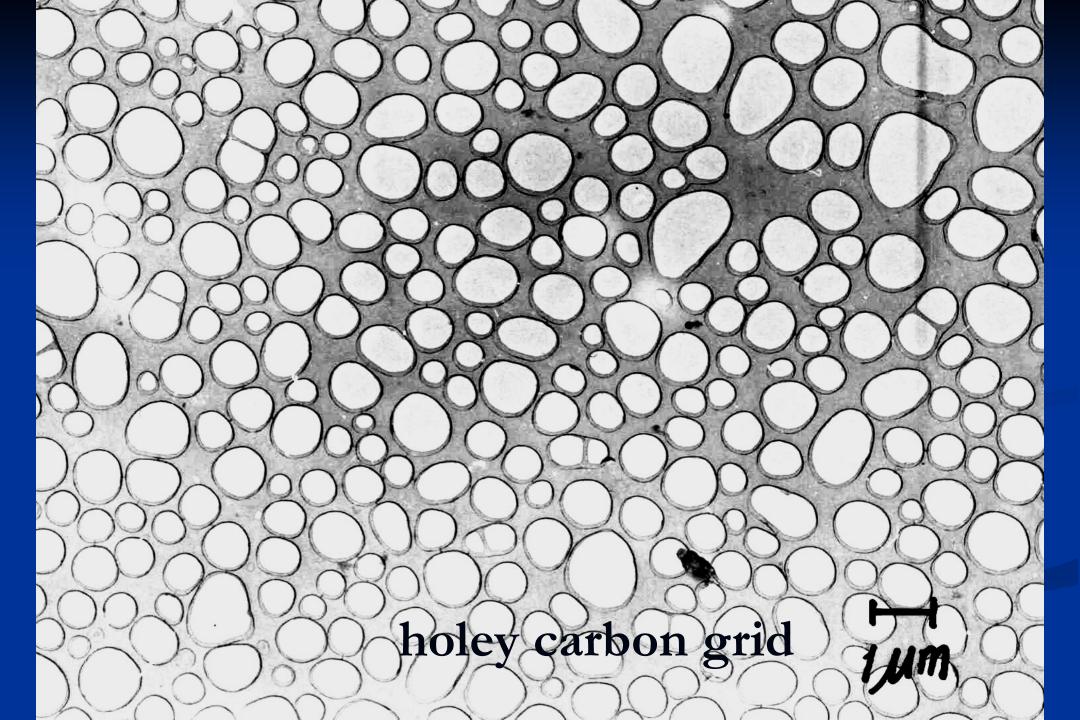
Tilt angle, working distance,





Movements and controls of the specimen





correction:

The holey carbon grid used for supporting powder specimen is NOT made by carbonizing the organic film. Rather the holey organic film is coated with a thin film of carbon. The carbon coating provides electrical conductivity. Therefore it can be used under the electron beam. For high resolution electron microscopy the carbon coated organic film is often too thick as a support. Then the organic film is dissolved with a solution such as acetone, leaving only the very thin carbon film.

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.

VACUUM

Thirty spokes share the wheel's hub; It is the center hole that makes it useful. Shape clay into a vessel; It is the space within that makes it useful. Cut doors and windows for a room; It is the holes which make it useful. Therefore profit comes from what is there; Usefulness from what is not there.

> Tao Te Ching, by Lao Tsu Translation & Caligraphy by Gia-fu Feng & Jane English

HIGH VACUUM = LOW PRESSURE

```
Why vacuum?
How to evacuate?
How to measure the vacuum?
Why the electron microscope has to be
evacuated?
  Stability of the speciman ·
  Filament life ·
  Sufficient mean free path of the electrons ·
   (for the required electron optical design.)
```

Physics of gases: Elastic gas molecules.

Constant motion of gas molecules, colliding each others and walls of container.

System in equilibrium.

Negligible external force (magnetic, gravity).

Physical phenomena under various pressures:

Boyle's Law : $p_1V_1 = p_2V_2$ p : pressure V : volume

Gas Law: pV = nRT n: number of moles

R: gas constant, 8.314 J/K·mol

T: temperature

Number of molecules per unit volume at T and p:

nA/V = p/(RT) A: Avogadro number, 6.022x10²³

```
Unit of pressure : 1 \text{ Pa} = 1 \text{ N/m}^2
                            = 1.45 \times 10^{-4} \text{ psi}
                            = 7.50 \times 10^{-3} \text{ torr (mm-Hg)}
Mean free path L under pressure p torr :
     L = 5x10^{-5}/p (m)
Types of gas flow:
     Turbulent: irregular, many vortices.
     Smooth: regular, no vortices.
     Knudsen: L < tube diameter.
     Molecular: L > tube diameter;
          Molecules do not interact with each others
          except collisions.
          (such is the case inside the electron microscope.)
```

Pumping:

When evacuating a chamber one does not draw molecules. One allows gas molecules to diffuse out and prevent them from going back in.

Various pumps: mechanical, diffusion, turbo-molecular, ion, sorbtion....

Multi-state pumping is necessary for 10⁻³ torr or below. Pumping rate: (torr)-liter/sec.

To maintain vacuum, keep on pumping to balance the leak and outgas.

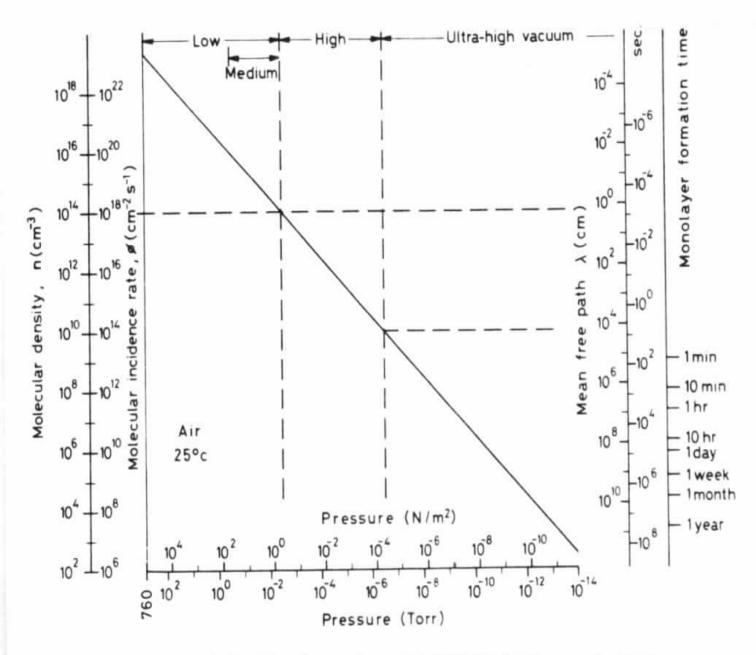
leak: (torr)-liter/sec.

outgas: torr-liter/cm²-sec.

measurement of vacuum: based on physical phenomena at various pressure.

Vacuum (pressure) gauges : Mercury column, thermo couple, ionization current.

Maintain good vacuum:
Instrument design and operation procedure.
Operator's good practice and skill.
Environment of the lab.



degree of vacuum.

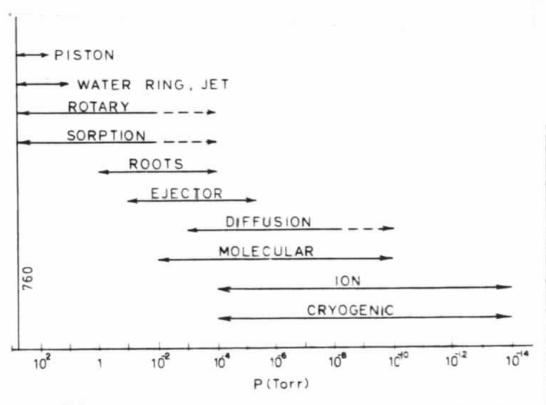
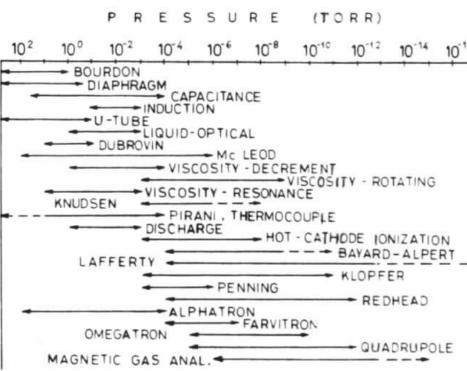


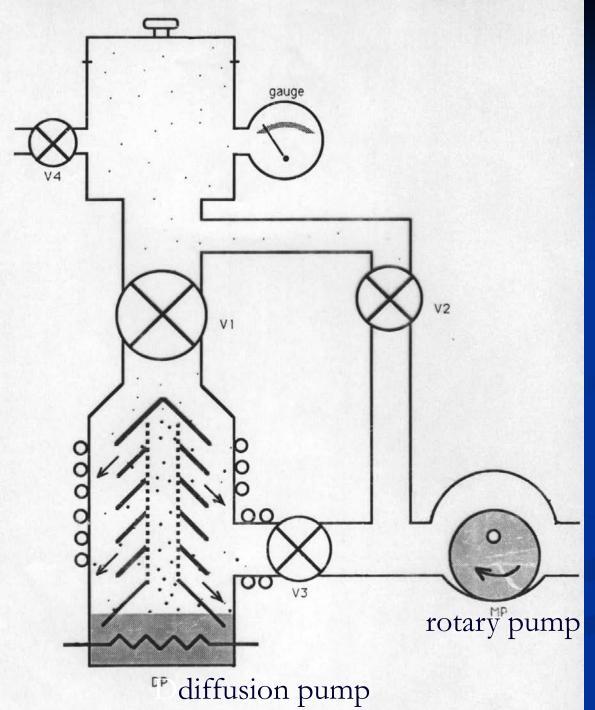
Fig. 5.1 Pressure ranges of vacuum pumps.

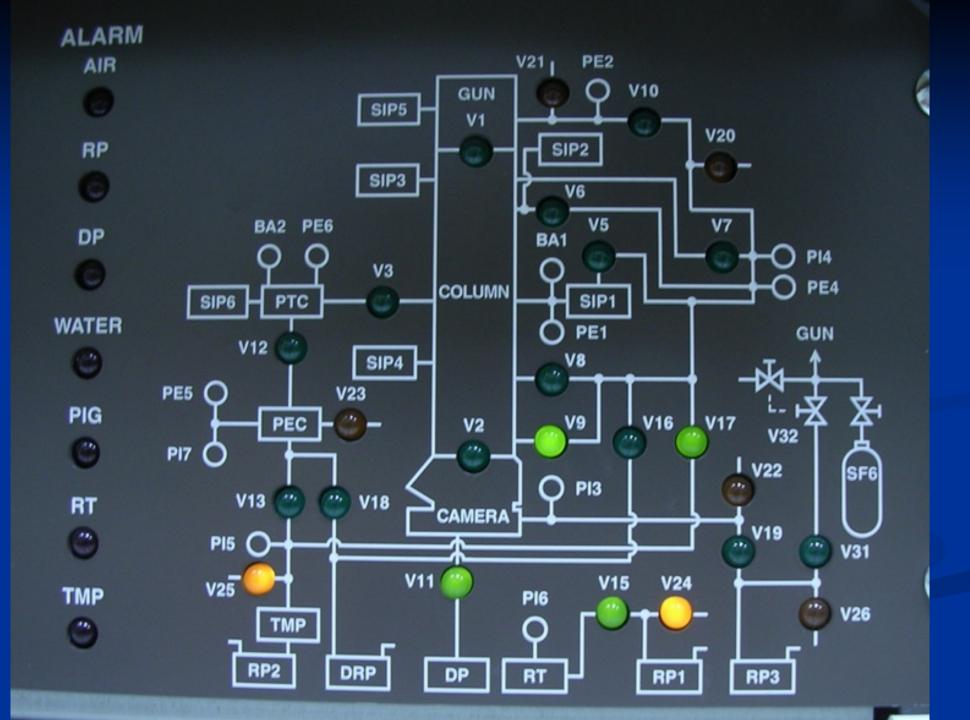


MEASUREMENT OF LOW PRESSURES

Fig. 6.1 Pressure ranges of vacuum gauges.

A. Roth, "Vacuum Technology", 2"d ed. 1982 North Holland.







A turbo pump

scanning electron microscope (SEM)

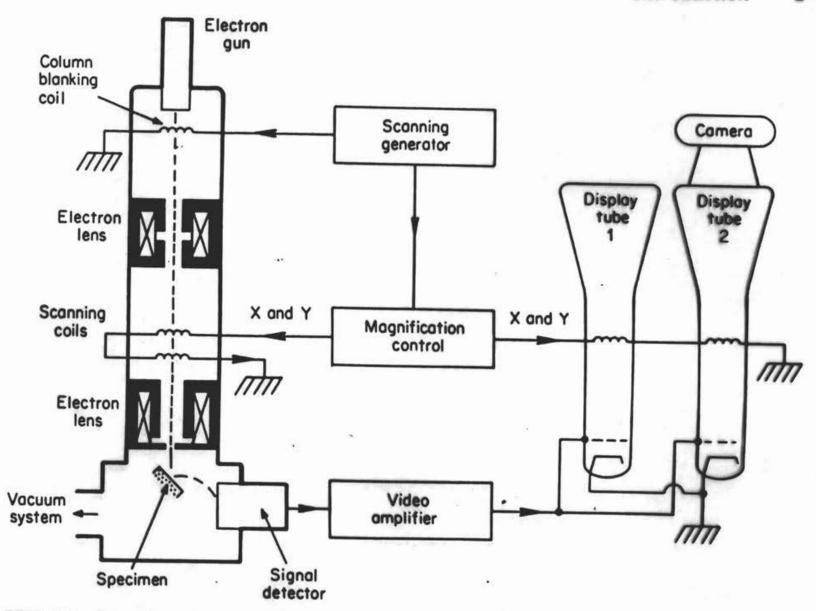


FIG. 1.2 Scanning electron microscope.

...

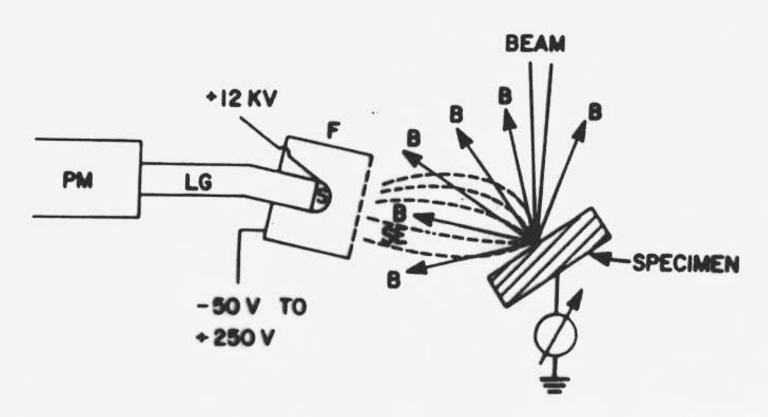
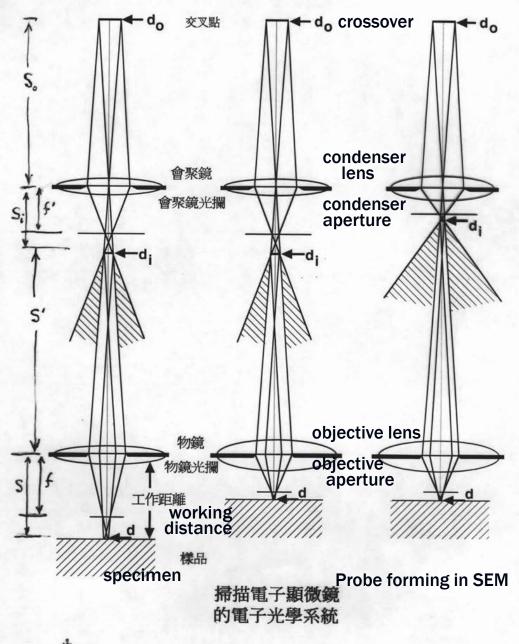


Figure 4.17. Schematic diagram of Everhart-Thornley scintillator-photomultiplier electron detector. B, backscattered electron; SE, secondary electron; F, Faraday cage; S, scintillator; LG, light guide; PM, photomultiplier.



sem©Tung Hsu, 1999
Ref. "Scanning electron microscopy and x-ray microanlalysis",
Goldstein, et al, 1981, Plenum, Ch. 2.

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

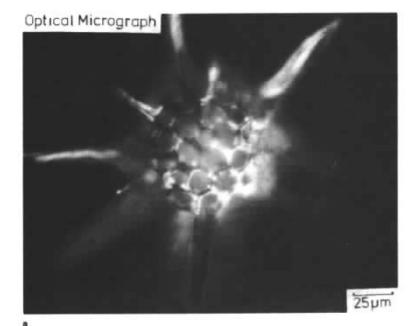
The column

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



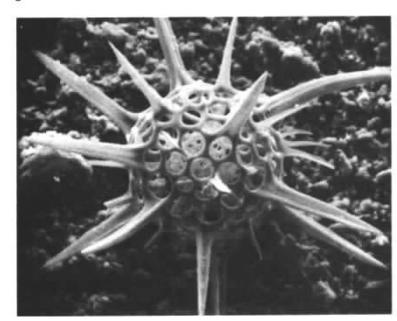
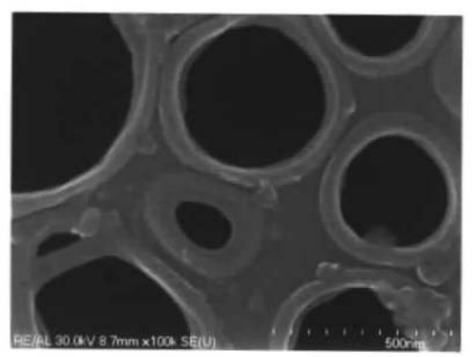
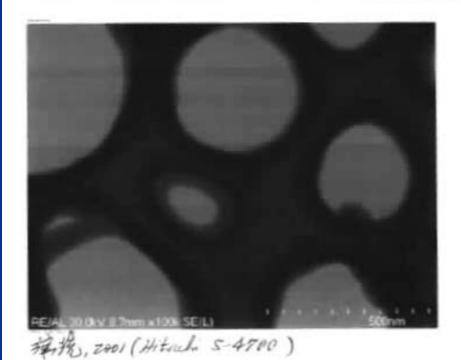


Figure 1.3. (a) Optical micrograph of the radiolarian Trochodiscus longispinus. (b) SEM micrograph of same radiolarian. The greater depth of focus and superior resolving capability of the SEM are apparent.





Examples of SEM images

The following material was not covered.

Electron microprobe / Analytical electron microscopy: (EPMA)

Energy dispersive (X-ray) spectrometer, EDS (EDX)
Wavelength dispersive (X-ray) spectrometer, WDS (WDX)
Electron energy loss spectroscopy, EELS
Quantitative analysis
etc.

Ref. Dr. Yoshi Iizuka

Institute of Earth Science

Academia Sinica

He has two cutting edge EPMAs.

Co Ni Te 1
Mendeleev: A = 58.9 58.6 127.7 126.9
Moseley: Z = 27 28 52 53

Parameter	nλ	2d	sin0
diffraction	known	calculated	measured
spectrometry(WDS)	calculated	known	measured
spectrometry(EDS)	E: measured		

E=hc/λ, λ = C'(Z - σ)⁻² (for the same spectral line, K \propto , K β , ...)

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

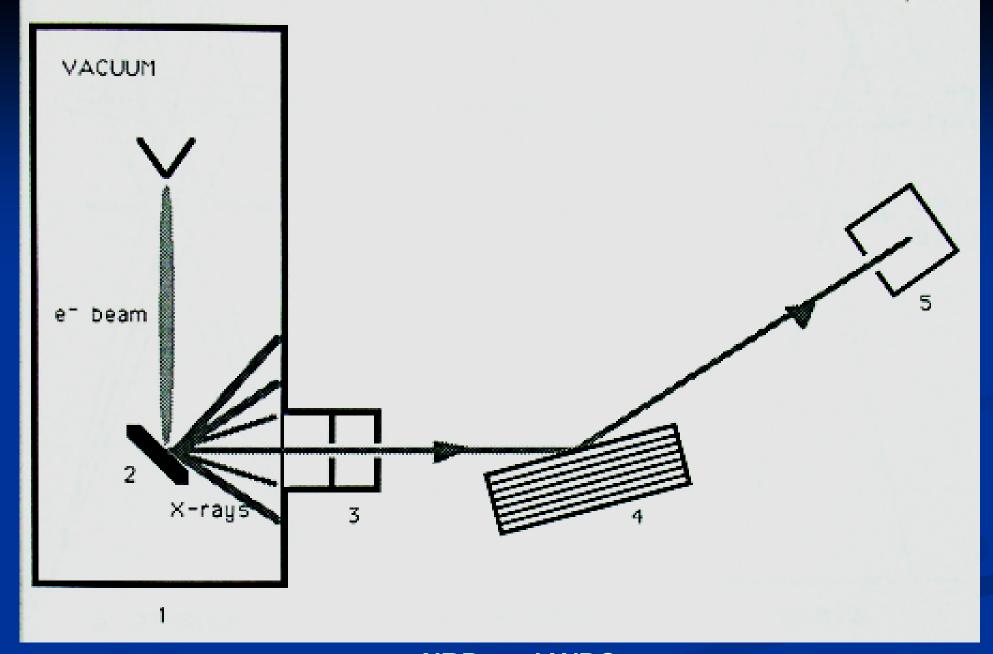
EDS: Si(Li) detector

Multi-channel analyzer (MCA)

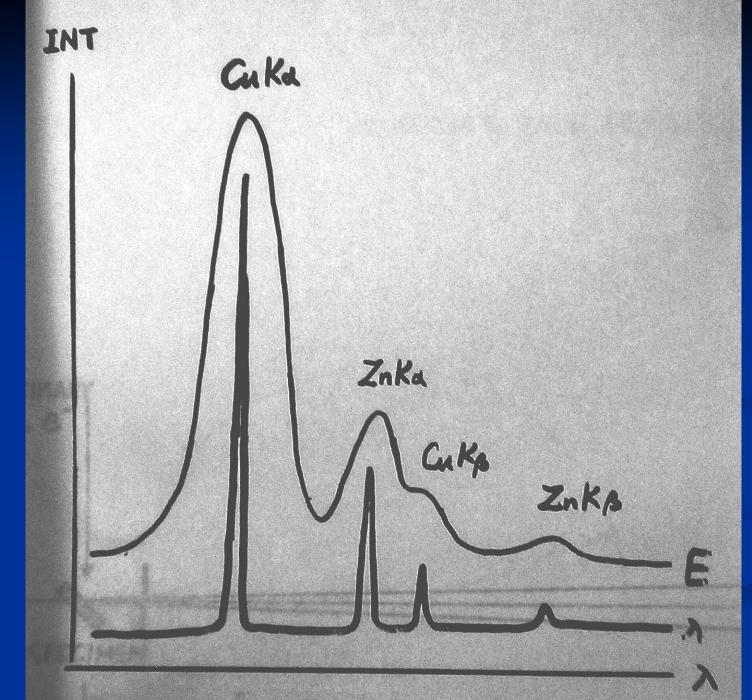
Be window ⇒ Na and up

Ultra-thin window or Windowless ⇒ B and up

Dead layer in Si(Li) detector is the limit



XRD and **WDS**





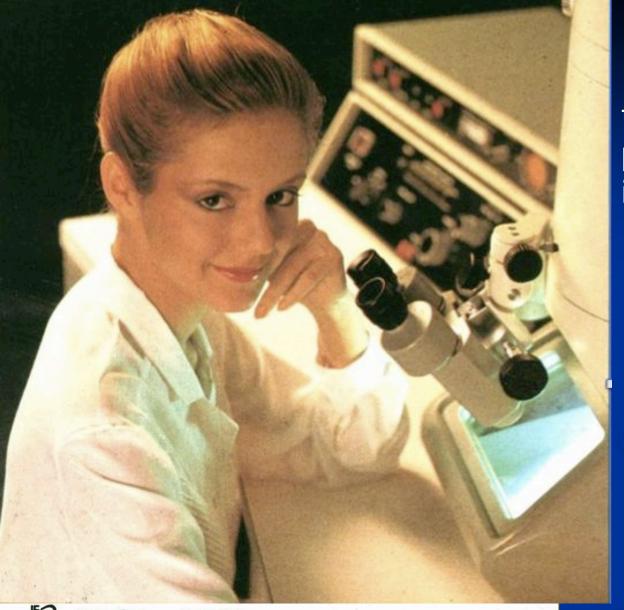
Microscopy Society of America Position on Ethical Digital Imaging

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



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