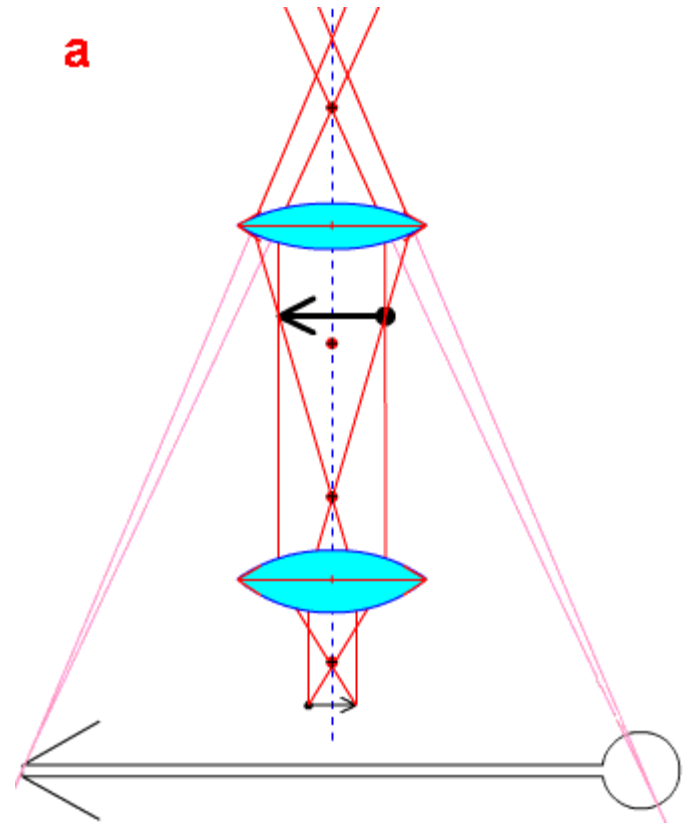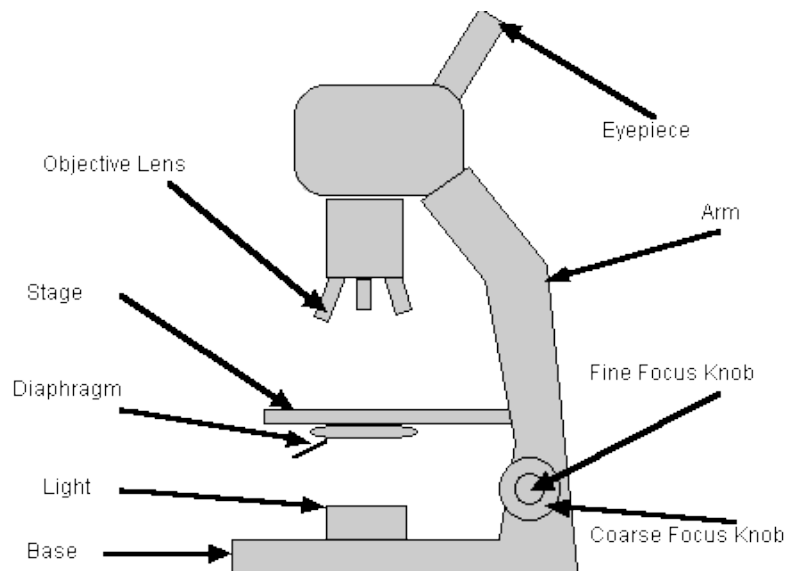- Optical Microscope;

- Scanning Electron Microscope (SEM);

- Transmission Electron Microscope (TEM);

- Comparison with scanning probe microscope (SPM)

Robert Hooke's "Micrographia" (1665)
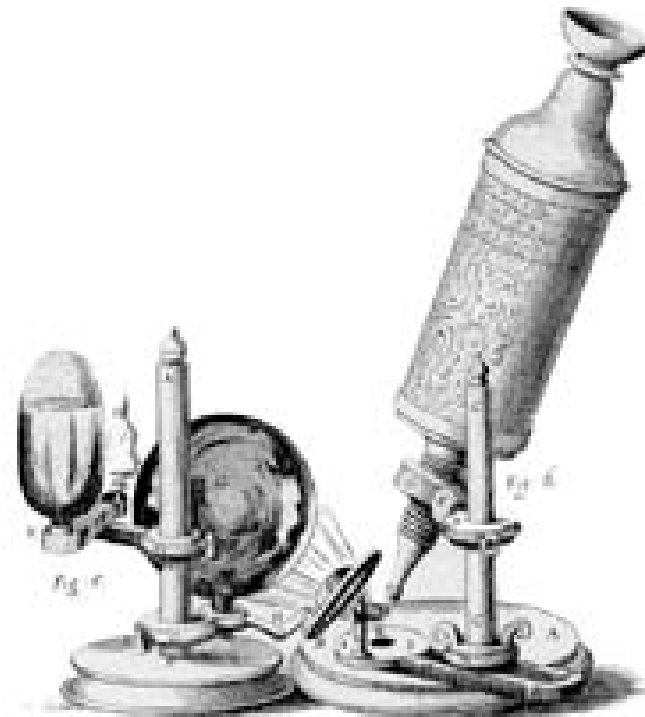
Principles of an optical microscope

Advantages and Disadvantages of Optical Microscope

Advantages:

- Direct imaging with no need of sample pre-treatment, the only microscopy for real color imaging.

- Fast, and adaptable to all kinds of sample systems, from gas, to liquid, and to solid sample systems, in any shapes or geometries.

- Easy to be integrated with digital camera systems for data storage and analysis.

Disadvantages:

- Low resolution, usually down to only sub-micron or a few hundreds of nanometers, mainly due to the light diffraction limit.

Electronic Microscope for higher resolution

- Resolution limit of optical microscopes is due to the light diffraction; roughly optical resolution can be estimated as wavelength λ/2NA (NA is the numerical aperture of lens, usually ~ 1.0): for white light, average wavelength is around 500 nm, the best resolution is thus a few hundreds nm.

- Decreasing the wavelength is the way to improve the resolution, though nobody would deal with UV light.

- Electron wave is a unique medium that can be used in imaging. By accelerating the electrons into high energy beam (via high voltage), the wavelength thus created is far shorter than white light. For example, for an electron beam produced from a 20 kV gun, the wavelength is only 1240.7/20,000 (eV) = 0.06 nm = 0.6 Å, corresponding to a resolution limit of λ/2 = 0.3 Å --- *theoretically, it can be used to image a species as small as 0.3 Å.* Most atoms are in size of 2-3 Å.

History of Electronic Microscope

- The first electromagnetic lens was developed in 1926 by Hans Busch.

- According to Dennis Gabor, the physicist Leó Szilárd tried in 1928 to convince Busch to build an electron microscope, for which he had filed a patent.

- The German physicist Ernst Ruska and the electrical engineer Max Knoll constructed the prototype electron microscope in 1931, capable of four-hundred-power magnification; the apparatus was the first demonstration of the principles of electron microscopy. Two years later, in 1933, Ruska built an electron microscope that exceeded the resolution attainable with an optical (light) microscope. Moreover, Reinhold Rudenberg, the scientific director of Siemens-Schuckertwerke, obtained the patent for the electron microscope in May 1931.

- In 1932, Ernst Lubcke of Siemens & Halske built and obtained images from a prototype electron microscope, applying concepts described in the Rudenberg patent applications. Five years later (1937), the firm financed the work of Ernst Ruska and Bodo von Borries, and employed Helmut Ruska (Ernst's brother) to develop applications for the microscope, especially with biological specimens. Also in 1937, Manfred von Ardenne pioneered the scanning electron microscope. The first *practical* electron microscope was constructed in 1938, at the University of Toronto, by Eli Franklin Burton and students Cecil Hall, James Hillier, and Albert Prebus; and Siemens produced the first *commercial* transmission electron microscope (TEM) in 1939. Although contemporary electron microscopes are capable of two million-power magnification, as scientific instruments, they remain based upon Ruska's prototype.

http://en.wikipedia.org/wiki/Electron_microscope

History of Electronic Microscope

The Nobel Prize in Physics 1986
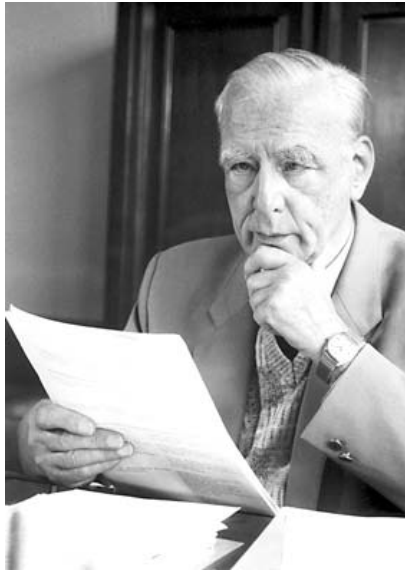
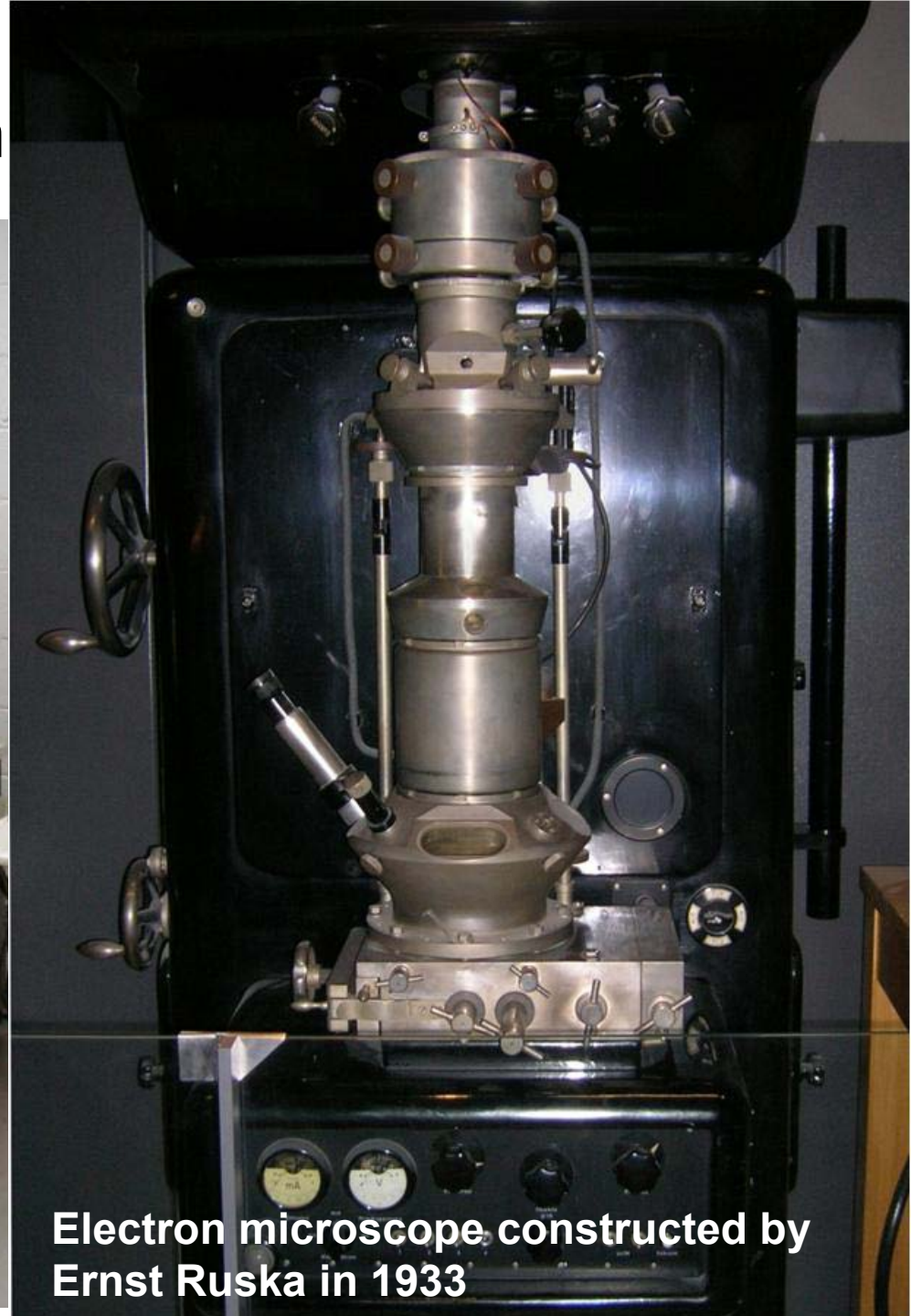"for his fundamental work in electron optics, and for the design of the first electron microscope"

Ernst Ruska
Born: 25 December 1906, Heidelberg, Germany
Died: 27 May 1988, West Berlin, Germany
Affiliation at the time of the award: Fritz-Haber-Institut der Max-Planck-Gesellschaft, Berlin, Federal Republic of Germany

Scanning Electron

Electron microscope constructed by Ernst Ruska in 1933

What is SEM?

- In scanning electron microscopy (SEM) an electron beam is focused into a small probe and is <u>rastered</u> across the surface of a specimen.
- Several interactions with the sample that result in the emission of <u>electrons</u> or <u>photons</u> occur as the electrons penetrate the surface.
- These emitted particles can be collected with the appropriate detector to yield valuable information about the material.
- The most immediate result of observation in the scanning electron microscope is that it displays the <u>shape</u> of the sample.
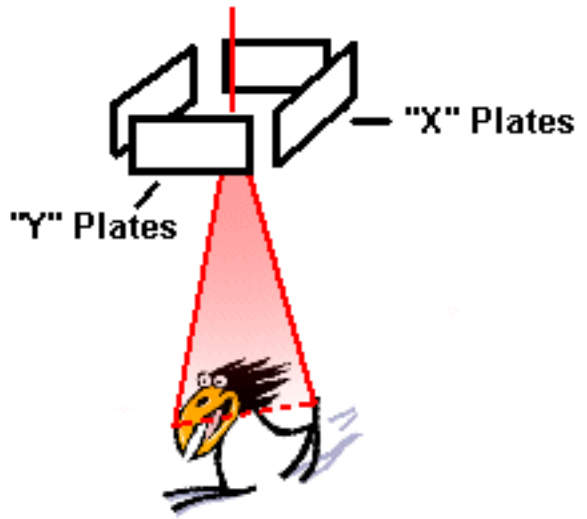- The resolution is determined by <u>beam diameter</u>.
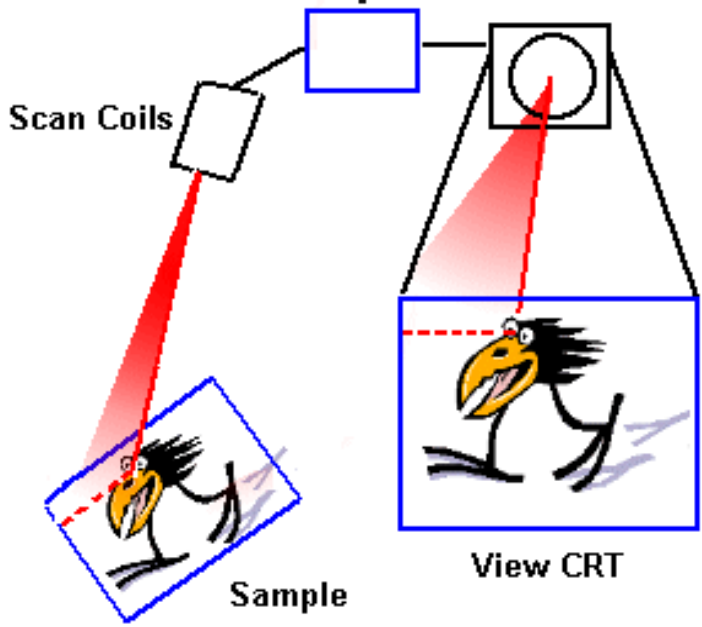
Brief schemes showing how SEM works
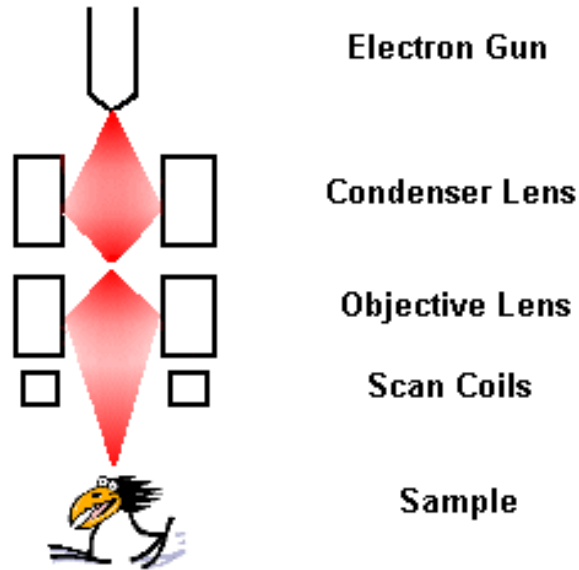
Brief schemes showing how SEM works

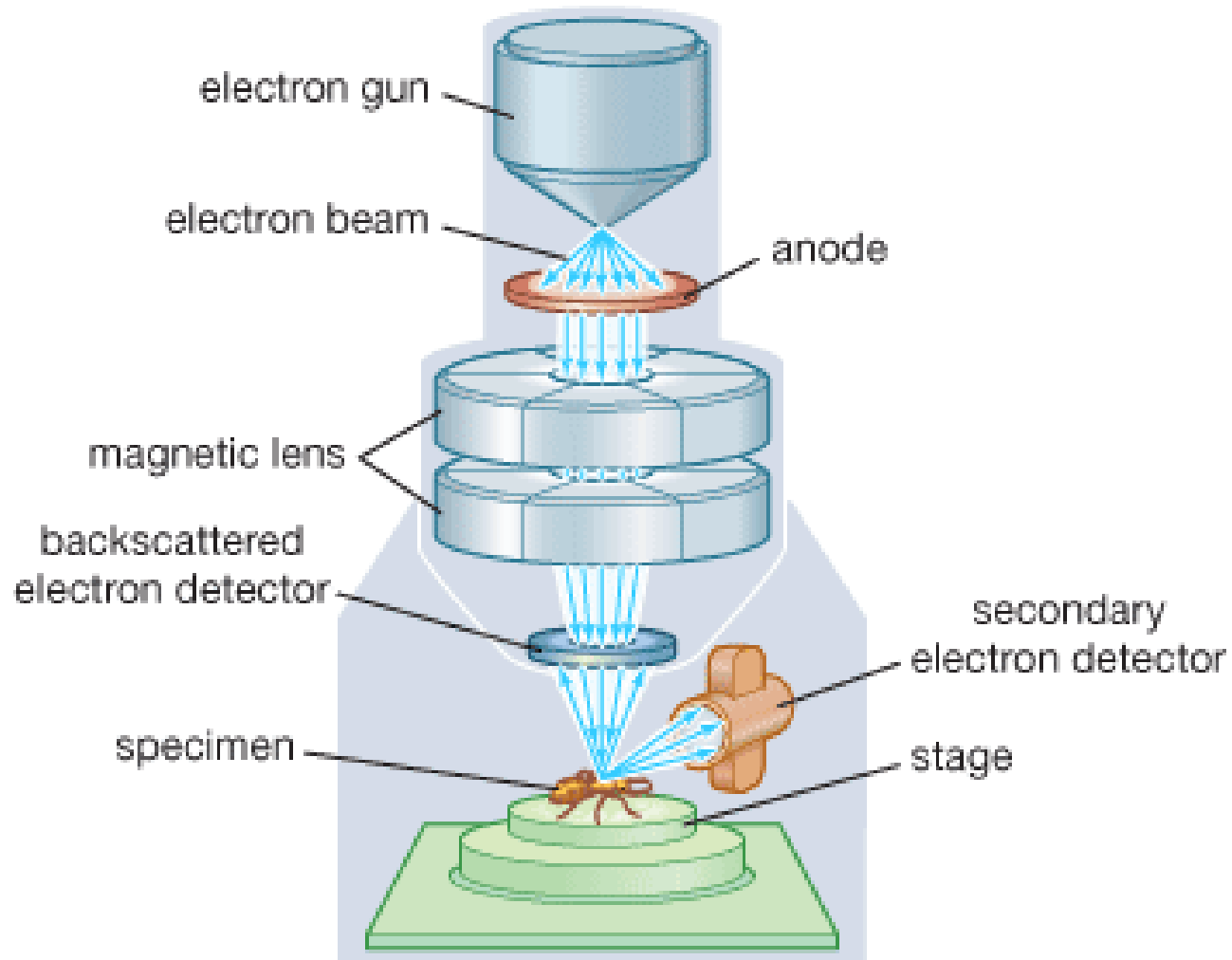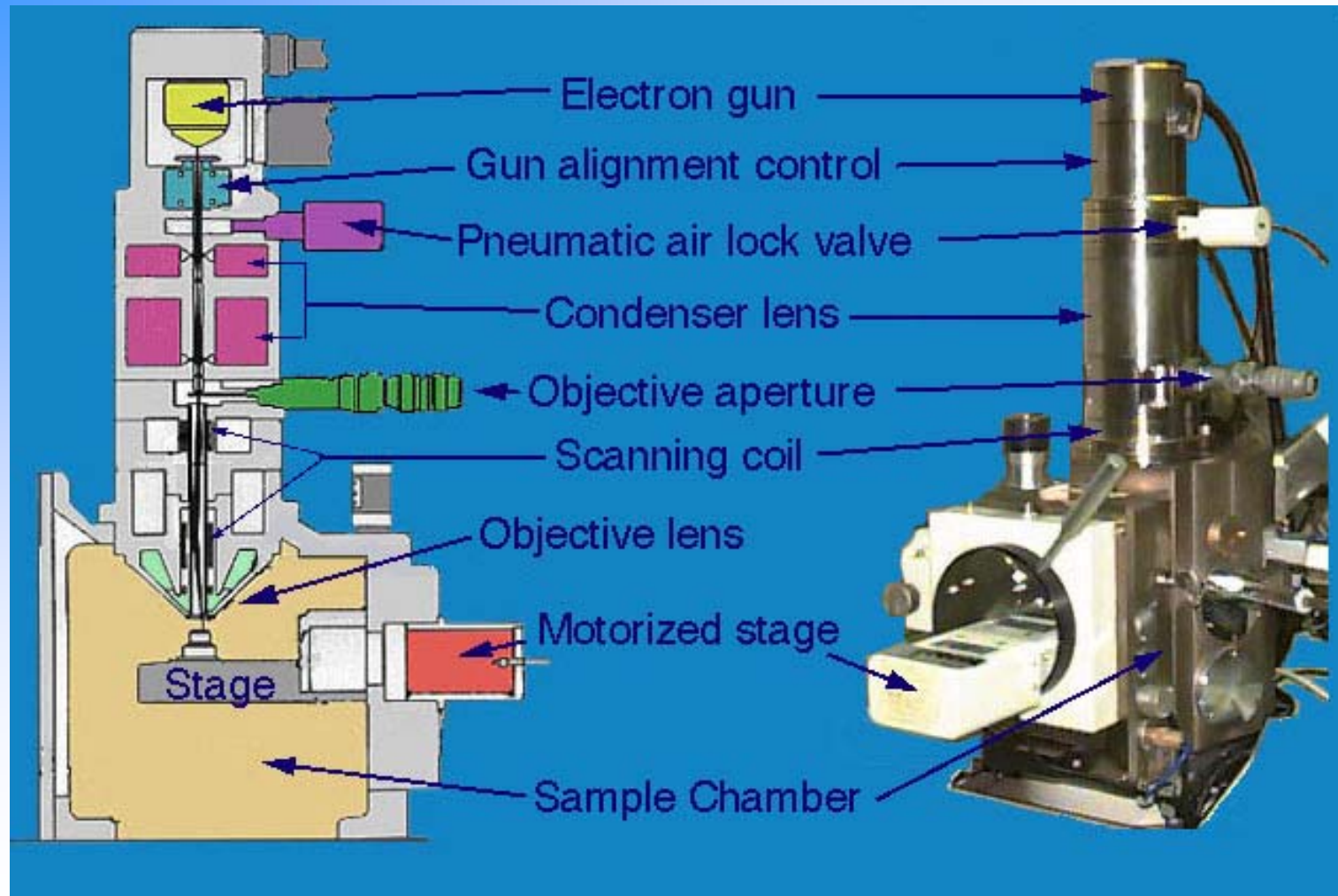# Scanning Electron Microscope (SEM)

- Electron microscope follows the same ideas of optical microscope, but uses electrons instead of light;
- "Lens" here are not the optical materials (like glass), but electrical field.
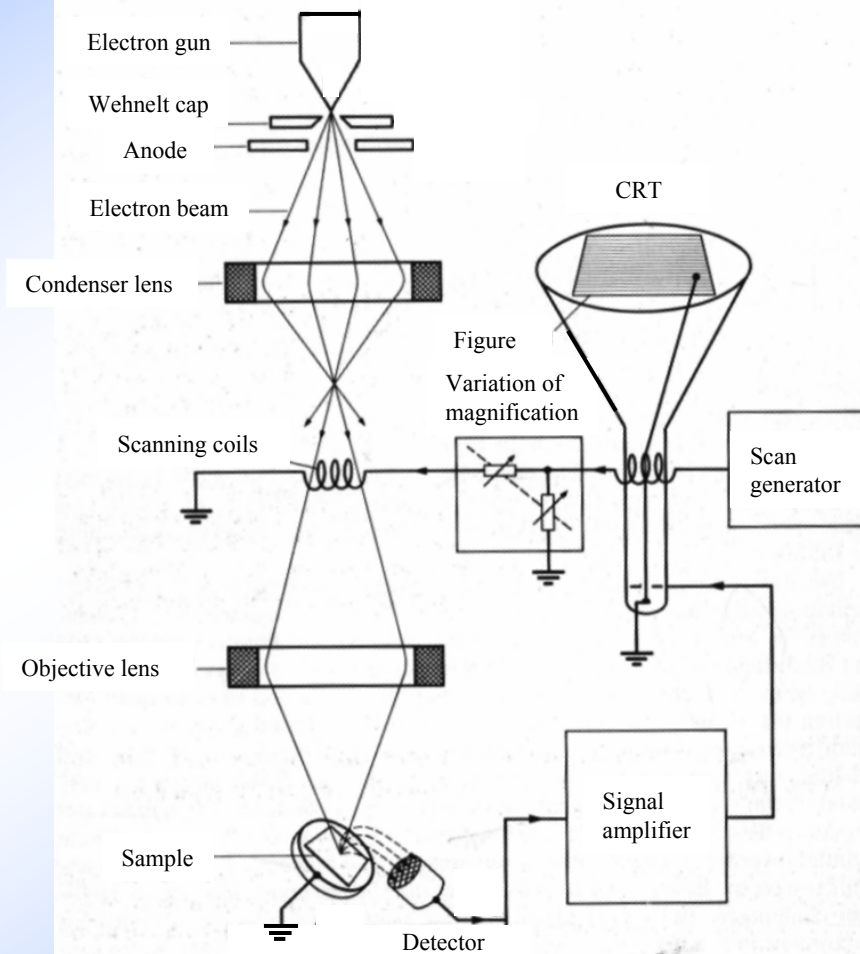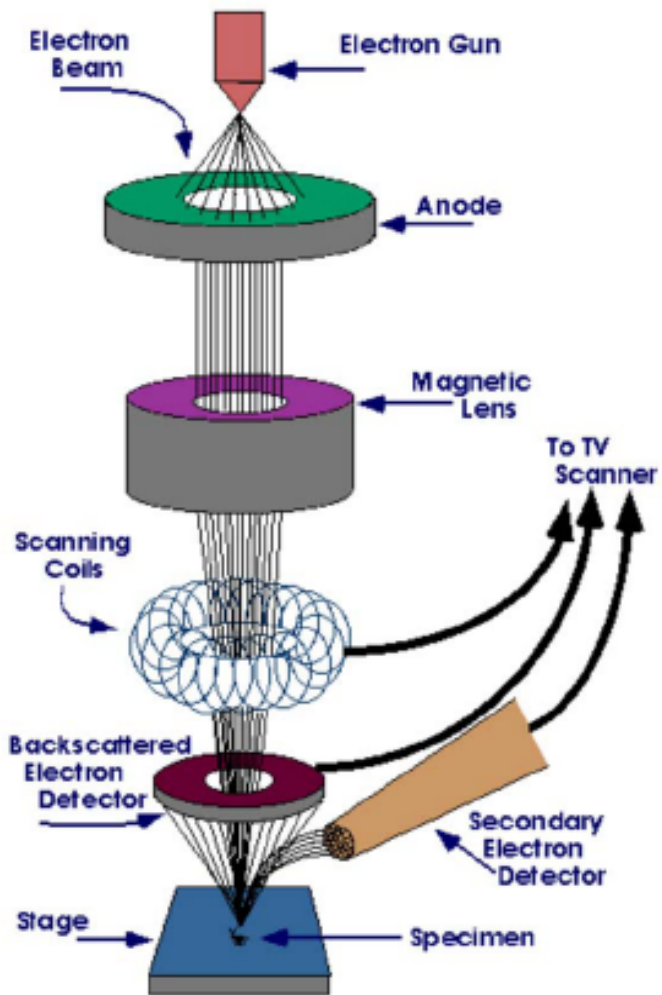
SEM: electron optical column

Scheme of Scanning Electron Microscope

Basic components of SEM

Electron optics:
  1. condenser lens --- focusing the electron beam to the objective lens.
  2. objective lens --- responsible for the size of electron beam impinging on sample surface
  3. electromagnetic coils --- responsible for driving the raster scanning by deflecting the electron beam.

• Sample and sample holder:
  1. size --- centimeters.
  2. rotation --- sample can be rotated freely at three dimensions, x, y, z, to achieve imaging at all directions.
  3. conductivity --- required for sample to be measured. For non-conducting samples, like biological specimens, metallic coating is required.

• Transducers (detectors):
  1. scintillation device --- doped glass or plastic target that emits a cascade of visible photons when struck by electrons.
  2. semiconductor transducers --- when struck by electrons, electron-hole pairs are generated, thus increasing the conductivity.

Basic signals of SEM

The electrons interact with the atoms at or close to sample surface produces signals that contain information about the sample's surface topography, composition, and other properties such as electrical conductivity. The types of signals produced by an SEM include secondary electrons, back-scattered electrons (BSE), characteristic X-rays, light (cathodo-luminescence), specimen current and transmitted electrons.

Secondary electrons: they are electrons generated as ionization products. They are called 'secondary' because they are generated by other radiation (the primary radiation). This radiation can be in the form of ions, electrons, or photons with sufficiently high energy, i.e. exceeding the ionization potential. Secondary electron detectors are common in all SEMs. A SEM with secondary electron imaging or SEI can produce very high-resolution images of a sample surface, revealing details less than 1 nm in size.

Back-scattered electrons (BSE): they are beam electrons that are reflected from the sample by elastic scattering. BSE are often used in analytical SEM along with the spectra made from the characteristic X-rays. Because the intensity of the BSE signal is strongly related to the atomic number (Z) of the specimen, BSE images can provide information about the distribution of different elements in the sample.

Characteristic X-rays: they are emitted when the electron beam removes an inner shell electron from the sample, causing a higher energy electron to fill the shell and release energy. These characteristic X-rays are used to identify the composition and measure the abundance of elements in the sample.

Advantages and Disadvantages of SEM

<u>Advantages:</u>

- Almost all kinds of samples, conducting and non-conducting (stain coating needed);

- Based on surface interaction --- no requirement of electron-transparent sample.

- Imaging at all directions through **x-y-z (3D)** rotation of sample.

<u>Disadvantages:</u>

- Low resolution, usually above a few tens of nanometers.

- Usually required surface stain-coating with metals for electron conducting.

Note: Specialized SEM works directly on non-conductive samples

- Nonconductive specimens tend to charge when scanned by the electron beam, and especially in secondary electron imaging mode, this causes scanning faults and other image artifacts. So, for usual SEM imaging, the samples must be coated with an ultrathin coating of electrically-conducting material, commonly gold.

- However, nonconducting specimens may be imaged uncoated using specialized SEM instrumentation such as the "Environmental SEM" (ESEM) or field emission gun (FEG) SEMs operated at low voltage, and low vacuum. field emission guns (FEG) is capable of producing high primary electron brightness and small spot size even at low accelerating potentials. In a regular SEM, the electron source (gun) is a tungsten filament cathode.

- At the Physics Department of UU, there is a FEI NanoNova SEM, with a field emitter electron source. Feature sizes of 2nm can be resolved. There is a low vacuum feature which allows imaging of non-conducting samples (biological samples need no special preparation).

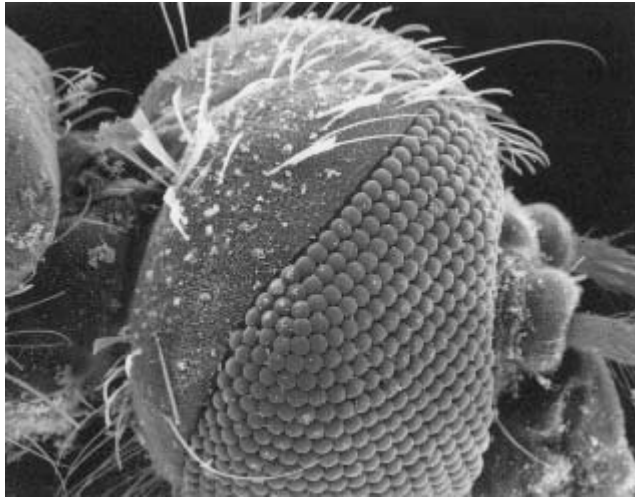# SEM images conducting and semiconducting materials
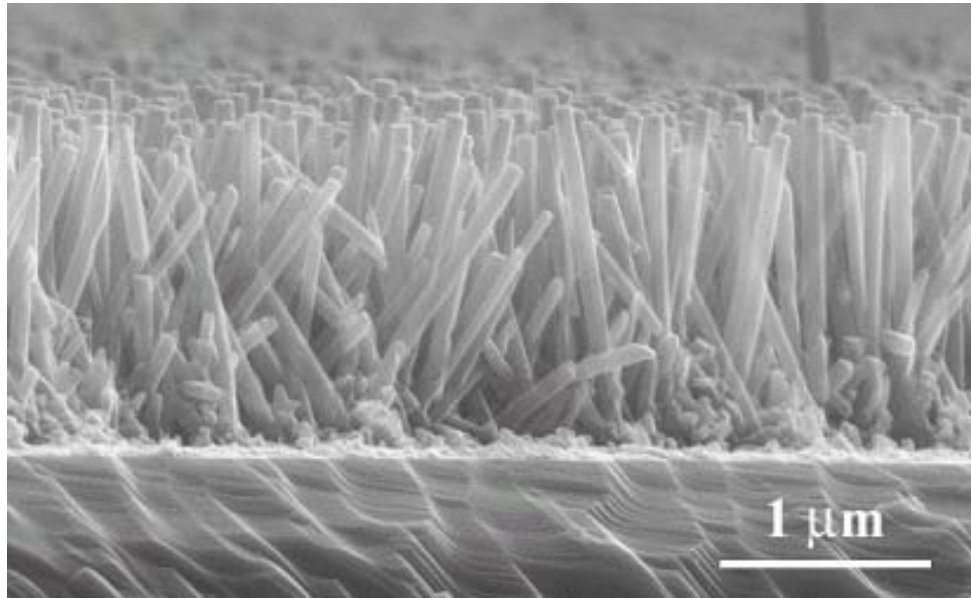
ZnO nano-wires
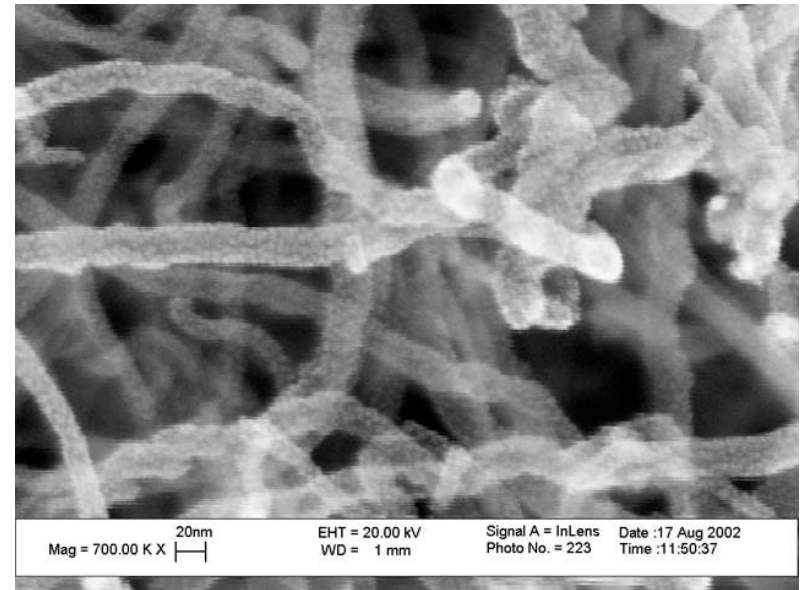
Carbon nano-tubes
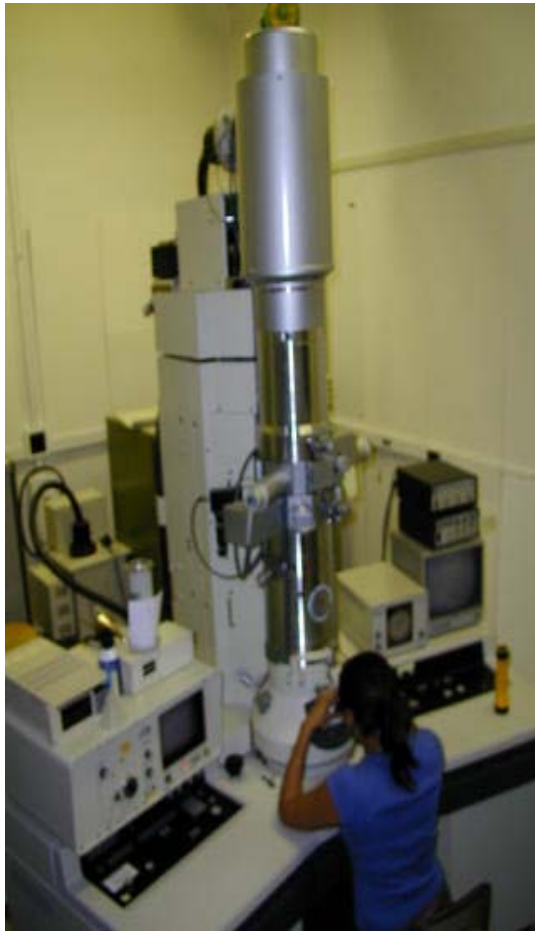
SEM images of bio-species

The head of a mosquito is mostly eye. The eyes are compound eyes, made up of many tiny lenses.
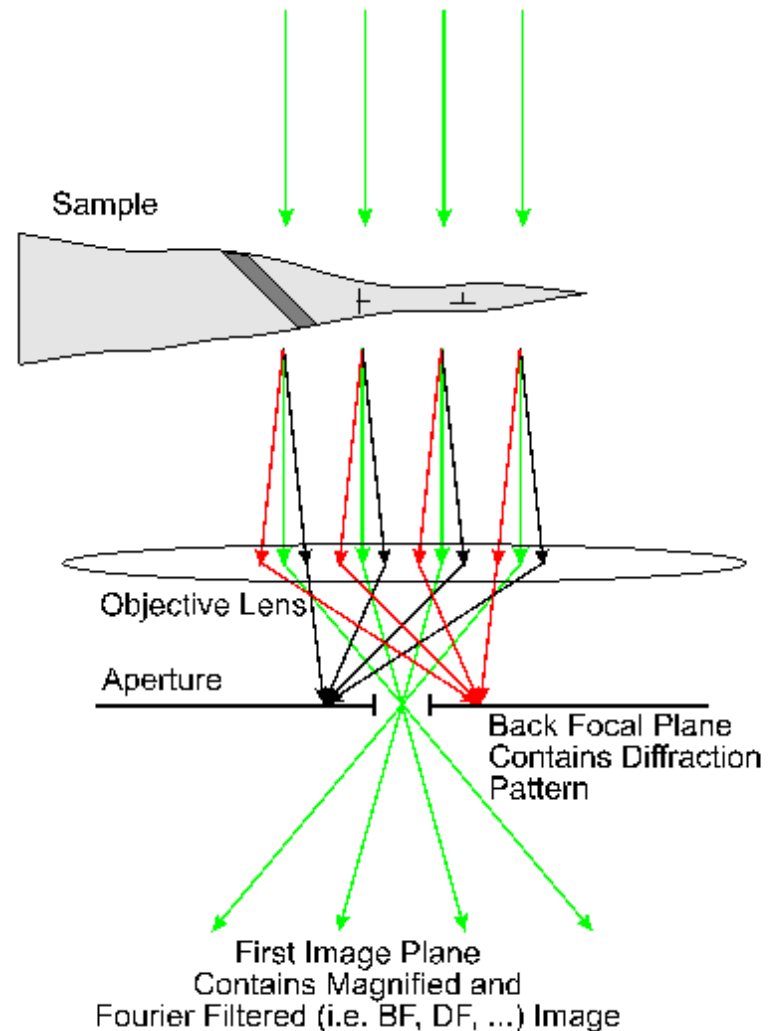

Claw of a Black Widow spider. The claw has three hooks, the middle one used to work the silk.

Transmission electron microscopy (TEM)

Hitachi H9000 UHR TEM:
a dedicated HREM TEM,
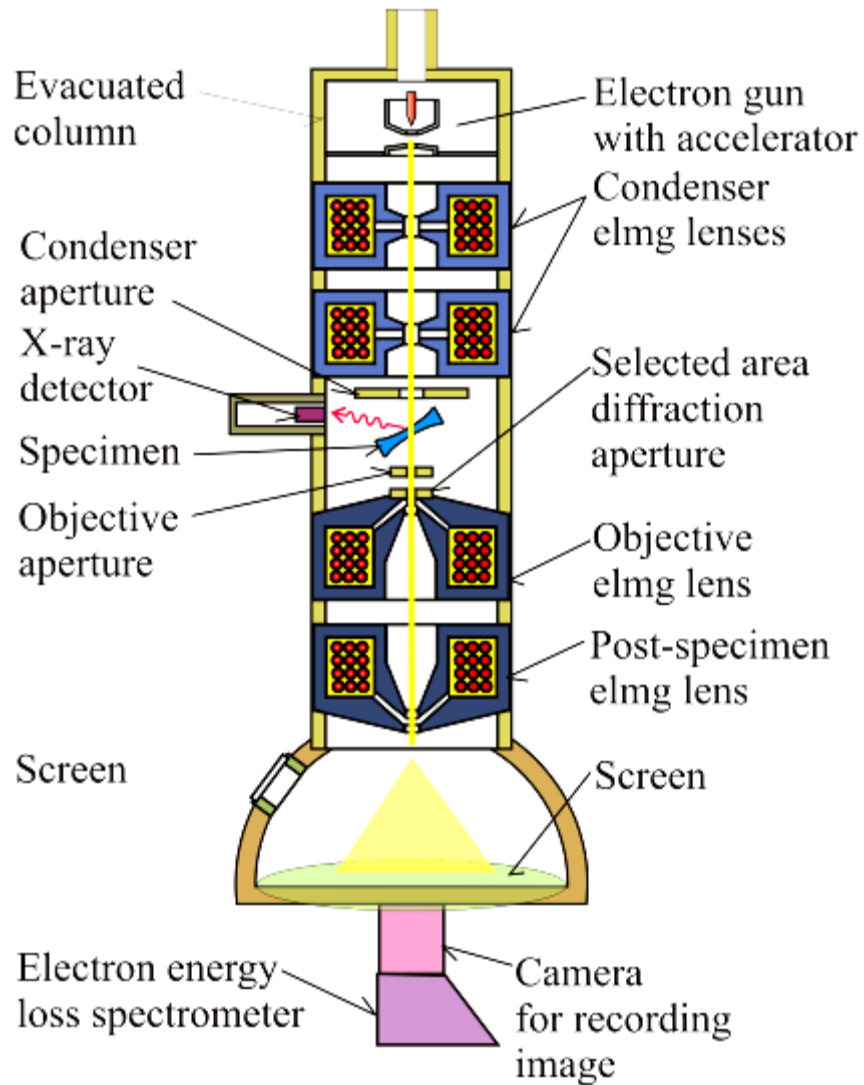capable of 0.18nm point
resolution operating at 300kV

Sample

Objective Lens

Aperture

Back Focal Plane
Contains Diffraction
Pattern

First Image Plane
Contains Magnified and
Fourier Filtered (i.e. BF, DF, ...) Image

Detector: CCD (2D imaging)

TEM is more complicated

Evacuated column
Electron gun with accelerator
Condenser elmg lenses
Condenser aperture
X-ray detector
Selected area diffraction aperture
Specimen
Objective aperture
Objective elmg lens
Post-specimen elmg lens
Screen
Screen
Electron energy loss spectrometer
Camera for recording image

What is TEM?

- In transmission electron microscopy (TEM), a beam of highly focused electrons are directed toward a thinned sample (<200 nm). Normally no scanning required --- helps the high resolution, compared to SEM.

- These highly energetic incident electrons interact with the atoms in the sample producing characteristic radiation and particles providing information for materials characterization.

- Information is obtained from both deflected and non-deflected transmitted electrons, backscattered and secondary electrons, and emitted photons.

Advantages and Disadvantages of TEM

<u>Advantages:</u>

- High resolution, as small as 0.2 nm.

- Direct imaging of crystalline lattice.

- Delineate the defects inside the sample.

- No metallic stain-coating needed, thus convenient for structural imaging of organic materials,

- Electron diffraction technique: *phase identification, structure and symmetry determination, lattice parameter measurement, disorder and defect identification*

<u>Disadvantages:</u>

- To prepare an electron-transparent sample from the bulk is difficult (due to the conductivity or electron density, and sample thickness).

SEM: General Information

Accelerating voltage 100 V – 30000 V

Resolution 0.7 nm – 5 nm (depends on the kind of electron gun)

Samples should be electrically conductive and

also be mounted electrically conductive on the holder

Size of sample is limited by the specimen chamber

Kind of Information

Topography
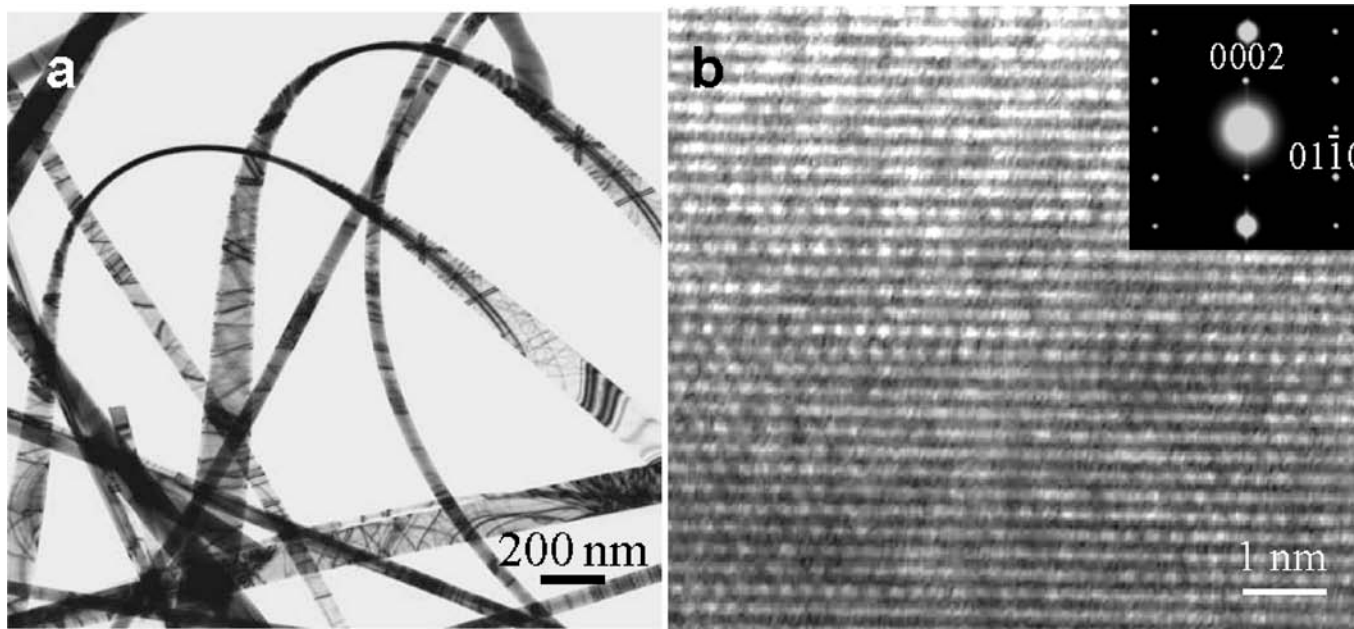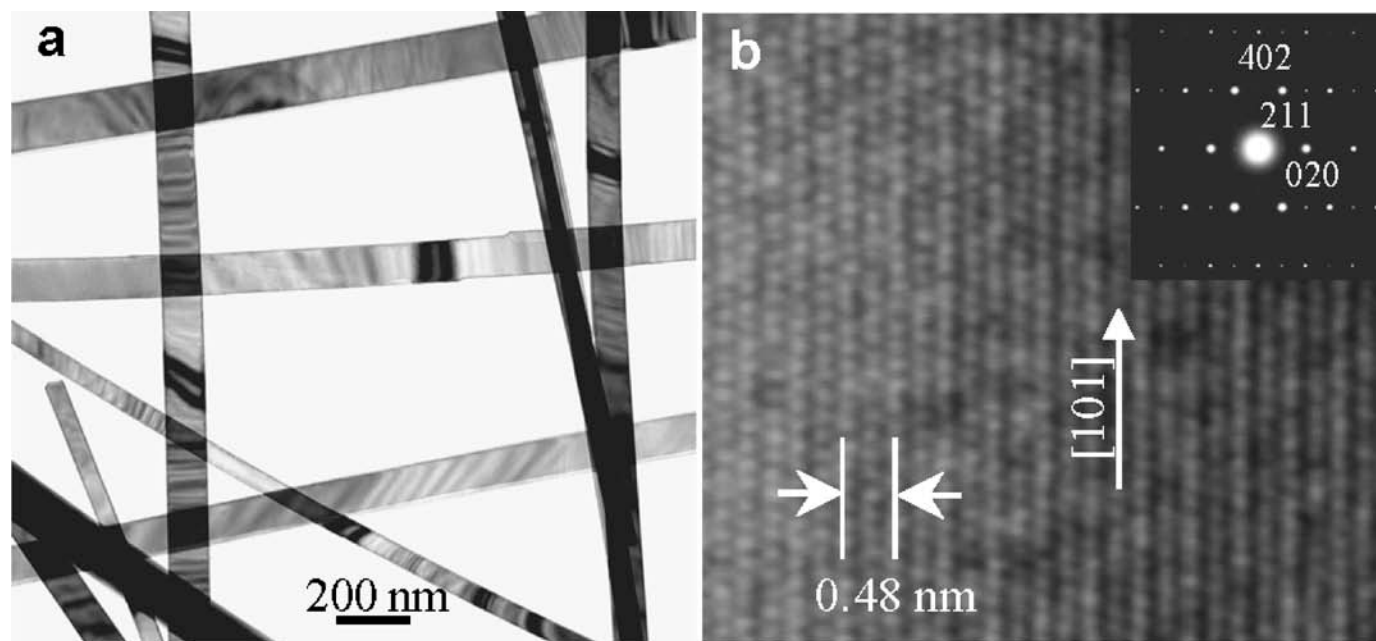
Morphology

Composition

Crystallographic structure

(*a*) Transmission electron microscopy (TEM) image of the as-synthesized **ZnO** nanobelts. (*b*) High-resolution TEM image recorded with the incident electron perpendicular to the top surface of the nanobelt.

Z. L. Wang, Annual Review of Physical Chemistry, Vol. 55: 159-196

TEM images: nanobelt

(*a*) Transmission electron microscopy (TEM) image of the as-synthesized **SnO2** nanobelts. (*b*) High-resolution TEM image recorded with the incident electron perpendicular to the top surface of the nanobelt.
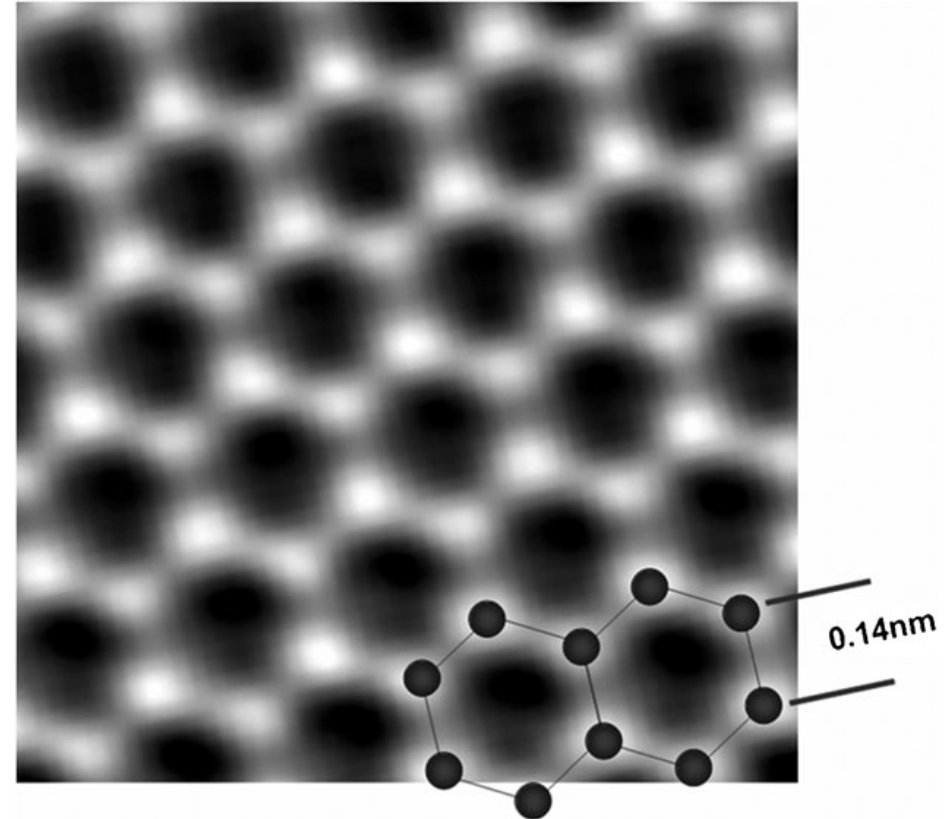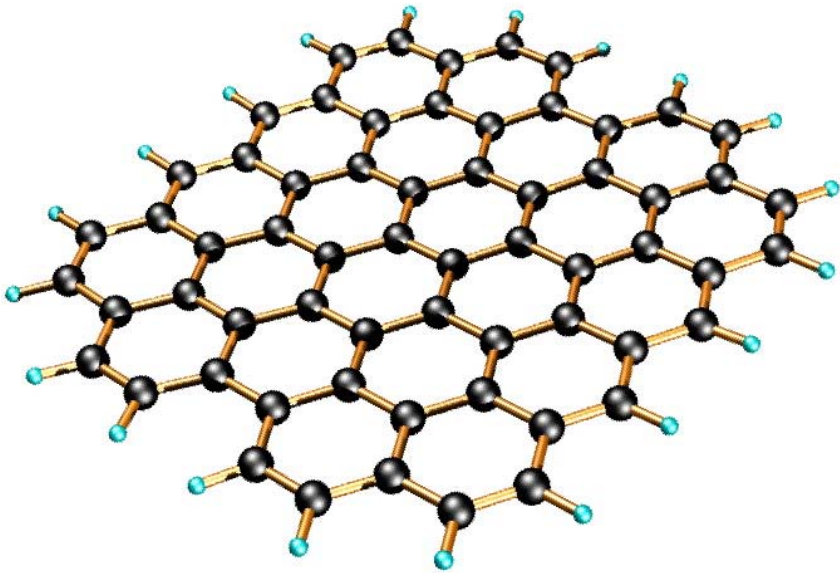
TEM images: nanotube
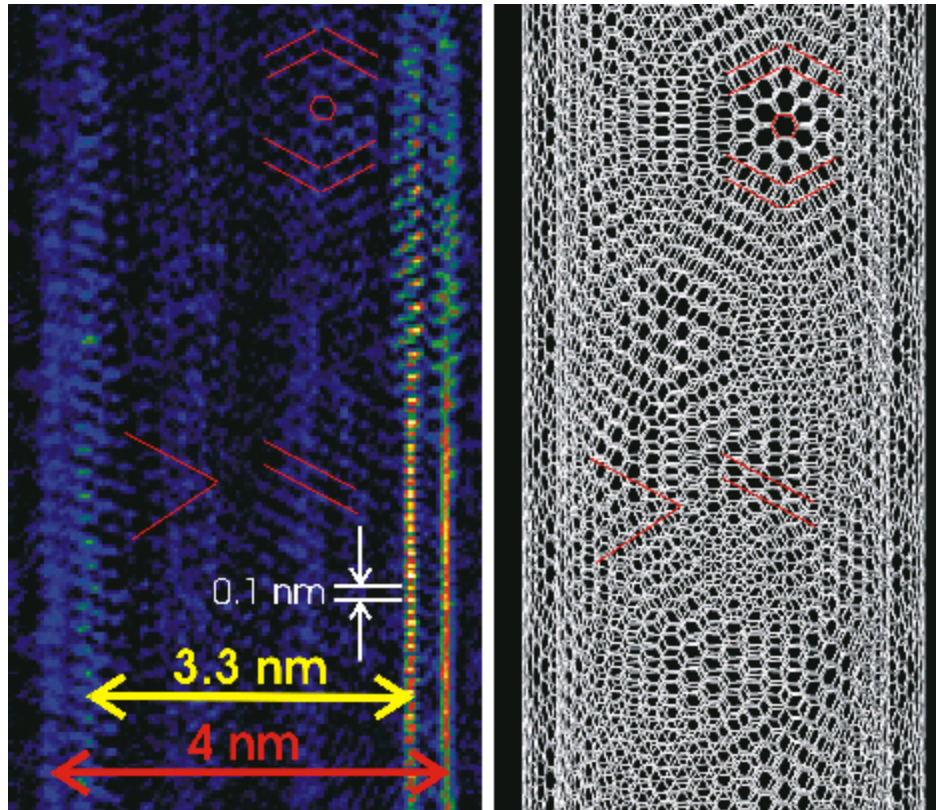
Imaging single-wall carbon nanotube

TEM imaging of graphene

0.14nm

Scanning Probe Microscopy (SPM)

Double functions: **scanning** and **probing**.

Scanning: piezo raster 2D (X-Y) scanning;

Probing: sharp tip mounted to a Z-scanner.

Tip Contour

Comparison between traditional optical and electron microscopes and SPM

	probe	Mechanism	Sample	Resolution
Traditional	Light/electron	Using properties of waves: diffraction, deflection, scattering	High vacuum chamber, Strict sample pre-treatment (e.g. conducting stain) required	Å – μm, good for X-Y lateral imaging
SPM	Tip	Using interaction between tip and sample: mechanic, electrostatic, meganetic.	Usually under ambient conditions, Highly flexible with other techniques	Å – nm, good for Z-height measurement, thus topography imaging

Note:
SPM cannot replace electron microscopes, but complementary each other.