

## Lecture 3 Brief Overview of Traditional Microscopes

- Optical Microscope;
- Scanning Electron Microscope (SEM);
- Transmission Electron Microscope (TEM);
- Comparison with scanning probe microscope (SPM)

# General philosophy

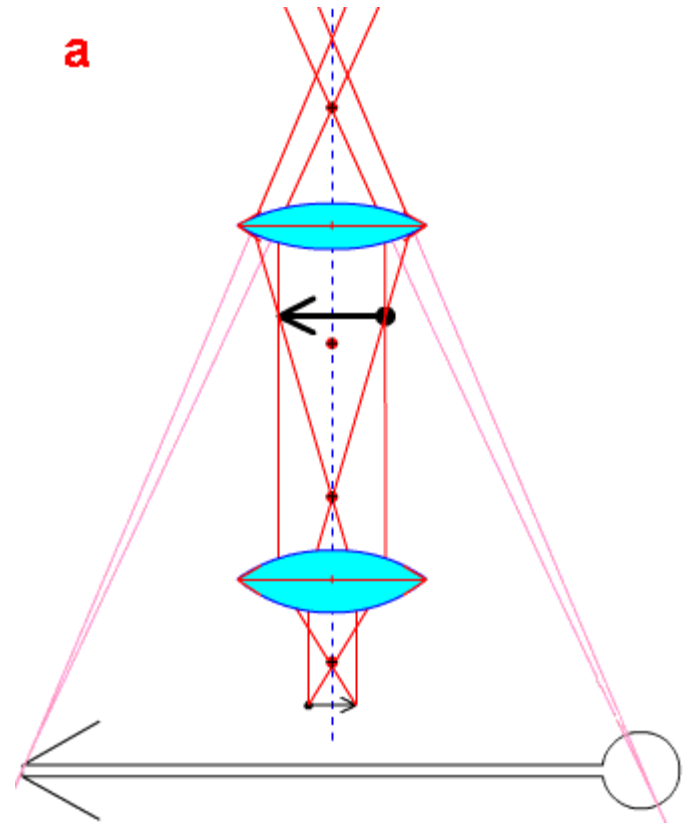
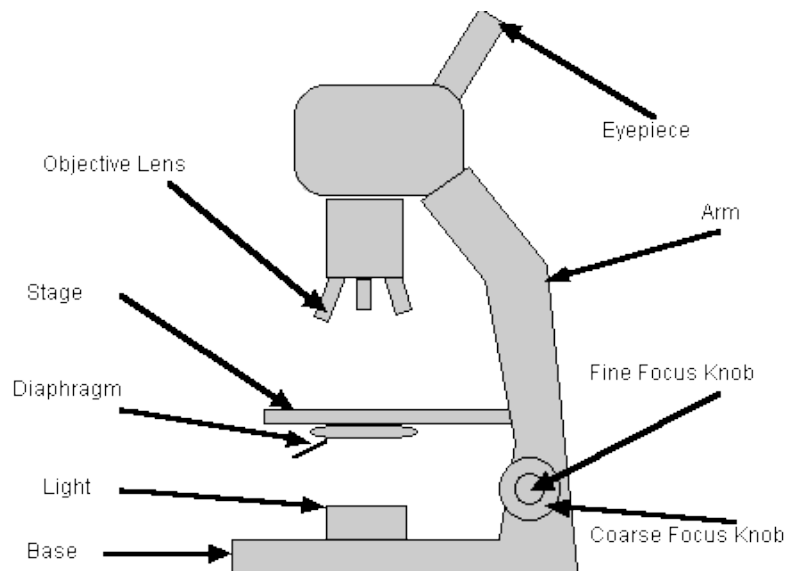
Human beings use two kinds of means to gauge objectives:

**Seeing** --- through eyes (light) ↔ optical microscope  
↔ electronic microscope (*higher resolution due to short wavelength*);

**Touching** --- through hands (probe) ↔ SPM.

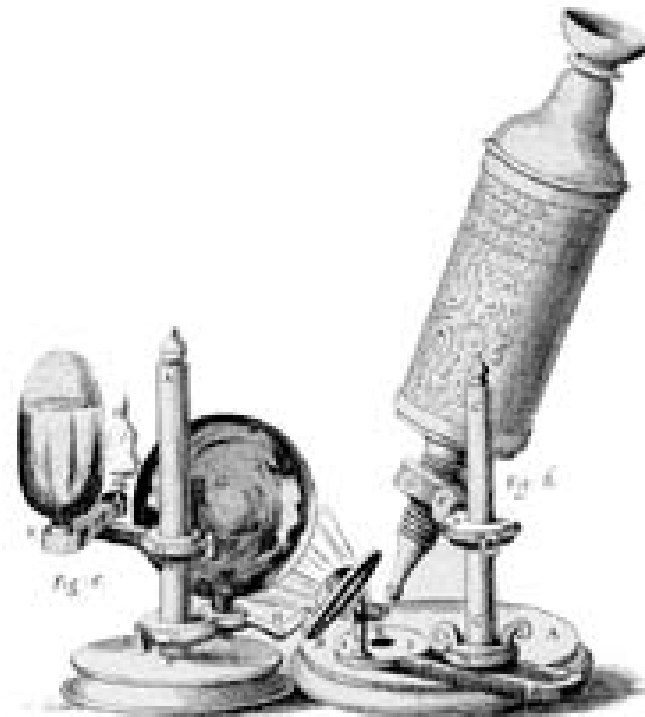
Converting wavelength (nm) to electron energy (eV):  $1240.7/\lambda(\text{nm})$ , for example, a 500 nm light corresponds to  $1240.7/500 = 2.48$  eV

# Principles of an optical microscope

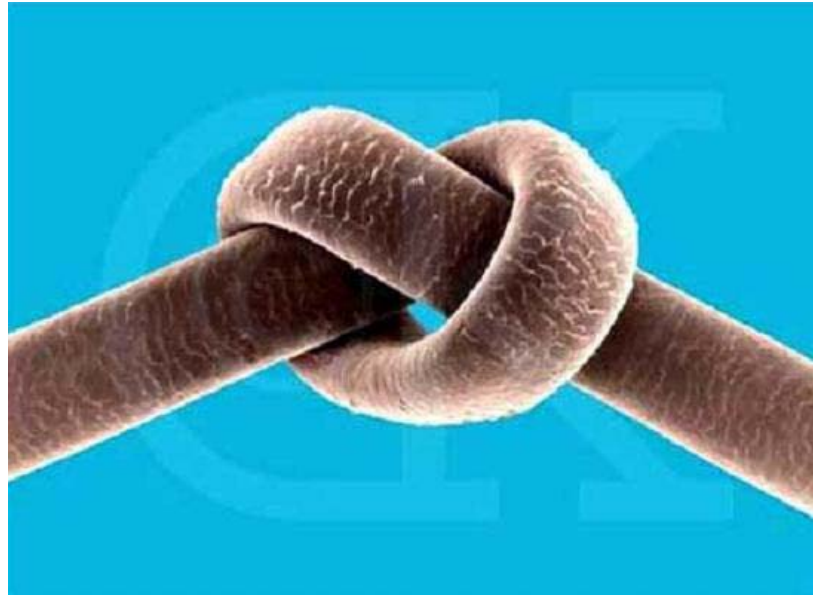


Development of microscope takes us back to 400 years ago

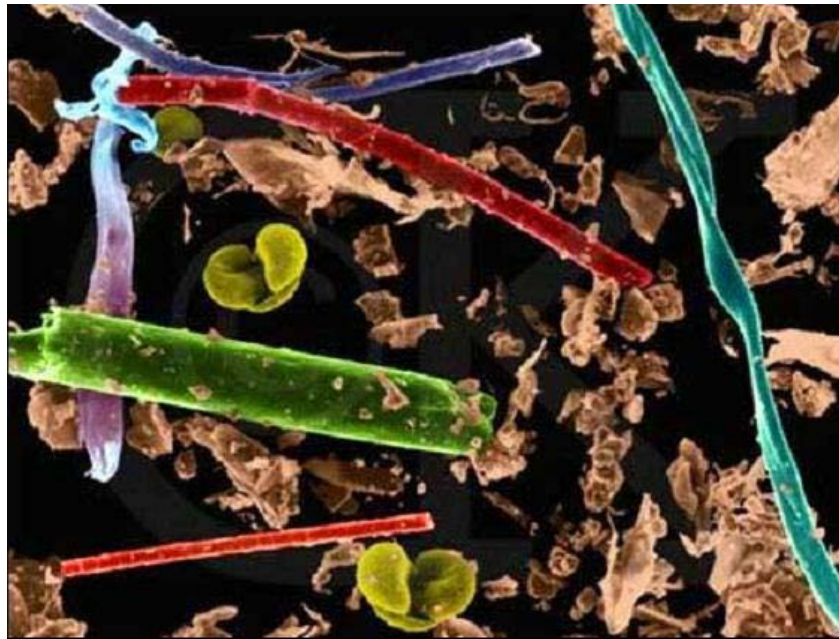
## Robert Hooke's "Micrographia" (1665)



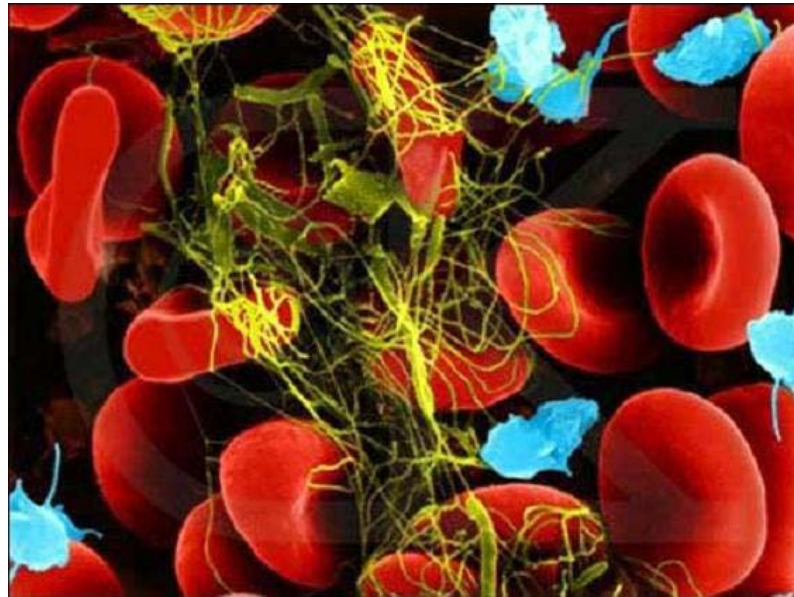
# A hair knot



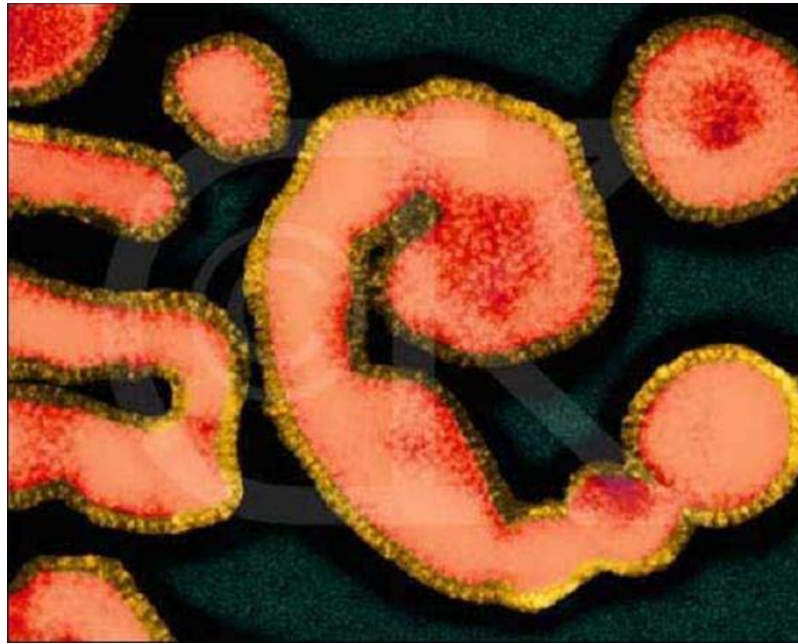
# Room dusts



# Red blood cell

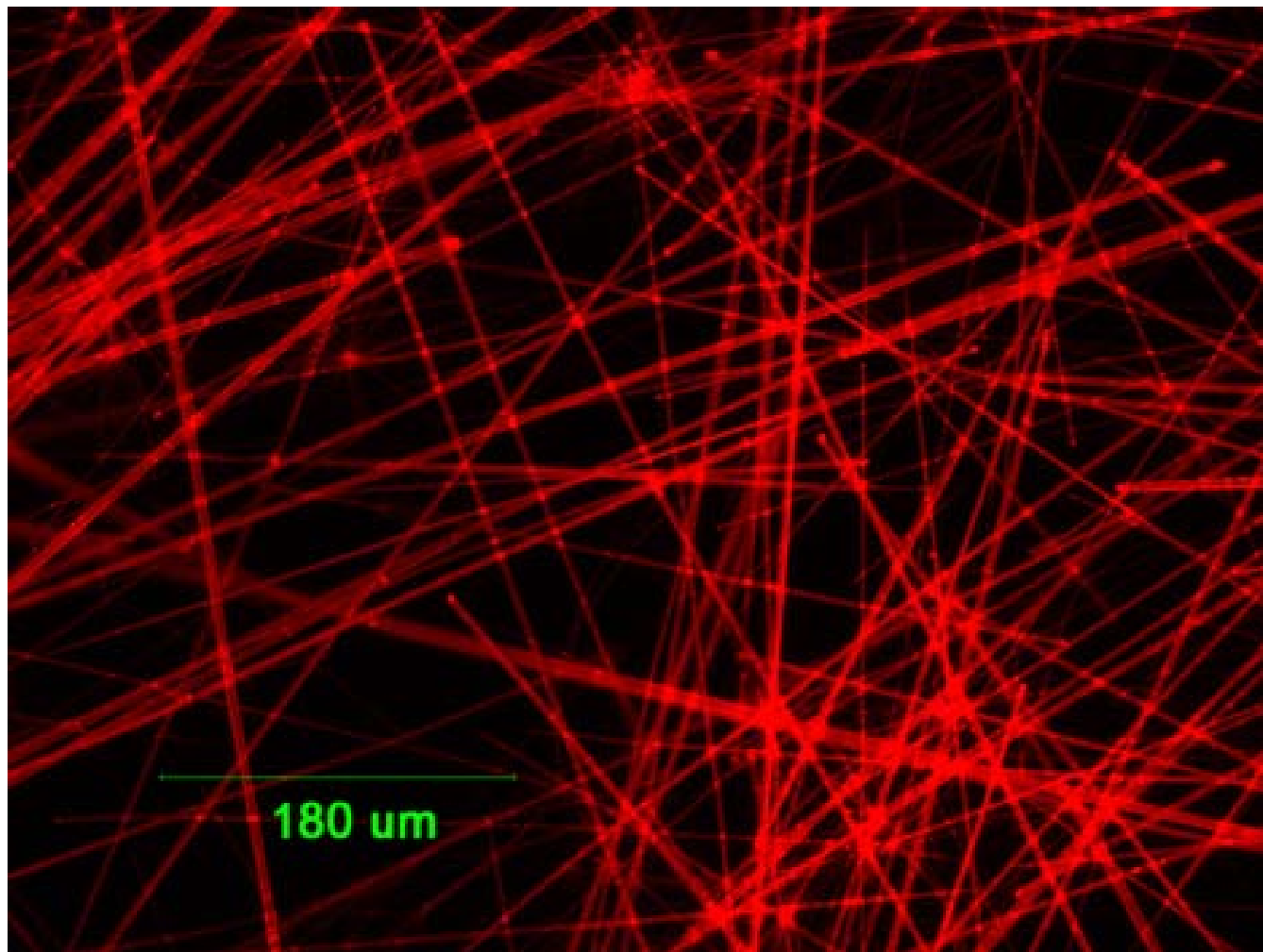


# Flu virus





# Organic nanobelts with strong emission



# 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  $\lambda/2NA$  (NA is the numerical aperture of lens, usually  $\sim 1.0$ ): 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  $\lambda/2 = 0.3$  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.

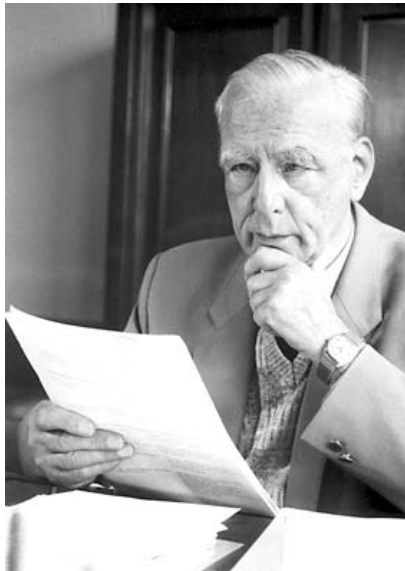
# History of Electronic Microscope

- 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.

# 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

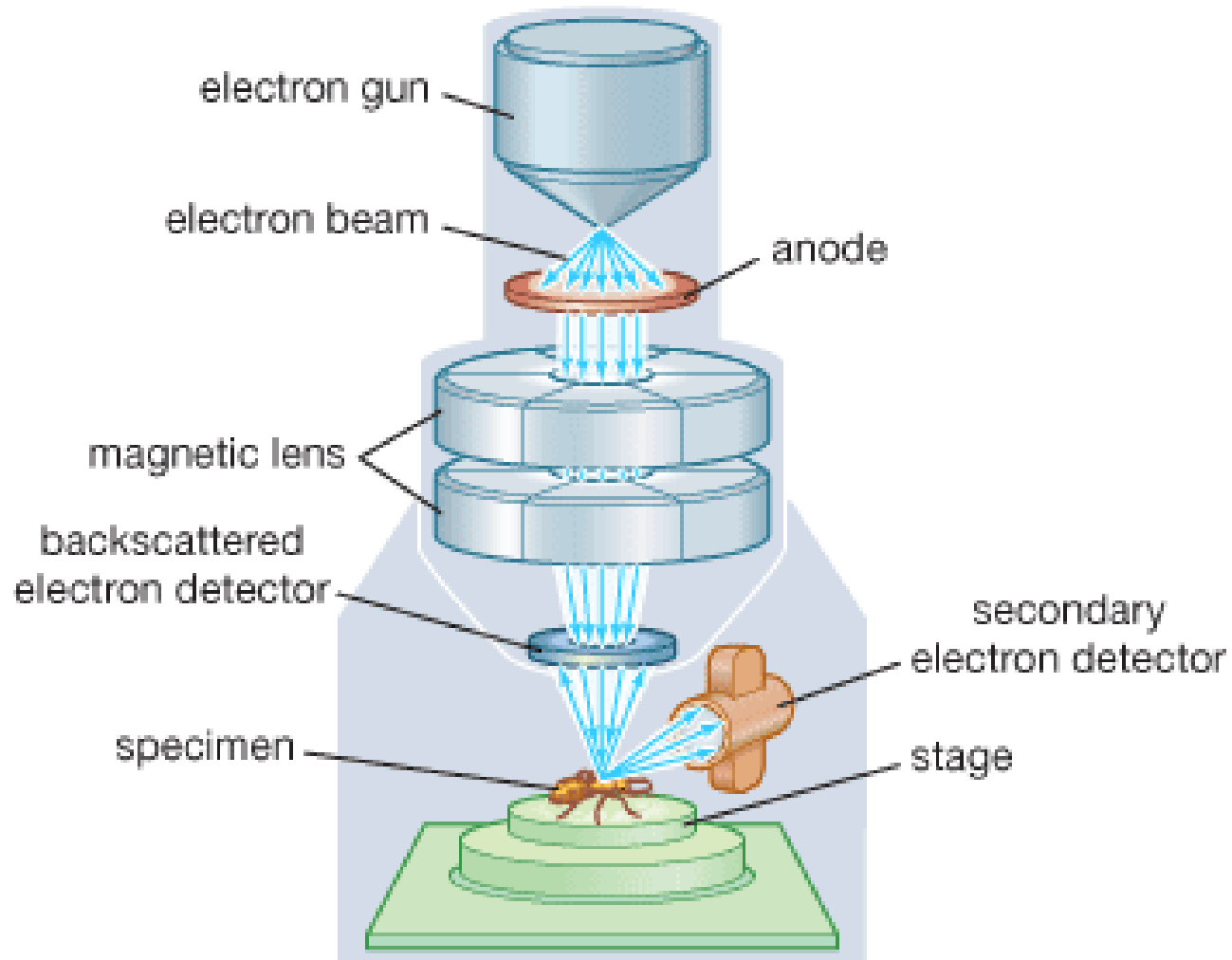
**Died:** 27 May 1988, West Berlin, Germany

**Affiliation at the time of the award:** Fritz-Haber-Institut der Max-Planck-Gesellschaft, Berlin, Federal Republic of Germany

# What is SEM?

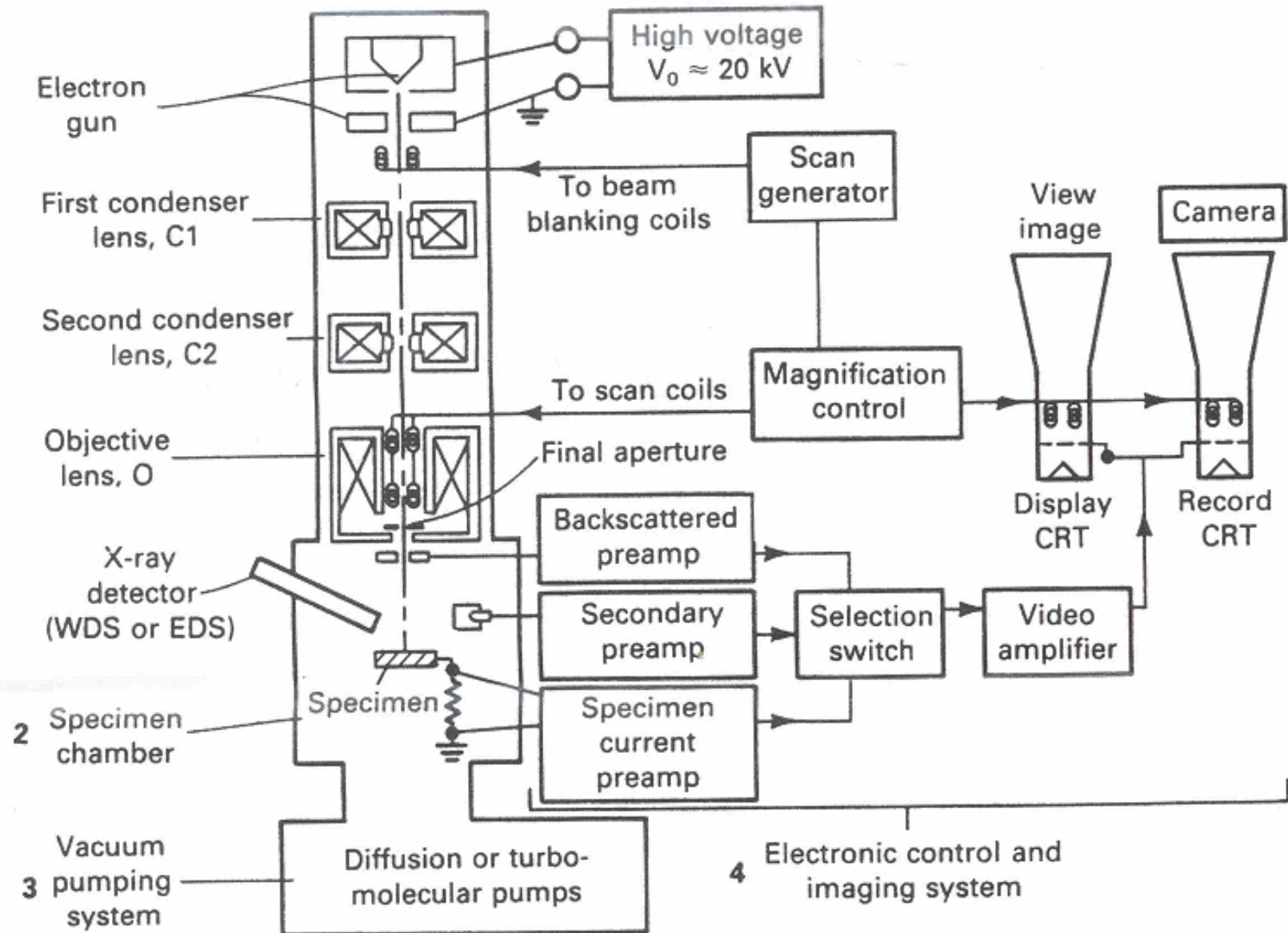
- In scanning electron microscopy (SEM) an electron beam is focused into a small probe and is rastered across the surface of a specimen.
- Several interactions with the sample that result in the emission of electrons or photons occur as the electrons penetrate the surface.
- These emitted particles can 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 shape of the sample.
- The resolution is determined by beam diameter.

# 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.

# Detailed working diagram of SEM





# 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

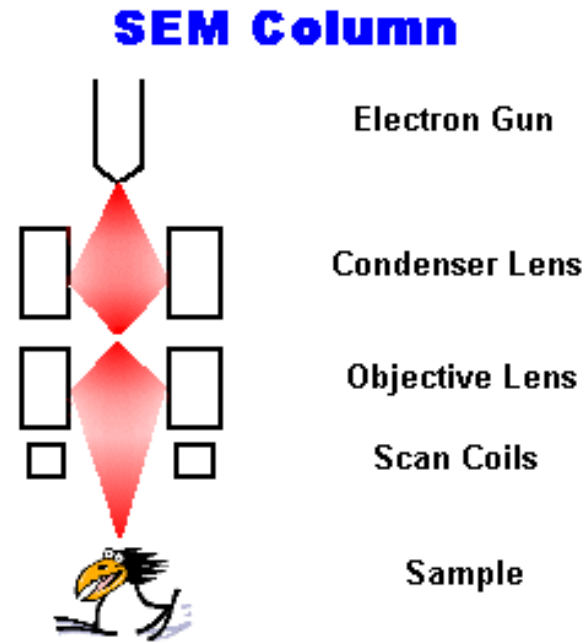
The electrons interact with the atoms at or close to sample surface produces signals that contain information about the sample's surface **topography**, **composition**, and other properties such as electrical **conductivity**. The types of signals produced by an SEM include secondary electrons, back-scattered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

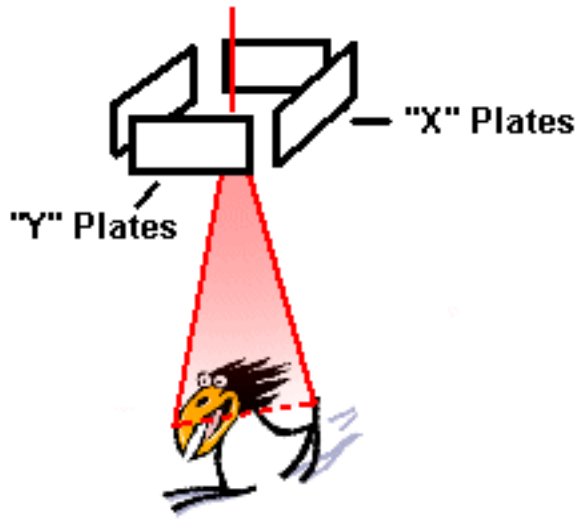
**Characteristic X-rays:** they are emitted when the electron beam removes an inner shell electron from the sample, causing a higher energy electron to fill the shell and release energy. These characteristic X-rays are used to identify the composition and measure the abundance of elements in the sample.

# Brief schemes showing how SEM works

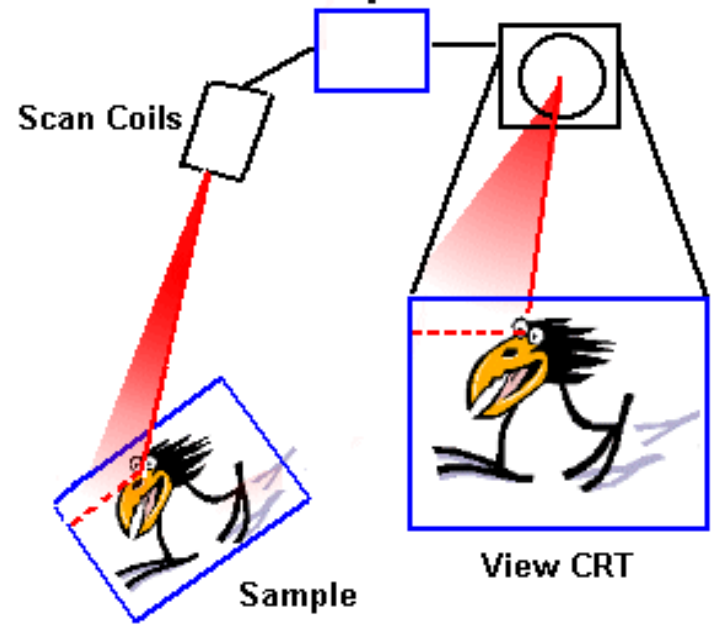


# Brief schemes showing how SEM works

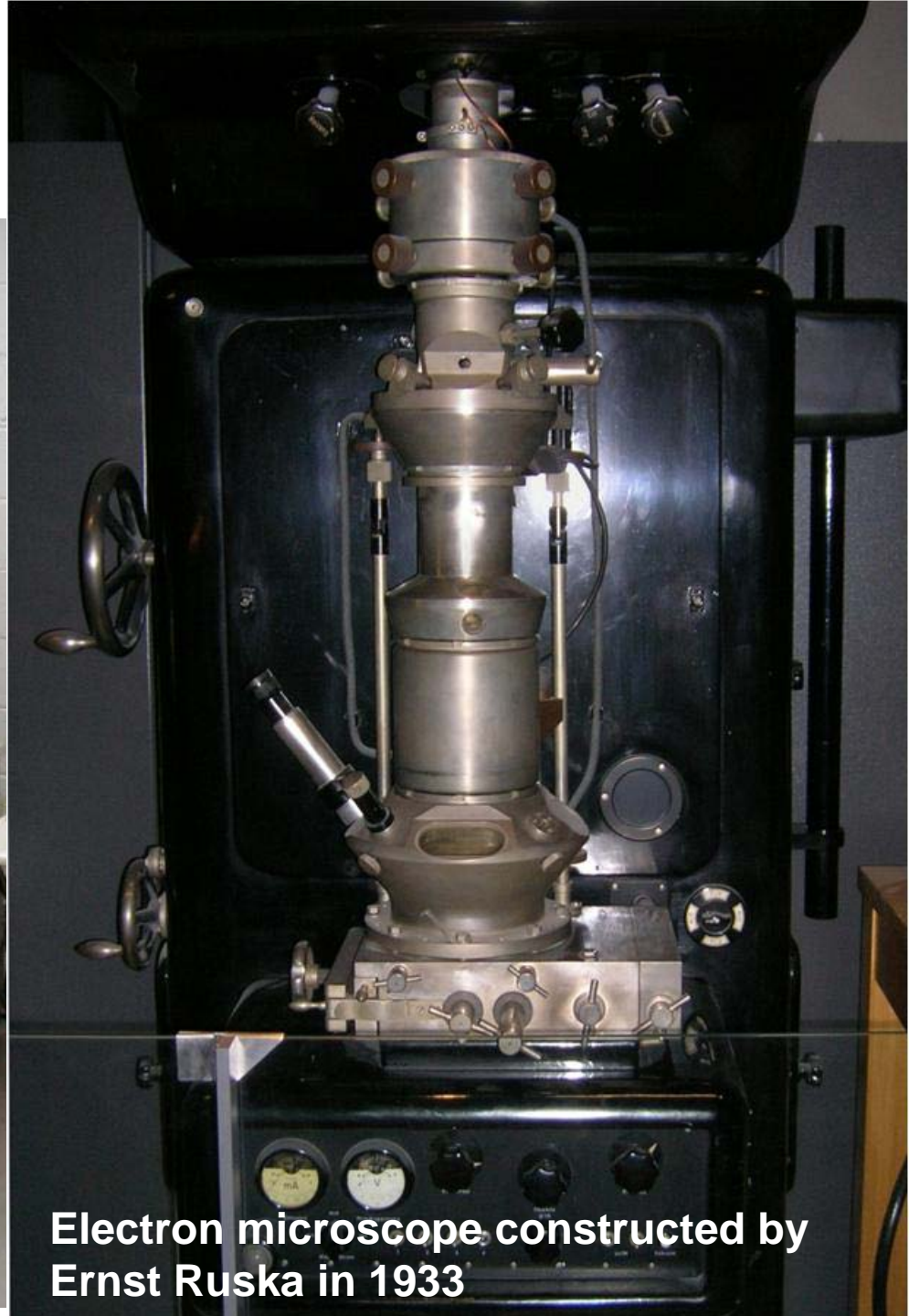
## Scan Coils



## Scan Generator



# Scanning Electron



**Electron microscope constructed by Ernst Ruska in 1933**

# Advantages and Disadvantages of SEM

## Advantages:

- 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.

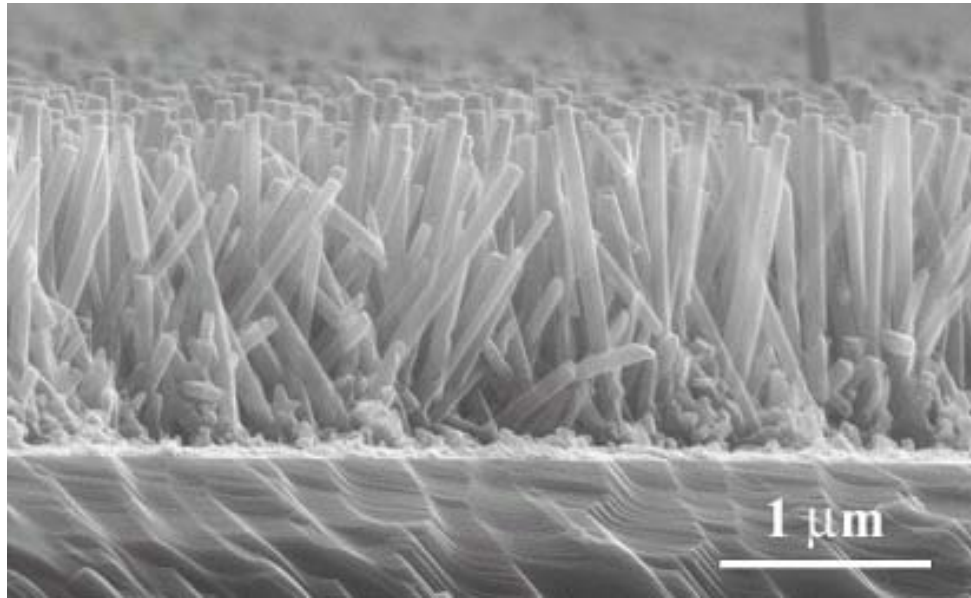
## Disadvantages:

- Low resolution, usually above a few tens of nanometers.
- Usually required **surface stain-coating** with metals for electron conducting.

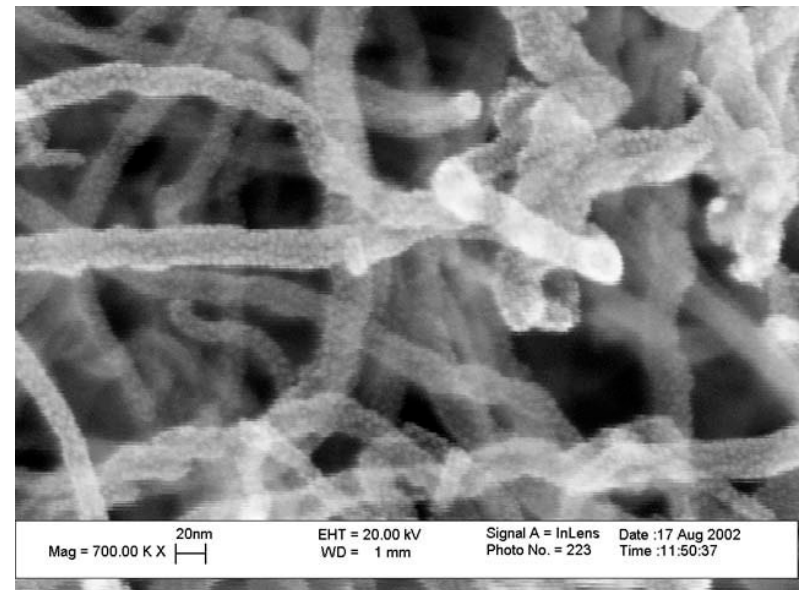
## Note: Specialized SEM works directly on non-conductive samples

- 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



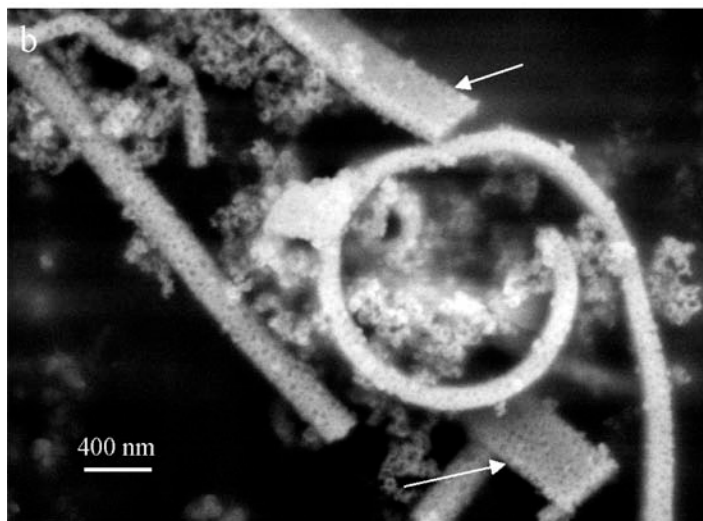
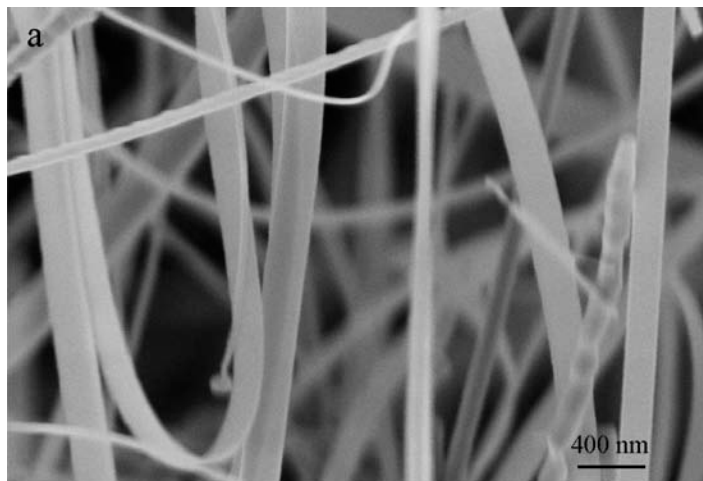
ZnO nano-wires



Carbon nano-tubes

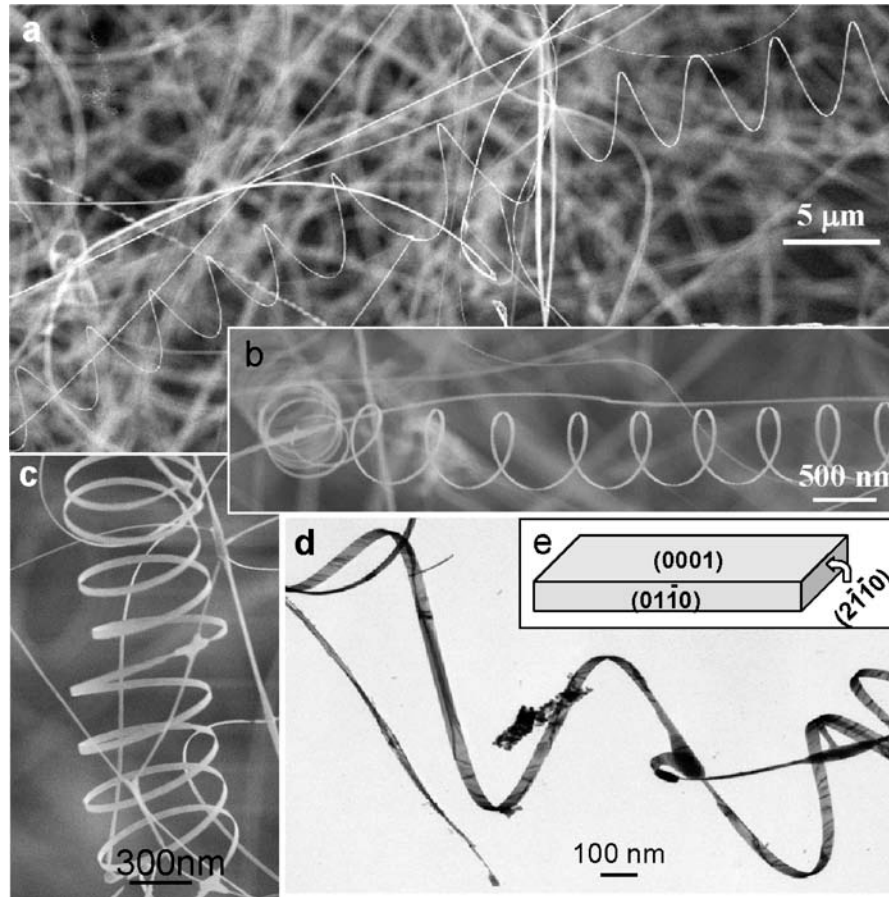


## Nanobelts



SEM images of (a) **ZnO** nanobelts and (b) the **ZnS** nanobelts converted through chemical reaction with  $\text{H}_2\text{S}$ .

# Nanosprings



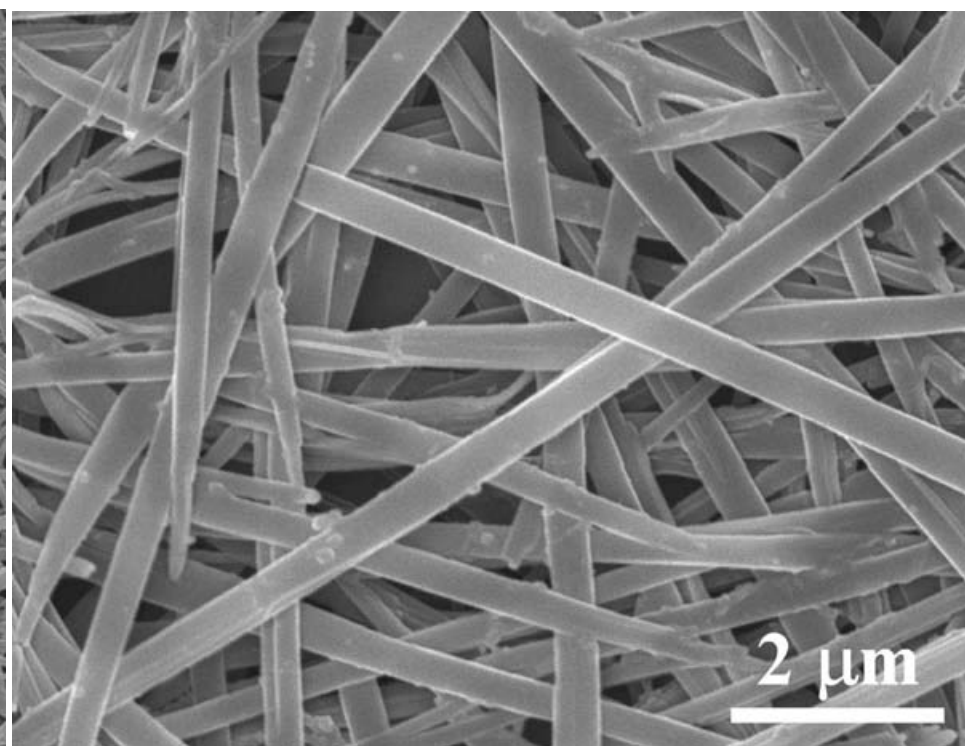
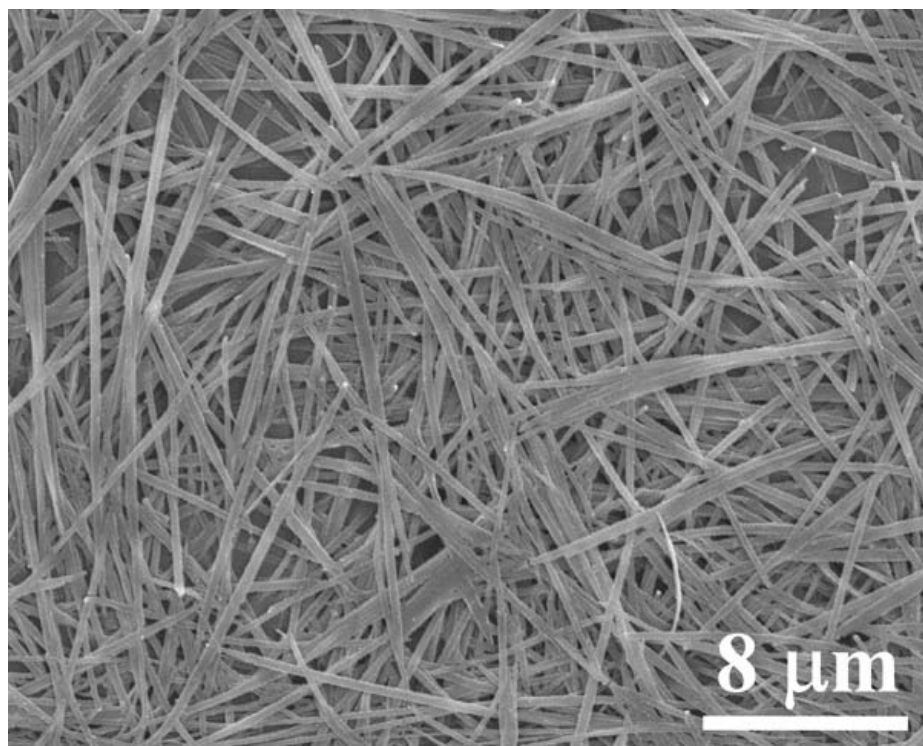
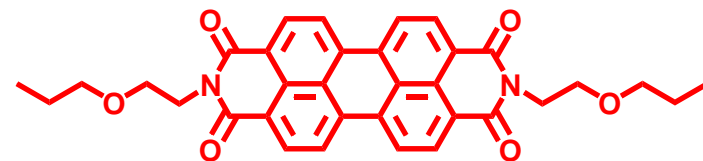
Nanosprings and nanorings of piezoelectric nanobelts. (a c) SEM images of the as-synthesized single-crystal **ZnO** nanobelts, showing helical nanosprings. The typical width of the nanobelt is 30 nm, and pitch distance is rather uniform. (d) TEM image of a helical nanospring made of a single-crystal ZnO nanobelt. (e) The structure model of the ZnO nanobelt.

# Nanosaw

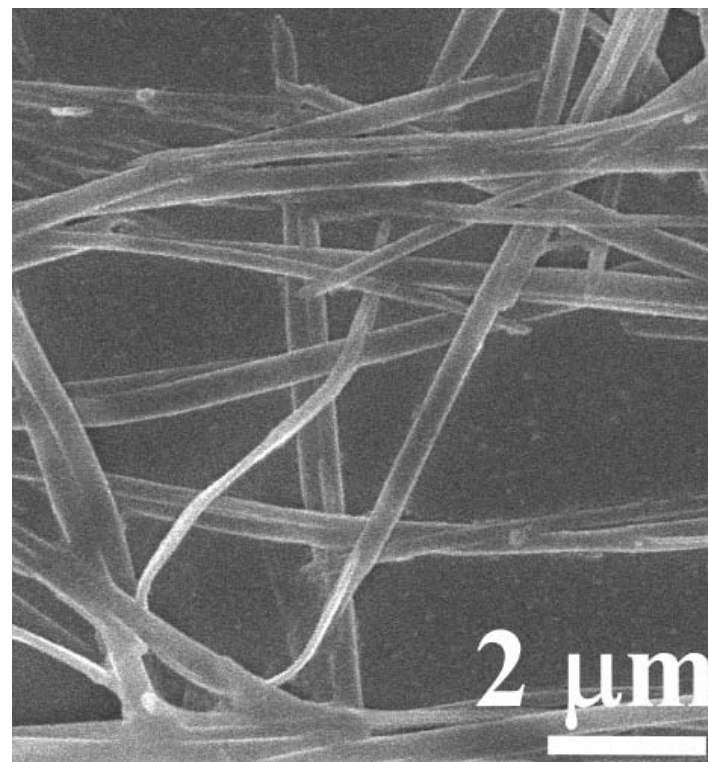
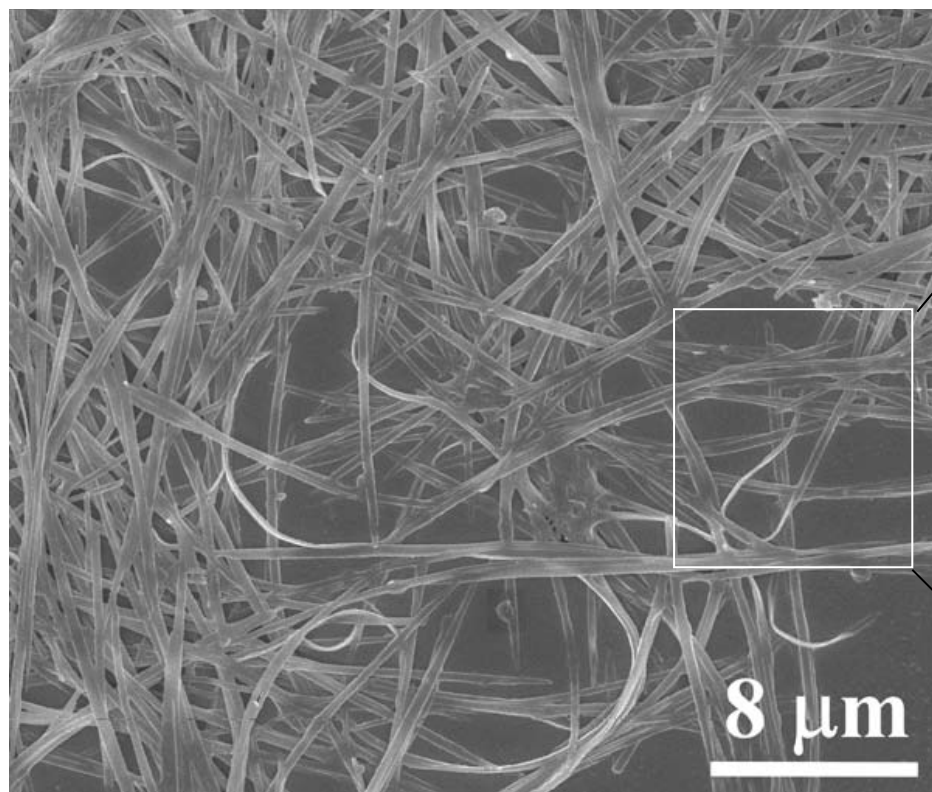


SEM image of comb-like nanostructure of **ZnO**, which is the result of surface polarization induced growth.

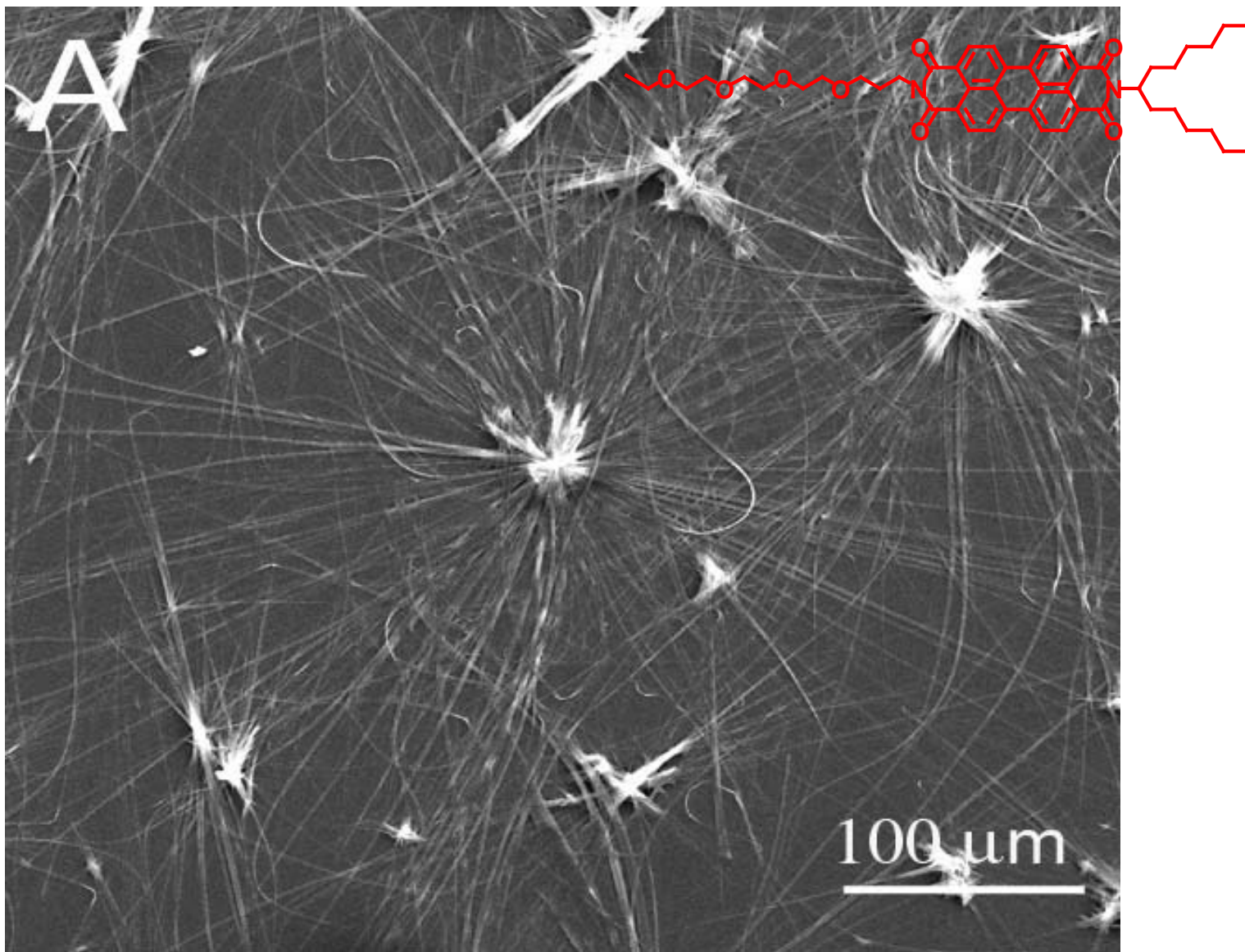
## Fabrication by simple injection into a poor solvent

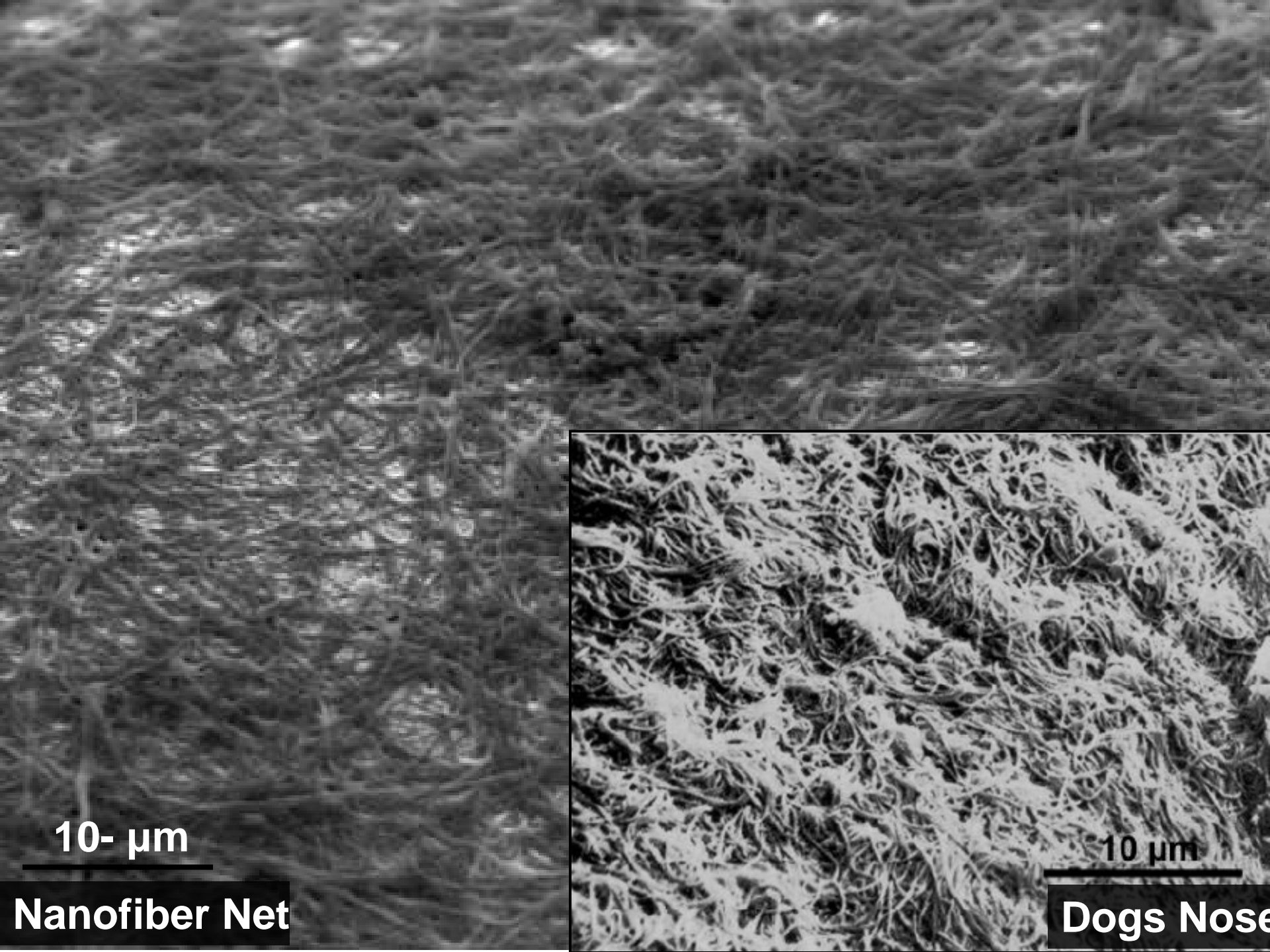


# Twisted Nanobelts



## Seeded growth of ultralong nanobelts





10-  $\mu\text{m}$

Nanofiber Net

10  $\mu\text{m}$

Dogs Nose

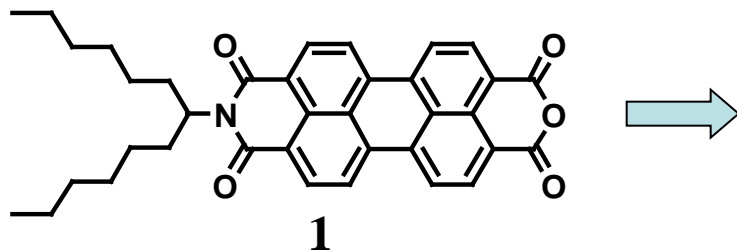


Video of nanofiber sensor response to 10 ug Meth simulant disperse in a filter paper

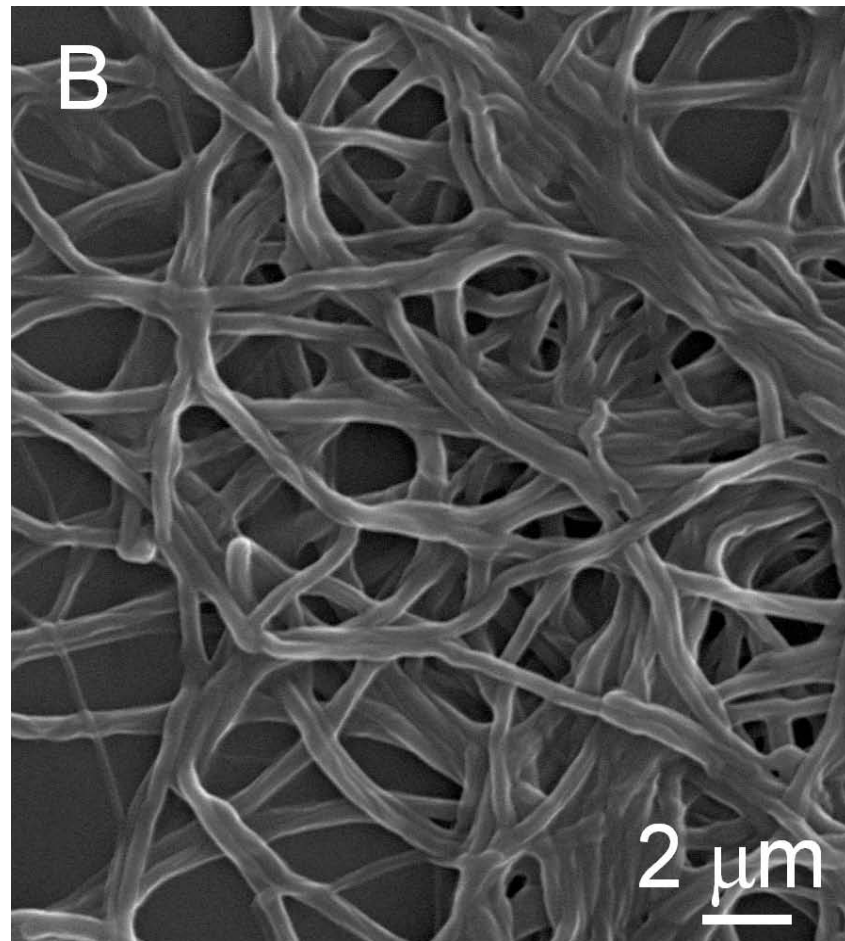
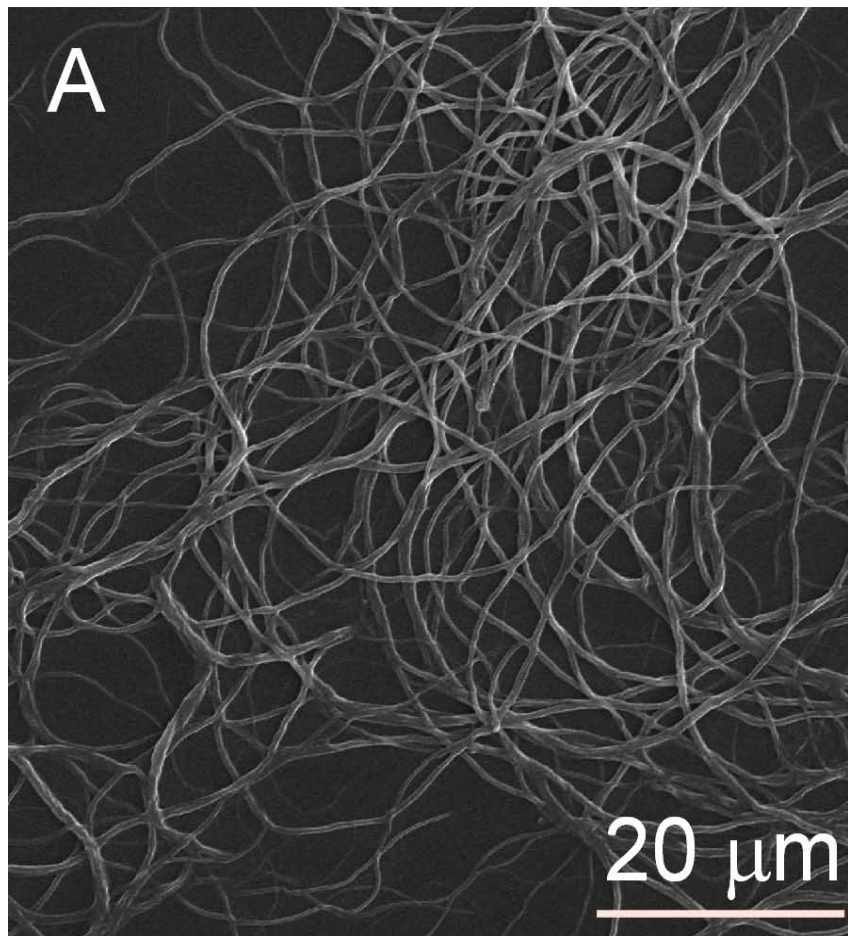


Video of nanofiber sensor response to 10 ug Meth  
simulant disperse in a filter paper

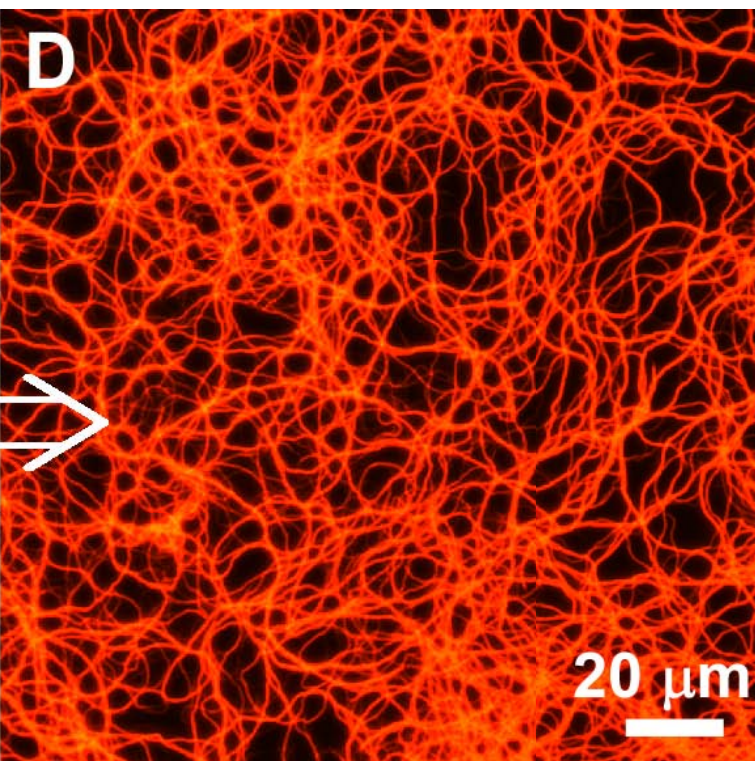
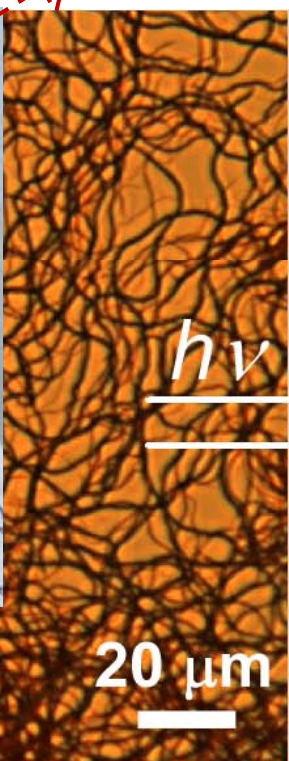
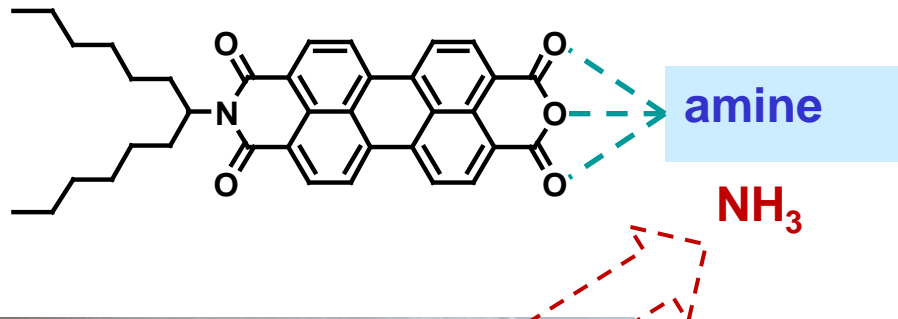
# Nanofibers fabricated from an amphiphilic molecules



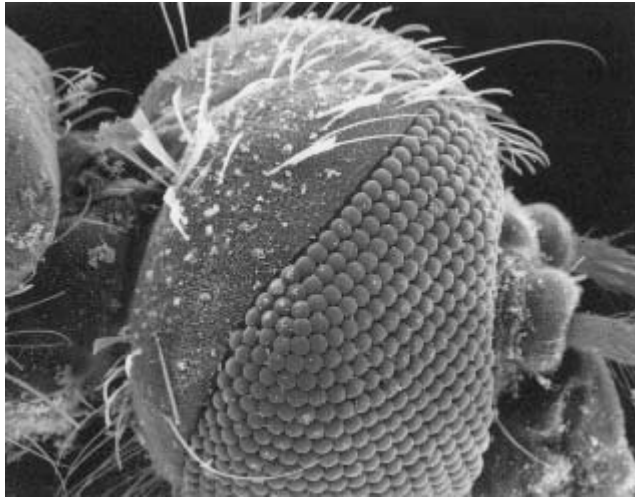
Nanofibers with anhydride moieties dominant on surface



# Efficient fluorescence sensing of amines vapor



# SEM images of bio-species

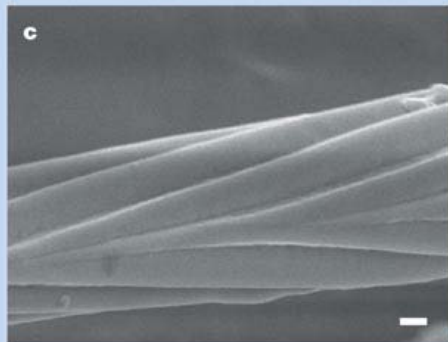
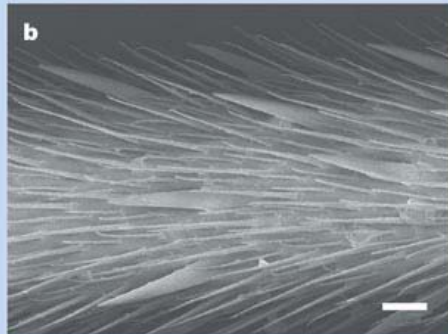
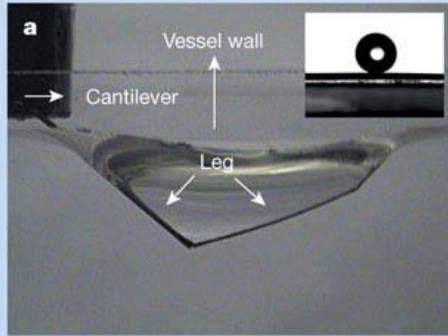


The head of a mosquito is mostly eye. The eyes are compound eyes, made up of many tiny lenses.



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

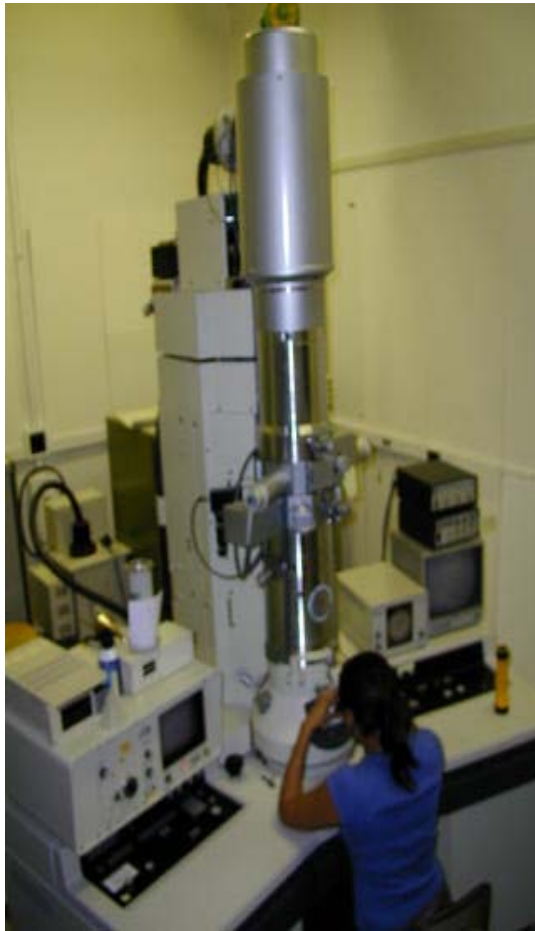
# Walking on water



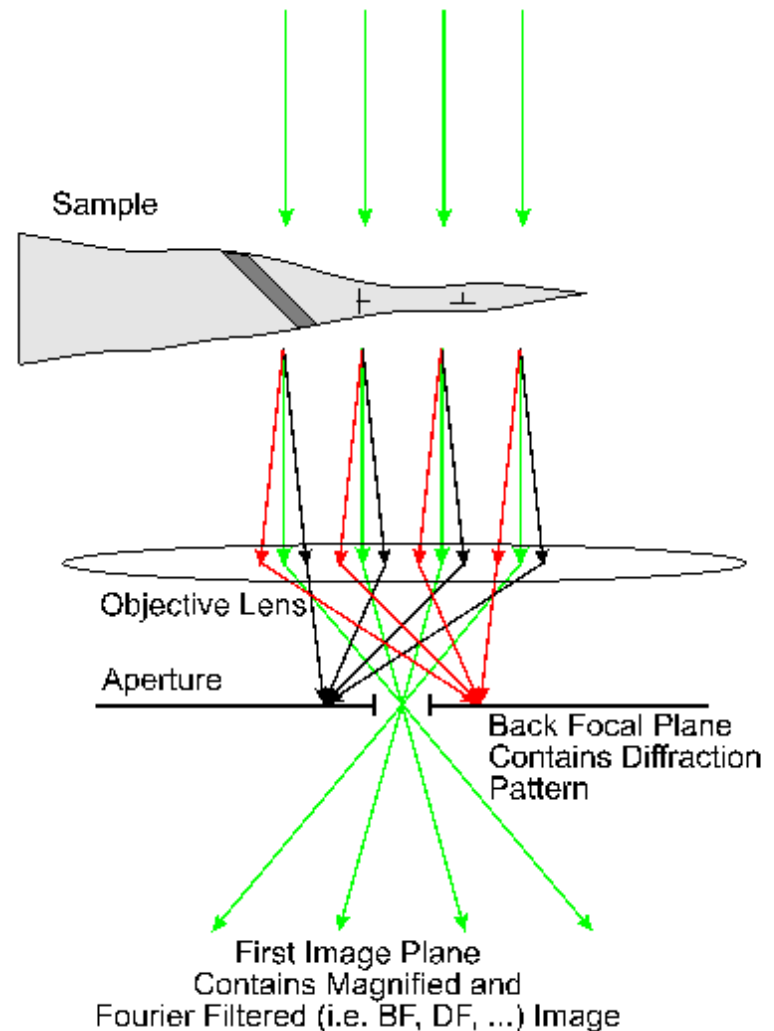
## The non-wetting leg of a **water strider**.

**a**, Typical side view of a maximal-depth dimple ( $4.38 \pm 0.02$  mm) just before the leg pierces the water surface. Inset, water droplet on a leg; this makes a contact angle of  $167.6 \pm 4.4^\circ$ . **b**, **c**, Scanning electron microscope images of a leg showing numerous oriented spindly micro-**setae** (**b**) and the fine nanoscale grooved structures on a seta (**c**). Scale bars: **b**, 20  $\mu\text{m}$ ; **c**, 200 nm.

# Transmission electron microscopy (TEM)



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



Detector: CCD (2D imaging)

# 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

## Advantages:

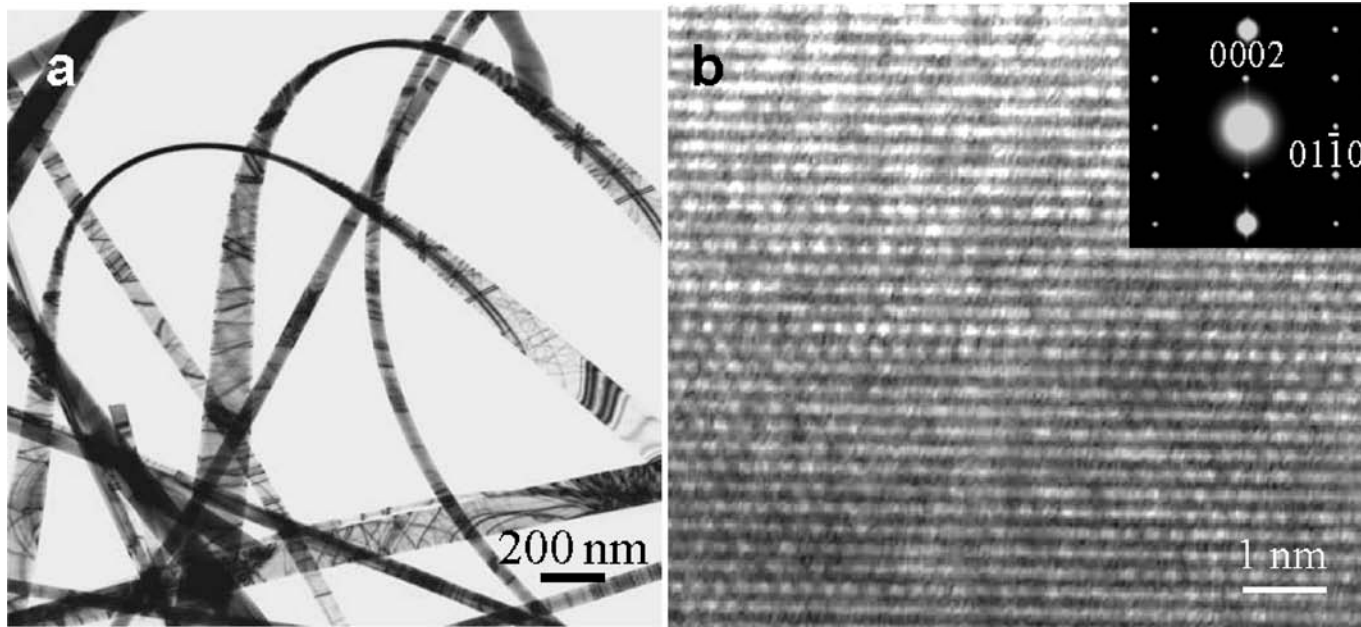
- High resolution, as small as 0.2 nm.
- Direct imaging of crystalline lattice.
- Delineate the defects inside the sample.
- No metallic stain-coating needed, thus convenient for structural imaging of organic materials,
- Electron diffraction technique: *phase identification, structure and symmetry determination, lattice parameter measurement, disorder and defect identification*

## Disadvantages:

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

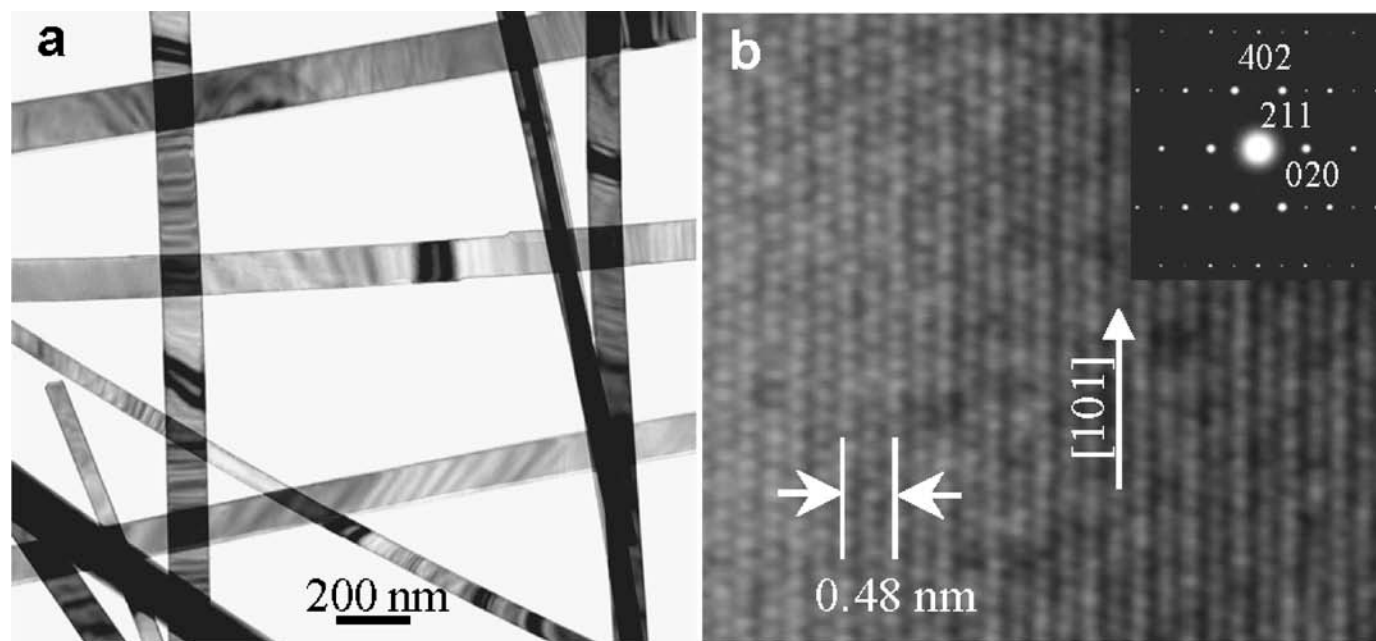


# TEM images: nanobelt



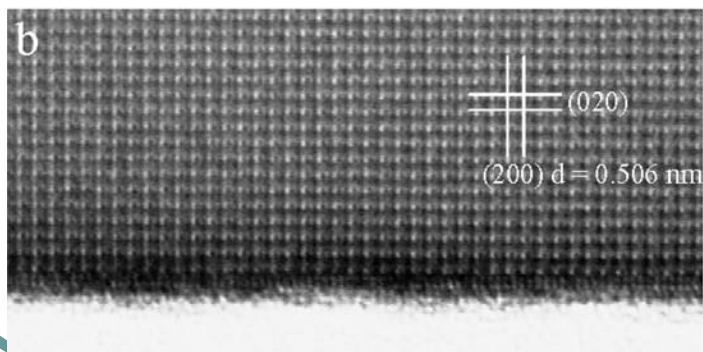
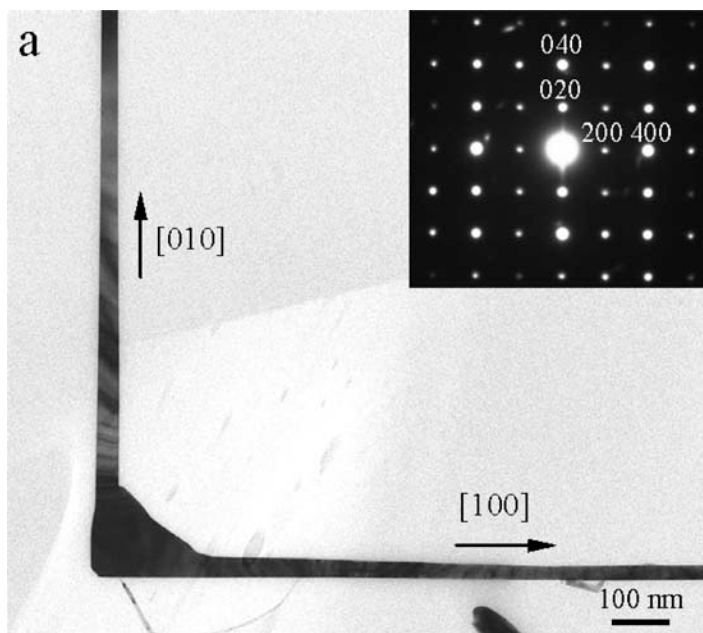
(a) Transmission electron microscopy (TEM) image of the as-synthesized **ZnO** nanobelts. (b) High-resolution TEM image recorded with the incident electron perpendicular to the top surface of the nanobelt.

# TEM images: nanobelt



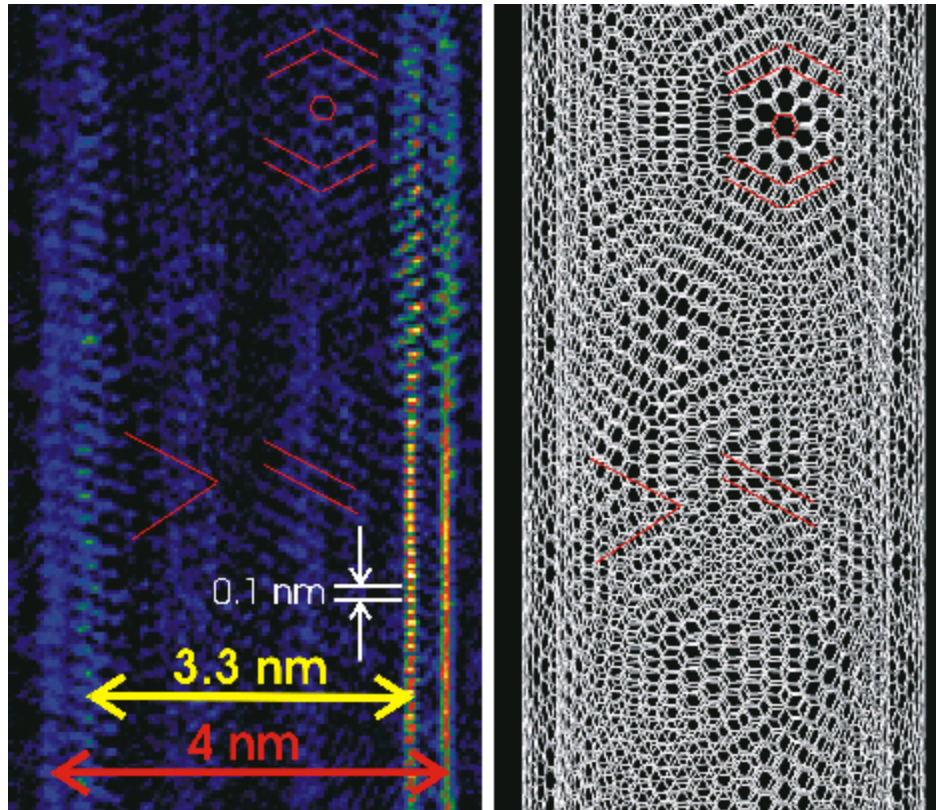
(a) Transmission electron microscopy (TEM) image of the as-synthesized **SnO<sub>2</sub>** nanobelts. (b) High-resolution TEM image recorded with the incident electron perpendicular to the top surface of the nanobelt.

# TEM images: nanobelt



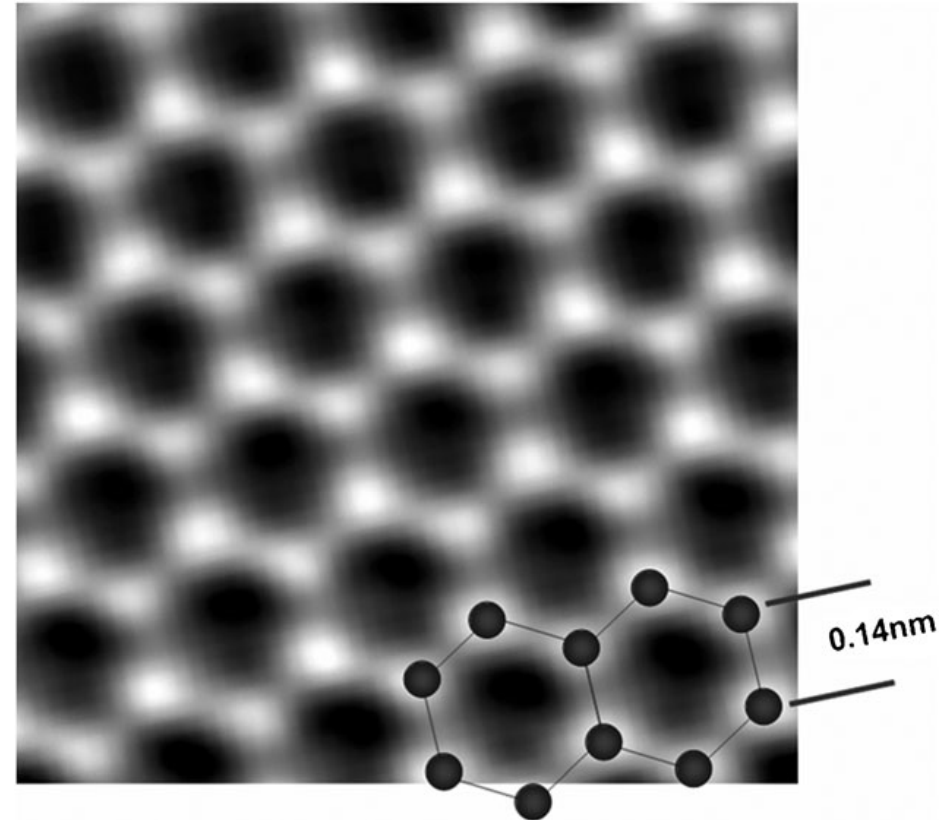
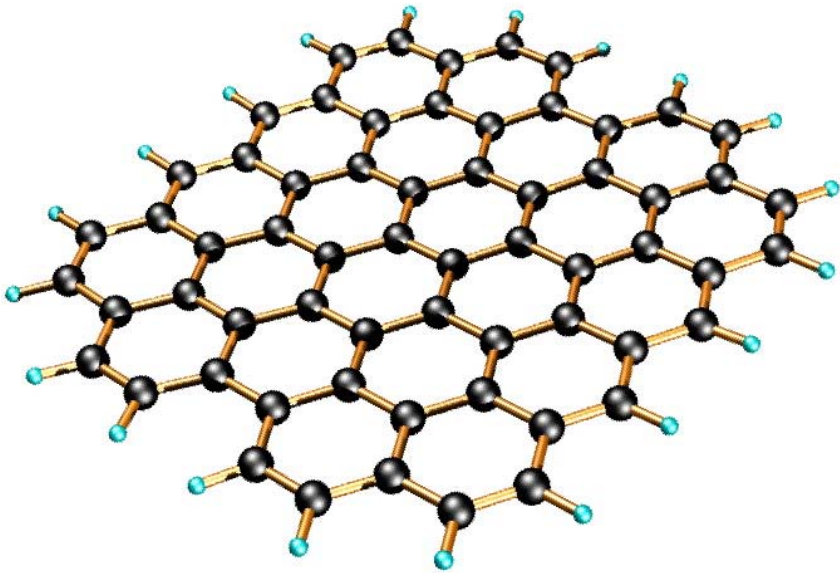
(a) Transmission electron microscopy (TEM) image of the as-synthesized L-shaped  $\text{In}_2\text{O}_3$  nanobelts. The inset is the electron diffraction pattern recorded from the nanobelt. (b) High-resolution TEM image recorded with the incident electron perpendicular to the top surface of the nanobelt.

# TEM images: nanotube

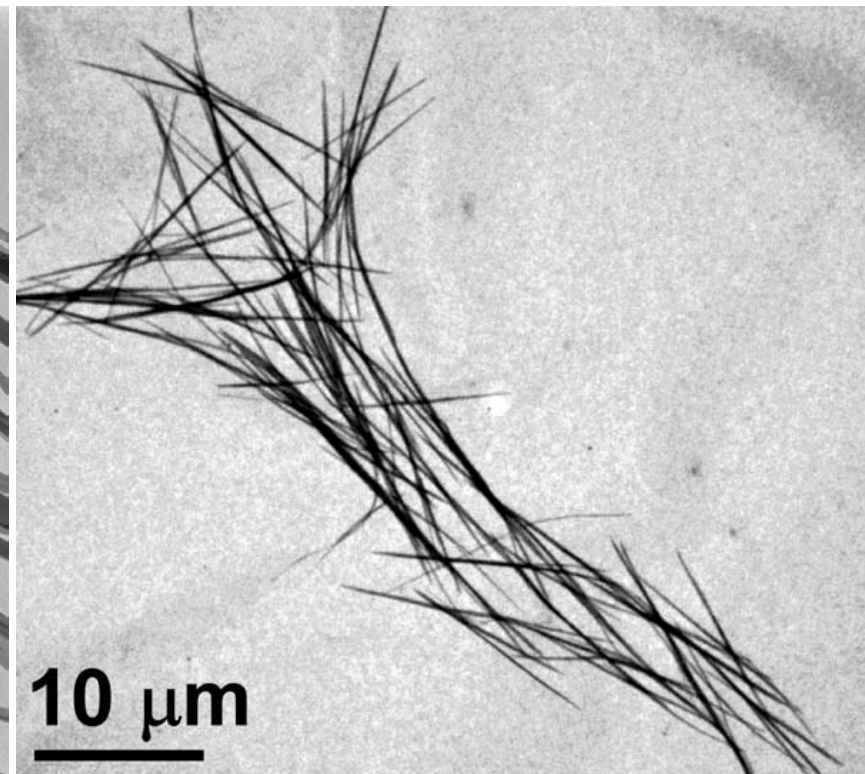
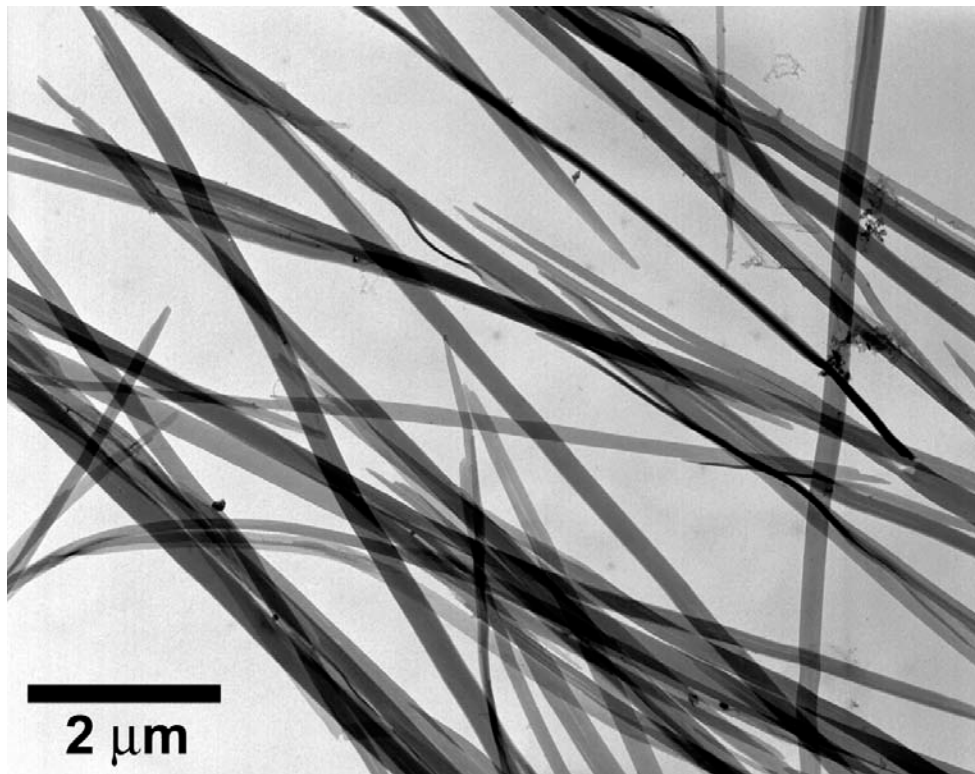


Imaging single-wall carbon nanotube

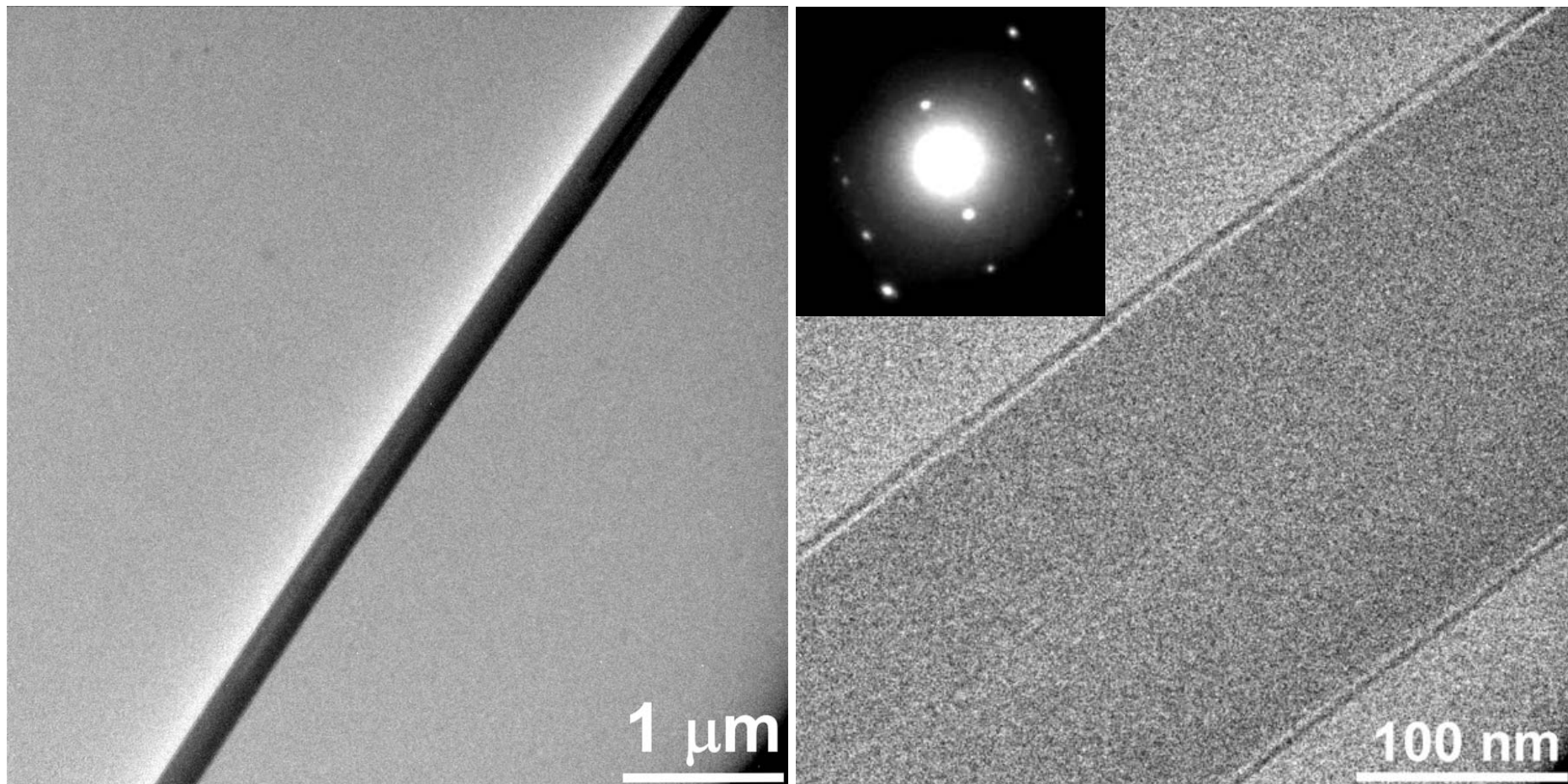
# TEM imaging of graphene



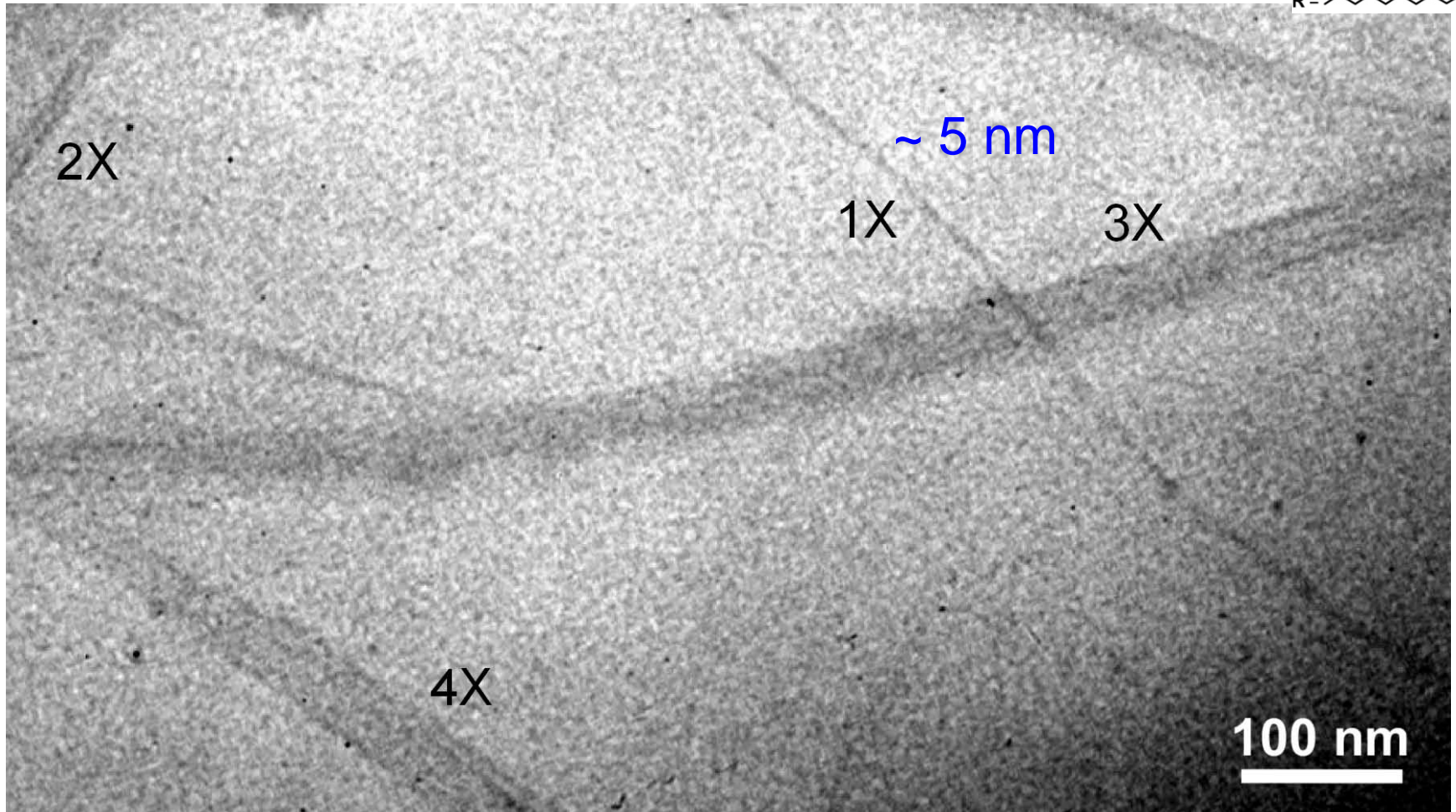
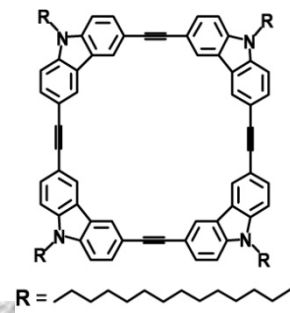
# Highly Uniform Nanobelts by TEM



# E-diffraction reveals stacking conformation



# Improved fabrication of single, uniform nanofibrils: *deposited from hot toluene solution*



Parallel alignment forms fibril arrays.



# Scanning Probe Microscopy (SPM)

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

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

Probing: sharp tip mounted to a Z-scanner.



# 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 <b>X-Y lateral imaging</b>
SPM	Tip	Using interaction between tip and sample: mechanic, electrostatic, meganetic.	Usually under ambient conditions, Highly flexible with other techniques	Å – nm, good for <b>Z-height measurement</b> , thus topography imaging

## Note:

SPM cannot replace electron microscopes, but complementary each other.