

Physical bases of dental material science

Methodes of structure analysis

Irén Bárdos-Nagy

Why it is important?

The macroscopic and microscopic structure strongly determine the physical, chemical and the biological properties and behaviour of materials.



for the goal of proper application we should know the structure

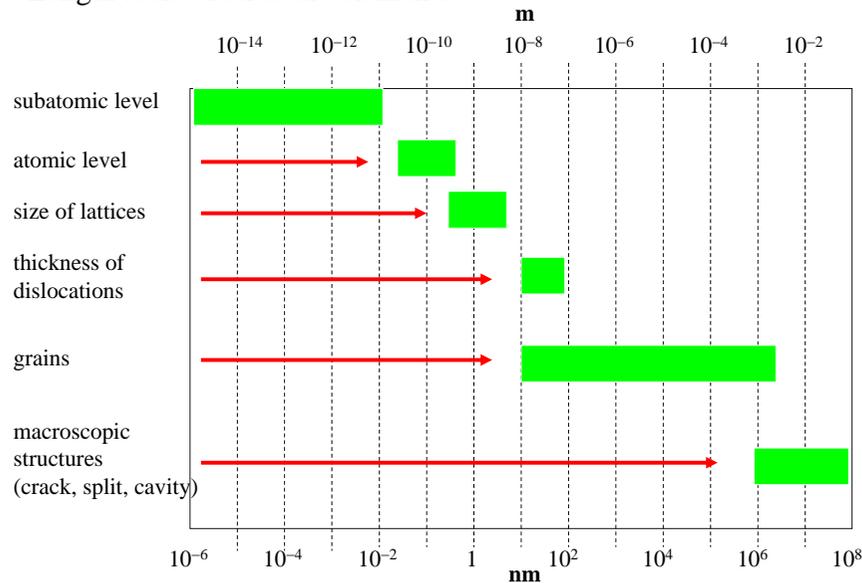
The materials can failure: fatigue
fracture
rupture
thermal shock → we have to recognize it
wear
buckling

To improve our knowledge to develop the properties of materials

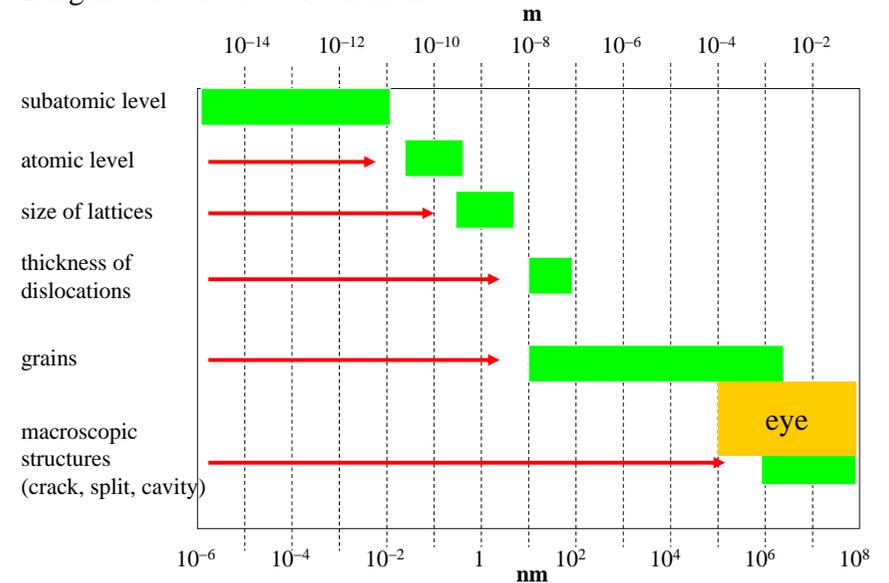


we have to analyse the structure

Length scale of structure elements



Length scale of structure elements



How can we see the smaller details?

Light microscope

real image of the specimen on the retina

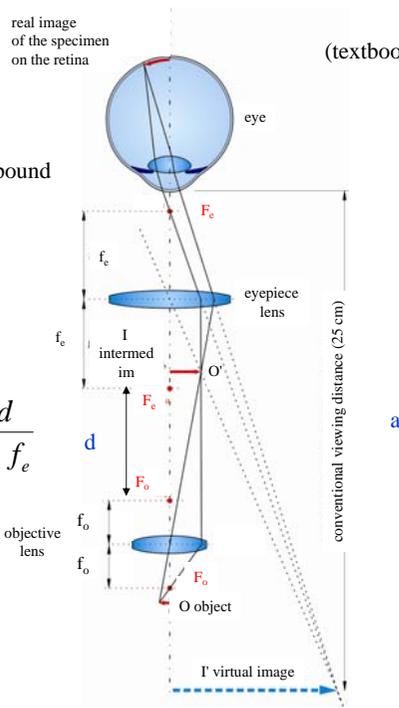
(textbook ch. VI/2.1, VI/2.2.)

image formation of the compound microscope

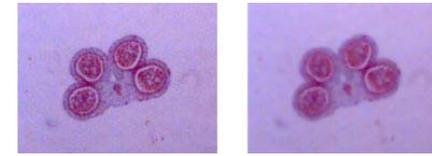
magnification:

$$M = M_{obj} \cdot M_e = -\frac{a \cdot d}{f_{obj} \cdot f_e}$$

(cc. 2000)



the smallest resolved distance

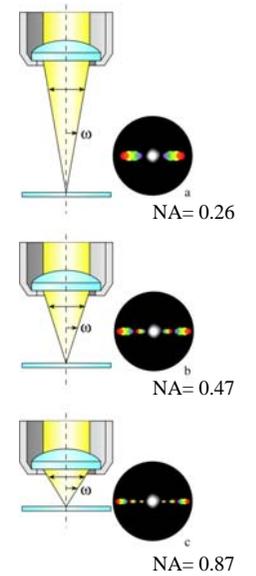
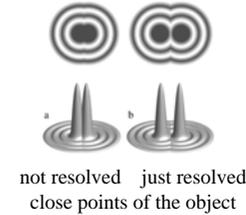
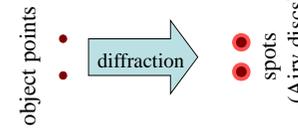


$$\delta = 0,61 \frac{\lambda}{n \cdot \sin \varphi} \approx \frac{\lambda}{NA}$$

wavelength of illumination light (λ)
 refractive index (n)
 half angle of the objective lens (φ)
 numerical aperture (NA)
 δ ≈ 200 nm

the reason of limited resolution:

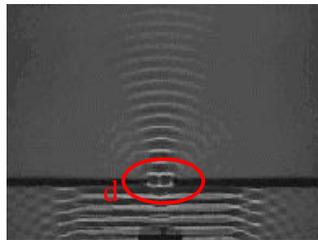
diffraction of light



Diffraction (Hygens principle)

(textbook ch. II/1.4, - II/1.6.)

Every point of a wave front may be considered the source of secondary wavelets that spread out in all directions with a speed equal to the speed of propagation of the waves.



d/λ >> 1
weak diffraction



d/λ ~ 1
strong diffraction

Conditions of constructive and destructive interference

constructive interference:

$$\Delta s = k \cdot \lambda$$

destructive interference:

$$\Delta s = (1 + k/2) \cdot \lambda$$

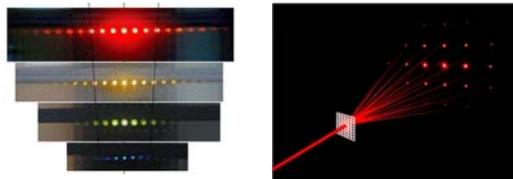
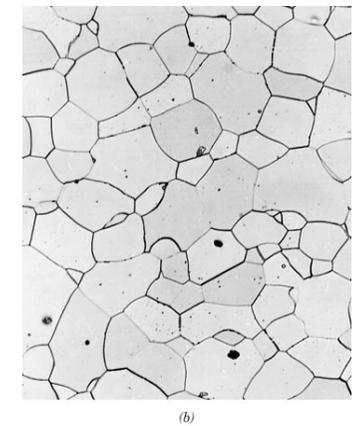
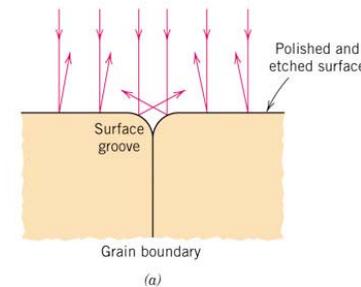
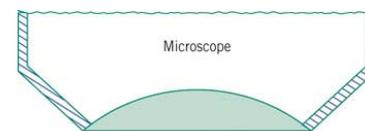
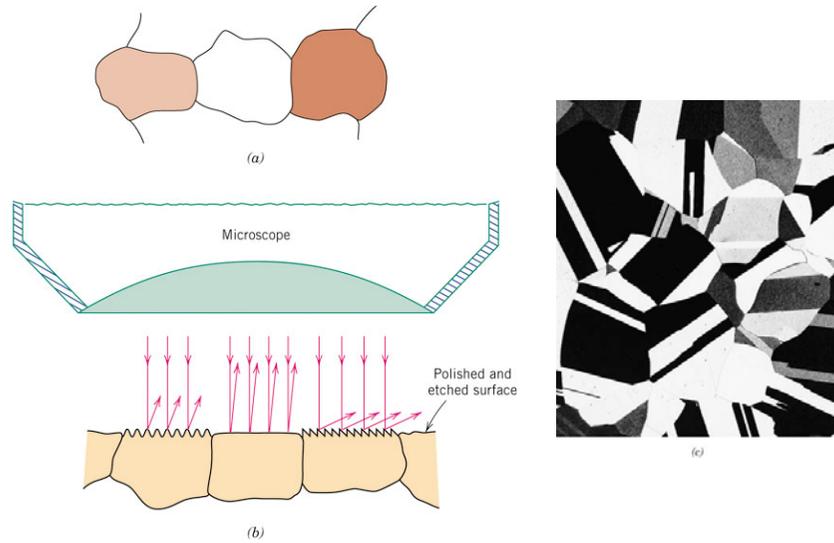


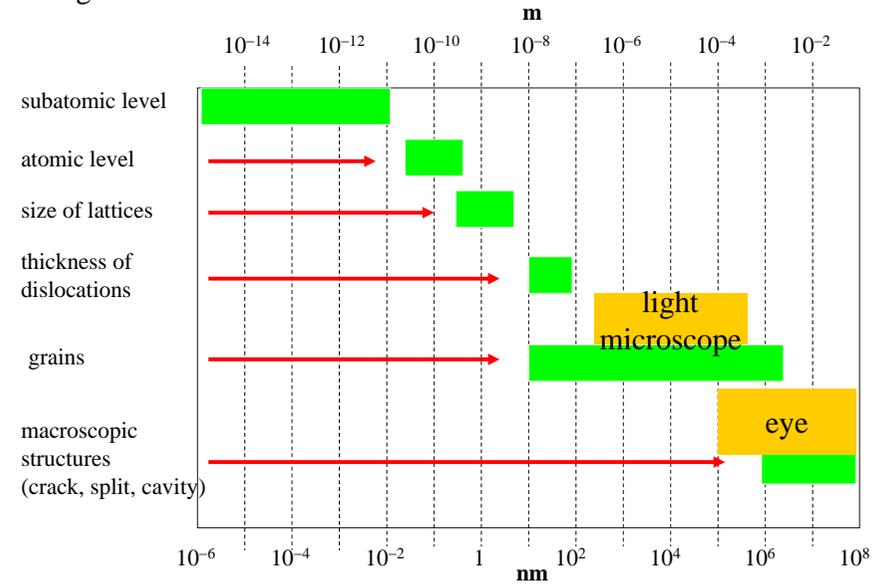
Image formation of a light microscope from a polished reflecting surface



The microscopic image of a partly polished reflecting surface



Length scale of structure elements



Electron microscope

(textbook ch. X/5.)

Theoretical bases of electron microscope

memo: the resolution depends on the wavelength!

Has the electron wave character?

$$\delta \approx \frac{\lambda}{NA}!$$

de Broglie's idea:
(1923)

$$\lambda = \frac{h}{m \cdot v} = \frac{h}{I}$$

Planck constant
(6.63×10^{-34} Js)

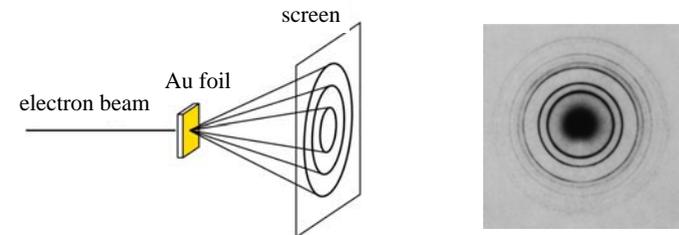
momentum of the electron

The wave nature and a certain wavelength have to be associated to every material mass!



The electron beam should have diffraction!

Experimental verification: Davisson and Germer (1927)



How large can be the resolved distance?

$$\lambda \approx 0,005 \text{ nm}$$

$$NA \approx 0,03$$

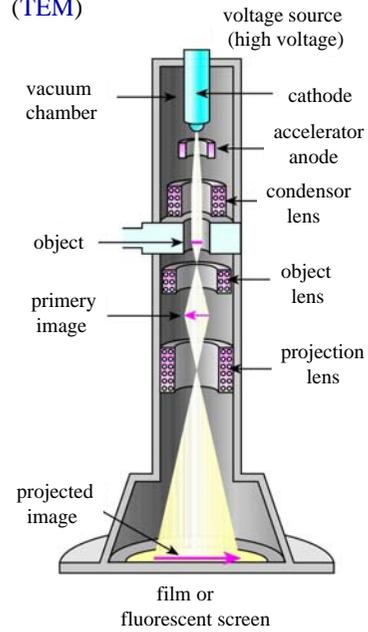


$$\delta \approx 0,2 \text{ nm}$$

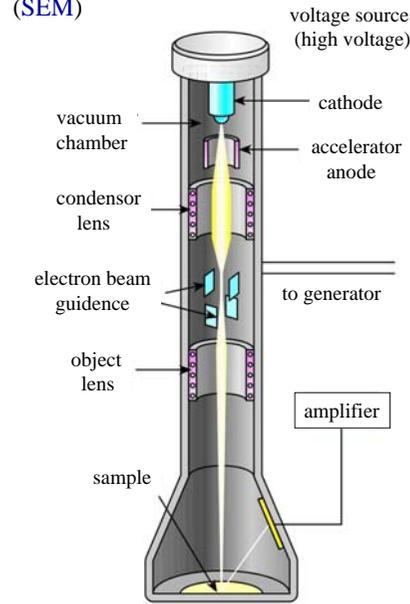
opens up the possibility of imaging sub-cellular details

How it is operating?

Transmission electron microscope (TEM)



Scanning electron microscope (SEM)



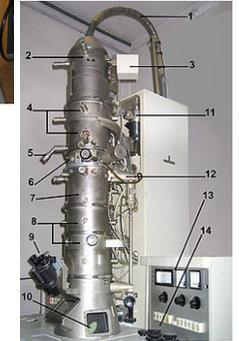
SEM in a Geological Survey



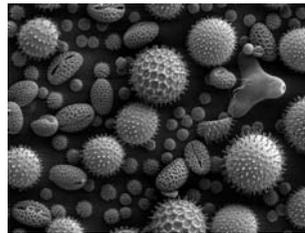
SEM opened sample chamber



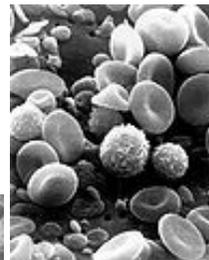
The first TEM (now on display at Deutsches Museum in Munich, Germany)



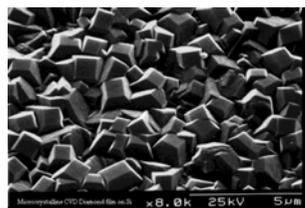
TEM applied nowadays



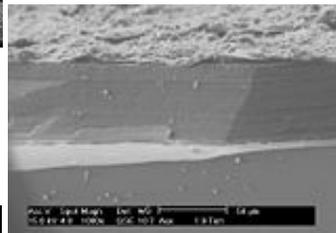
SEM image of pollen grains



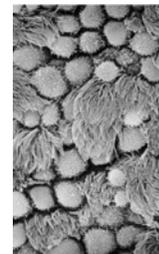
SEM image of normal circulating human blood



SEM image of microcrystalline diamond film on Si



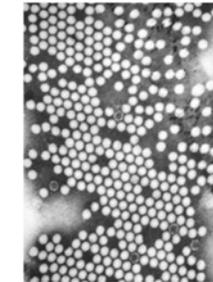
SEM image of corrosion layer on the surface of an ancient glass fragment



SEM image of trachea epithelium



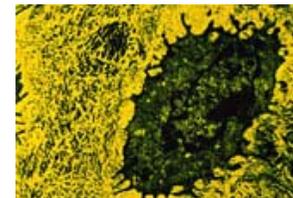
TEM image of heart muscle



TEM image of polio virus



TEM image of collagen fibers

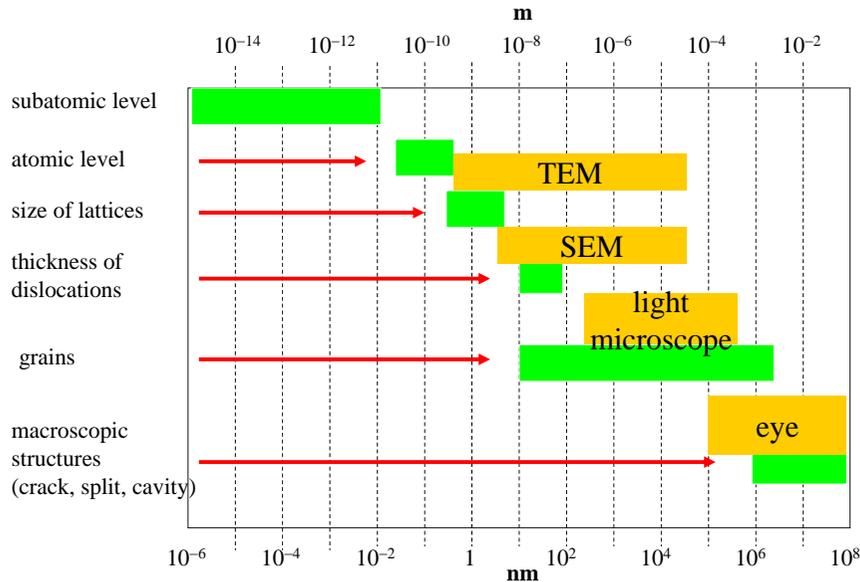


TEM image of bone cells



TEM image of skeletal muscle

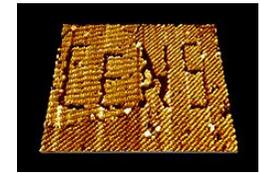
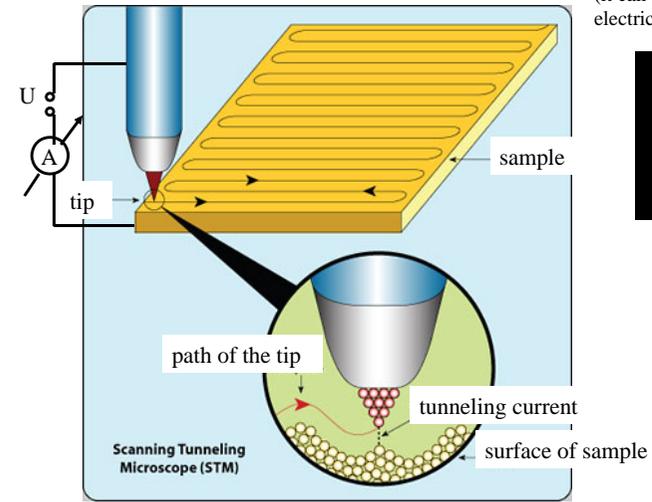
Length scale of structure elements



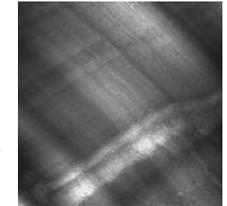
How to analyse the atomic and subatomic level?

Scanning tunneling microscope (STM)

exploits the tunneling effect of electrons between two conducting surfaces
(it can be used only for electrically conducting materials)



organic semiconductor monolayer on graphite

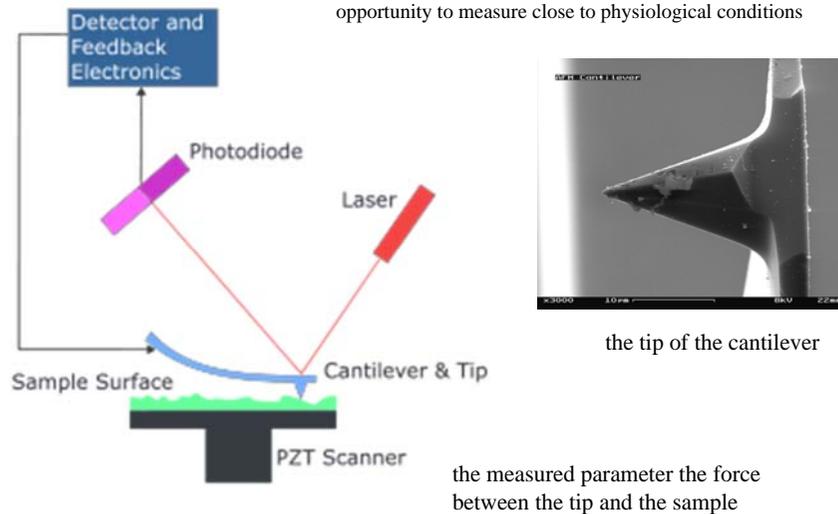


collagen

Atomic force microscope (AFM)

(textbook ch. X/2.)

advantages → information of surface topography of the sample
high resolution examination of various surface structures
investigation of structures under the plasma membrane of cell
opportunity to measure close to physiological conditions



the tip of the cantilever

the measured parameter the force between the tip and the sample

(Piezoelectricity)

(textbook ch. VIII/4.2.1)

1880 P. and J. Curie (piesmos = pressure, compression)

Piezoelectric effect

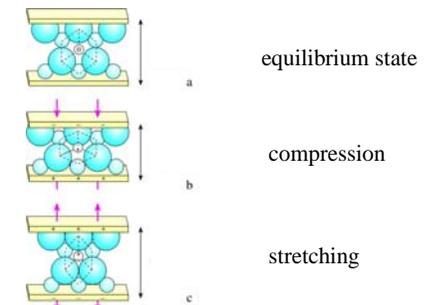
mechanical deformation (pressure) leads to charge separation



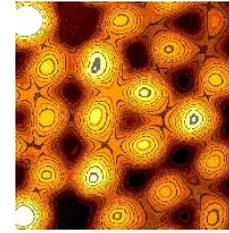
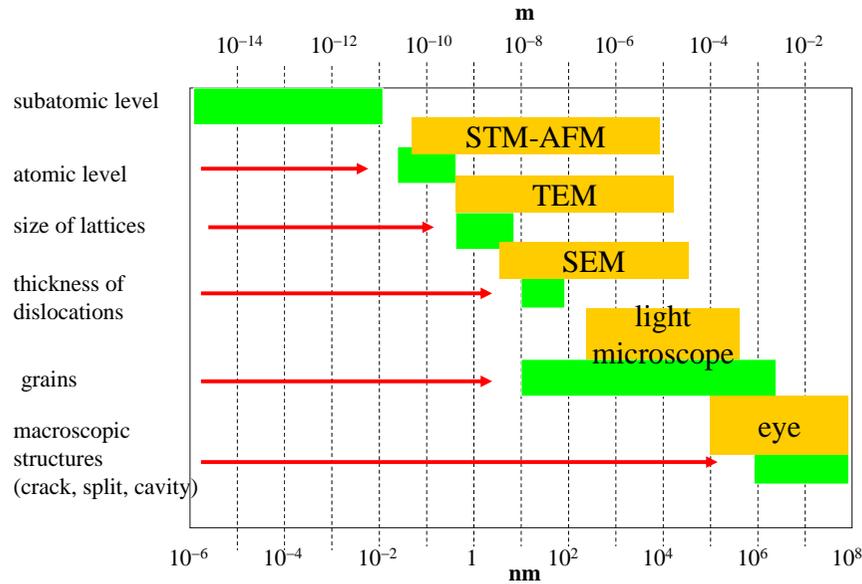
quartz crystal

Inverse piezoelectric effect

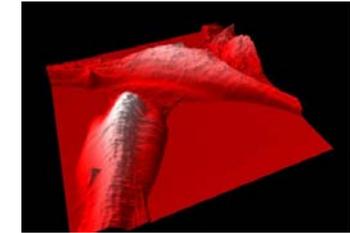
mechanical deformation of the crystal in the electric field



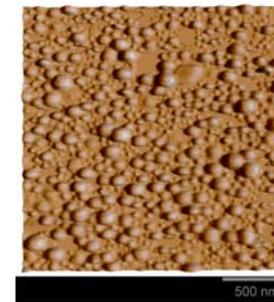
Length scale of structure elements



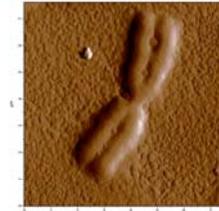
Si crystal (3*3 nm)



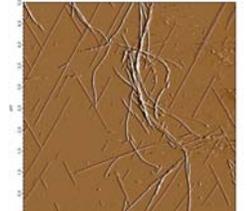
bone cells on Ti surface



liposomes on mica surface



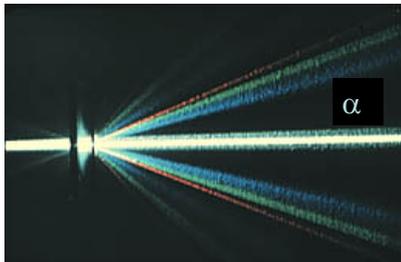
human chromosome



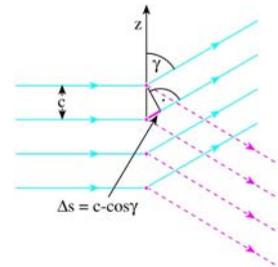
amiloid fibers

Diffraction methodes

(textbook ch. X/6.)



$$d \cdot \sin \alpha = k \cdot \lambda$$

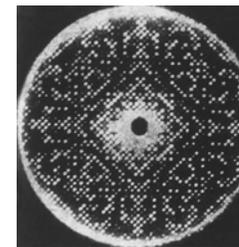


diffraction of X ray on a one dimensional crystal

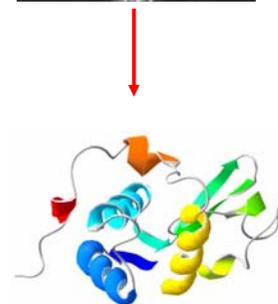
neutron diffraction ($\lambda \sim 0.1$ nm)

X-ray diffraction ($\lambda \sim 0.01$ - 0.1 nm)

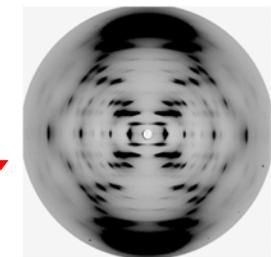
electron diffraction ($\lambda \sim 0.01$ nm)



X-ray diffraction image of the crystallized lysozyme enzyme



the molecular structure of lysozyme enzyme based on the X-ray diffraction

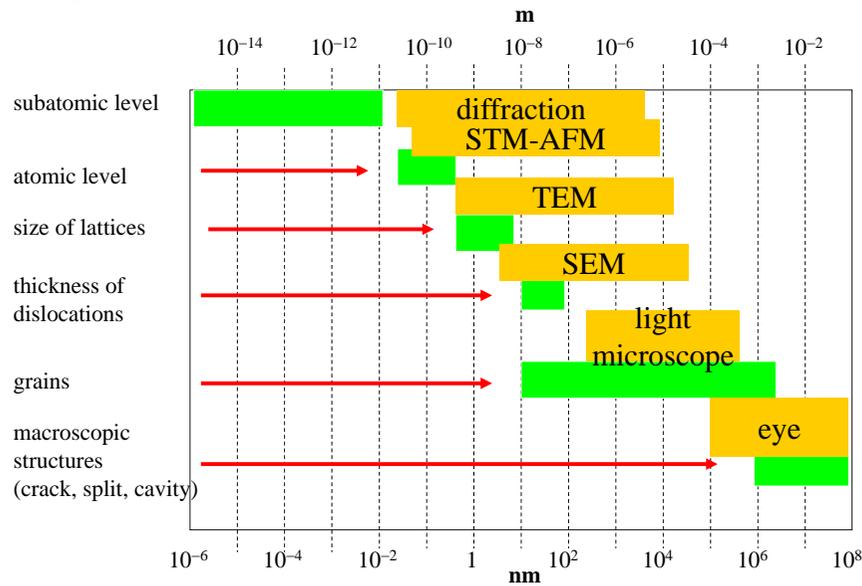


X-ray diffraction image of the crystallized DNA



the molecular structures of DNA based on the X-ray diffraction

Length scale of structure elements



Summary of structure analysis

- eye
- microscopy
 - light
 - electron (TEM, SEM)
 - surface scanning (STM, AFM)
- diffraction methods
 - neutron
 - X - ray
 - electron
- spectroscopic methods
 - absorption (UV, VIS, IR)
 - emission (fluorescence, phosphorescence, X-ray fluorescence)
 - Raman
 - magnetic resonance (NMR, ESR)
 - CD spectroscopy

How large is the smallest resolved distance in a microscope, if the wavelength of the illumination light is 510 nm, the half angle of the microscope is 72° ?

$$\delta = 0,61 \frac{\lambda}{n \cdot \sin \omega}$$

$$\delta = 0,61 \frac{515}{1 \cdot \sin 72^\circ} = 330,4 \text{ nm}$$

How this distance will change, if we use a 1,54 refractive index immersion oil instead of the air?

$$\delta = 0,61 \frac{515}{1,54 \cdot \sin 72^\circ} = 214,5 \text{ nm}$$

How large is the smallest resolved distance in an electron microscope, if the wavelength of the electron beam is 0,01 nm, and the numerical aperture of the microscope is 0,02?

$$\delta = \frac{\lambda}{NA} = \frac{0,01}{0,02} = 0,5 \text{ nm}$$

How large is the speed of the electrons in this microscope?

$$\lambda = \frac{h}{m \cdot v} = \frac{6,6 \cdot 10^{-34}}{9,1 \cdot 10^{-31} \cdot v} = 0,01 \cdot 10^{-9} \text{ (m)}$$

$$v = 7,25 \cdot 10^7 \text{ (m / s)}$$