T E M —

the Transmission Electron Microscopy,

a Powerful Tool for Imaging, Diffraction and Spectroscopy in Materials Science

Lecture -- I

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A quick review of last two lessons

Let's review what have impressed you the most by far:

- **What is this course (PHYS-E0525) about?**

  Keywords: *Microscopy of Nanomaterials*

- **What is "Microscopy" about ?**

  Keywords: *Optical (light); SPM (STM, AFM); EM; …*

- **What is "Electron Microscopy" about ?**

  Keywords: *SEM; FIB; TEM; STEM; Tomography…*

- **How does "Electron Microscopy" work?**

  Keywords: *electron microscopes; guns, lens, aperture, aberrations, vacuum, camera / detectors….*

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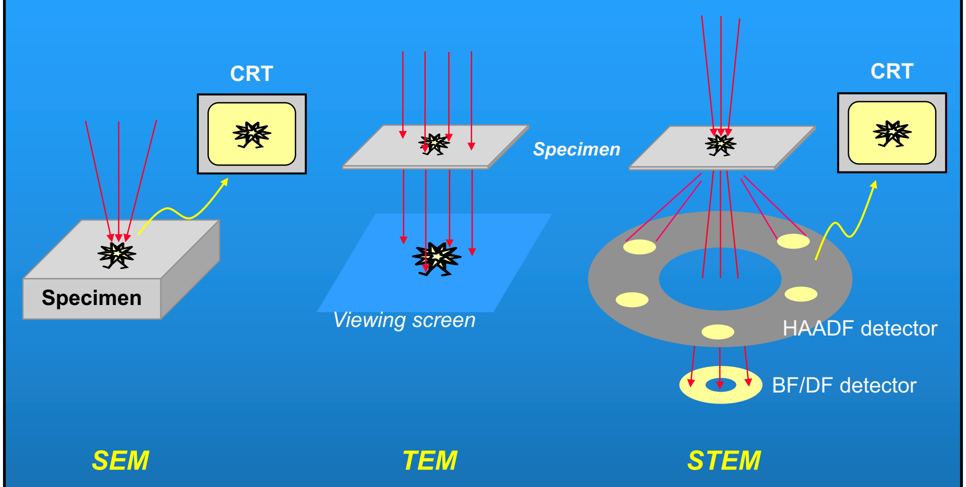
The big picture

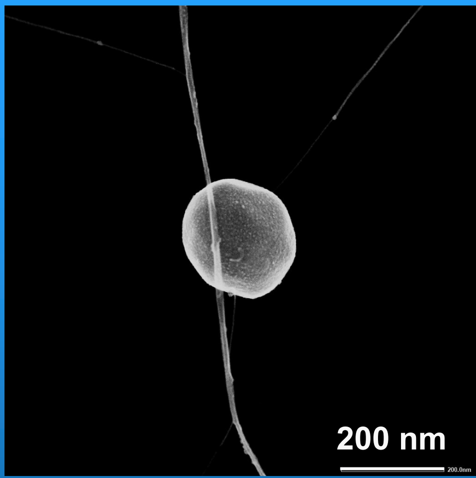
- **Microscopy** (PHYS-E0525)
  - Optical (light) Microscopy (from high school)
  - Scanning Probe Microscopy (SPM) (Dr. Peter Liljeroth)
    - STM
    - AFM
    - ….
  - Electron Microscopy (EM)
    - SEM (Dr. Ramzy Abdelaziz)
    - FIB (Dr. Lide Yao)
    - TEM
      - HRTEM (Dr. Hua Jiang)
      - STEM (Dr. Hua Jiang)
      - Diffraction (Dr. Hua Jiang)
      - Spectroscopy (Dr. Hua Jiang)
      - Cryo-TEM (Dr. Janne Ruokolainen)
      - Electron tomography (Dr. Jani Seitsonen)
    - ….
  - ….

Electron Microscopy



Electron Microscopy images

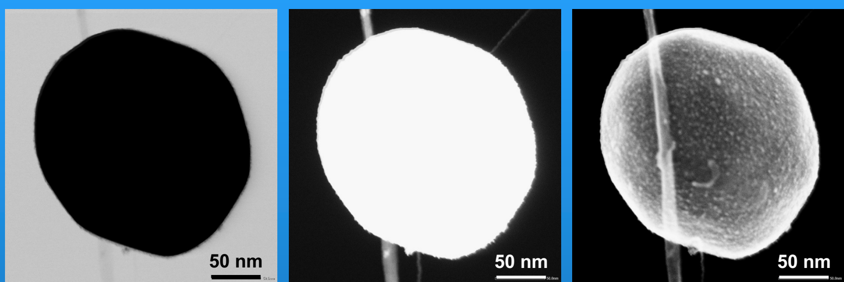
This is a CNT sample



Are you able to tell what types of microscopy images they are?

Electron Microscopy images

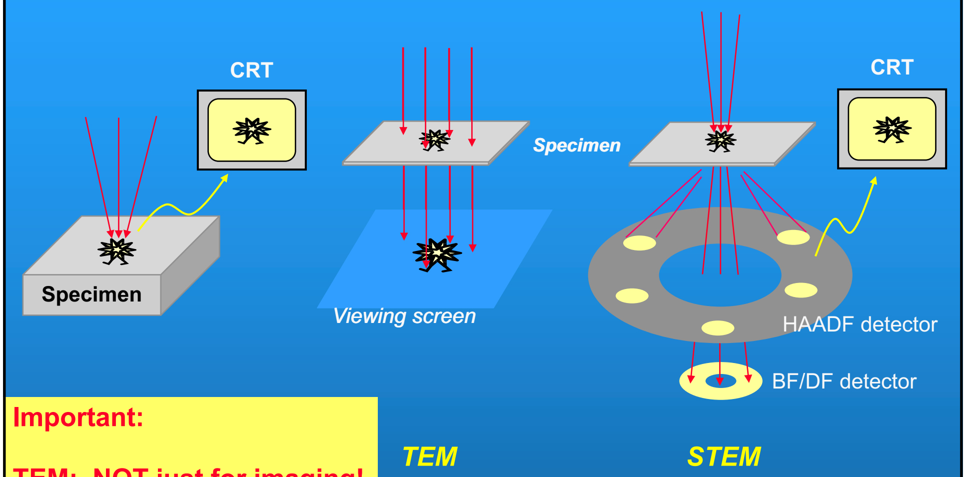
This is a CNT sample



TEM BF **STEM DF** **SEM**

This lecture is designed for you to recognize and analyze those images, and extract as much structural info as possible from the sample.

Transmission Electron Microscopy



Important:

TEM: NOT just for imaging!

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Outline of the lectures

- **TEM basics – a brief overview**
  - why we use electrons for microscope?
  - why it is possible to use electrons?
  - How to use electrons: electron-sample interactions
  - basics of the instrument
  - sample requirements and preparation
  - what to learn from this lecture
- **Diffraction in TEM**
- **Imaging in TEM**
- **STEM imaging**
- **Spectroscopy in TEM**

Lect. 1 Jan. 31 (TEM basics – a brief overview)

Lect. 2 Feb. 7 (Diffraction in TEM; Imaging in TEM)

Lect. 3 Feb. 14 (STEM imaging; Spectroscopy in TEM)

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TEM Basics --- a quick review

- Basics of the TEM instrument --- the *Microscope*
- *Electrons* and their interaction with *samples*
- TEM sample preparation
- Electron microscopy of a number of aspects

Why electrons

☞ Limitations of a light microscope (LM) ☹

$$d = \frac{0.61\lambda}{n \sin \alpha} \cong \frac{1}{2}\lambda$$

d --- resolution; λ --- light wavelength

$n \sin \alpha$: may be as large as 1.3 (Numerical Aperture or N.A.)

λ as short as $400nm$

In brief: *the resolution limit of a light microscope (LM) cannot be better than 200 nm, far away from seeing the nano-world.*

Why electrons

- nature of electrons ☺
  - **Moving electrons are electron wave**

$$\lambda = \frac{h}{m_e v} \propto \frac{1}{v}$$

v --- speed; h --- Planck constant; m_e --- mass

  - **The higher electron energy, the shorter electron wavelength**

$$\lambda = \frac{12.25}{\sqrt{U(1+0.9788 \quad 10^{-6}U)}} \propto \frac{1}{\sqrt{U}}$$

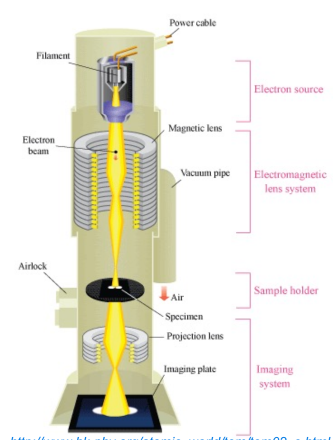
U --- accelerating voltage

80kV:	0.00418nm
120 kV:	0.00335nm
200 kV:	0.00251nm
300 kV:	0.00197nm

In brief: *resolution supposed to be improved by using electron to image.*

The basic units in an electron microscope

- ***Electron gun***
  --- a source for producing high energy electrons
- ***Electron lens***
  --- a medium for deflecting or focusing electrons
- ***Cameras / Detectors***
- ***High vacuum***
  --- an environment that frees the interaction of electrons from anything else but your sample and the lenses.



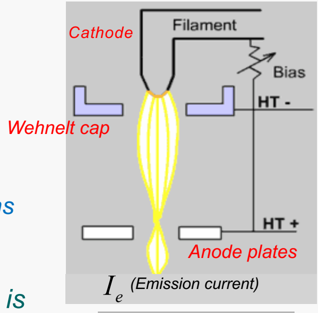
http://www.hk-phy.org/atomic_world/tem/tem02_e.html

In brief: *using electron as illumination source is not just* ***beneficial*** *but also* ***possible****.*

Electron Gun

- **Function**
  --- providing electron illumination at high current in a small probe size

- **Configuations**
  --- a scheme of a typical thermionic-emission gun (right)
  - Cathode: the filament
  - Anode plates: accelerating electrons

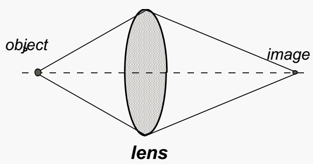


- **Accelerating voltage (U)**
  --- the electron energy (wavelength) is given by the accelerating voltage

- **Emmision current (I_e)**
  --- the total current leaving the gun, typically ~100 μA

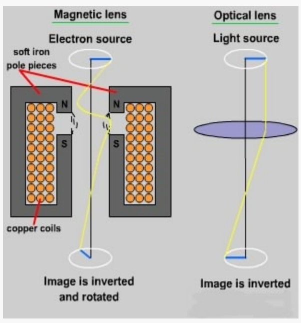
Electron Lens

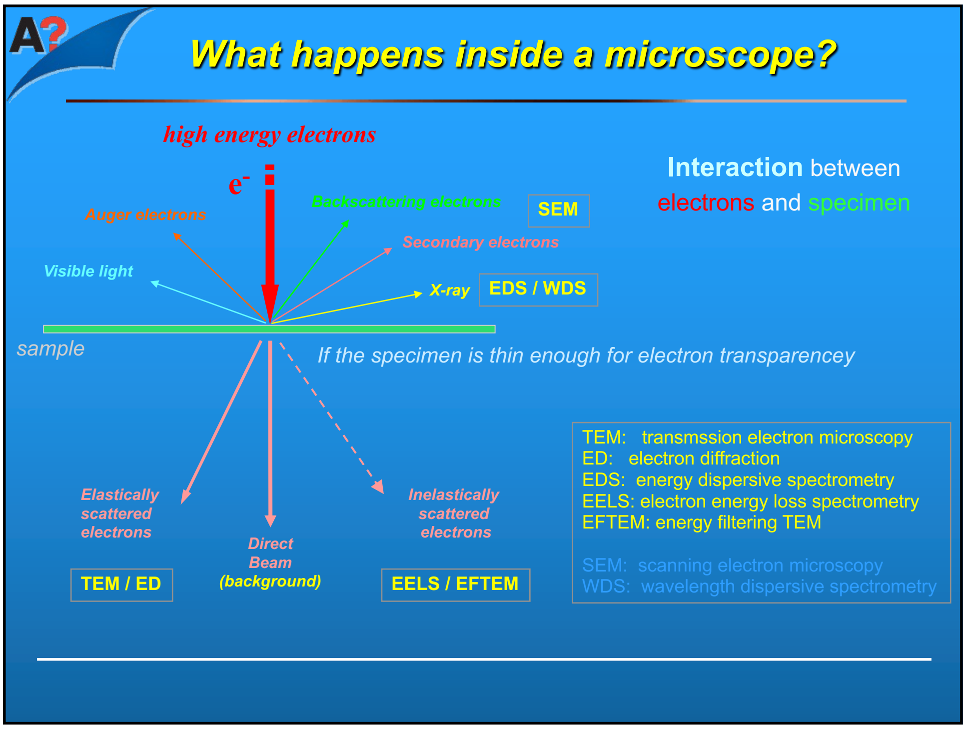
- **Function**
  --- a lens forms an image of an object
  - an optical microscope uses glass to refract light and form images.
  - The electron beam is focused using electromagnetic lenses, which employ magnetic or electrostatic fields as the refracting medium.

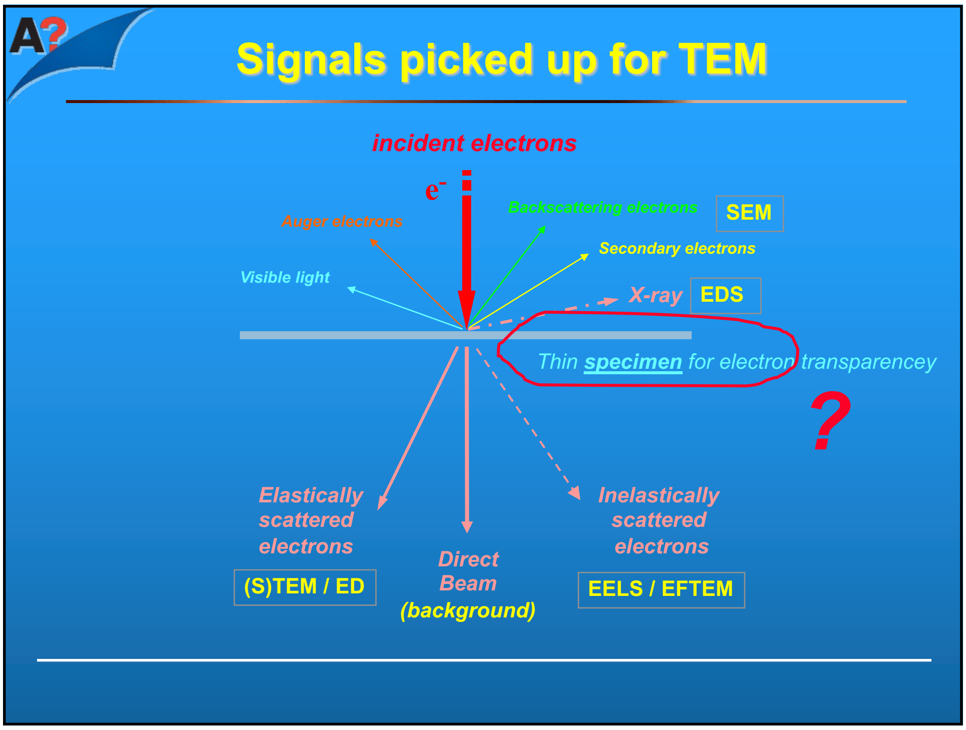


- **Magnetic lens**
  --- including bacially lens coil which produce the magnetic field.

  --- the focal length f can be changed by varying the lens excitation (the current or the potential)




What happens inside a microscope?
high energy electrons
e-
Interaction between
electrons and specimen
Auger electrons
Backscattering electrons
SEM
Secondary electrons
Visible light
X-ray
EDS / WDS
sample
If the specimen is thin enough for electron transparencey
Elastically scattered electrons
Direct Beam (background)
Inelastically scattered electrons
TEM / ED
EELS / EFTEM
TEM: transmssion electron microscopy
ED: electron diffraction
EDS: energy dispersive spectrometry
EELS: electron energy loss spectrometry
EFTEM: energy filtering TEM
SEM: scanning electron microscopy
WDS: wavelength dispersive spectrometry


Signals picked up for TEM
incident electrons
e-
Auger electrons
Backscattering electrons
SEM
Secondary electrons
Visible light
X-ray
EDS
Thin specimen for electron transparencey
?
Elastically scattered electrons
(S)TEM / ED
Direct Beam (background)
Inelastically scattered electrons
EELS / EFTEM

Good specimen for TEM research

General requirements ??

Be small: 3mm in diameter

Be thin: <100 nm in thickness

Remember: ***Your microscope (TEM) is only as good as the sample that you put into it !!!***

* *1 nm = 10^{-9} meter*
1 nm = 10 Å

TEM sample preparation

General rule: ***the thinner, the better,*** **for *"Electron transparency"***

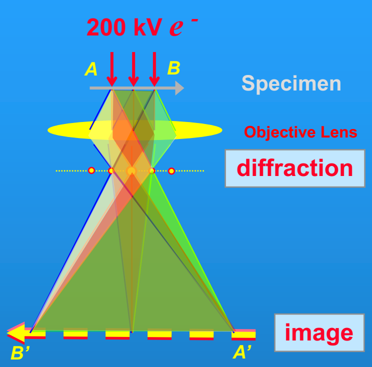
☞ **Basic requirements**

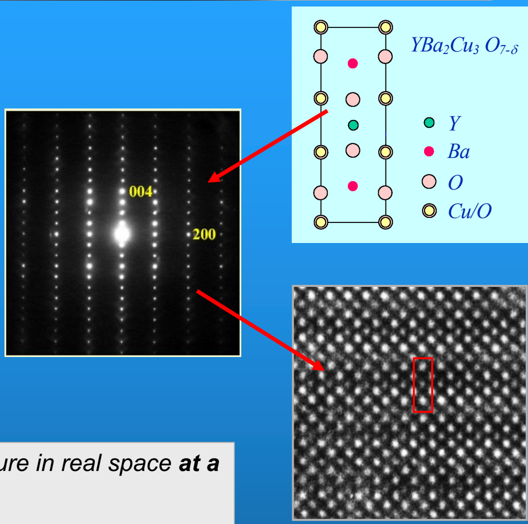
- **diameter** ϕ ~ 3 mm
- **thickness** t < 100 - 150 nm, < ~ 50 nm for HREM and spectrum

☞ **Ways out:** *highly depend on the materials you are working on*

- **powders, nanoparticles:** direct TEM sample is possible
- **ion-milling:** a sputtering method by kicking out atoms from the surface of the specimen by bombing with accelerated ions, such as Ar ions. Better for ceramics, semiconductor in particular.
- **FIB** (Focus ion beam): originally developed for semiconductor devices. The specimen is thinned rapidly by sputtering with a sharply focused ion beam, such as Ga ions. Especially suitable for specimens containing a boundary between materials. A SEM image can be displayed while thinning.

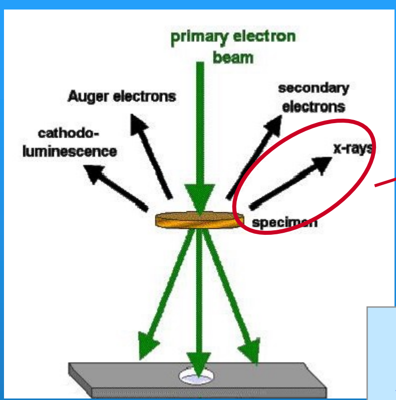
What can a TEM do? --- Diffraction and Imaging

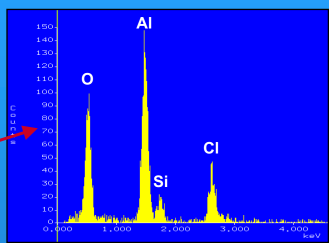




- An *image* represents the structure in real space **at a certain resolution**;
- The *diffraction* is an reproduction of the structure in reciprocal space.

What else can be picked up in TEM? --- EDS

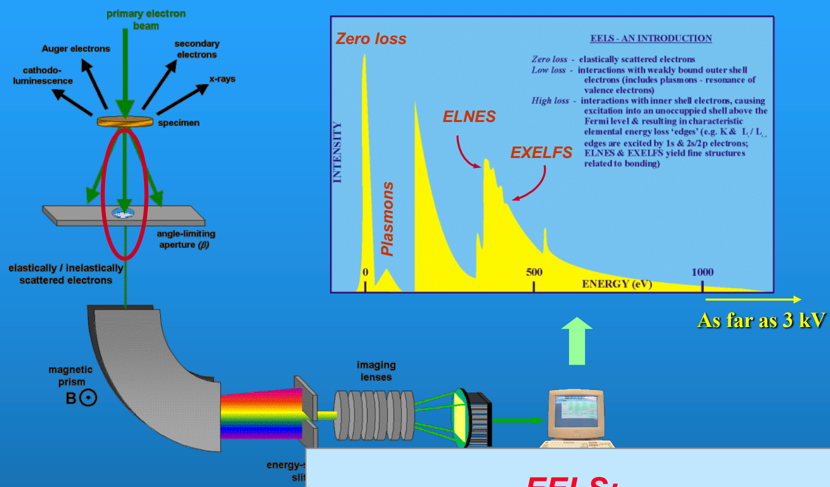




EDS:

X-ray **E**nergy **D**ispersive **S**pectroscopy

What's more? --- EELS



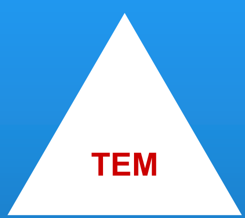
EELS:

Electron **E**nergy **L**oss **S**pectroscopy

Three viewing angles

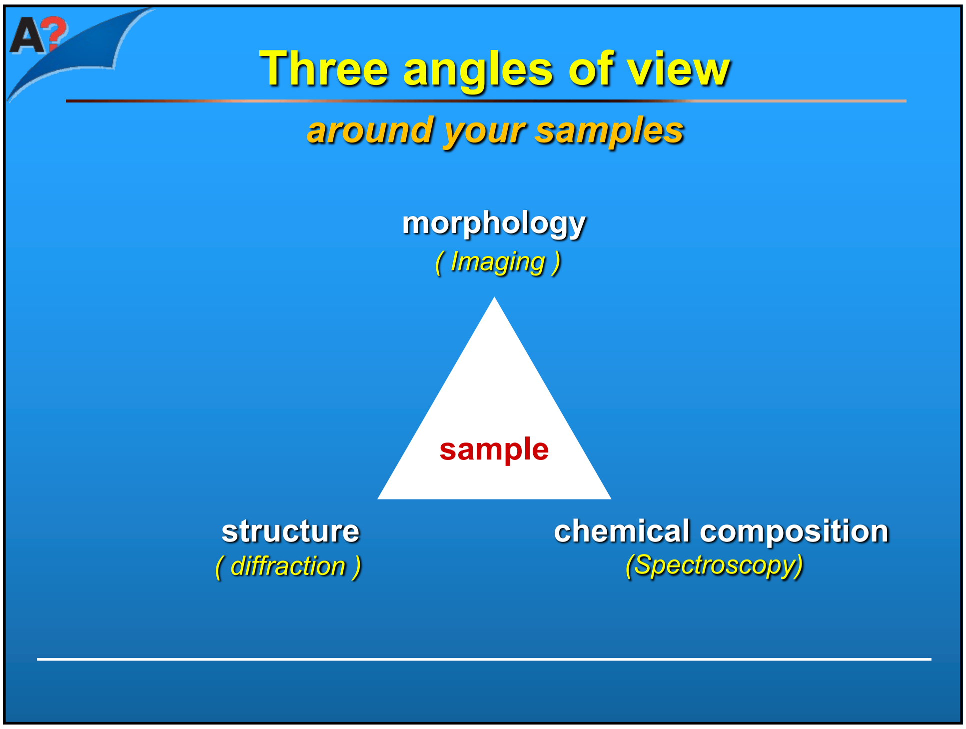
from Electron Microscopy

Imaging



Diffraction

Spectroscoopy


Three angles of view
around your samples
morphology
(Imaging)
sample
structure
(diffraction)
chemical composition
(Spectroscopy)

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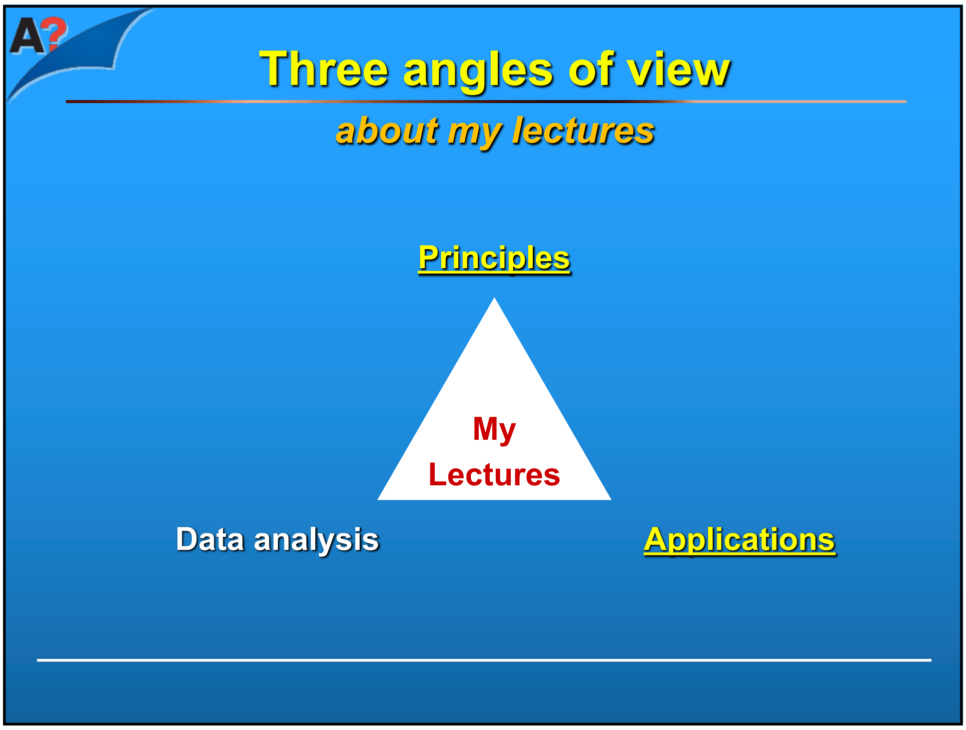
Three angles of view
about my lectures
Principles
My
Lectures
Data analysis
Applications

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What are you expected to learn from this lecture?

☞ **Be aware of:**

What is TEM about in general?

☞ **Be capable of:**

planning your experiments and working out your ideas by using TEM techniques !!

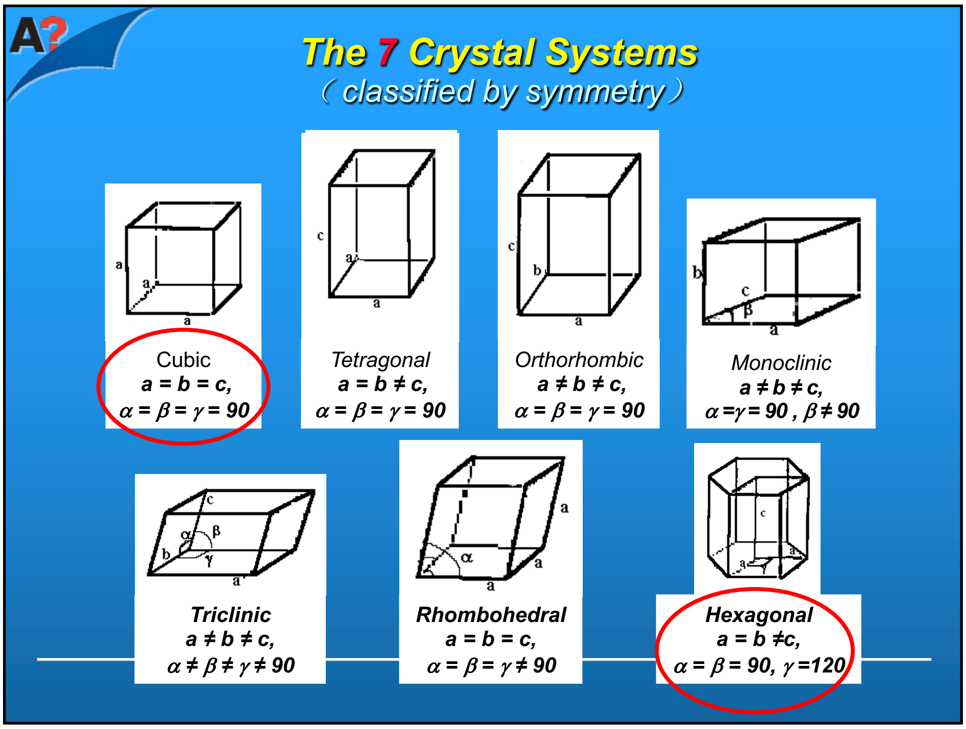
"Diffraction" in TEM

"Diffraction" in TEM

- Basic crystallography concept
- Diffraction principles
  - diffraction direction
  - diffraction intensity
- Application Examples
  - Structure analysis of SWCNTs
  - Oxygen behavior in YBCO

Basic crystallography

for a basic understanding of diffraction principle


The 7 Crystal Systems
(classified by symmetry)
Cubic
a = b = c,
α = β = γ = 90
Tetragonal
a = b ≠ c,
α = β = γ = 90
Orthorhombic
a ≠ b ≠ c,
α = β = γ = 90
Monoclinic
a ≠ b ≠ c,
α =γ = 90 , β ≠ 90
Triclinic
a ≠ b ≠ c,
α ≠ β ≠ γ ≠ 90
Rhombohedral
a = b = c,
α = β = γ ≠ 90
Hexagonal
a = b ≠c,
α = β = 90, γ =120

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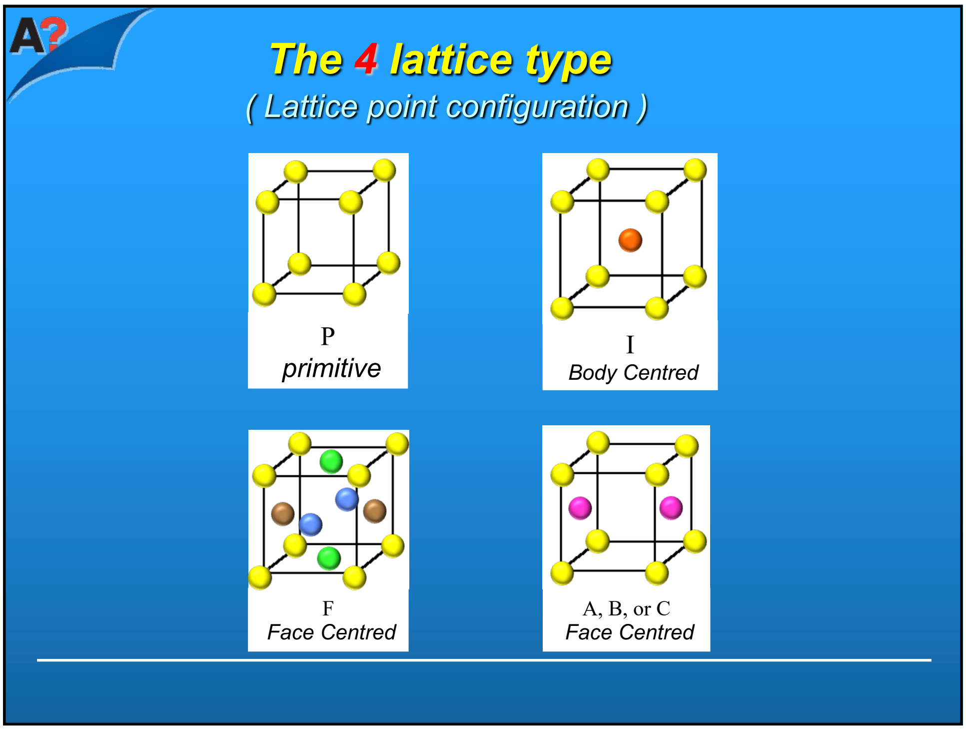
The 4 lattice type
(Lattice point configuration)
P
primitive
I
Body Centred
F
Face Centred
A, B, or C
Face Centred

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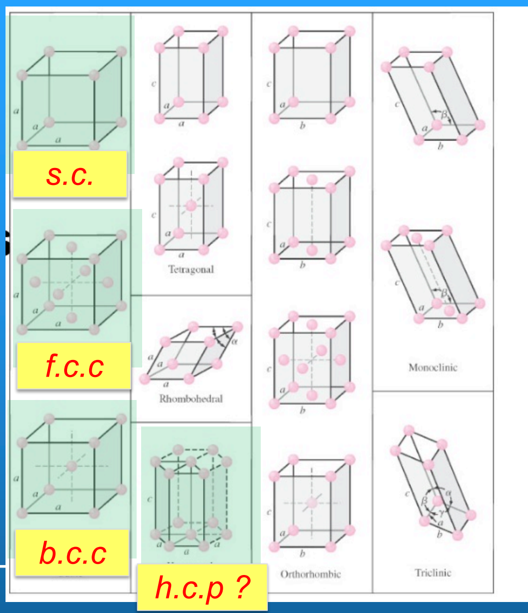
The 14 Bravais Lattices



The combination of the 4 lattice types (P, I, F, C) and the the 7 crystal classes give rise to all 14 possible variations, which are known as the 14 Bravais Lattices.

Miller indices: *Directions [u v w]*

Grouping symbols:

() are called parentheses or rounded brackets;

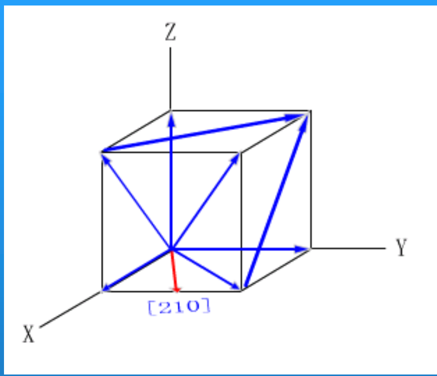
[] are called brackets or square brackets;

{} are called braces or curly brackets;

⟨⟩ are called bra and ket or angular brackets.

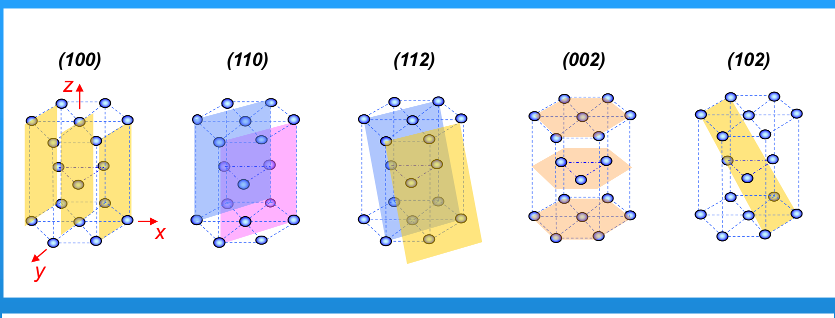
A crystallographic direction is expressed in terms of its ratio of unit vectors in the form **[u v w]** where u, v and w are integers. A family of crystallographically equivalent directions is expressed as **<u v w>**

Miller indices: *Directions [u v w]*



A crystallographic direction is expressed in terms of its ratio of unit vectors in the form **[u v w]** where u, v and w are integers. A family of crystallographically equivalent directions is expressed as **<u v w>**

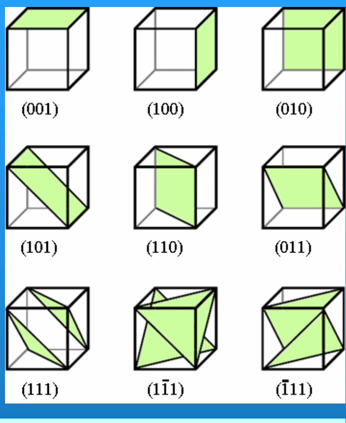
Miller Indices: *Lattice planes (h k l)*



- In a crystal, there are numerous sets of lattice planes which are characterized by their 1) *orientations* and 2) *lattice spacings*.
- A set of lattice plane is labelled by using its ***Miller Indices (h k l)***.
- Electron beam is diffracted by lattice planes in a crystal structure.

A lattice structure is thus composed of a number of sets of lattice planes.
(or say, a crystal structure can be decomposed into many lattice planes of…)

Common lattice planes in a simple cubic structure



Simple cubic (*s.c*) structure

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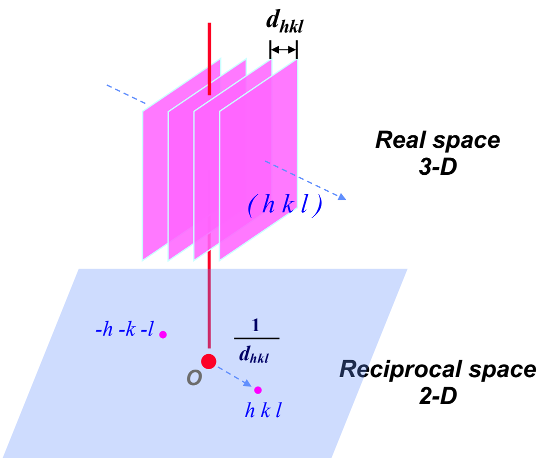
"reciprocal space" --- *Construction*

A lattice plane (h k l) → F T → One single point



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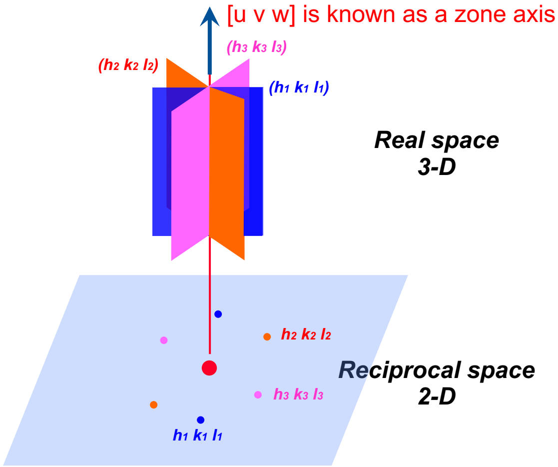
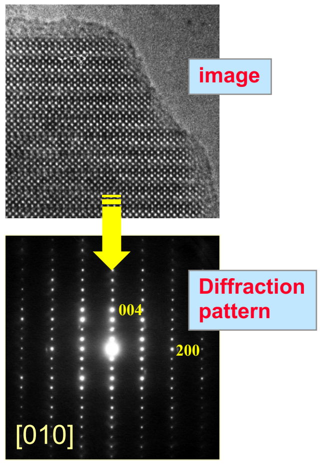
"reciprocal space" --- *Construction*

Diffraction : a description of a structure in reciprocal space !!





Fourier transform (FT):

A tool to bridge "Real space" and "Reciprocal space "

FFT Simulation

Electron Diffraction in TEM

Principles and applications

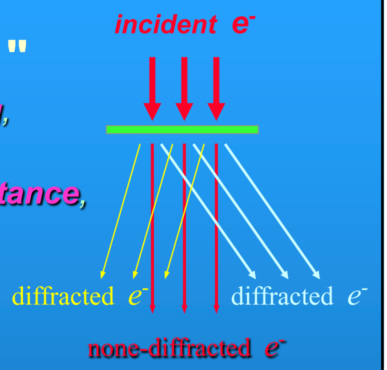
Transmission electron microscopes create magnified images of samples and are, in contrast to the light microscope, even able to resolve individual atoms. When transmitting a beam of electrons through a crystalline sample such as a complex mineral or a crystallized protein, the electrons are being diffracted in a specific way. Collecting such diffraction patterns of a sample from several different directions uniquely identifies a specific crystal structure.

Basic laws for electron diffraction

☞ What is "diffraction" " 衍射 "

*Diffraction is, firstly, a **scattering**, which follows certain **rules**, and features a basic nature of **inheritance**, namely, the **genetics** !*

- Diffraction directions ?
- Diffraction intensities ?

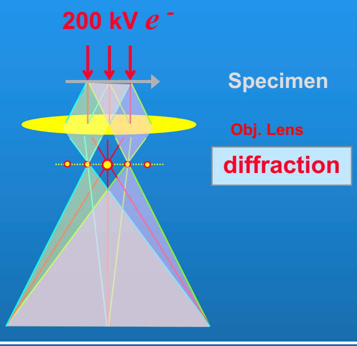


How the direction and the intensity of a diffraction beam inherit the structure information of your specimen ??

Diffraction: *directions ?*

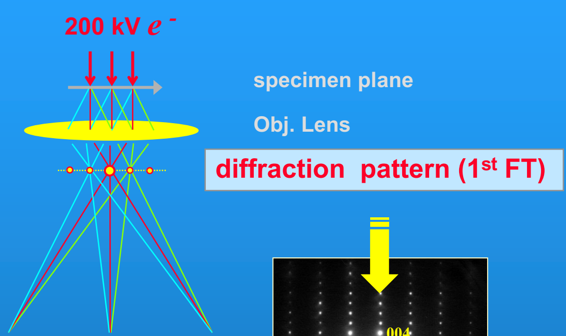
- Bragg's law --- *diffraction direction*

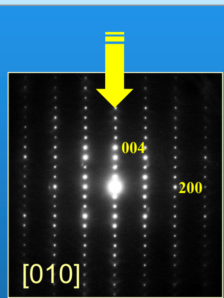
$$2\,d\sin\theta = \lambda \quad \Rightarrow \quad d_{hkl} \Rightarrow \theta_{hkl}$$



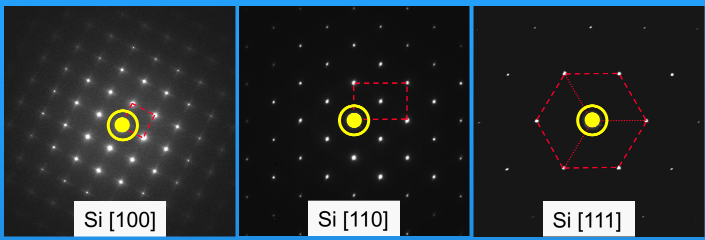
- One set of lattice planes (d) will diffract electrons along a certain direction (θ).
- Electrons coming from the same direction will be focused at one spot on the back-focal-plane of the objective lens.
- So, one diffraction spot represents one set of lattice plane.
- Mathematically, the objective lens performs a Fourier Transform!!

Electron diffraction produced in a TEM



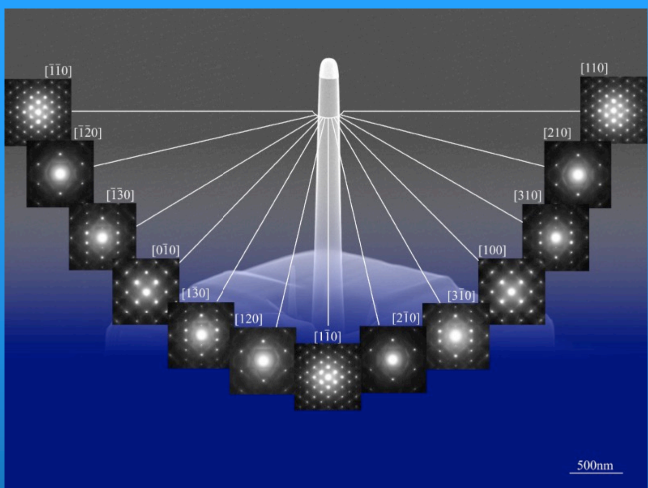


Electron Diffraction Patterns from a single crystal



Selected-area electron diffraction patterns from a Si single crystal taken along three different zone axis.

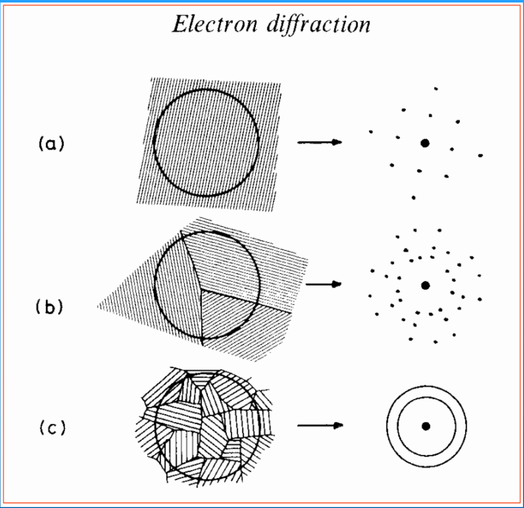
Diffraction Arts: Panorama Diffraction Pattern



Specimen: Si pillar prepared by FIB
TEM: Hitachi H-9500
Accelerating Voltage: 300kV

http://www.hitachi-hitec.com/global/em/nanoart/2005/index.html

Diffraction from a polycrystalline



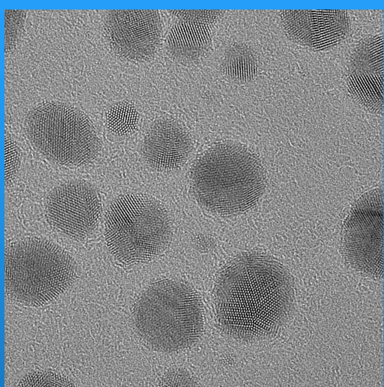
a single crystal

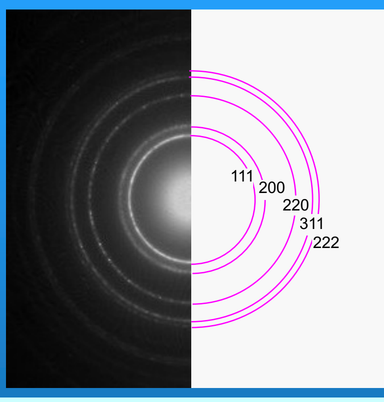
a small number grains

poly-crystalline
a large number of randomly oriented grains

An example: from f.c.c Au polycrystalline

Au nanoparticles: *d_{111} = 0.235nm, d_{200} = 0.204nm, … …*



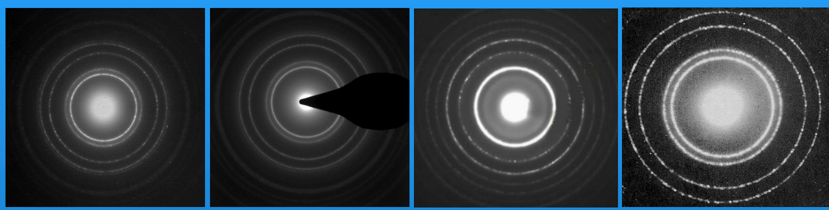


$$|R_{hkl}| = K \cdot \frac{1}{d_{hkl}}$$

For calibration of K factor !!
(Note: “camera constant”)

Learn to be an expert to recognize things!

The following four EDPs are taken from Au, Cu, Al and α-Fe polycrystalline respectively. Au, Cu and Al have f.c.c structure but α-Fe has b.c.c structure. Their lattice constants are given: a(Au)=0.408nm, a(Cu)=0.361nm, a(Al)=0.405nm, a(α-Fe)=0.286nm.



Questions:

1. *Can you recognize the EDP from b.c.c α-Fe structure simply by a quick glance of those patterns? .*
2. *How would you distinguish EDPs from f.c.c Au, Cu and Al?*
3. *What other information would you need to answer Question 2 ?*

See you next Tuesday….