Limitation of human eyes:

Color

390 to 700 nm

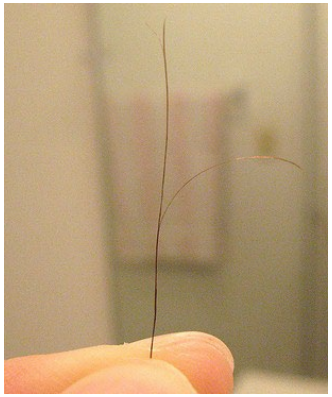
Color	Frequency	Wavelength
violet	668–789 THz	380–450 nm
blue	606–668 THz	450–495 nm
green	526–606 THz	495–570 nm
yellow	508–526 THz	570–590 nm
orange	484–508 THz	590–620 nm
red	400–484 THz	620–750 nm

Limitaton of human eyes:

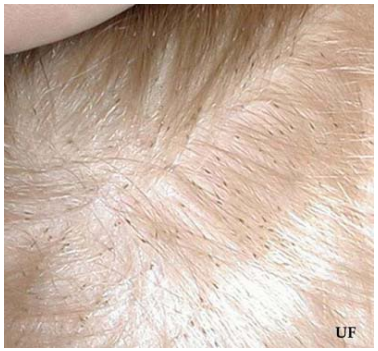
What Is the Smallest Thing You Can See?

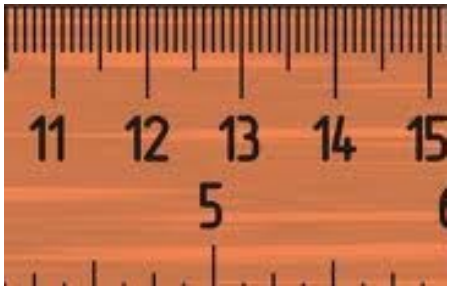
0.1 millimeters

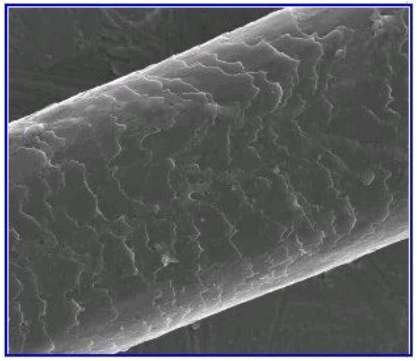
human hair

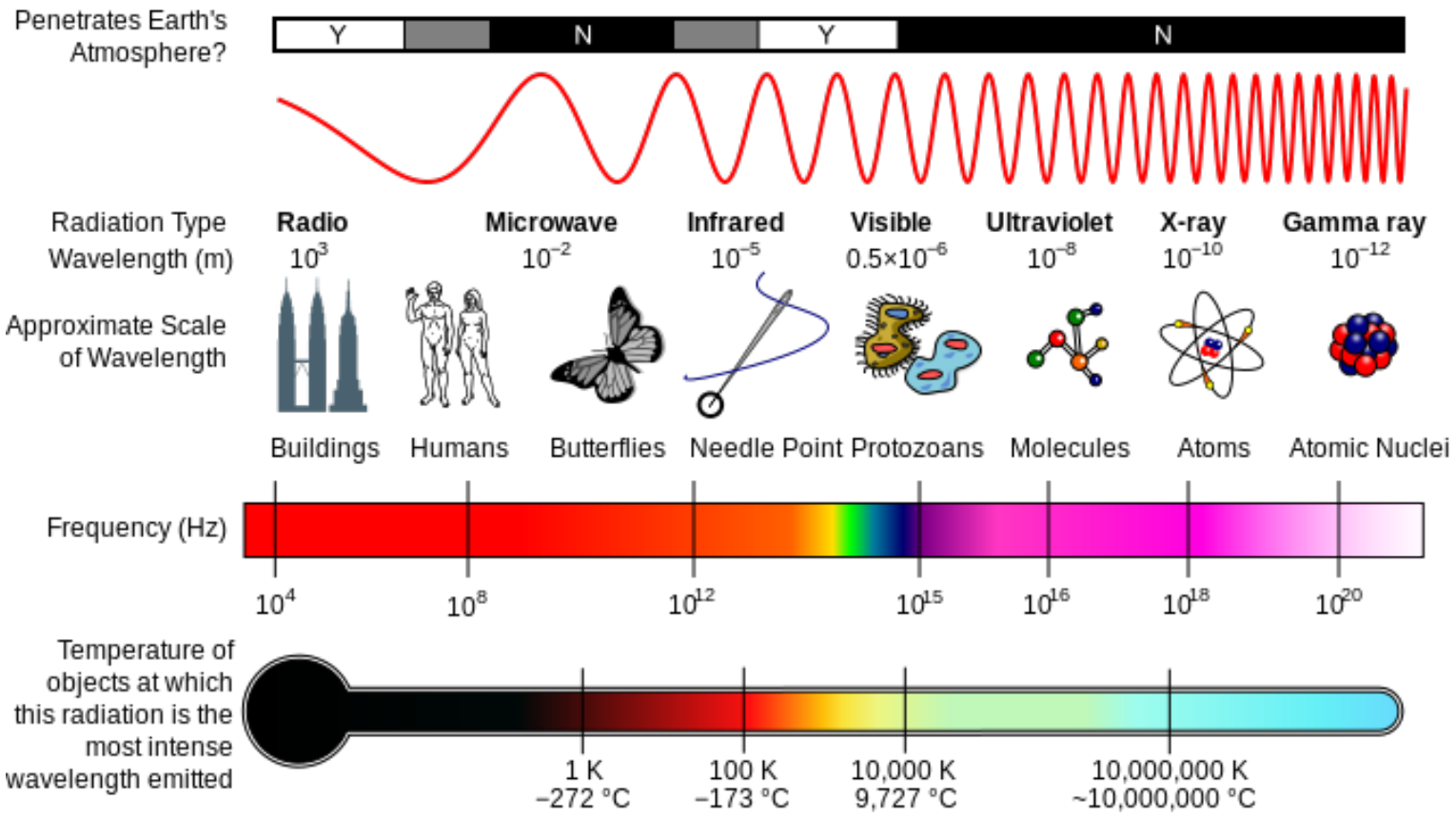


lice







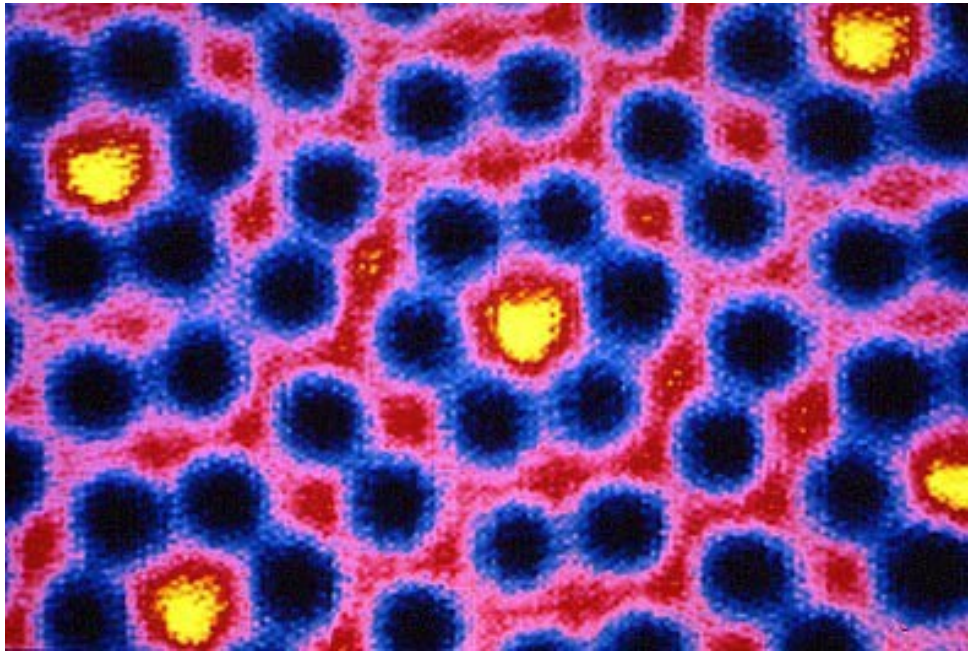

Penetrates Earth's Atmosphere?
Y
N
Y
N
Radiation Type
Wavelength (m)
Approximate Scale of Wavelength
Radio
10^{3}
Microwave
10^{-2}
Infrared
10^{-5}
Visible
0.5×10^{-6}
Ultraviolet
10^{-8}
X-ray
10^{-10}
Gamma ray
10^{-12}
Buildings
Humans
Butterflies
Needle Point
Protozoans
Molecules
Atoms
Atomic Nuclei
Frequency (Hz)
10^{4}
10^{8}
10^{12}
10^{15}
10^{16}
10^{18}
10^{20}
Temperature of objects at which this radiation is the most intense wavelength emitted
1 K
−272 °C
100 K
−173 °C
10,000 K
9,727 °C
10,000,000 K
~10,000,000 °C

Where the original idea of creating a better microscope came from (breaking free from optics, out of the box thinking)?

The story behind the birth of (STM--AFM)

The birth of STM at IBM:

Silicon surface atoms (color--enhanced by computer).



The world's first images of individual surface atoms and the bonds that hold them in place were produced by STM at IBM.

A little story behind the invention of AFM by Binnig: Gazing at the ceiling of house

According to Binnig, the inspiration for AFM came as he lay on the floor of his house.

He looked up and noticed the subtle structure of the ceiling, which reminded him of the topography seen in scanning tunneling microscope images.

He wondered why the surface must always be imaged with a current; why not a force?

Atomic Force Microscope
G. Binnig, C. F. Quate, Ch. Gerber

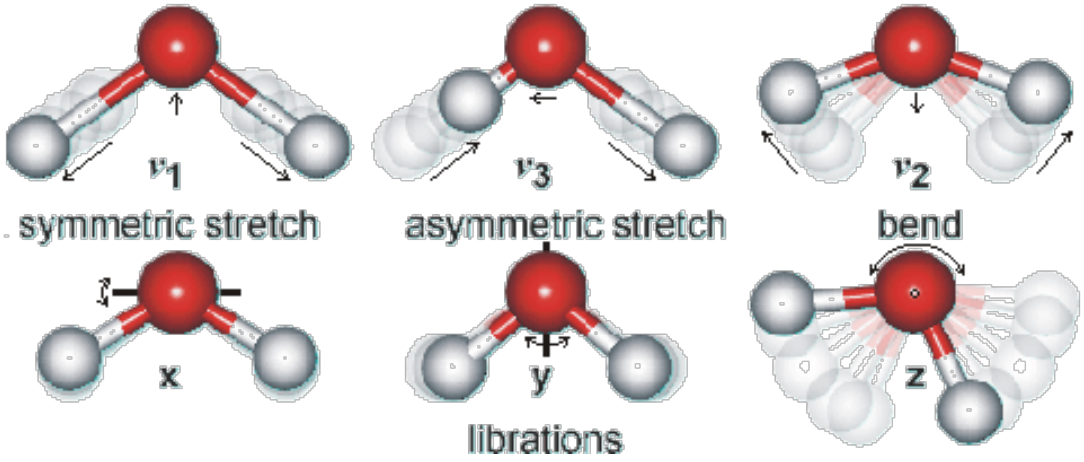
Phys. Rev. Lett. **56**, 930
Published 3 March, 1986

DOI: https://doi.org/10.1103/PhysRevLett.56.930

For example, the vibrational frequencies ω of atoms bound in a molecule or in a crystalline solid are typically 10^{13} Hz (10 THz) or higher.

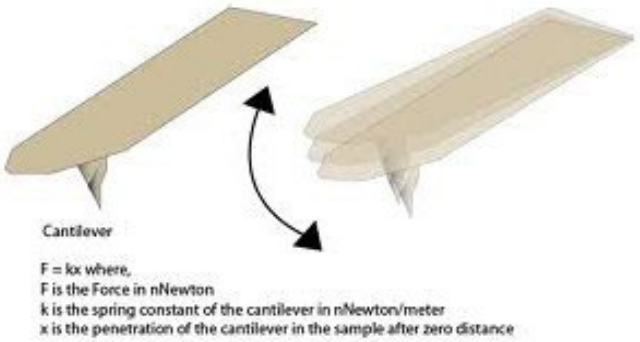
Combining this with the mass of the atoms, of order 10^{-25}kg, gives interatomic spring constants k, given by $\omega^2 m$, on the order **of 10 N/m.**

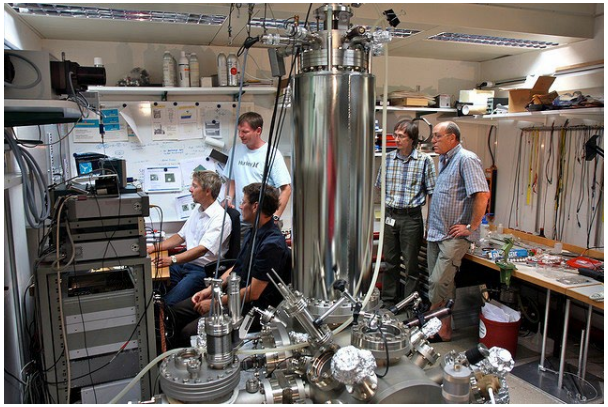
$$f = \frac{1}{2\pi}\sqrt{\frac{k}{m}}$$

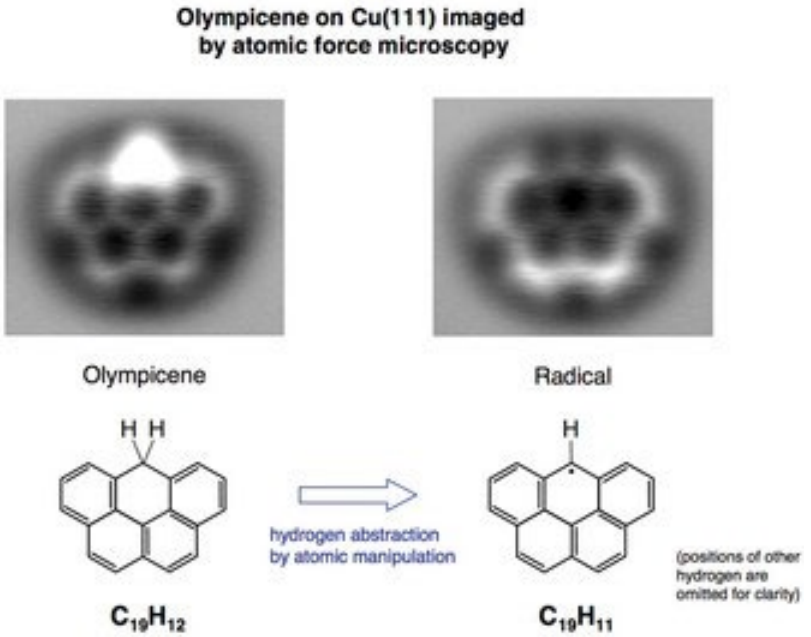


A little story behind the invention of AFM by Binnig:

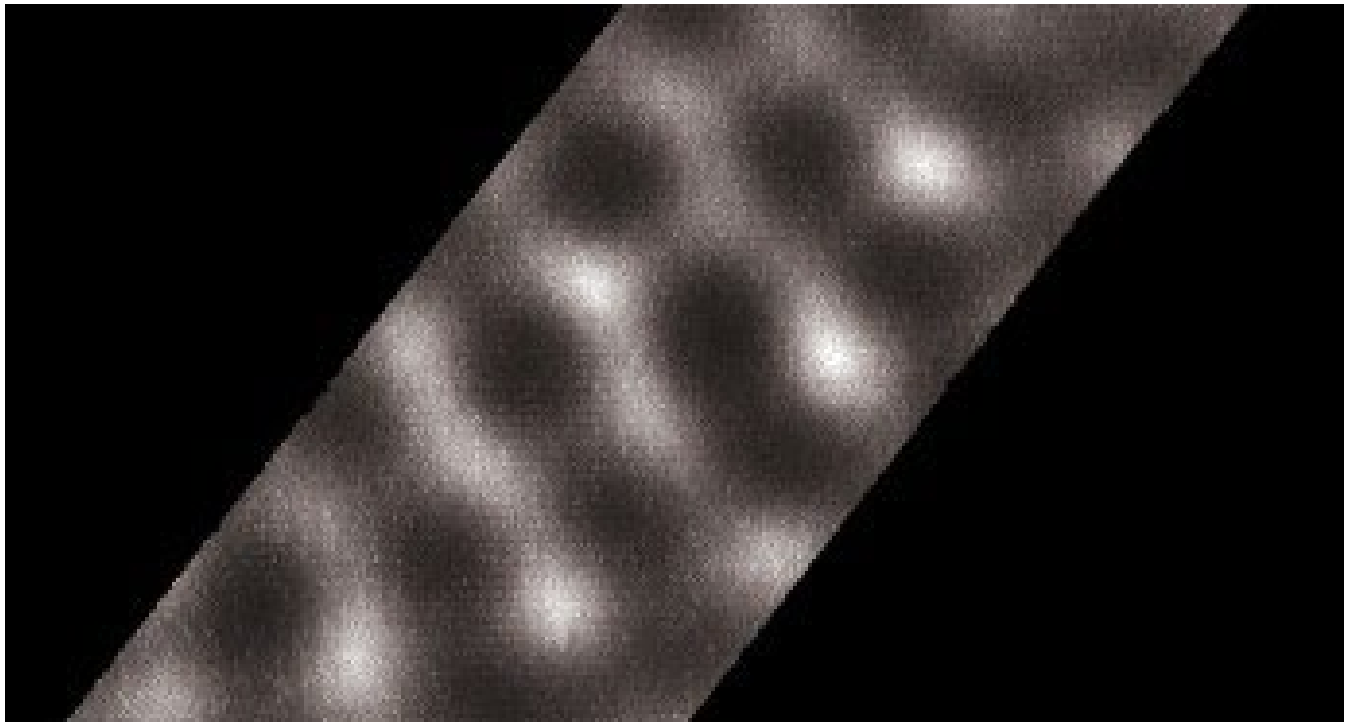
They believed that by sensing angstrom--size displacements of such a cantilever spring, one could image atomic--scale topography.







(b)

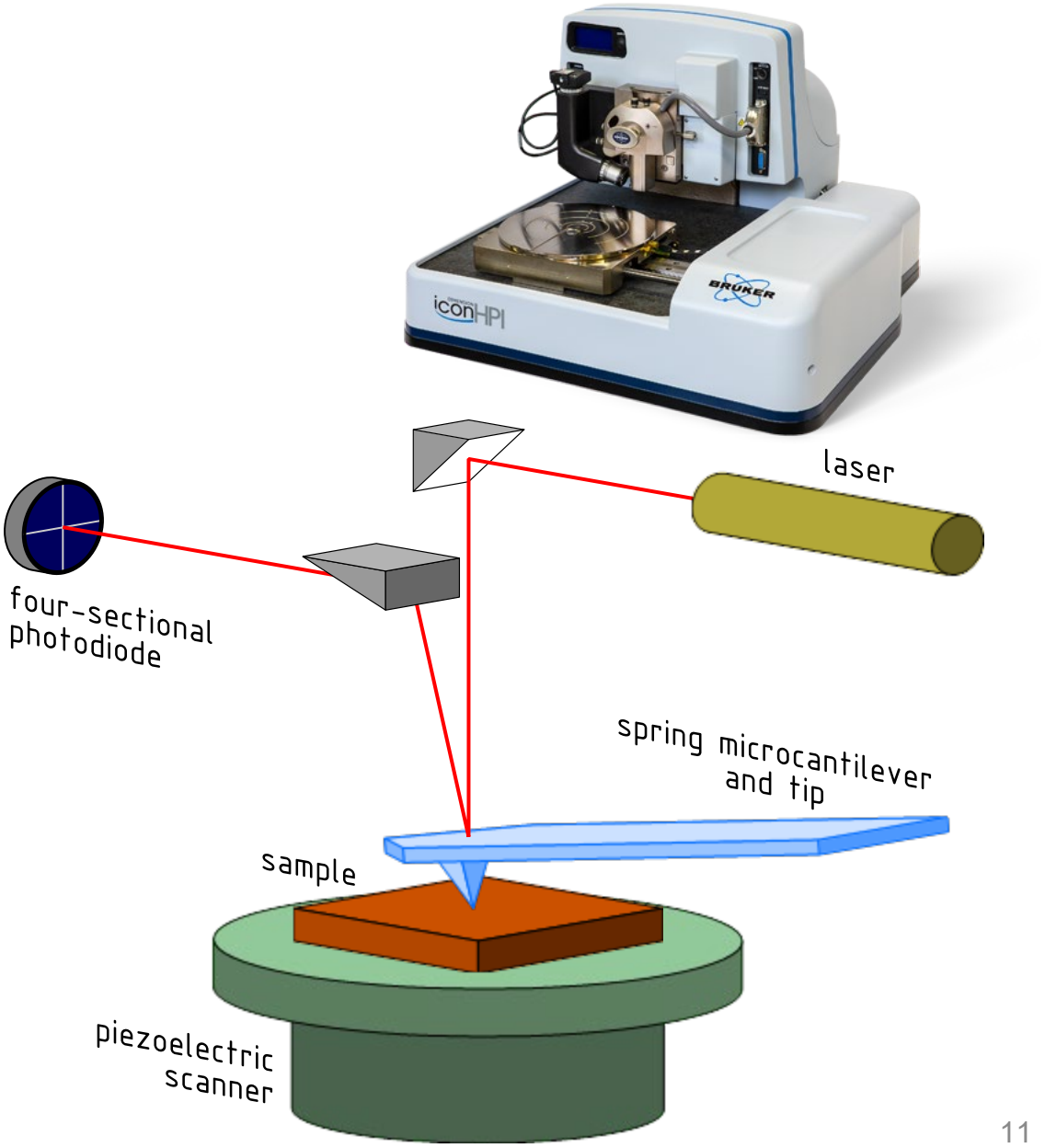


First atomic resolution with AFM on highly oriented pyrolytic graphite (HOPG)

AFM

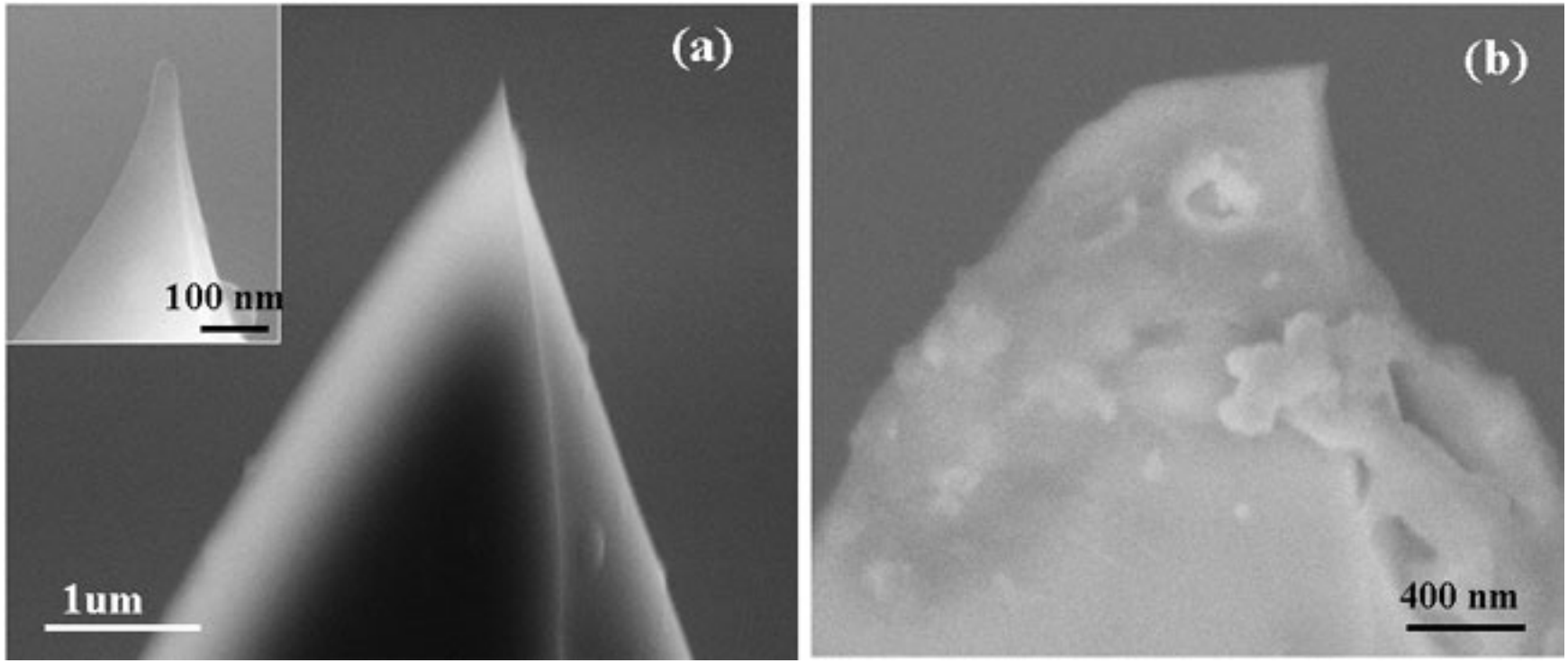
One kind of scanning probe microscopes (SPM).
SPMs are designed to measure local properties, such as height, friction, magnetism, with a probe.

To acquire an image, the SPM raster-scans the probe over a small area of the sample, measuring the local property simultaneously.



AFMs operate by measuring force between a probe and the sample. Normally, the probe is a sharp tip, which is a 3-6 micrometer tall pyramid with 15-40 nm end radius (Figure 1).

AFM tips and cantilevers are typically micro-fabricated from Si or Si_3N_4.



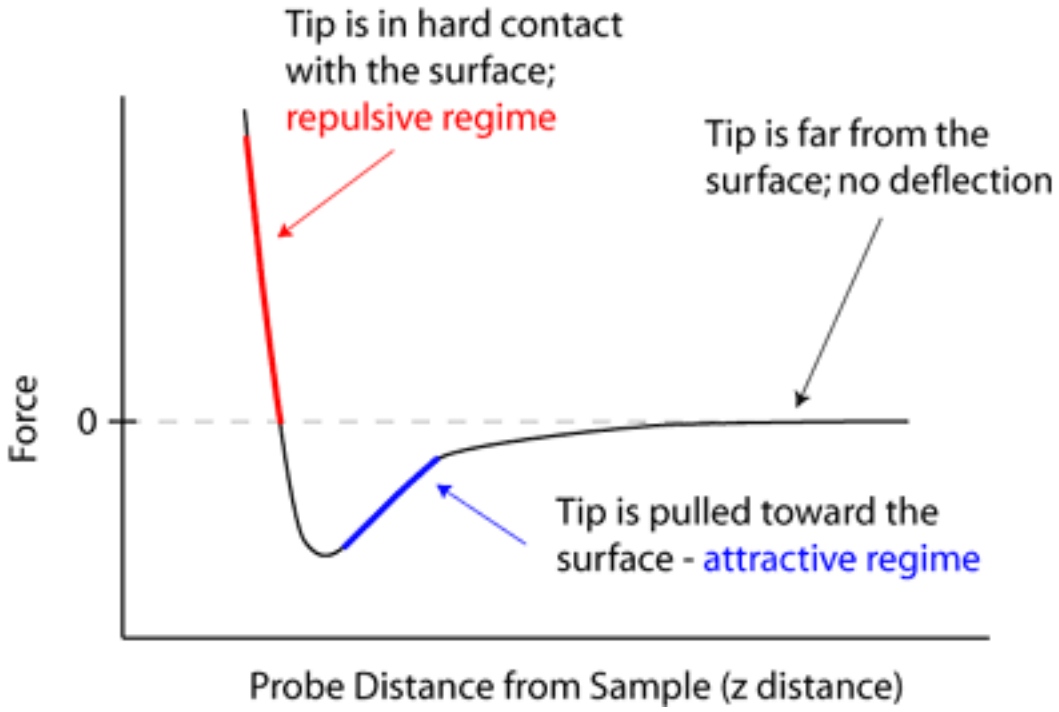
Measuring Forces

Because the Atomic Force Microscope relies on the forces between the tip and sample, these forces impact AFM imaging.

The force is not measured directly, but calculated by measuring the deflection of the lever, knowing the stiffness of the cantilever. Hooke's law gives:

$$F = -kz$$

where F is the force, k is the stiffness of the lever, and z is the distance the lever is bent.



Contact mode

In contact mode the tip contacts the surface through the adsorbed fluid layer on the sample surface. The detector monitors the changing cantilever deflection and the force is calculated using Hooke's law:

$F = -kx$ (F = force, k = spring constant, x = cantilever deflection)

The feedback circuit adjusts the probe height to try and maintain a constant force and deflection on the cantilever. This is known as the *deflection setpoint*.

Non-contact mode

In non-contact mode the cantilever oscillates near the surface of the sample, but does not contact it. The oscillation is at slightly *above* the resonant frequency. Van der Waals and other long-range forces decrease the resonant frequency just above the surface. This decrease in resonant frequency causes the amplitude of oscillation to decrease.
In ambient conditions the adsorbed fluid layer is often significantly thicker than the region where van der Waals forces are significant. So the probe is either out of range of the van der Waals forces it attempts to measure, or becomes trapped in the fluid layer. Therefore non-contact mode AFM works best under ultra-high vacuum conditions.

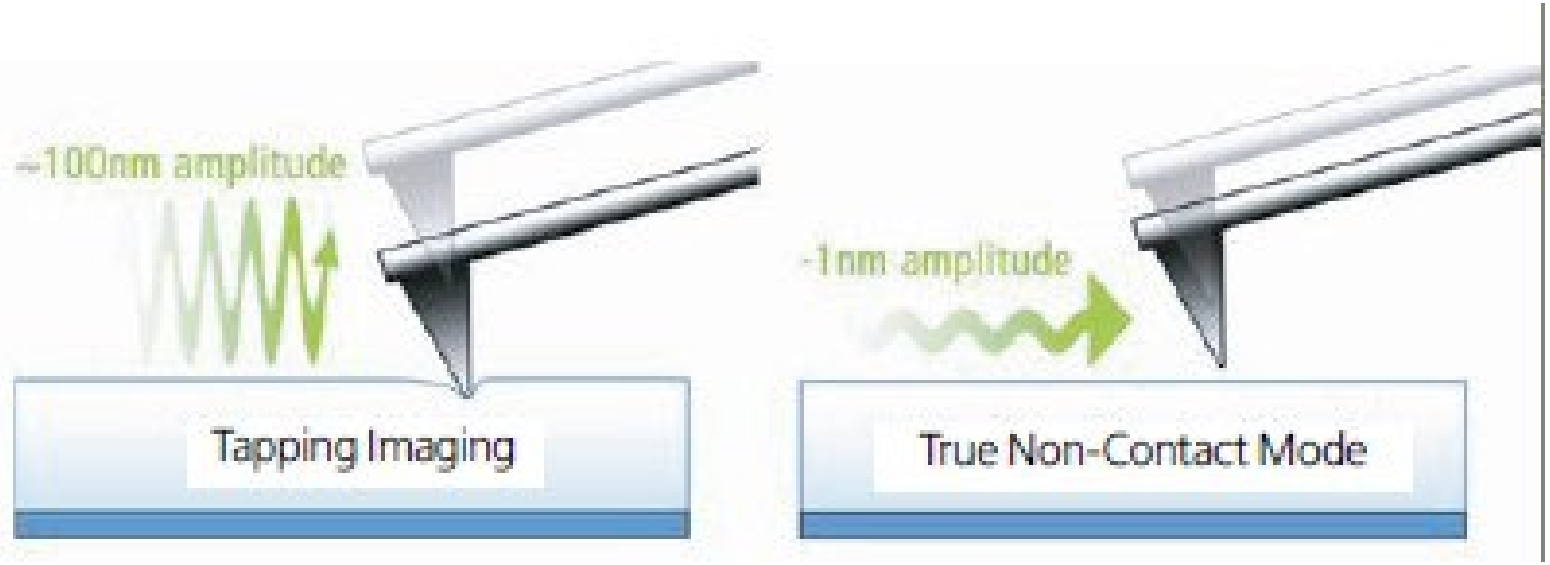
Difficulties in Non--Contact AFM and invention of tapping mode AFM (1993)

Difficulties in Non--Contact AFM and invention of tapping mode AFM (1993):

It is important to maintain the tip--sample distance at a certain constant value (~ a few nm) and to prevent the tip from contacting the sample surface.

If tip makes an accidental contact with the sample, it can stuck to the sample surface and stop vibrating due to the meniscus force of the liquid layer on the sample surface.

Therefore, a very high performing Z--servo system is required for NC--AFM. The mechanical response of the Z--scanner must be very fast.

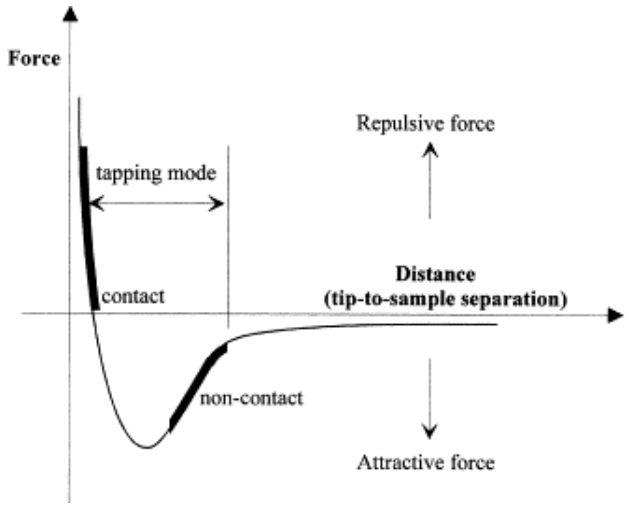


www.parkAFM.com

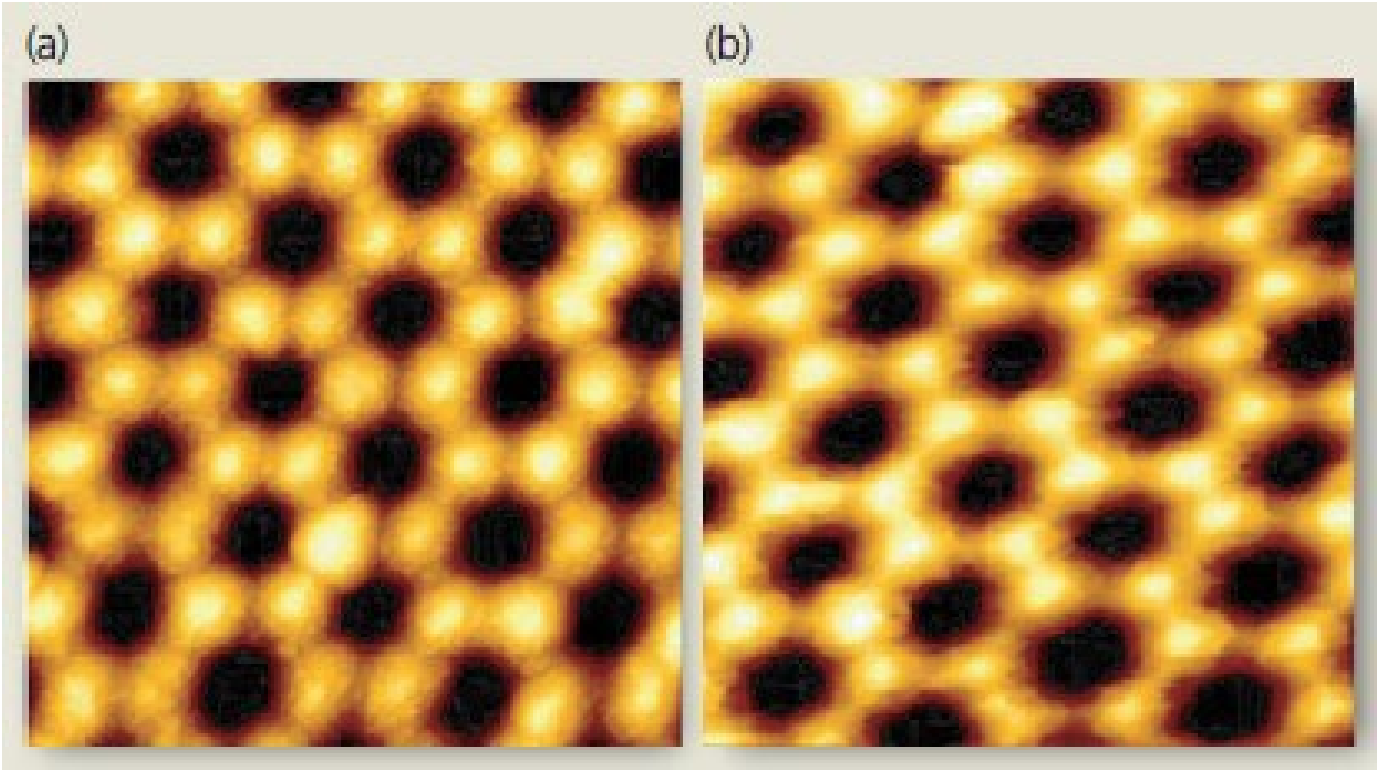
Invention of tapping mode AFM (1993):

To solve this problem, Zhong et al. introduced a 'tapping' imaging in which a tip strikes against the surface on each oscillation cycle and detaches from the sample surface by using a large vibration amplitude.

Poor resolution in comparison to NC–AFM since the very sharp end of a tip is extremely fragile and becomes blunt instantaneously as it makes aggressive contact with the sample.



True NonContact AFM vs. tapping imaging AFM.



www.parkAFM.com

Tapping Mode

In tapping mode, the cantilever oscillates at or slightly *below* its resonant frequency. The amplitude of oscillation typically ranges from 20 nm to 100 nm. The tip lightly “taps” on the sample surface during scanning, contacting the surface at the bottom of its swing.
Because the forces on the tip change as the tip-surface separation changes, the resonant frequency of the cantilever is dependent on this separation.

$$\omega = \omega_0 \sqrt{1 - \frac{1}{k}\frac{\mathrm{d}F}{\mathrm{d}z}}$$

The oscillation is also damped when the tip is closer to the surface. Hence changes in the oscillation amplitude can be used to measure the distance between the tip and the surface. The feedback circuit adjusts the probe height to try and maintain a constant amplitude of oscillation i.e. the *amplitude setpoint*.

	Advantage	Disadvantage
Contact Mode	•High scan speeds •Rough samples with extreme changes in vertical topography can sometimes be scanned more easily	•Lateral (shear) forces may distort features in the image •In ambient conditions may get strong capillary forces due to adsorbed fluid layer •Combination of lateral and strong normal forces reduce resolution and mean that the tip may damage the sample, or vice versa
Tapping Mode	•Lateral forces almost eliminated •Higher lateral resolution on most samples •Lower forces so less damage to soft samples or tips	•Slower scan speed than in contact mode
Non-contact Mode	•Both normal and lateral forces are minimised, so good for measurement of very soft samples •Can get atomic resolution in a UHV environment	•In ambient conditions the adsorbed fluid layer may be too thick for effective measurements •Slower scan speed than tapping and contact modes to avoid contacting the adsorbed fluid layer

Scanning Electron Microscopy

A **scanning electron microscope** (**SEM**) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons.

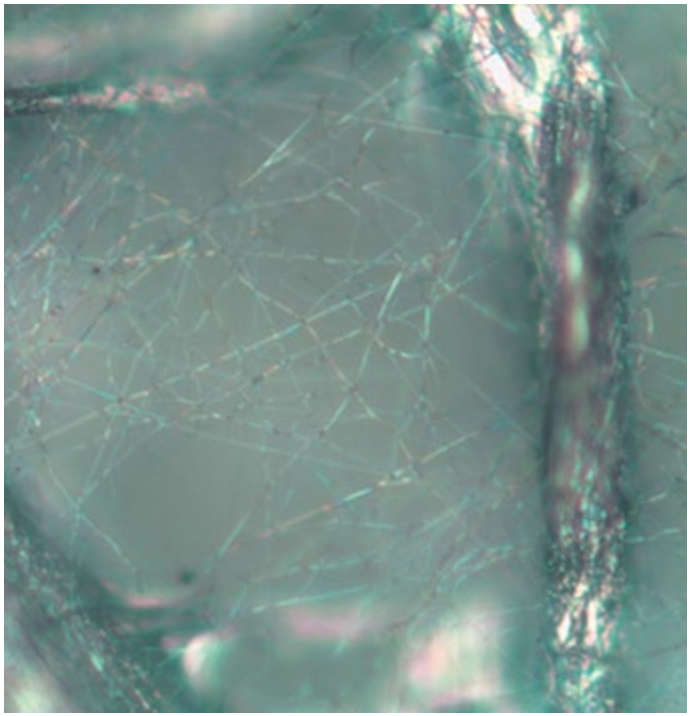
Max Knoll
M. Von Ardenne
Cecil E. Hall
Vladimir K. Zworykin
Charles Oatley
Cambridge Scientific Instrument Company 1965

Why use electrons?

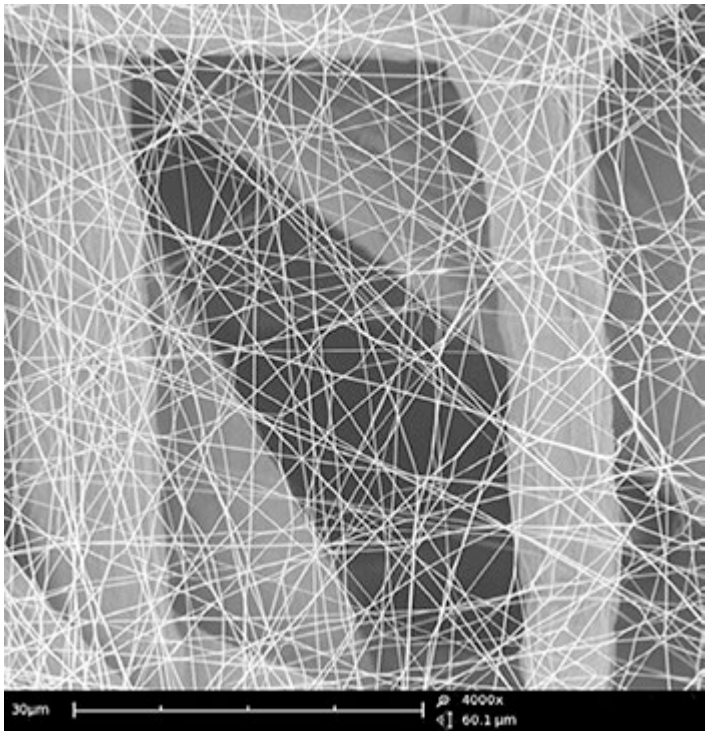
$$\lambda = \frac{h}{\sqrt{2meV}}$$

h = Planck's constant (6.626 x 10^{-34} m^2kg/s) | *m* = mass of electron (9.109 x 10^{-31} kg) | *e* = electron charge (1.60 x 10^{-19} C) | *V* = accelerating voltage

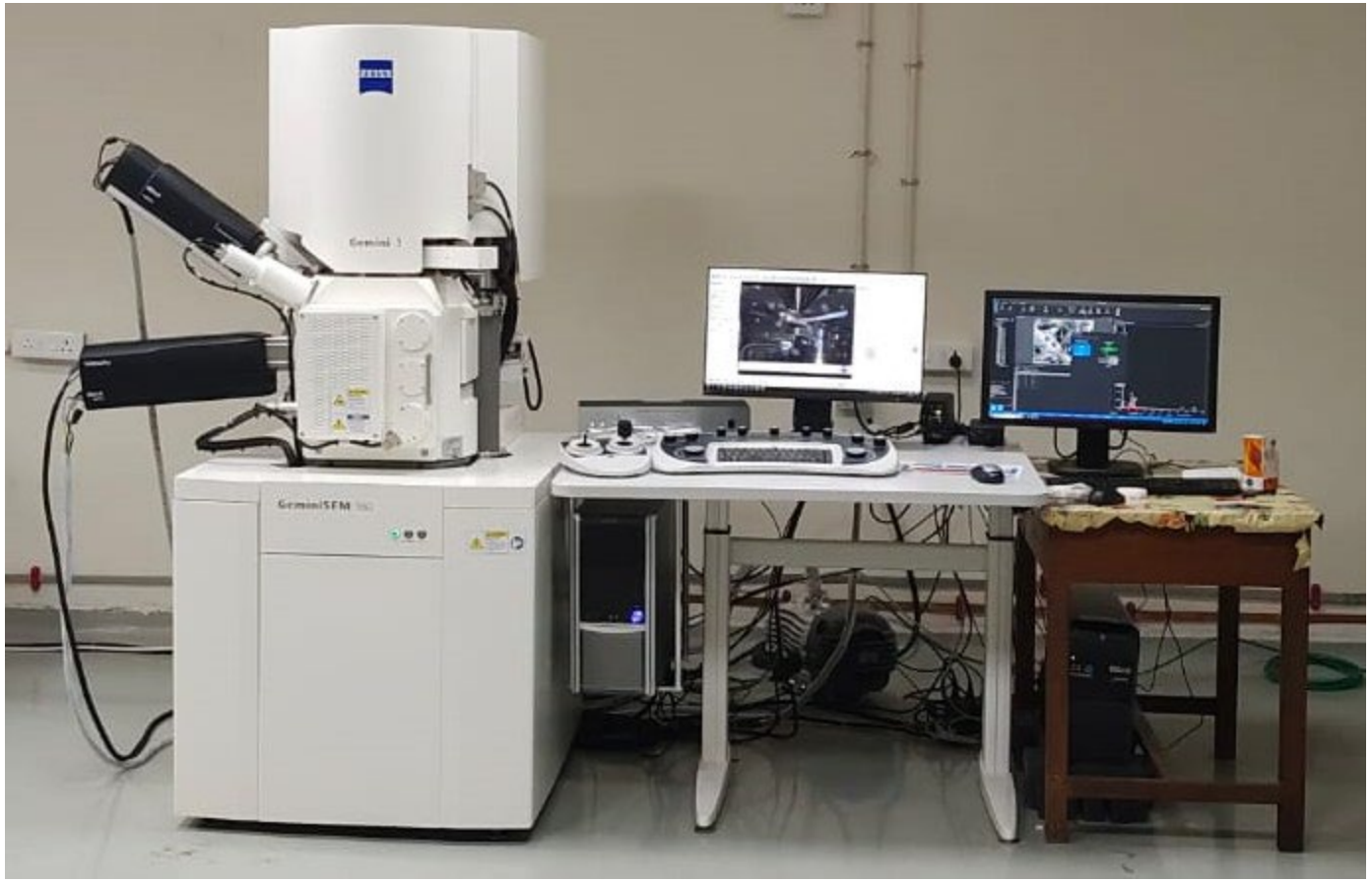
Accelerating Voltage	Type of Electron Microscope	Electron Wavelength, λ
5 kV	SEM	0.0172 nm
10 kV		0.0122 nm
20 kV		0.0086 nm
200 kV	TEM/STEM	0.0027 nm



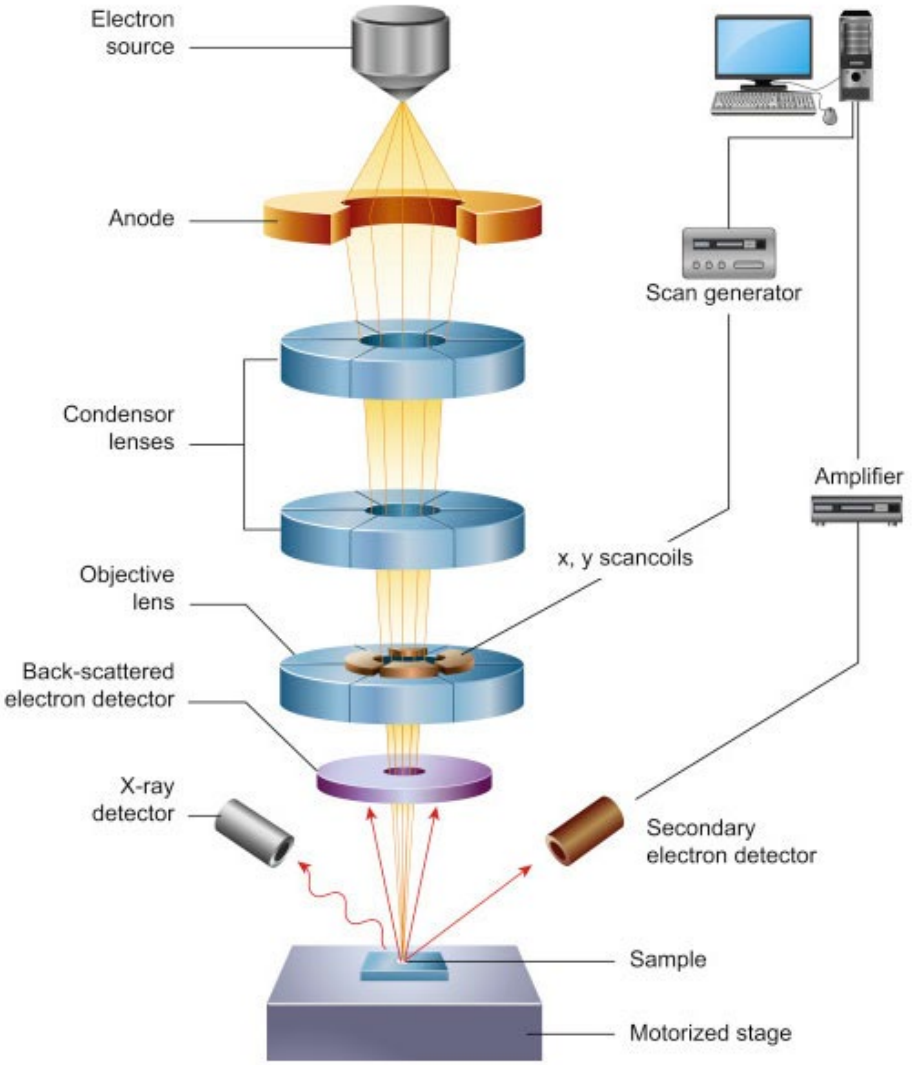
Optical microscope image of nanofibers



Scanning electron microscope image at 4000x magnification of the same nanofibers.



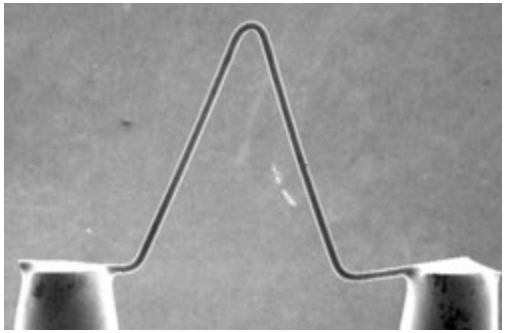
Zeiss GeminiSEM 560 at BHU Central Discovery Centre


Electron source
Anode
Condensor lenses
Objective lens
Back-scattered electron detector
X-ray detector
Scan generator
x, y scancoils
Amplifier
Secondary electron detector
Sample
Motorized stage

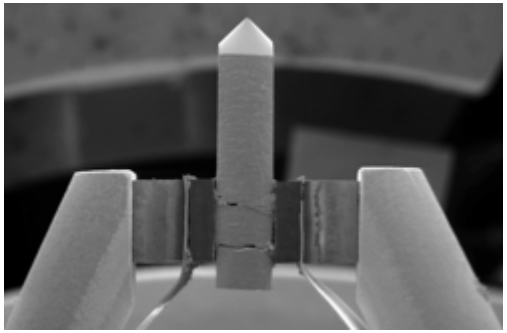

FEI
NOVA NANOSEM 450

Electron Source

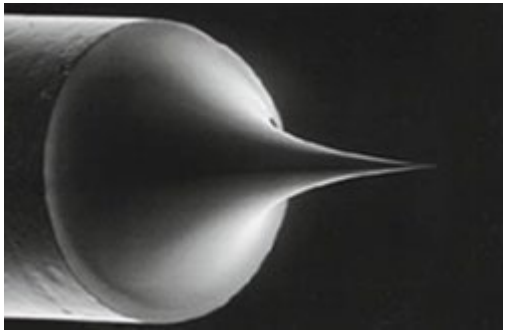
SEM image of the crystal



Tungsten Filament Source

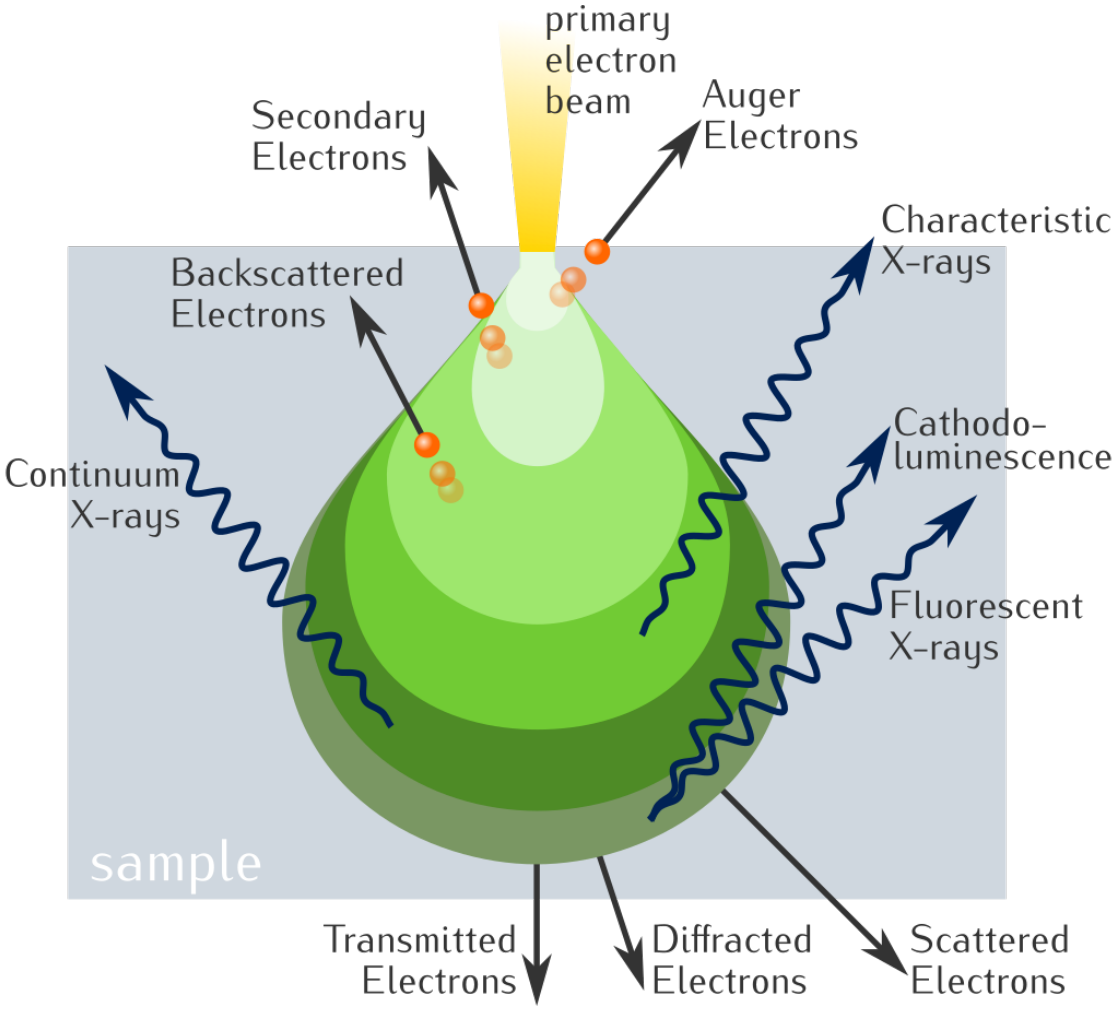


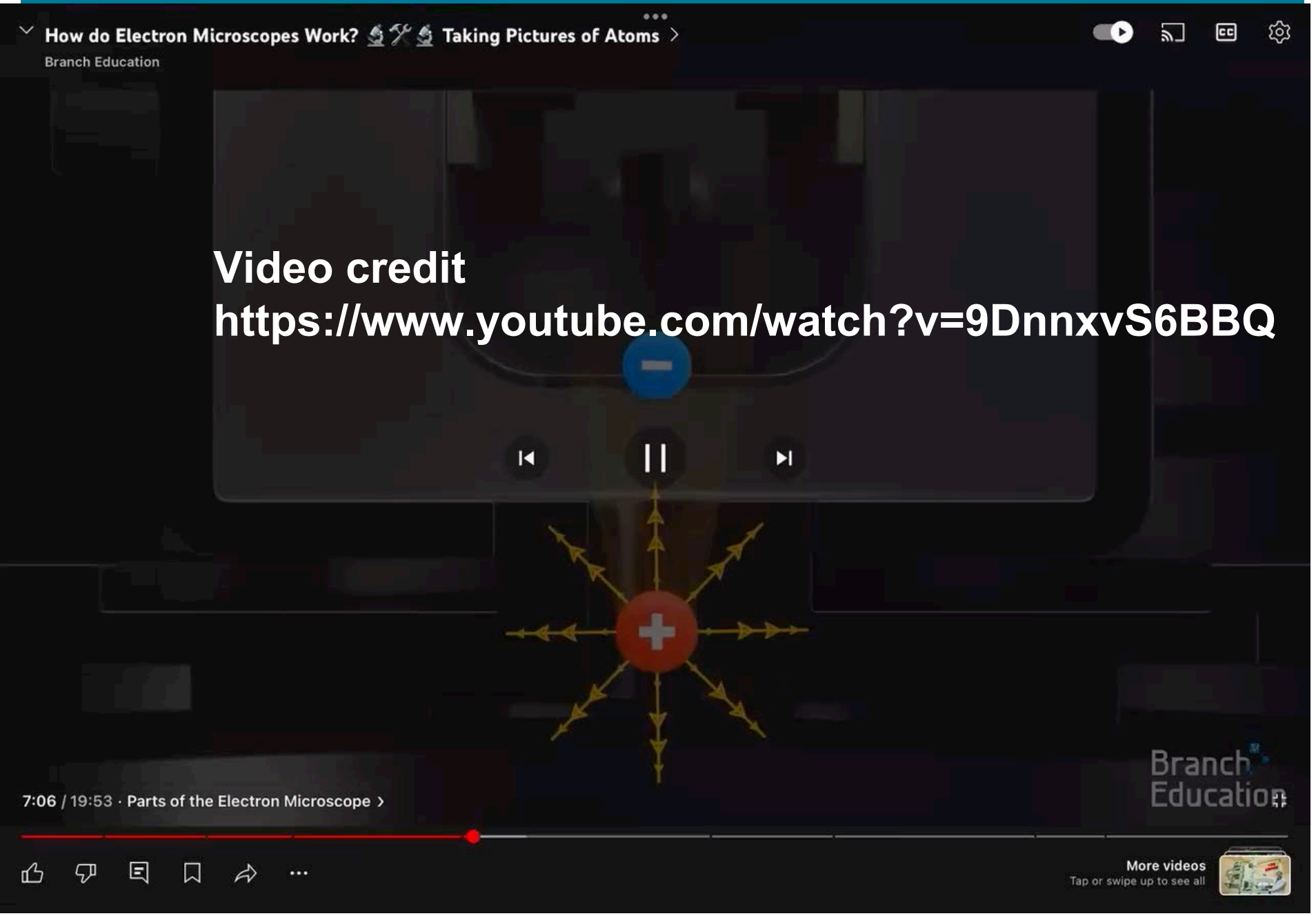
Lanthanum Hexaboride (LaB_6) or Cerium Hexaboride (CeB_6)



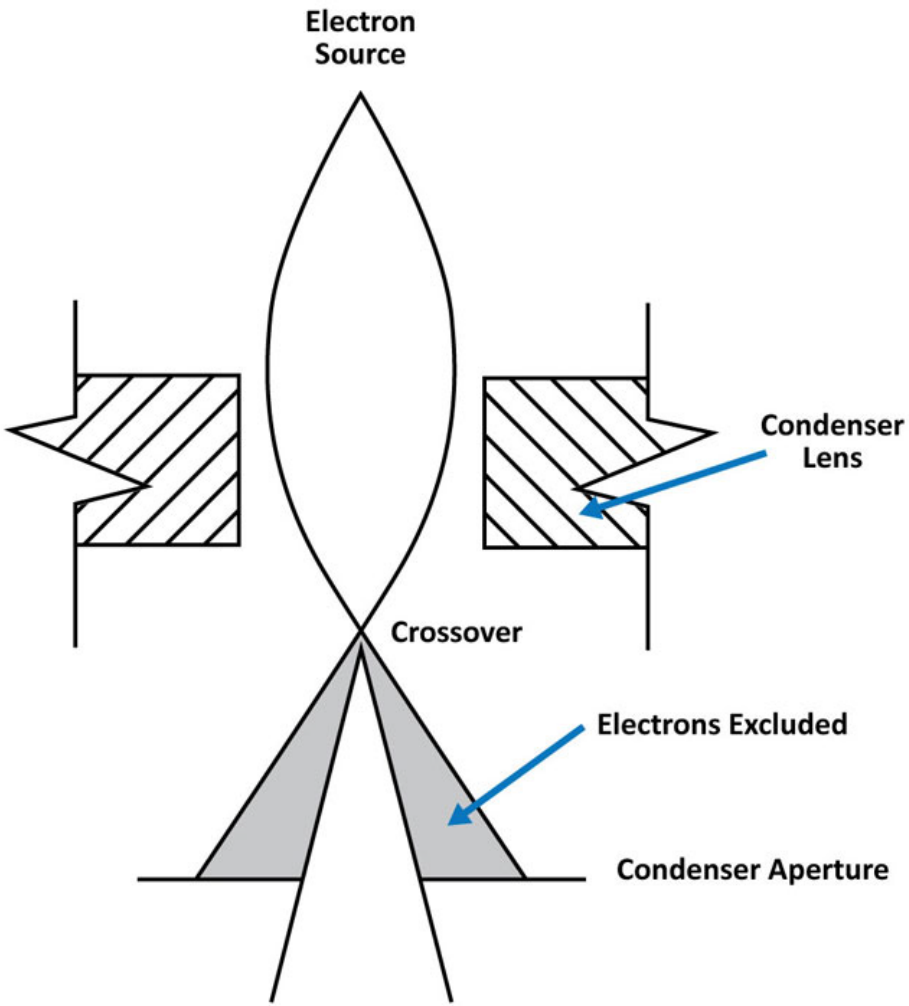
FEG source
sharp tip, less than 100 nm

Electron-Sample Interaction

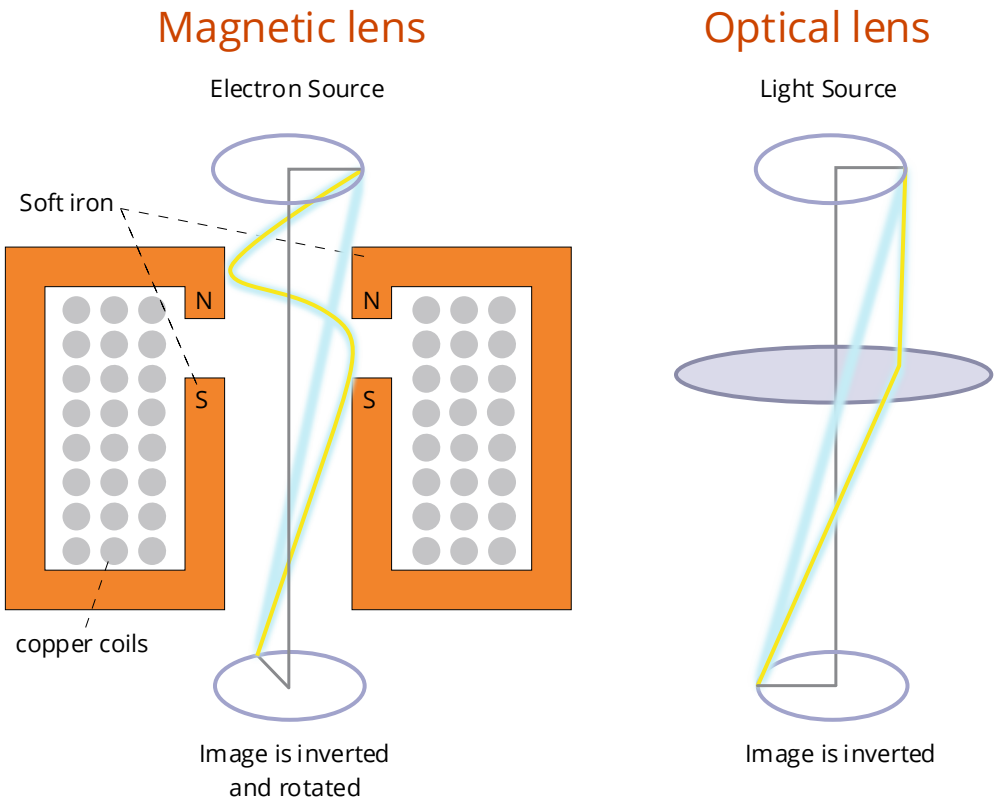



How do Electron Microscopes Work? 🔬🛠️🔬 Taking Pictures of Atoms
Branch Education
Video credit
https://www.youtube.com/watch?v=9DnnxvS6BBQ
7:06 / 19:53 · Parts of the Electron Microscope
Branch Education
More videos
Tap or swipe up to see all

Condenser Lens

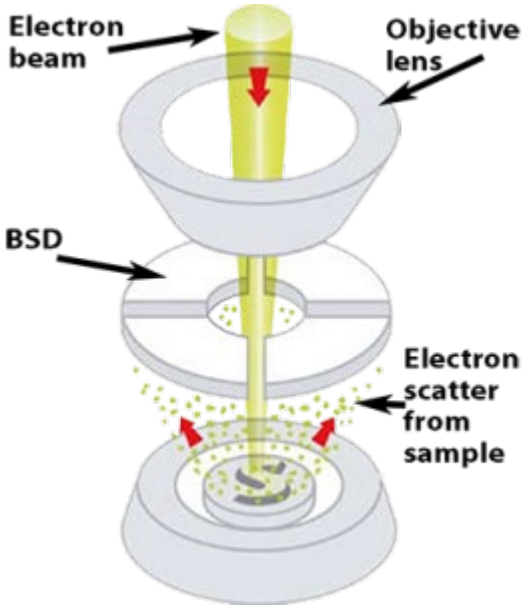


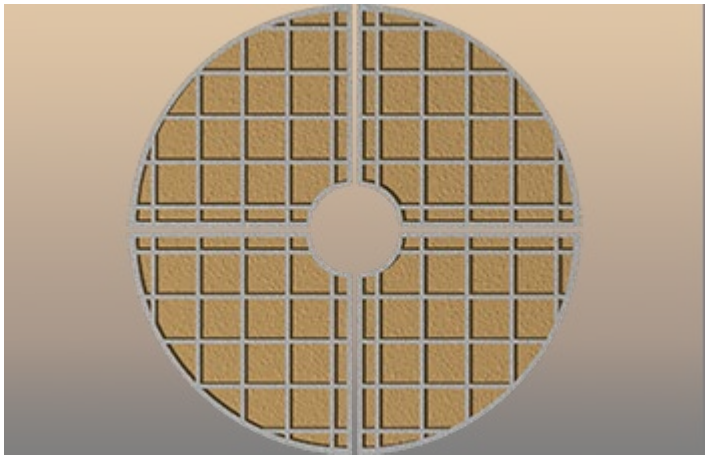
Electromagnetic lens vs Optical Lens



Video credit
https://www.youtube.com/watch?v=9DnnxvS6BBQ

Back Scattered Detector (BSD)





The most common type of BSD is composed of a solid-state sensor

The contrast of the backscattered electron image depends on multiple factors, including the atomic number (Z) of the sample material, the acceleration voltage of the primary beam, and the specimen angle (tilt) in relation to the primary beam.

Secondary electron detector

Everhart–Thornley detector (**E–T detector** or **ET detector**)

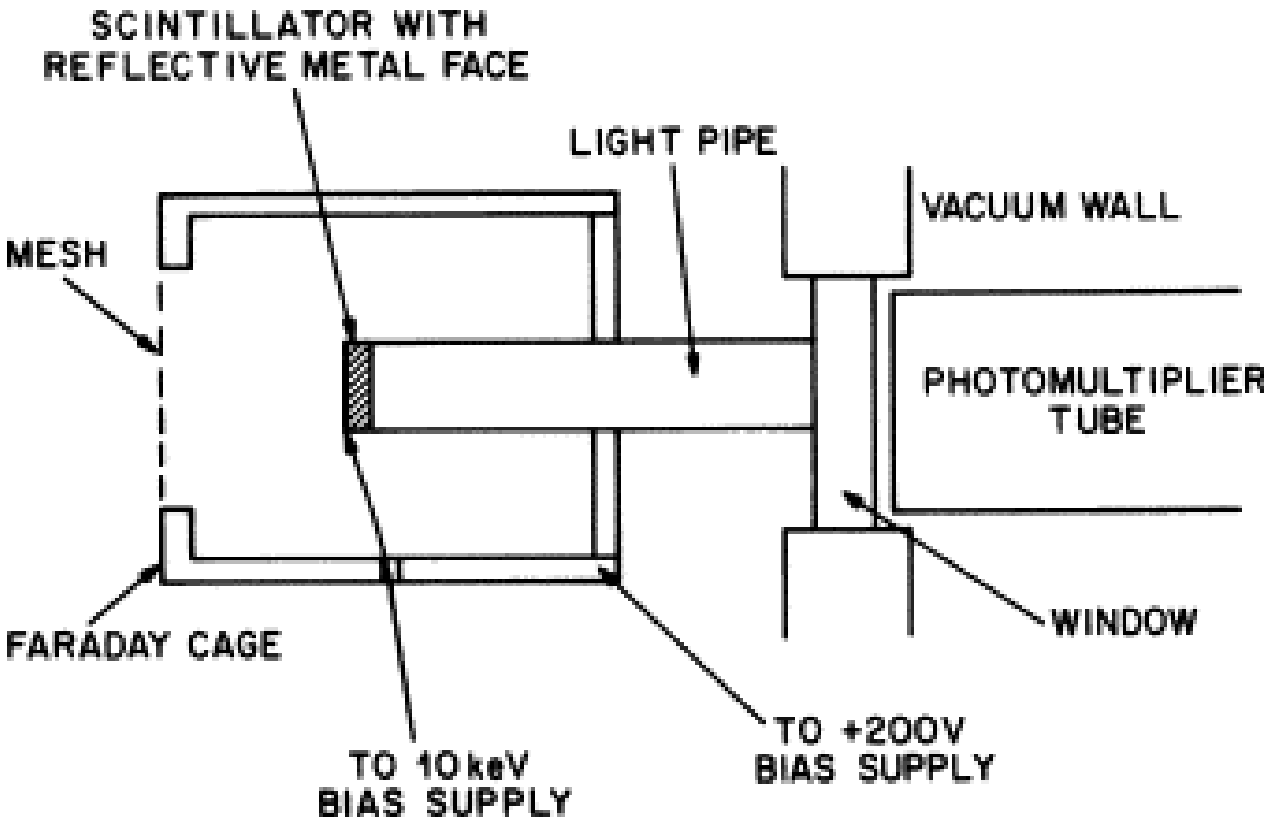
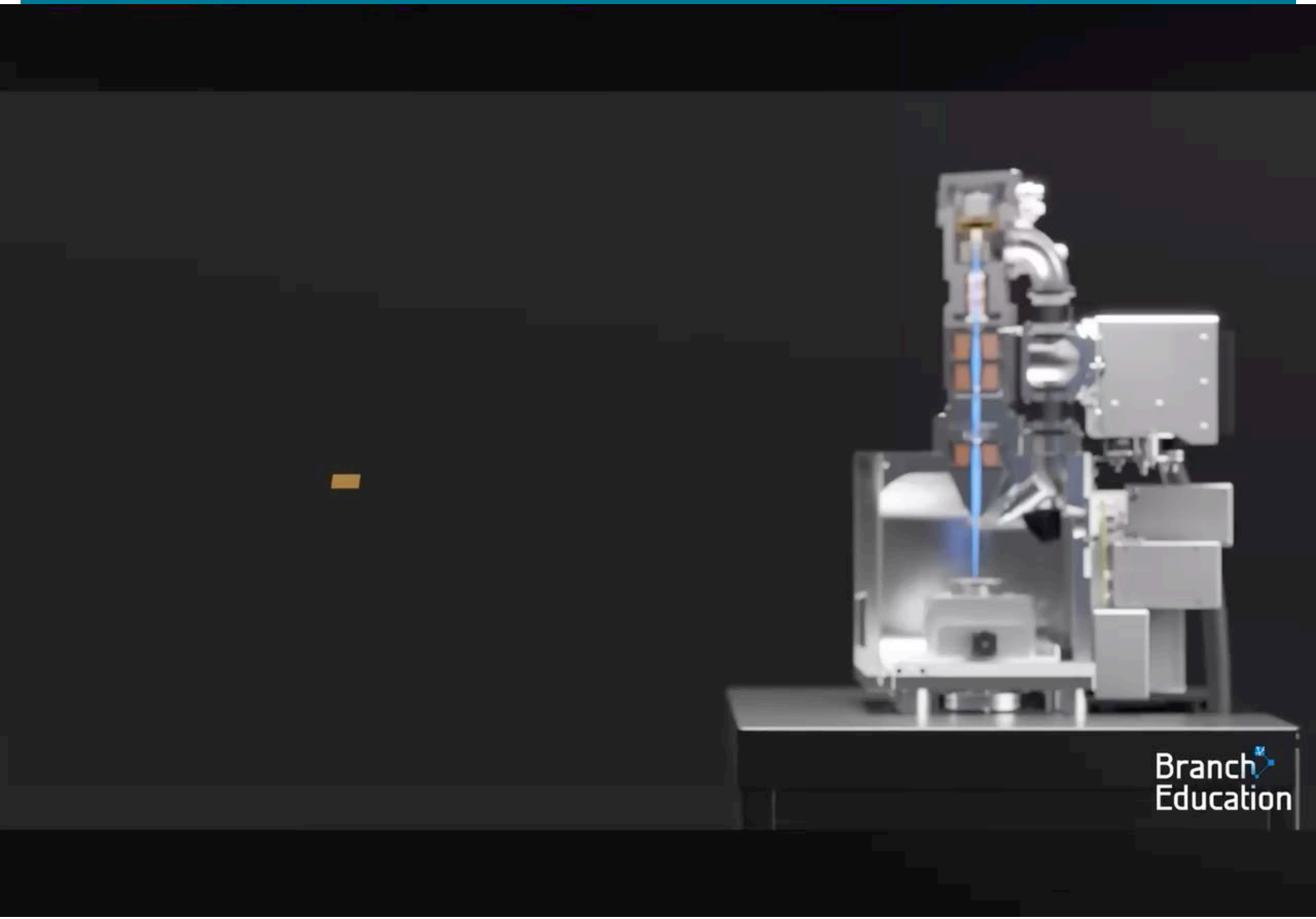
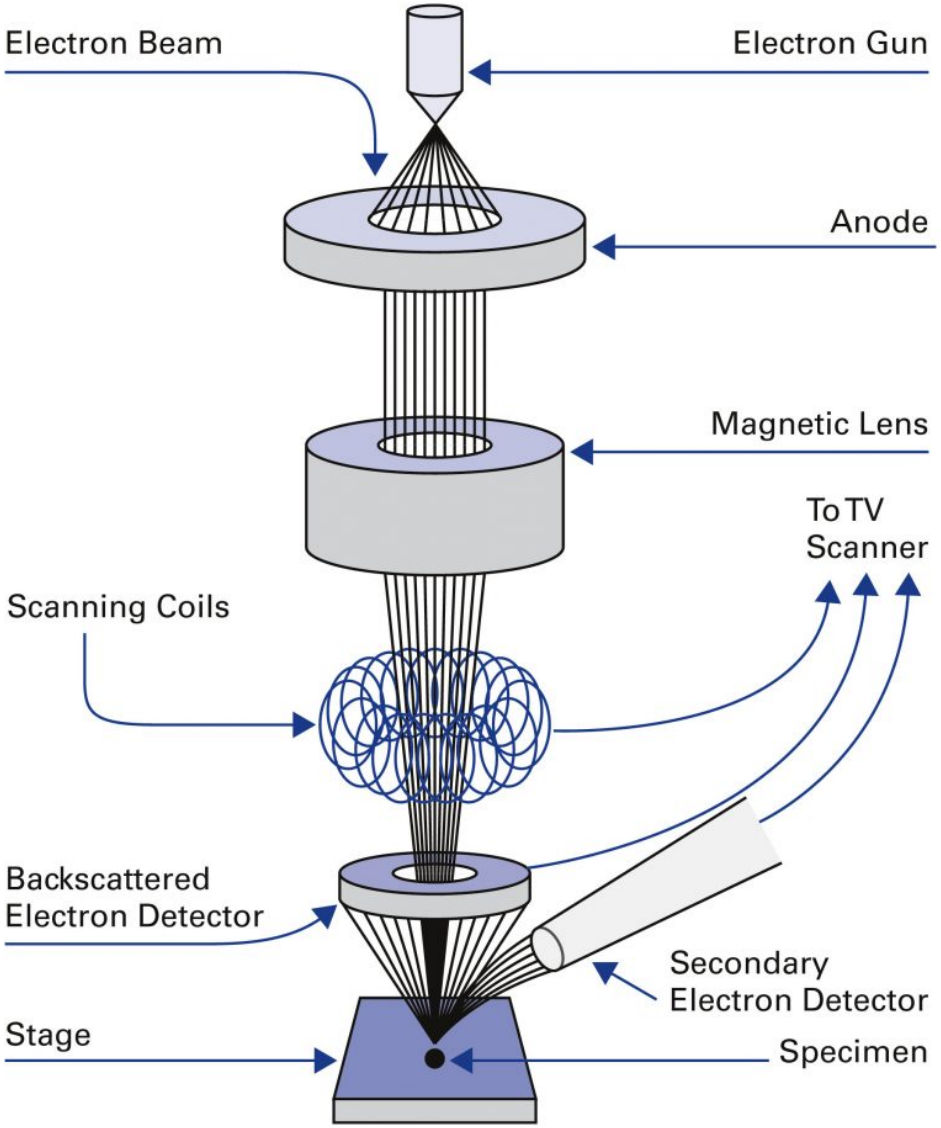
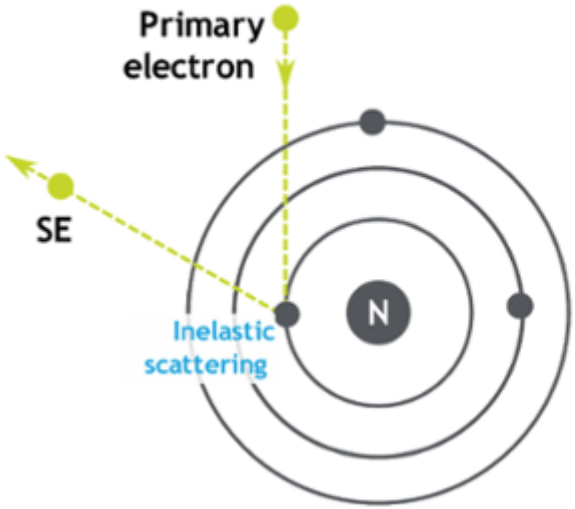
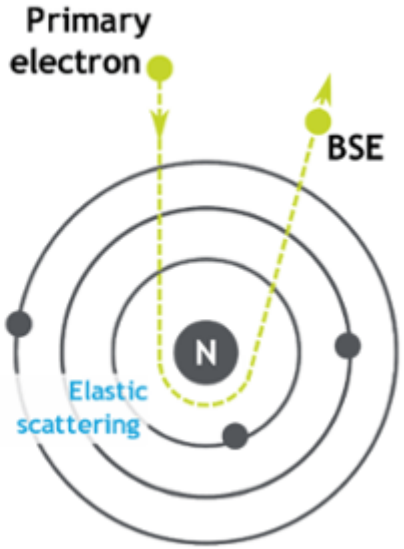


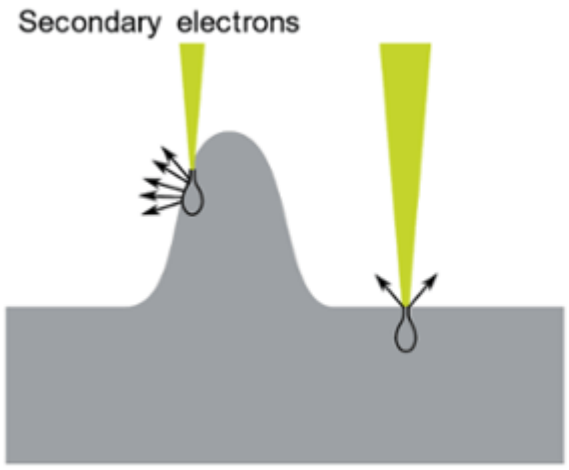
Image of an E-T detector

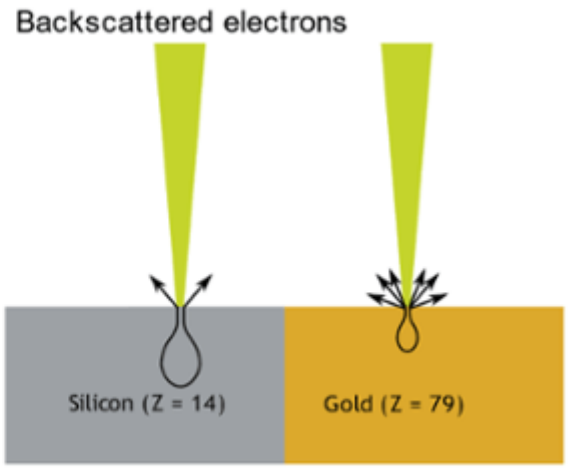

Branch
Education


Electron Beam
Electron Gun
Anode
Magnetic Lens
To TV
Scanner
Scanning Coils
Backscattered
Electron Detector
Secondary
Electron Detector
Stage
Specimen









More secondary electrons are emitted on edges and slopes, this causes brighter contrast than on flat areas, giving users useful information of the sample's morphology/ topography.

In contrast, the number of backscattered electrons emitted will very much depend on the material in question, as can be seen on the right of the figure. For example, if the beam hits silicon atoms (which have atomic number Z=14), less backscattered electrons will be formed than if the beam hits gold (which has atomic number Z=79).

Backscattered Electrons (BSE)

Backscattered electrons are **high-energy electrons** that originate from the electron beam and get **reflected (or backscattered)** out of the sample due to interactions with atomic nuclei. These electrons have higher energy than secondary electrons and carry important information about the composition of the sample.

Key Characteristics of BSE:

- **High Energy:** Retain a large portion of their original energy.
- **Composition Sensitivity:** Heavier elements (higher atomic number **Z**) backscatter more electrons, making them appear brighter in an SEM image.
- **Lower Resolution:** Due to their higher energy, BSE images tend to have lower resolution than SE images.
- **Useful for Material Contrast:** Great for distinguishing different elements in a sample.

When to Use BSE Imaging:

- Identifying **elemental composition** variations in a sample.
- Examining **multi-phase materials** where contrast between different materials is needed.
- Studying **geological samples**, metals, and semiconductors.

Secondary Electrons (SE)

Secondary electrons are **low-energy electrons** that are ejected from the sample's surface after being hit by the electron beam. These electrons provide detailed information about the topography of the sample.

Key Characteristics of SE:

- **Low Energy:** Typically less than 50 eV.
- **Surface Sensitivity:** Since SEs come from the topmost layer of the material, they provide **high-resolution** surface details.
- **Better Resolution than BSEs:** Due to their low energy and short escape depth, SE imaging gives sharp, detailed images.
- **Less Dependent on Atomic Number:** Unlike BSEs, SE contrast is primarily influenced by surface morphology, not composition.

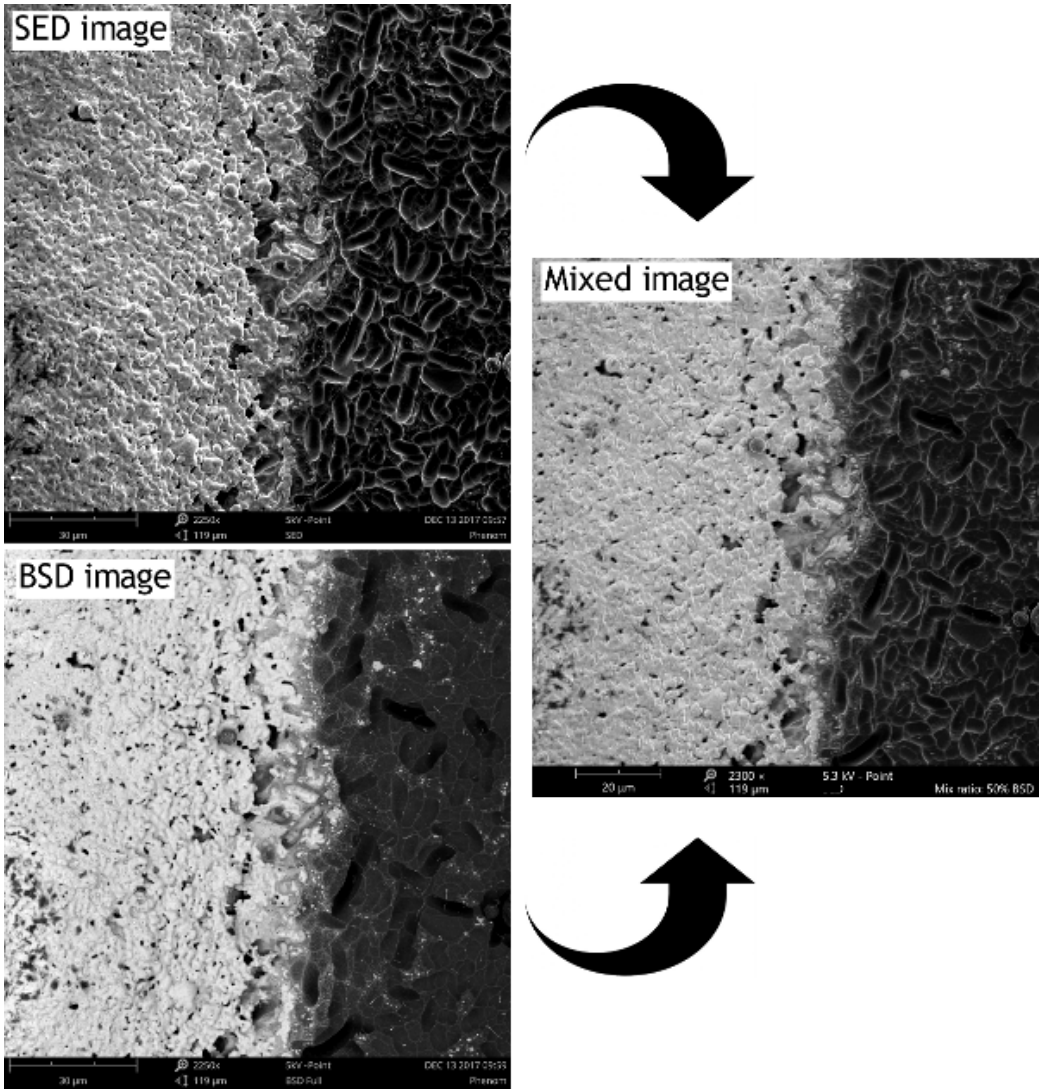
When to Use SE Imaging:

- Analyzing **fine surface structures** such as nanomaterials and biological specimens.
- Capturing **high-resolution textures** of metals, polymers, and microelectronics.
- Studying **fracture surfaces, coatings, and corrosion** effects.

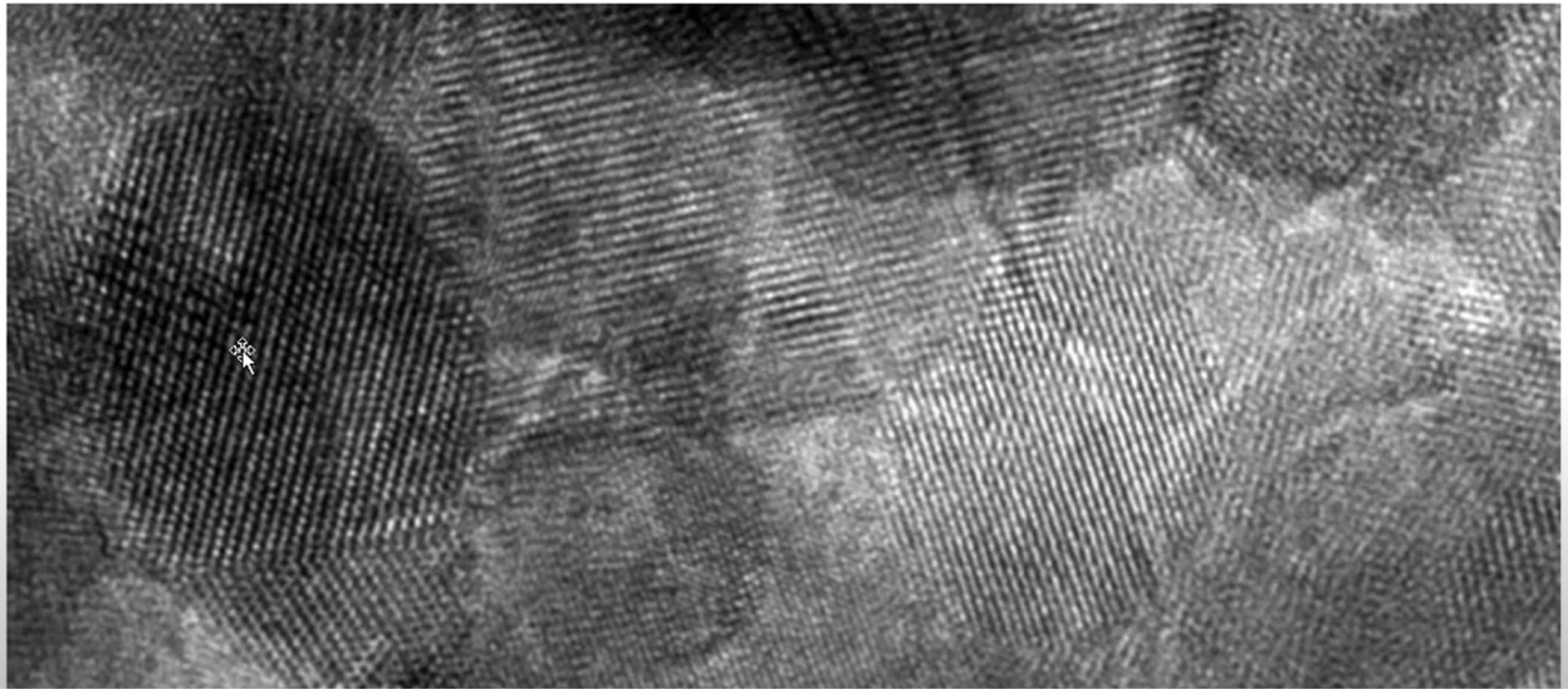
BSE vs. SE: A Side-by-Side Comparison

Feature	Backscattered Electrons (BSEs)	Secondary Electrons (SEs)
Energy Level	High energy (elastic scattering)	Low energy (inelastic scattering)
Origin	Interaction with atomic nuclei	Ejected from the sample surface
Image Contrast	Based on atomic number differences (Z Contrast)	Based on surface topography
Resolution	Lower	Higher
Best For	Material contrast & composition	Surface details & textures (topography)

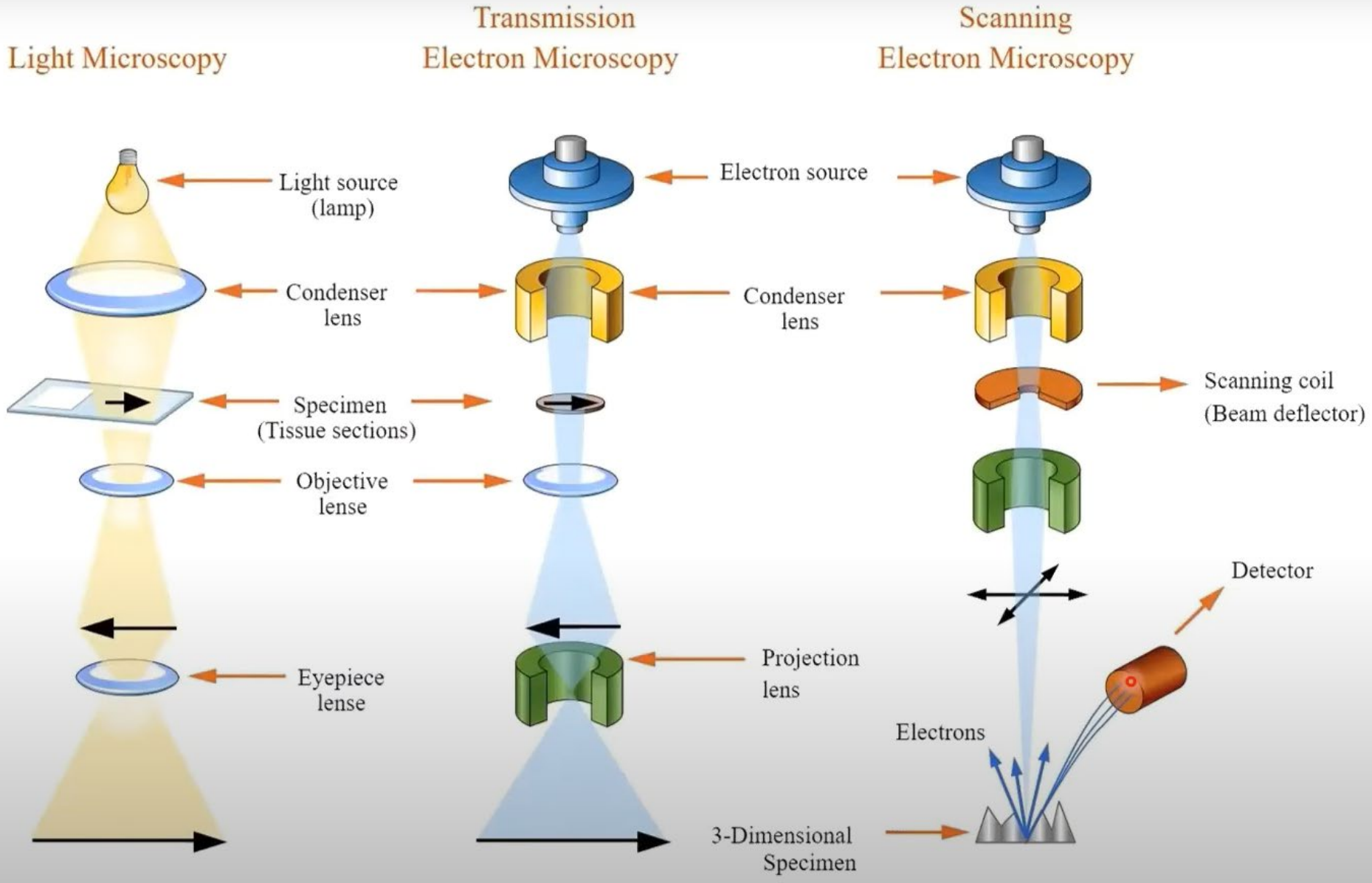
Post-processing
Mixing of images



Transmission Electron Microscopy

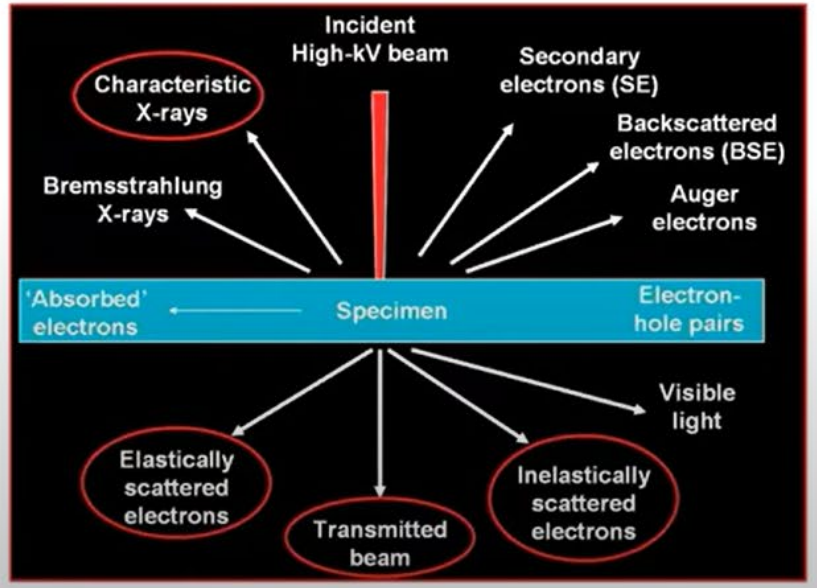


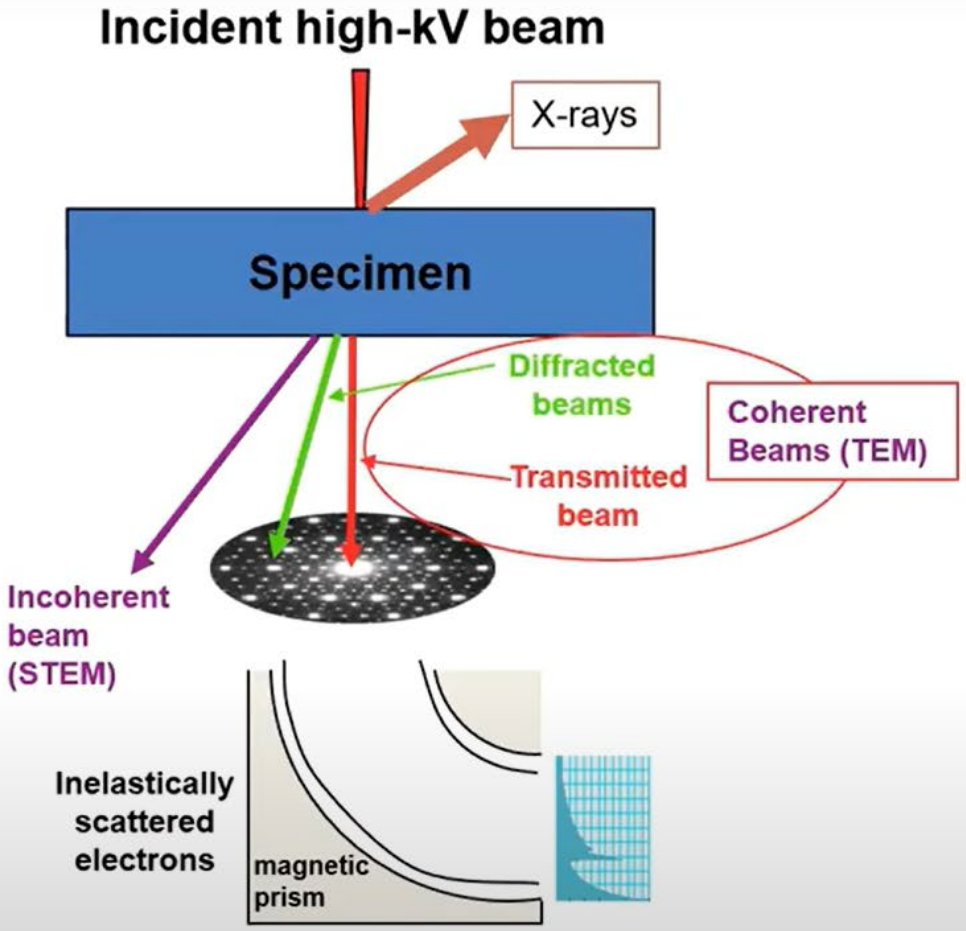
Schematics of instrumentation



Electron-Sample Interaction

1. **Transmitted electrons (beam)**
2. **Diffracted electrons (beams)**
   (Elastically scattered)
3. **Coherent beams**
4. **Incoherent beams**
5. **Inelastically scattered electrons**
6. **Characteristic X-rays**





TEM can acquire at resolutions of 50-1000 pm:

- Images
- Diffraction Patterns
- Spectroscopic and chemically resolving maps

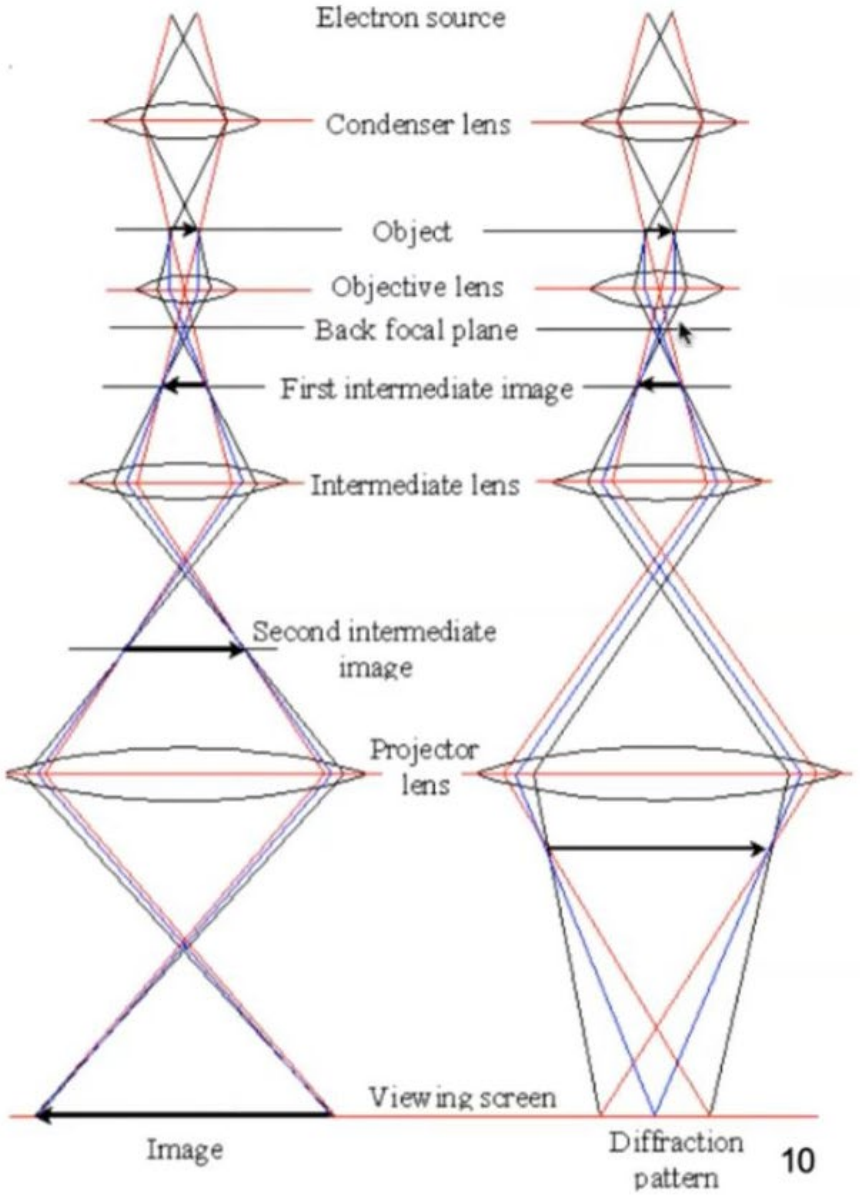
Mode of detection in TEM

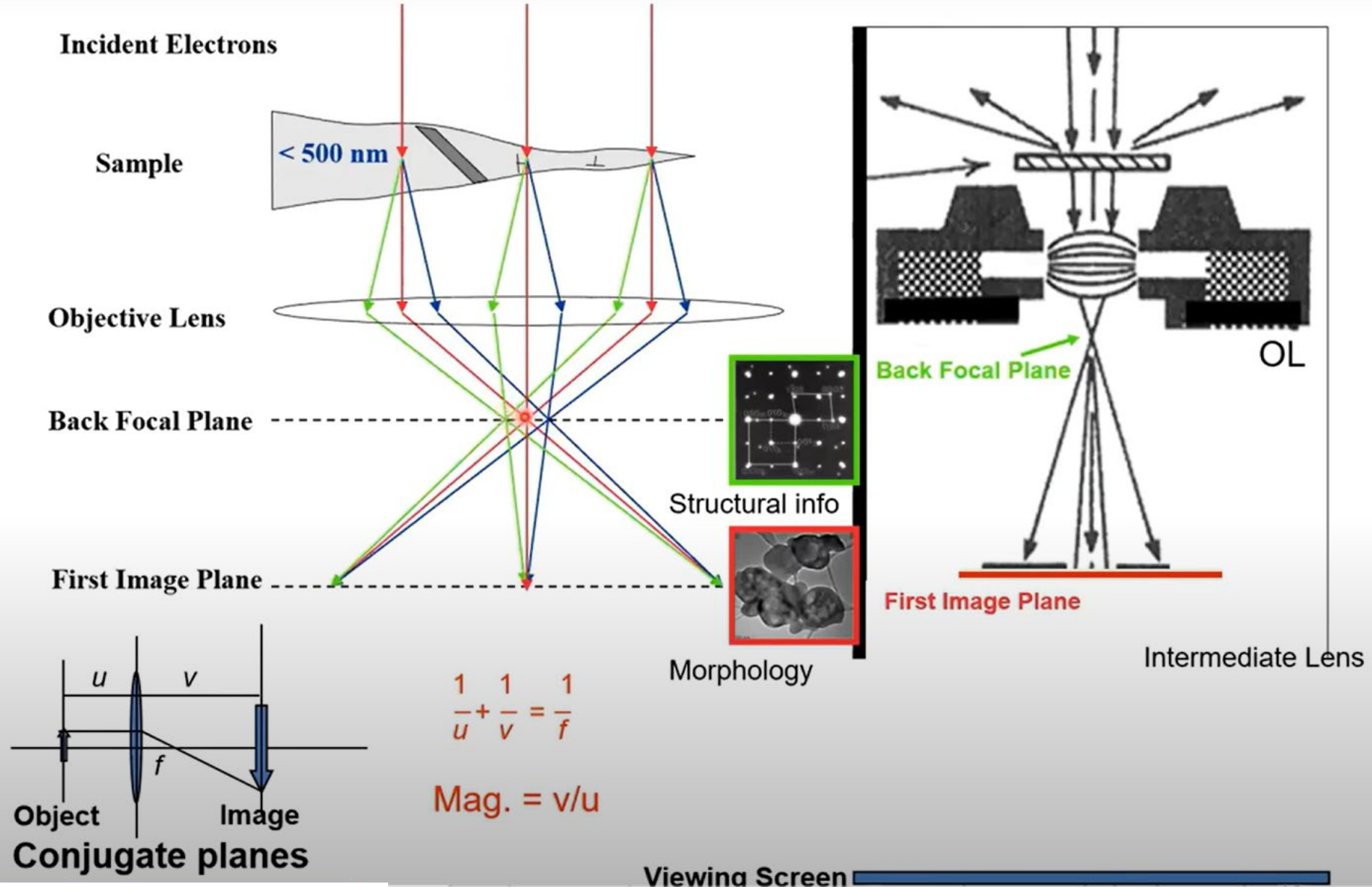
Imaging mode
Diffraction mode

Imaging mode Vs Diffraction mode

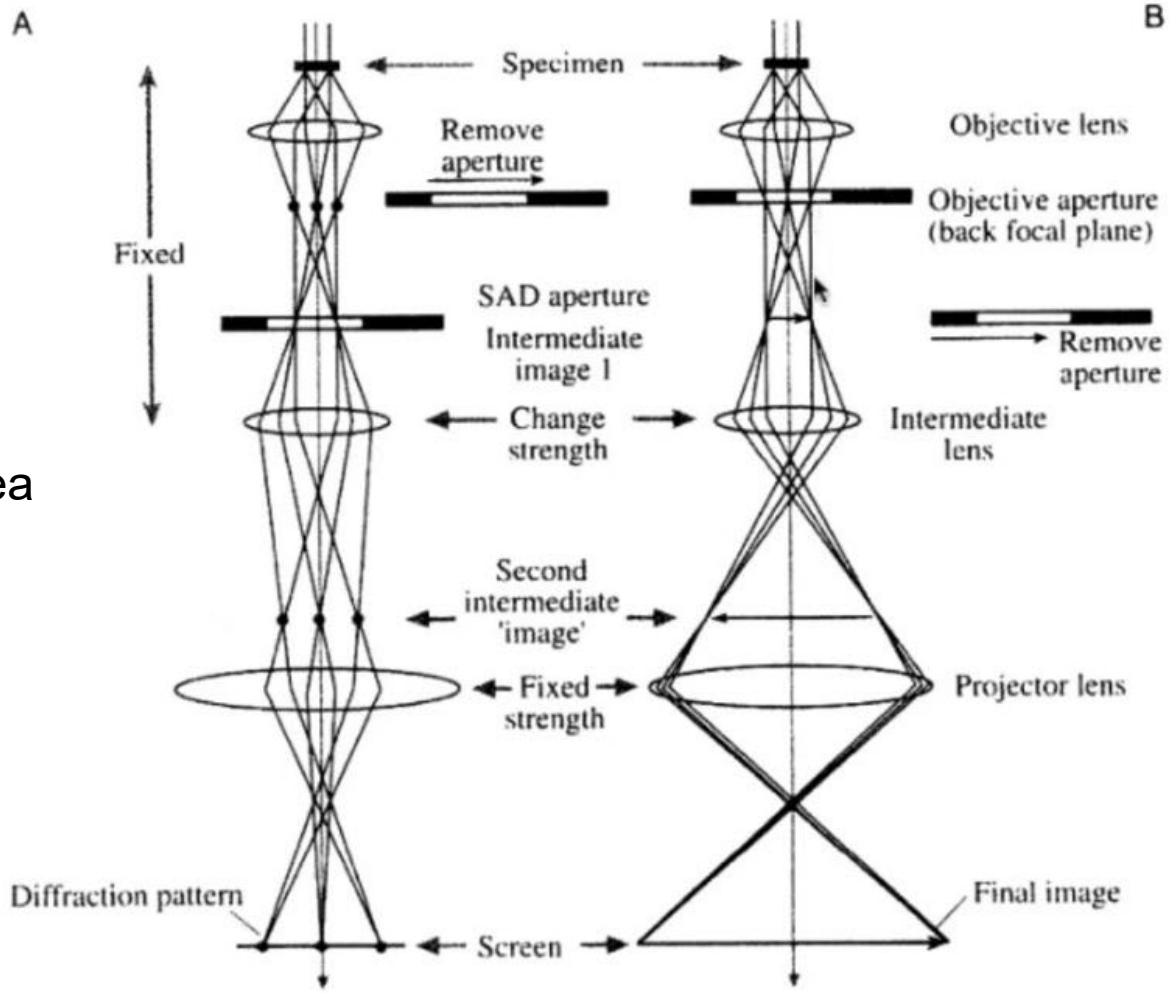
Feature	Imaging Mode	Diffraction Mode
Information	Provides a magnified image of the sample's physical microstructure (real space information).	Provides a diffraction pattern that reveals the sample's crystal structure and orientation (reciprocal space information).
Lens Settings	The intermediate lens is focused on the objective lens's image plane.	The intermediate lens is focused on the objective lens's back focal plane, where the diffraction pattern naturally forms.
Apertures	An **objective aperture** is inserted in the **back focal plane** to select specific beams (transmitted or diffracted) for image contrast (e.g., bright-field or dark-field imaging). A selected area aperture is used to define the region of interest in the sample plane from which the image is formed.	A **selected area aperture** is inserted in the image plane to define the specific area of the sample from which the diffraction pattern is collected.

Ray Paths in the TEM

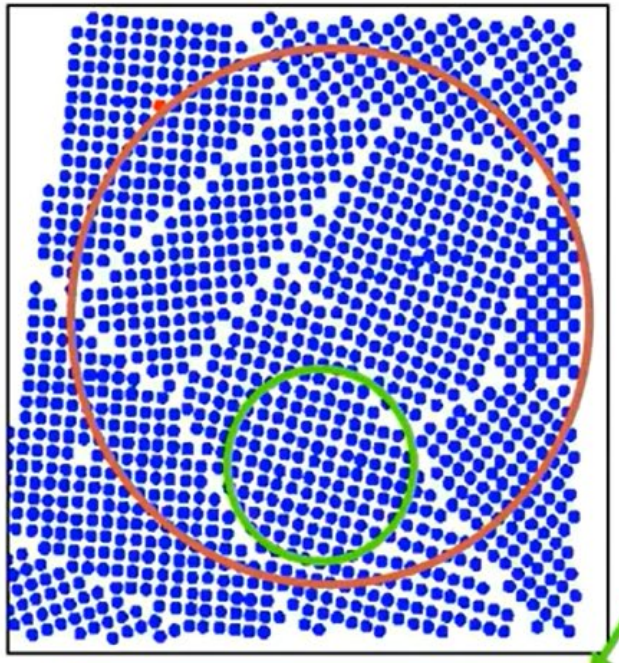


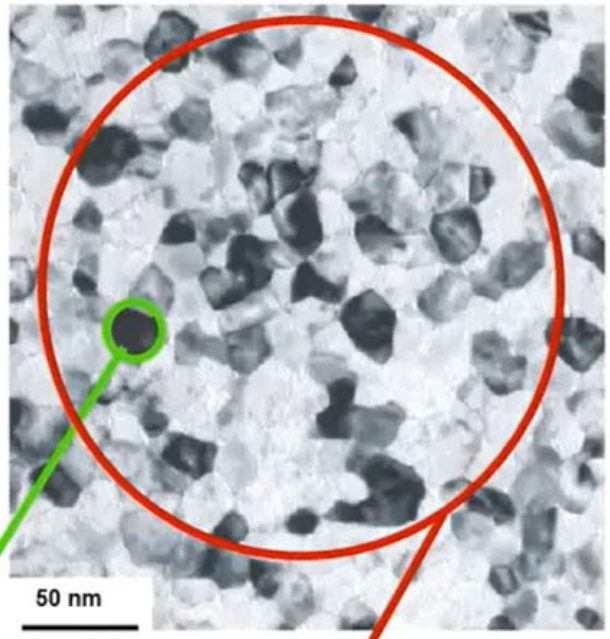

Incident Electrons
Sample
< 500 nm
Objective Lens
Back Focal Plane
First Image Plane
Structural info
Morphology
Back Focal Plane
OL
First Image Plane
Intermediate Lens
u
v
f
Object
Image
Conjugate planes
1/u + 1/v = 1/f
Mag. = v/u
Viewing Screen

TEM Imaging Modes: Diffraction vs Imaging

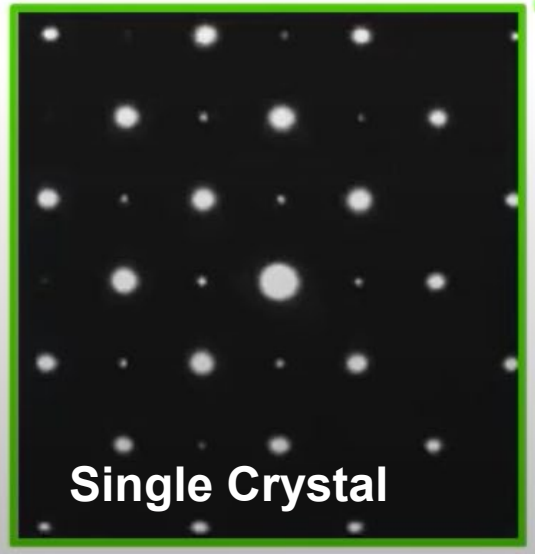


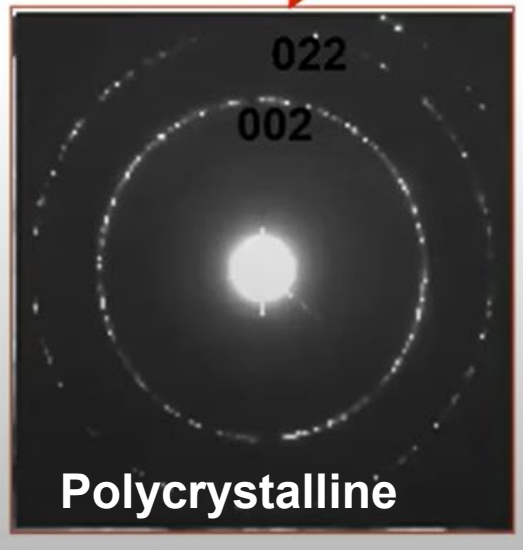
SAD= Small area diffraction



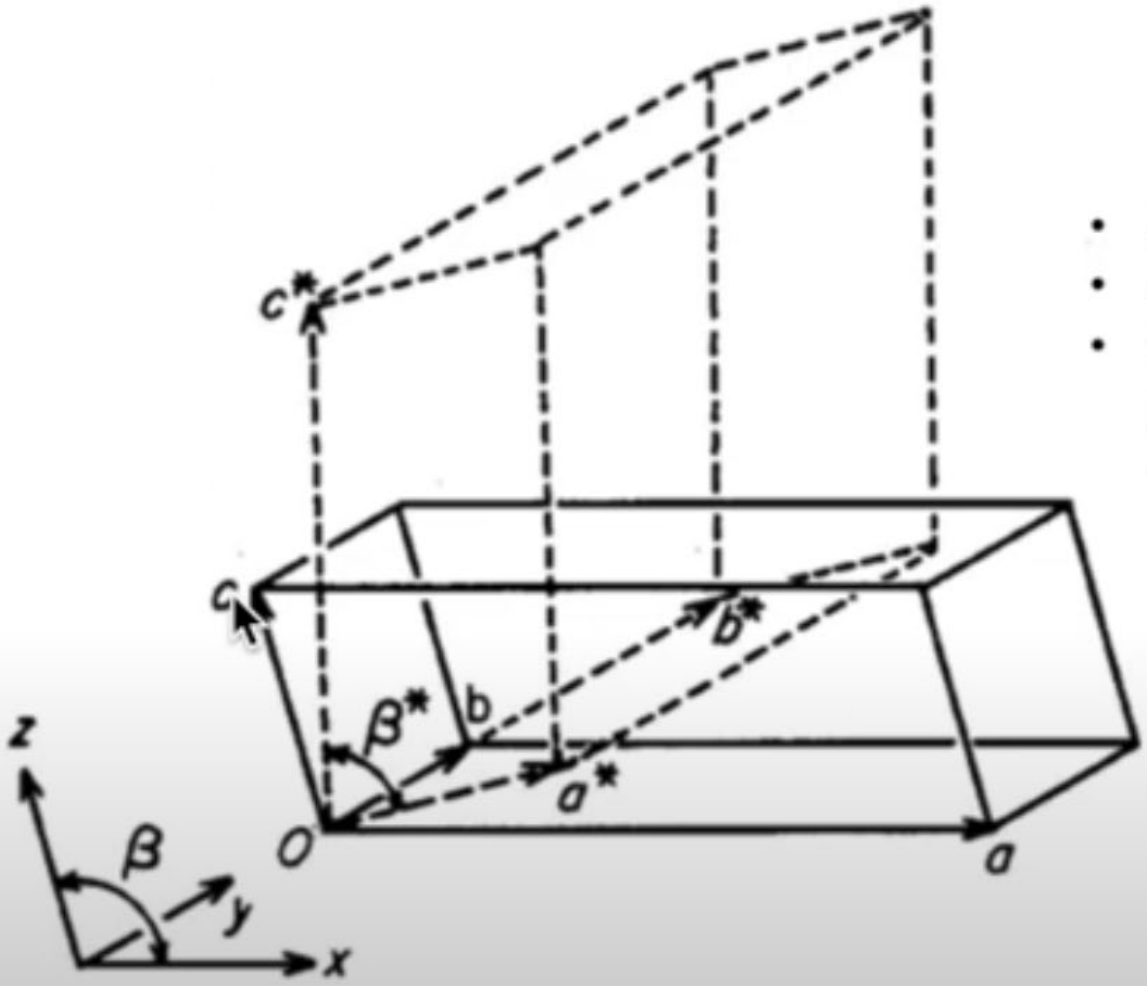


Diffraction patterns from single grain or multiple grains





Reciprocal lattice of a generic crystal



- *I Oc* I = 1/I Oc I*
- *Oc* is not parallel to Oc*
- *Oc* is perpendicular to the plane defined by Oa and Ob*

Electron scattering from a crystal

- Wave vectors:
  - k_I is the incident vector
  - k_D is the diffracted wave vector
- Elastic scattering:

$$|\mathbf{k}_I| = |\mathbf{k}_D| = \frac{1}{\lambda} = |\mathbf{k}|$$

- The difference vector K:

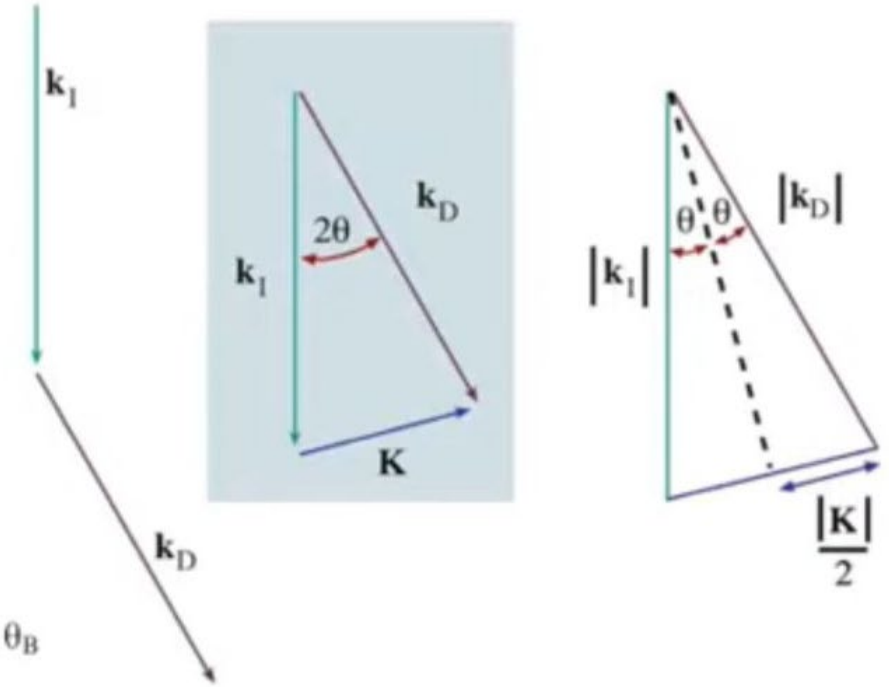
$$\mathbf{K} = \mathbf{k}_D - \mathbf{k}_I$$

- Simple trigonometry:

$$\sin\theta = \frac{|\mathbf{K}|/2}{|\mathbf{k}_I|} \Rightarrow |\mathbf{K}| = \frac{2\sin\theta}{\lambda}$$

- Bragg's condition:

$$|\mathbf{K}| = \frac{2\sin\theta_B}{\lambda}$$ And Bragg's law $n\lambda = 2d\sin\theta_B$



Williams and Carter, Transmission Electron Microscopy (2009), page 199

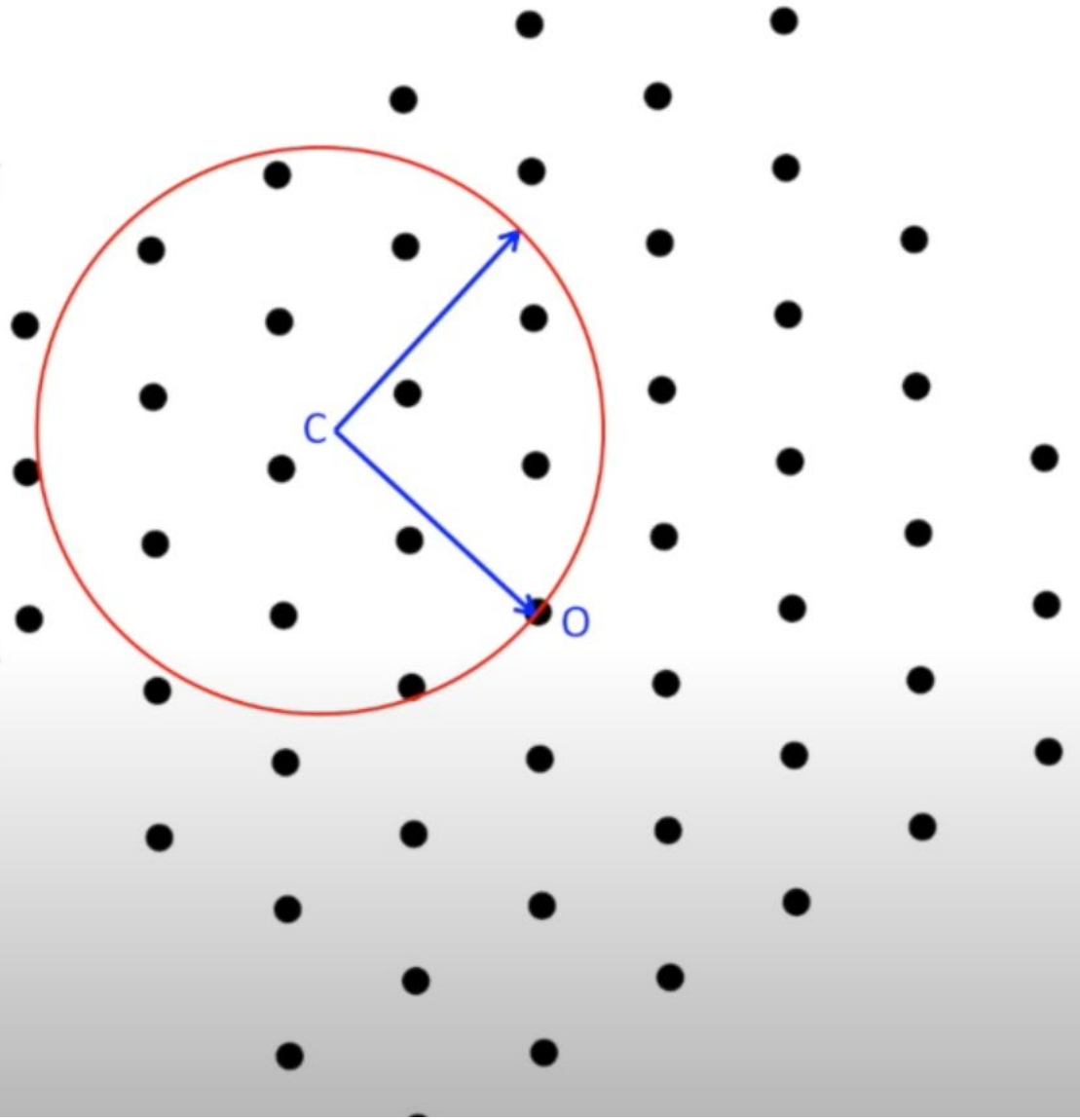
Construction of Ewald sphere

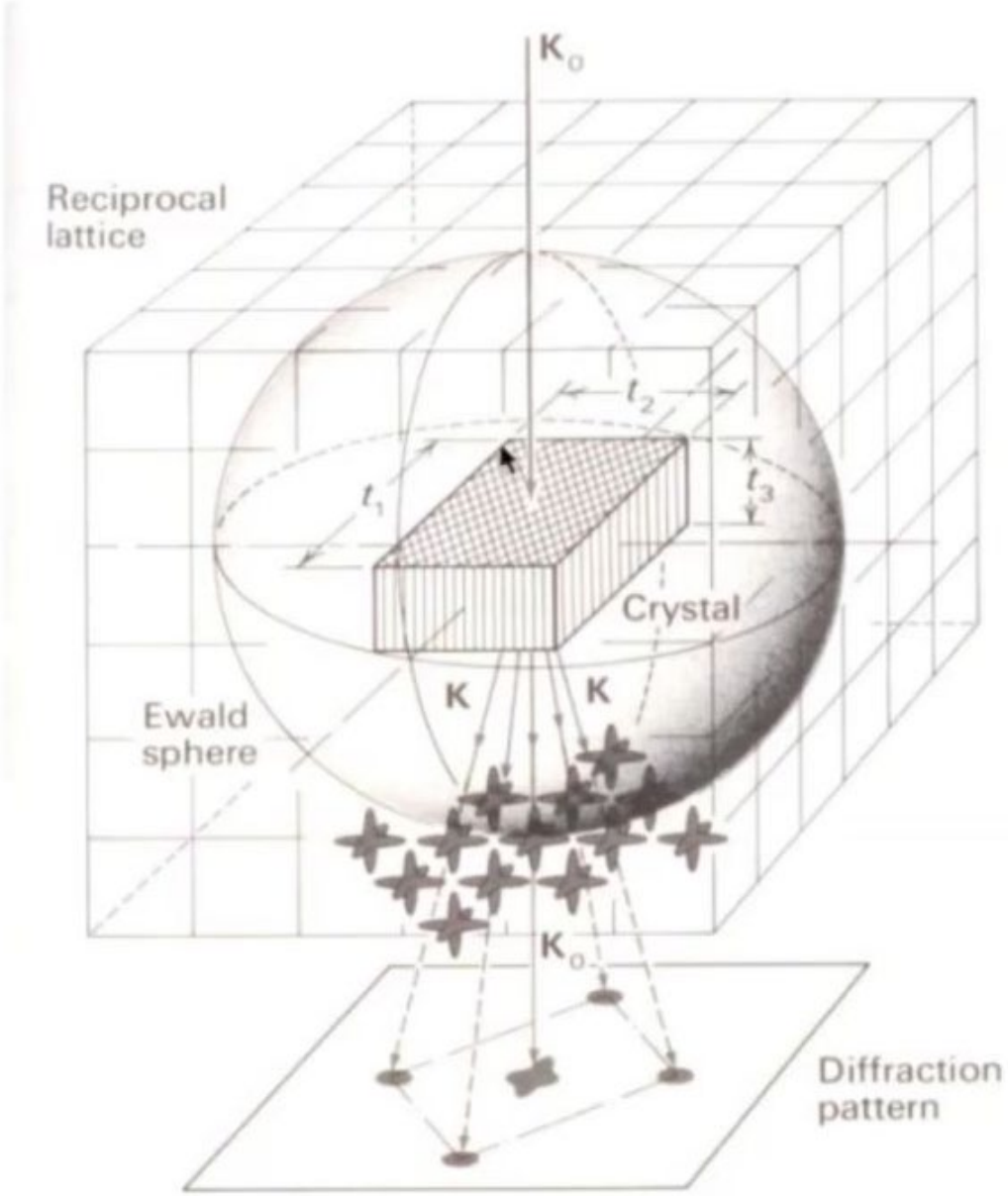
Note:

1. The points are a 2D projection of a 3D reciprocal lattice
2. The circle we draw is a a 2D projection of a 3D sphere

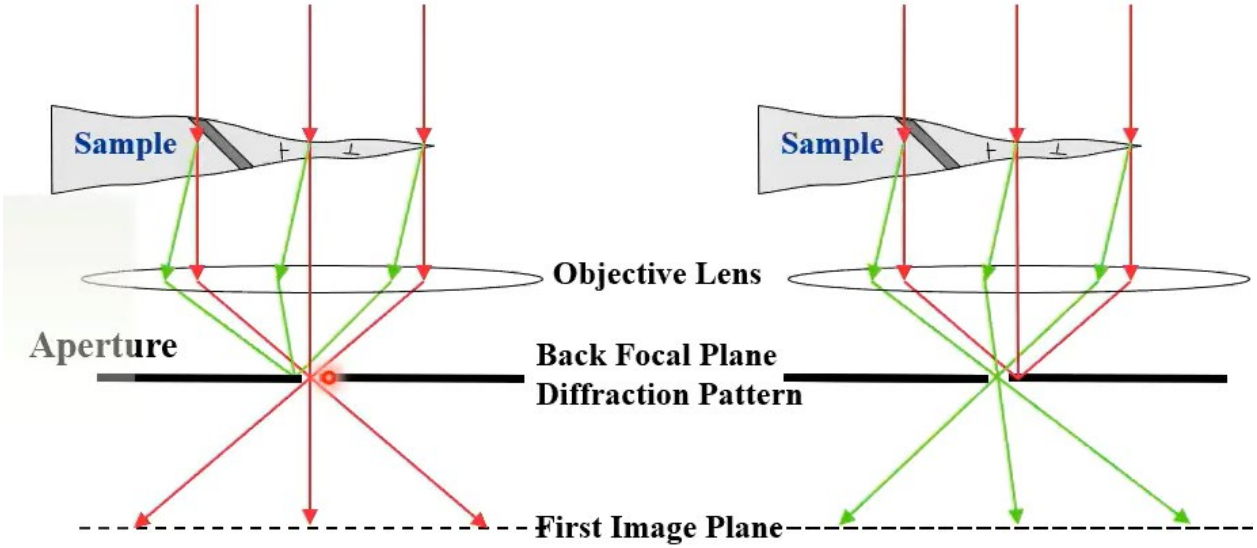
Ewald sphere construction:

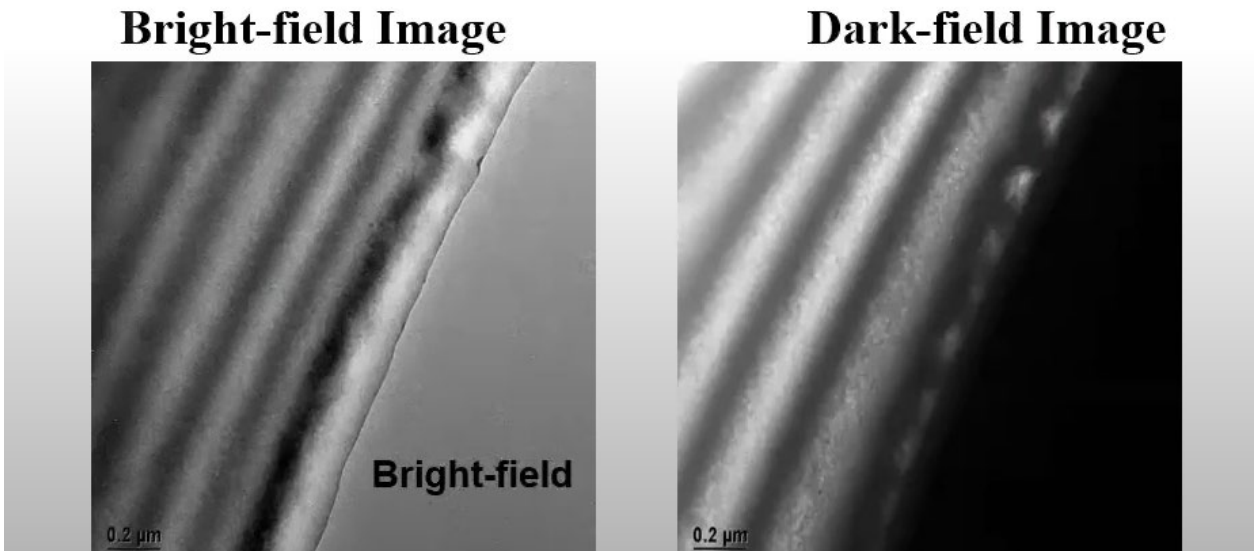
- C is the center of the circle/ sphere
- The magnitude of K_I or K_D ($1/\lambda$) is the radius
- The reciprocal lattice points that are intersected by the Ewald sphere has to satisfy Bragg's condition to strongly diffract

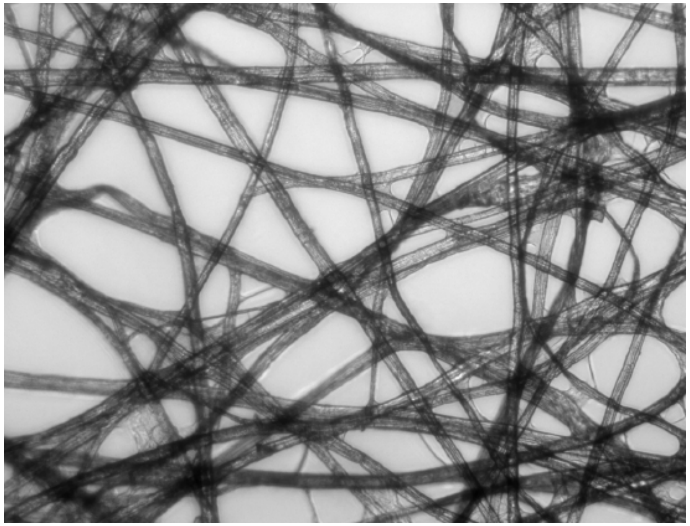


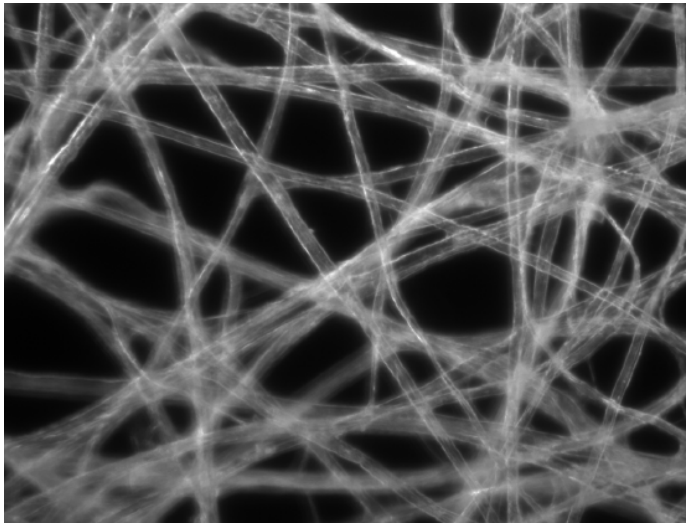

K_0
Reciprocal lattice
t_2
t_1
t_3
Crystal
K
K
Ewald sphere
K_0
Diffraction pattern

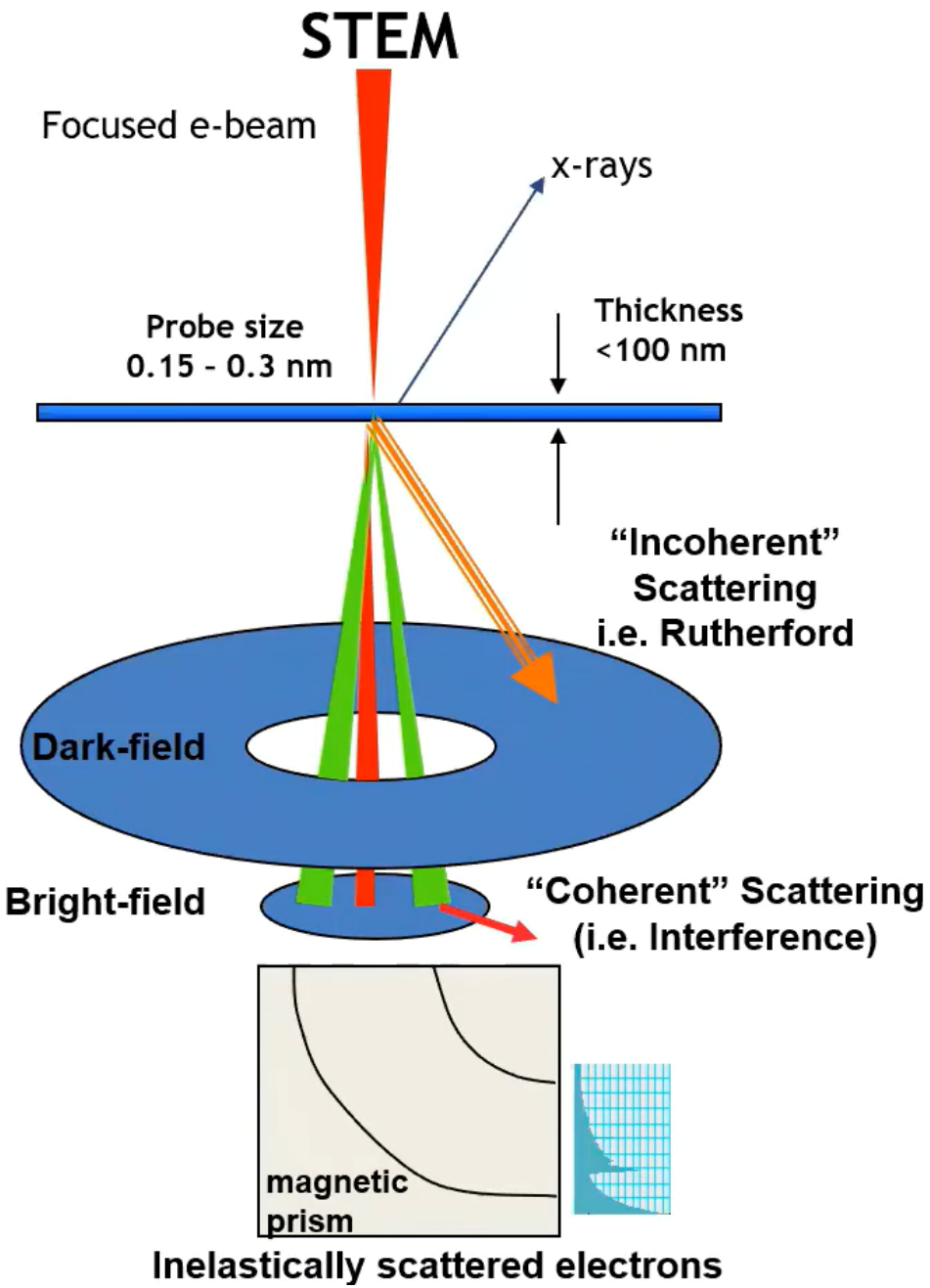
TEM Imaging Techniques











Technique

1. Raster a convergent probe across and collect the integrated signal on an **Annular** detector (**Dark Field**) or a circular detector (**Bright Field**).
2. An incoherent image is chemically sensitive (Z-contrast) under certain collection angles.
3. Annular Dark Field (ADF) STEM is directly interpretable and does not have contrast reversals or delocalization effects like HRTEM.
4. STEM resolution is determined by the probe size, which is typically 0.15 to 0.3 nm for a modern (uncorrected) S-FEG STEM.
5. Since STEM images are collected serially, the resolution is typically limited by vibrations and stray fields.