# Webinar Series in TEM: Introduction to Transmission Electron Microscopy Part 1


Riza Iskandar

Application Scientist

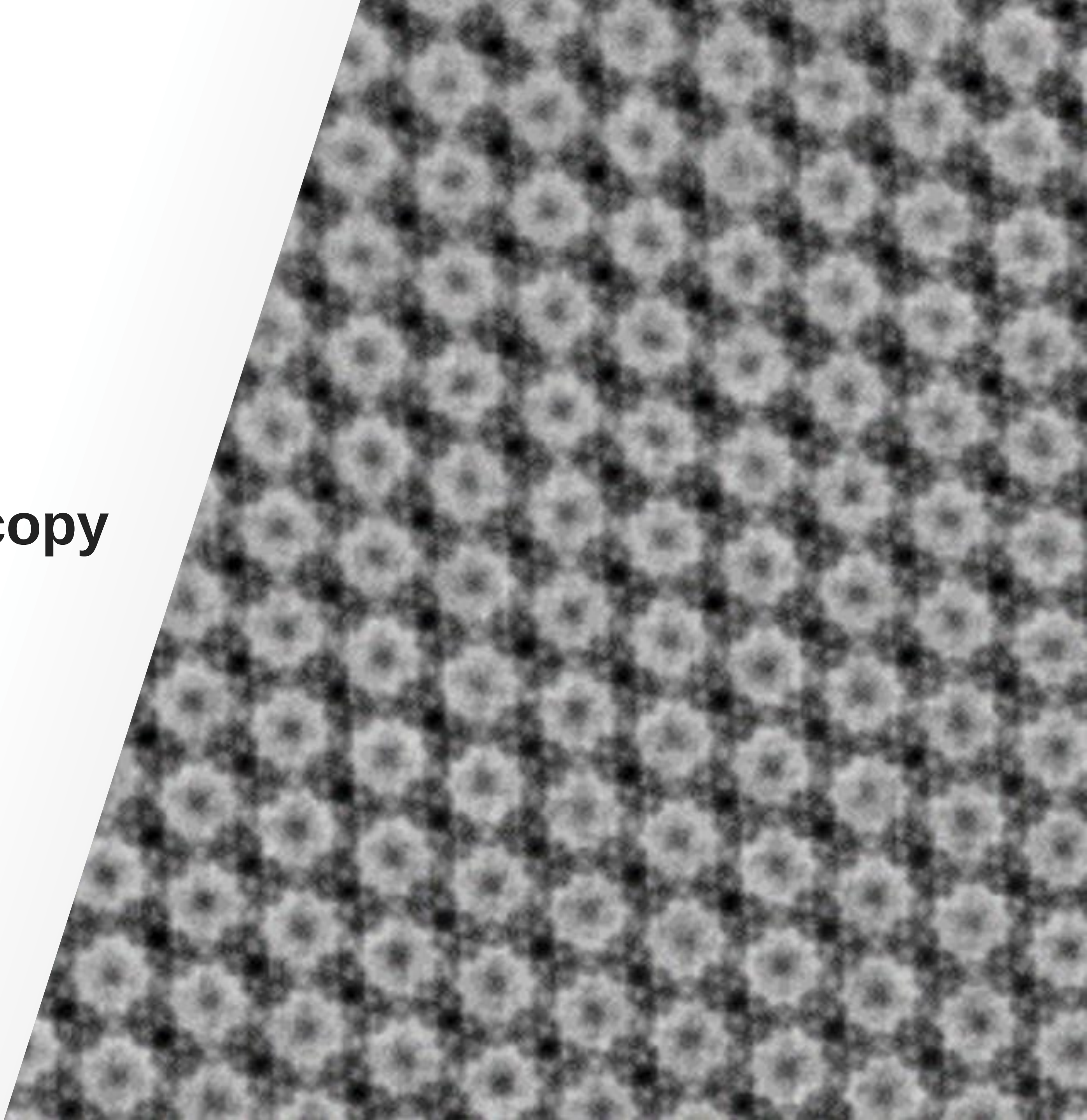
Material Science

Material and Structural Analysis - APAC


Universitas Indonesia | Direktorat Riset & Pengembangan

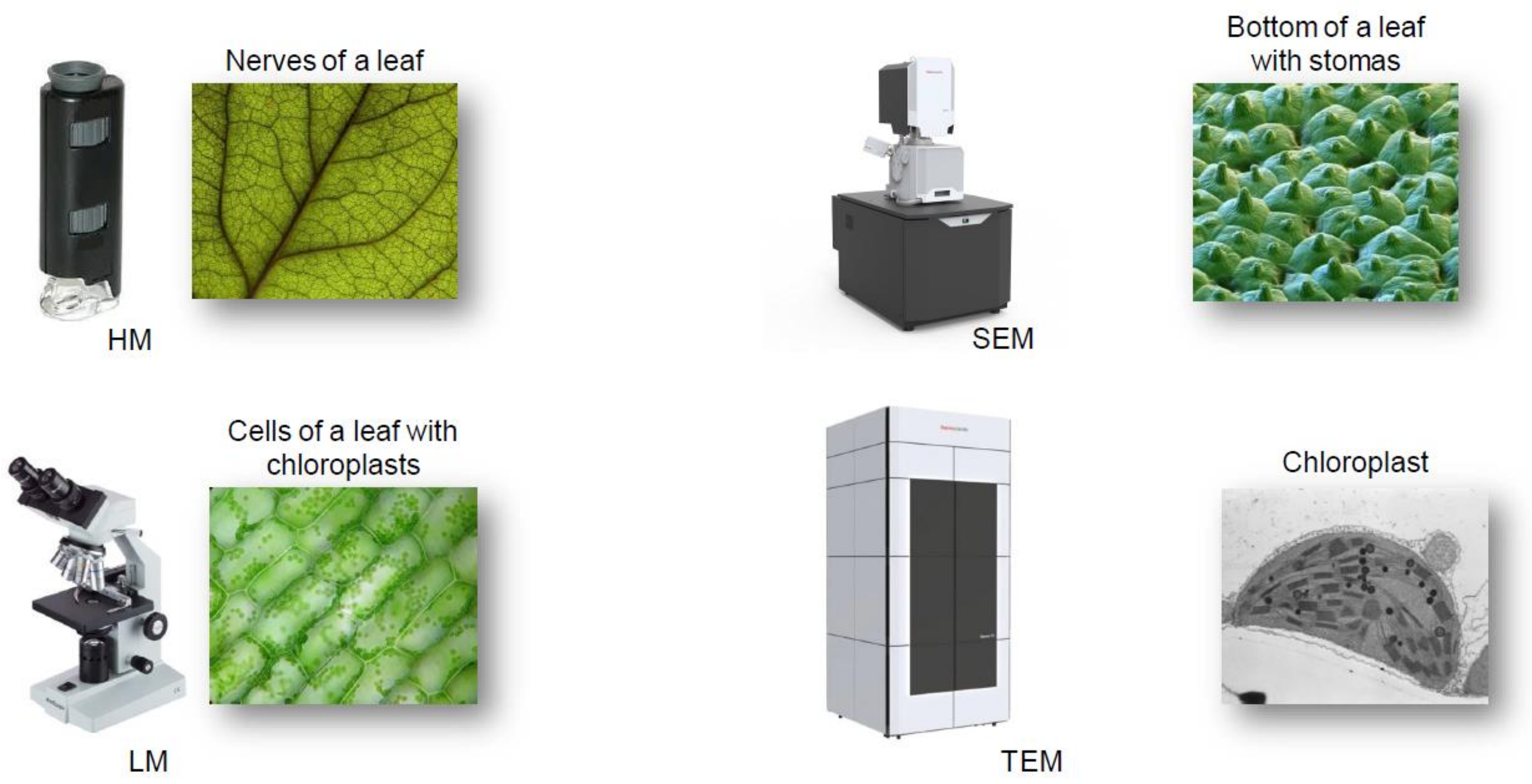
LabTEM High Resolution

The world leader in serving science



# Various Microscopes

Microscopy is one of the few methodologies applied to nearly every field of science and technology in use today



# Introduction to Transmission Electron Microscopy

Outline: Part 01

- TEM Basics
- SEM vs TEM: What are the differences?
- Conventional TEM Imaging
  - Bright-Field Imaging
  - Dark-Field Imaging
- Electron Diffraction
  - Selective Area Electron Diffraction
  - Convergence Beam Electron Diffraction
  - Nano Beam Electron Diffraction
  - High-resolution TEM

# Introduction to Transmission Electron Microscopy

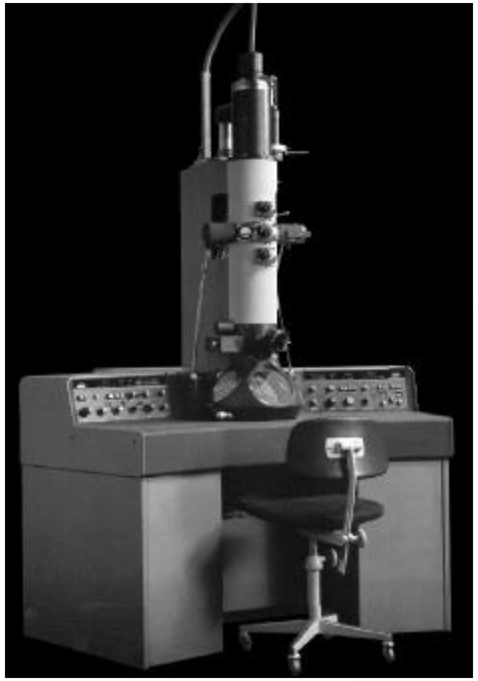
## Outline: Part 02

- Scanning Transmission Electron Microscopy (STEM)
- Spectroscopy in TEM
- Tomography in TEM: For 2D to 3D Imaging
- Sample Image Analysis
  - Good and Reliable TEM Data: How to produce and Analyze
  - Example of TEM Data
- TEM Facility in Universitas Indonesia

# TEST QUIZ

Which of the following is a figure of TEM?


A



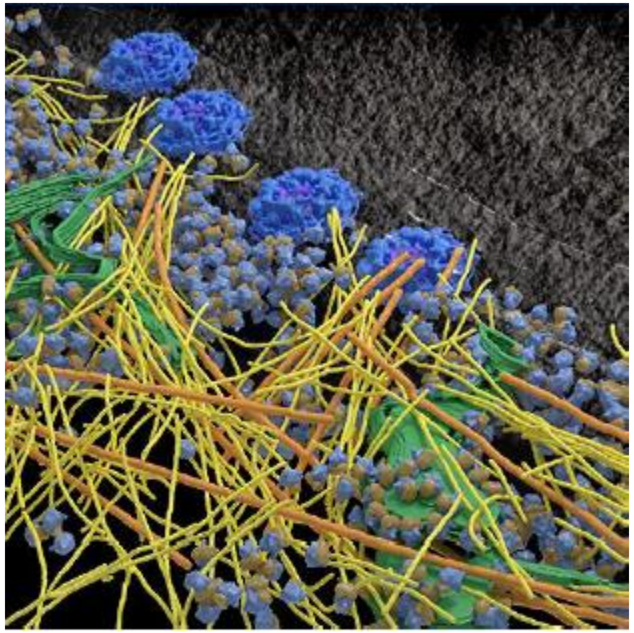
B


C


D

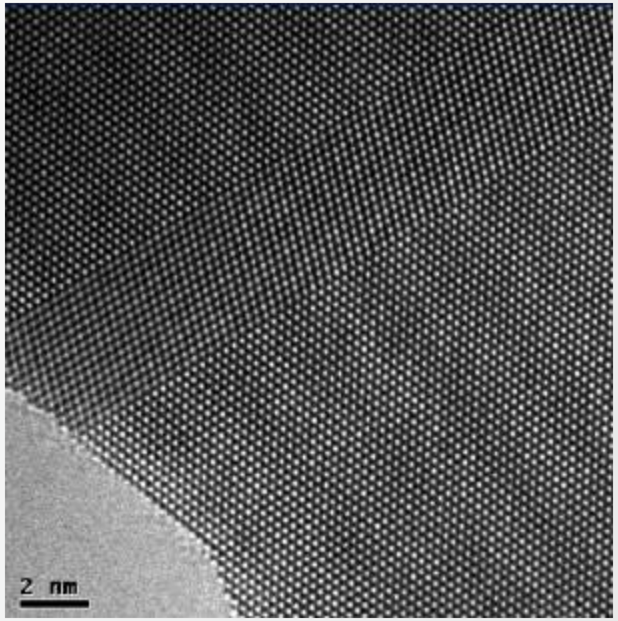
# TEST QUIZ

Which one is the image taken from TEM?



A

B




C

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- High-resolution TEM

# TEM Basics

Electron Microscope History

- 1897: Thompson describes the existence of negatively charged particles (electrons)
- 1925: De Broglie theorized that electrons have wave-like characteristics, addressing the wave/particle duality
- 1927: Thompson and Reid demonstrated the wave nature of electrons by diffraction experiments
- 1931: Ruska et al. build the first electron microscope (Nobel Prize in 1986)

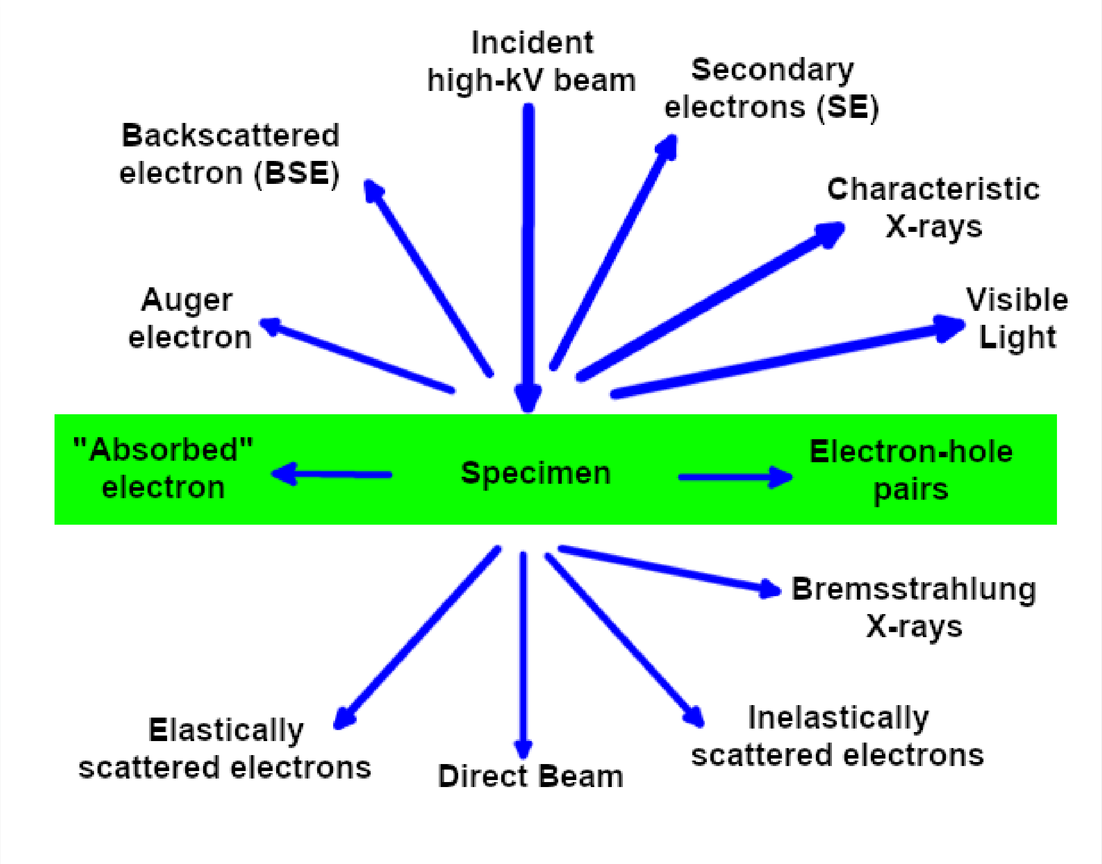
Knoll and Ruska (in the lab coat) with the first Transmission Electron Microscope in Berlin in the early 1930s

# TEM Basics

Thermo Fisher SCIENTIFIC

## What is TEM?

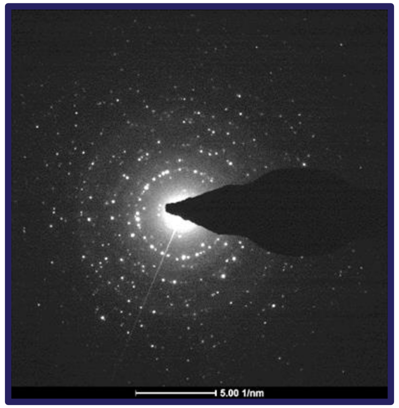

- To see **small objects** which cannot be seen with naked eyes, light microscope or even a SEM.
- To obtain structural information of **small objects**
- To analyze the chemical compositions of **small objects**

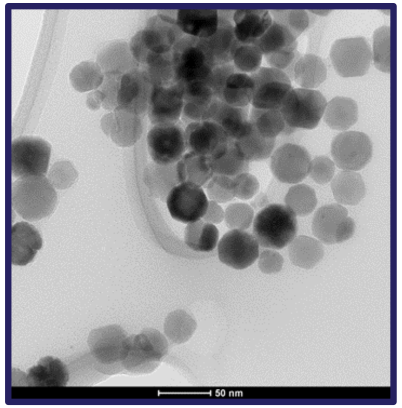



Electron – Sample Interactions

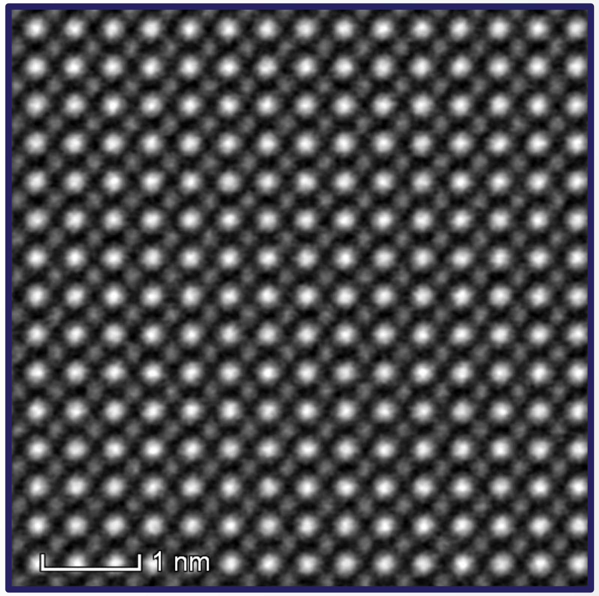
# TEM Basics

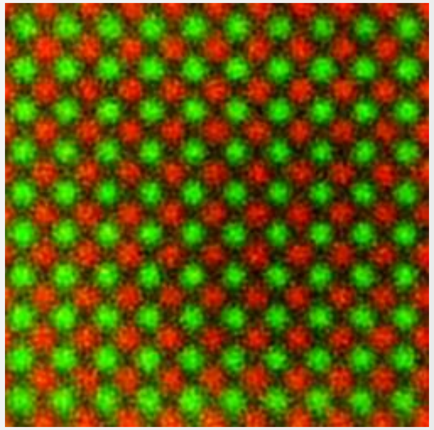
## What can TEM Do?



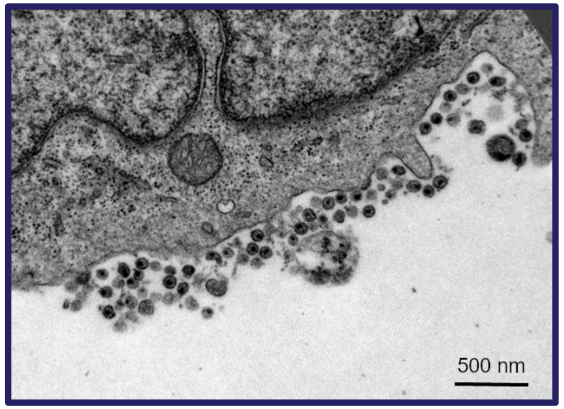




Electron Diffraction pattern (SAED) taken from nano particles (left) as shown in TEM-BF (right)

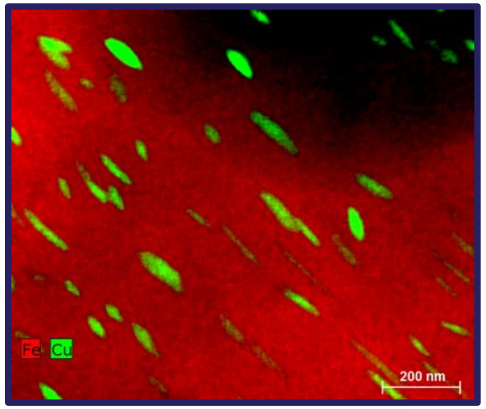


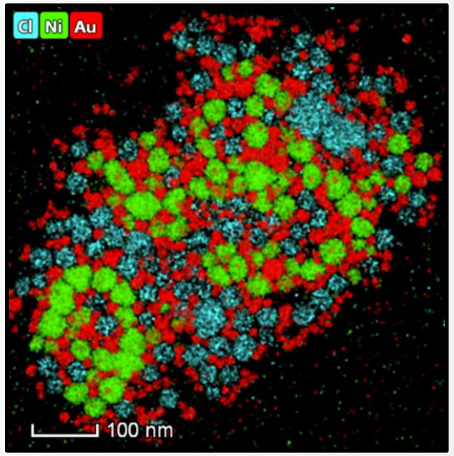
HRSTEM and HREDS $SrTiO_3$



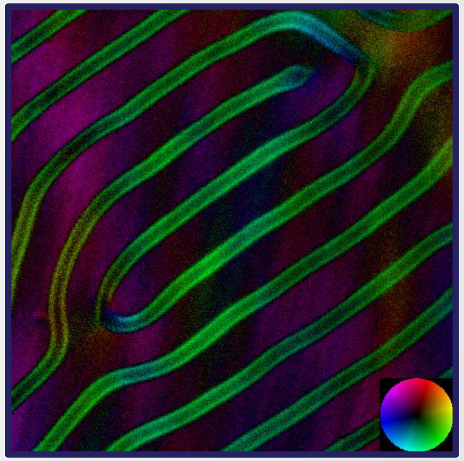

Cat bone marrow with
Feline Immunodeficiency Virus




EDX mapping of Cu and Fe elements distribution in steel




Au-Ni-Cl nanoparticles



Magnetic Hexa-ferrite

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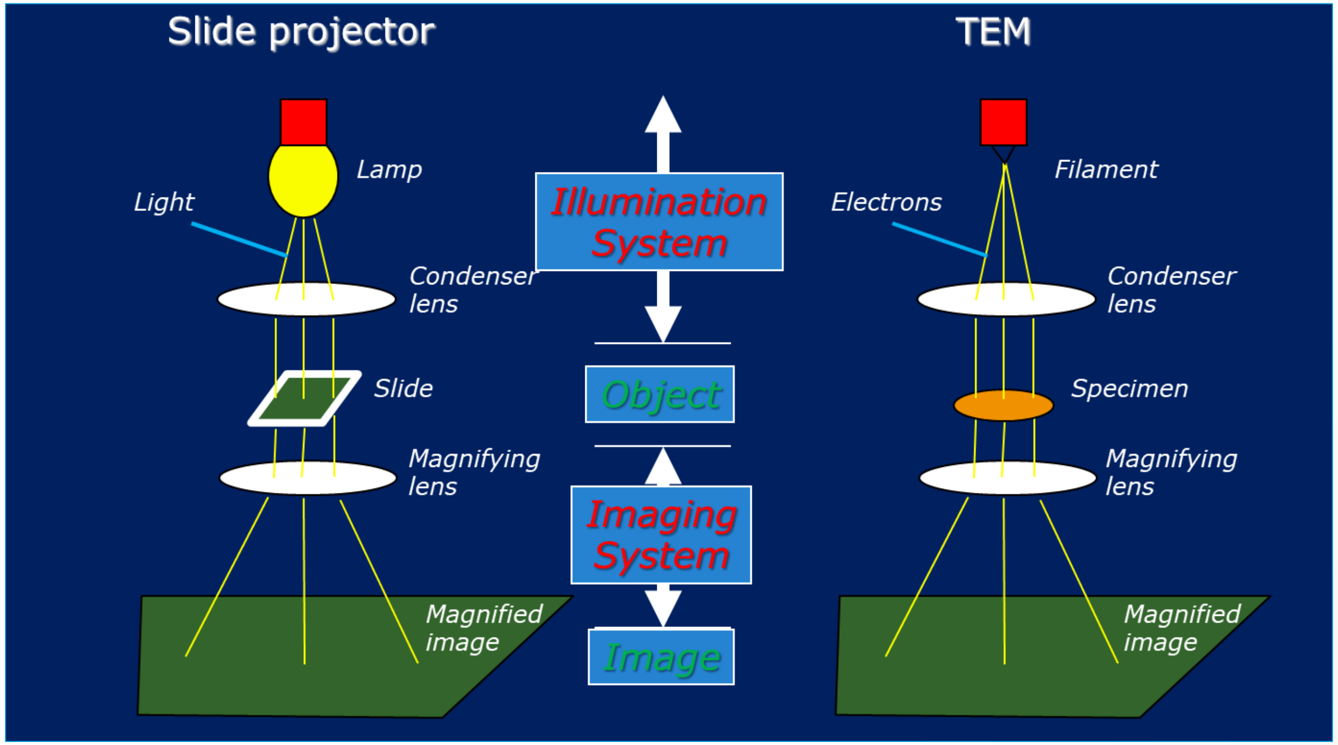
# TEM Basics

ThermoFisher SCIENTIFIC

## How does TEM Work?

### Slide Projector vs TEM




| Slide Projector | Transmission Electron Microscope |
|---|---|
| Visible light | electron |
| Glass lenses | Electron-magnetic lenses |
| $\lambda$ = 450-650 nm | $\lambda$ = 0.0025 nm (200 kV) |
| spatial resolution = 100 nm ($d > \lambda$) | spatial resolution = 0.24 nm ($d \cong \lambda$) |

# TEM Basics

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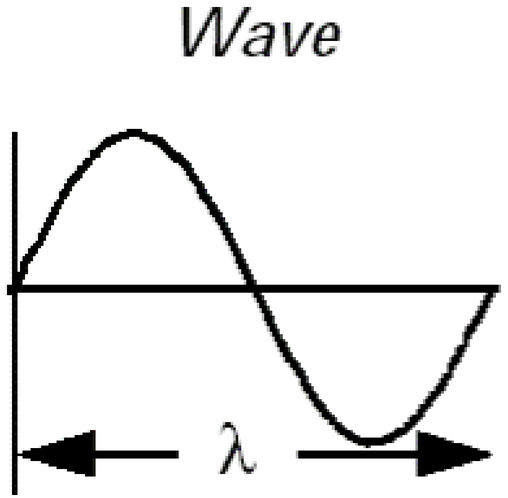
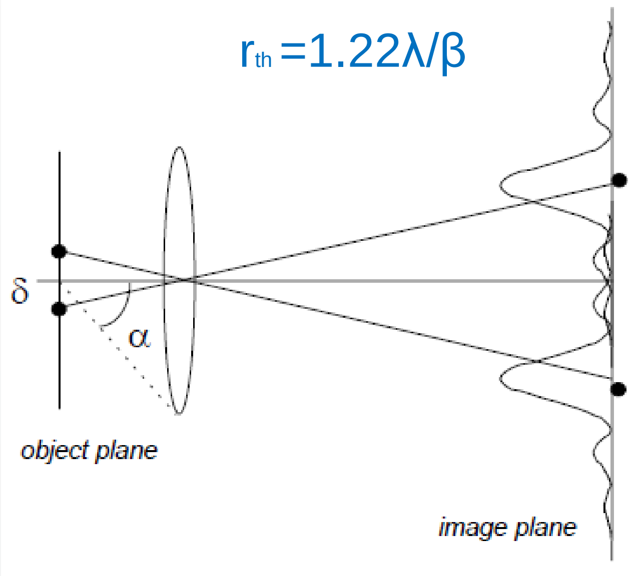
## Why Use Electrons?

- The resolution of light microscopy is limited by the wavelength of visible light (400 – 700nm)*
- Electrons, that are particles as well as a wave, have much shorter wavelength, which gives much better resolution
- De Broglie equation: $\lambda = h/mv$




| U (kV) | Relativistic (λ=nm) |
|---|---|
| 100 | 0.0037 |
| 120 | 0.0034 |
| 200 | 0.0025 |
| 300 | 0.0020 |

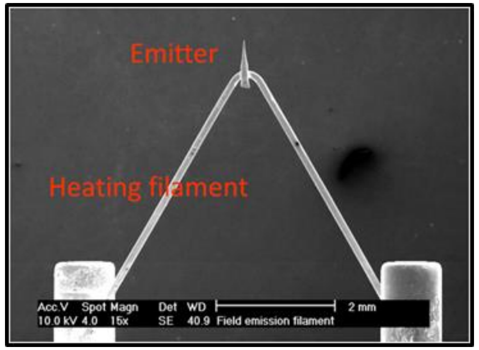



*X-ray wavelength is about 0.05-0.25 nm

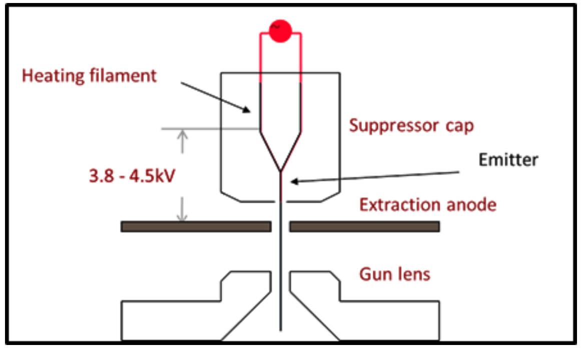
# TEM Basics

## Electron Sources

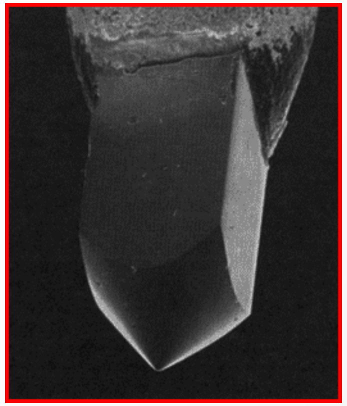
### Thermionic

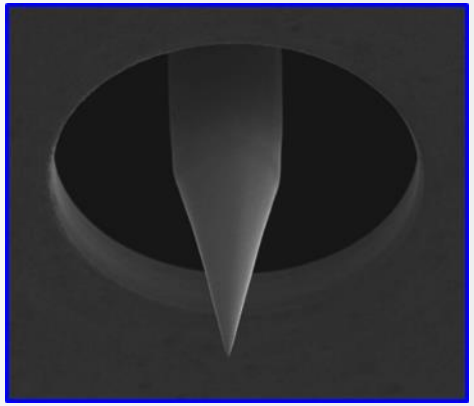


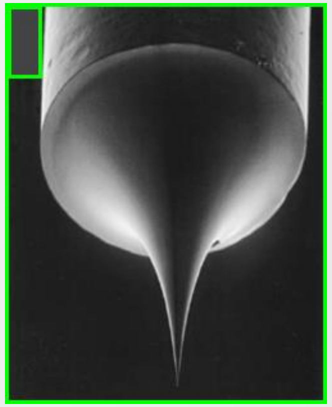

### Field Emission Gun (FEG)




| | $LaB_6$ | SFEG | XFEG | X-CFEG |
|---|---|---|---|---|
| Normalized brightness | 1-3 | 250 | 1250 | 1625 |
| Energy spread (eV) | 1.1-1.5 | ≤0.8 | ≤ 0.8 | ≤ 0.3 |







Tungsten(W)/$LaB_6$ → SFEG → XFEG → X-CFEG

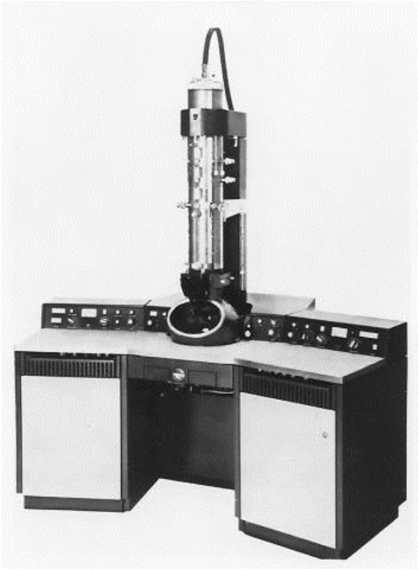
Lifetime SFEG is 5 times longer than $LaB_6$
$LaB_6$ = 1000 hours vs SFEG = 5000 hours

Brighter sources
Narrower energy spread
Smaller emitter tip

**Better Performance**

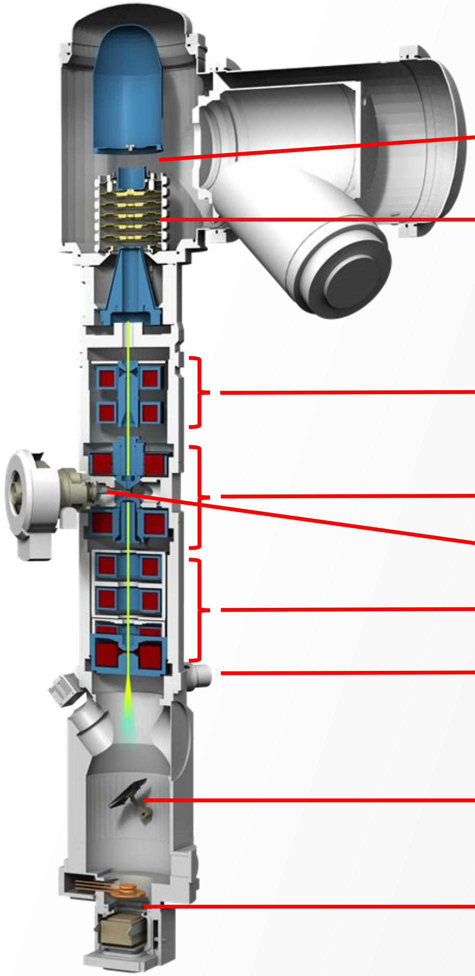
# TEM Basics

## Parts of the TEM



**Philips Eindhoven**
**1966**

**Talos F200X**

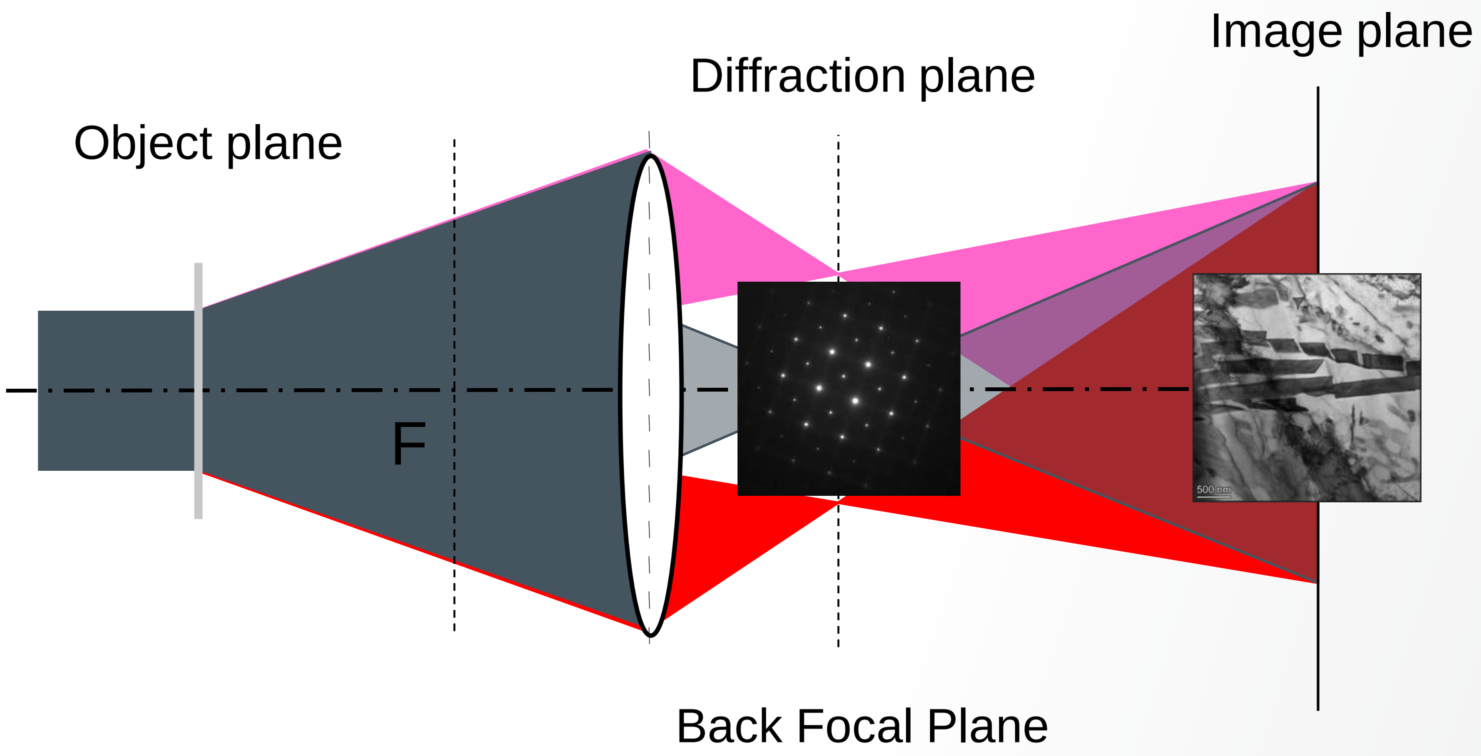


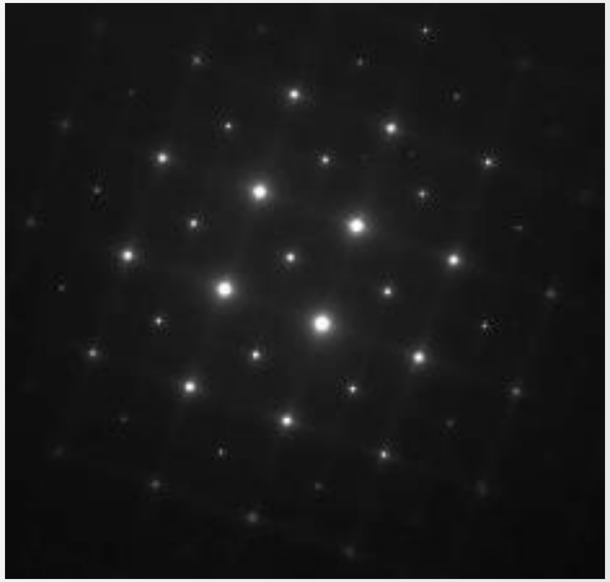
EELS

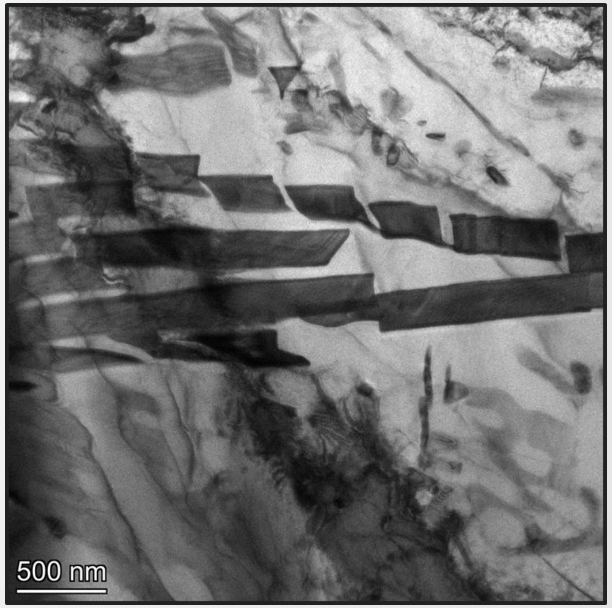
| | |
|---|---|
| Electron Gun – LaB6, FEG, XFEG (CFEG, X-CFEG, monochromator) | Produces electrons |
| Accelerator (200 kV)) | Accelerates the electrons to high energy |
| Condenser lens<br>Condenser aperture | Control illumination on the sample |
| Objective lens<br>Sample / Holder / EDS detectors | Sample and main imaging lens + EDS (objective lens) |
| Intermediate and Projection lens (magnification) | Controls magnification and image/diffraction mode |
| STEM detectors: HAADF, 4DSTEM<br>Detectors for imaging formation | |
| Flu-Screen | Flu-screen image viewed using a camera + monitor |
| TEM camera<br>STEM detectors (BF/DF) | TEM and STEM detectors |
| All in high vacuum | Whole column under high vacuum |

# Conventional TEM Imaging

Optics: Image and Diffraction Formation






- Object plane: a plane contains object points
- Back focal plane (BFP): the focal plane lies behind the lens
- Image plane: a plane contains image points

# TEM Basics

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QUIZ

1. A TEM is used to:
   A. See small objects that cannot be seen with other imaging tools
   B. Obtain structural information of small objects
   C. Analyze the chemical compositions of small objects
   **D. A, B and C**
2. A good TEM performance will depend on:
   A. A higher acceleration voltage
   B. Better electron sources
   C. An expert operator
   **D. A, B, C**
   E. A and B

# TEM Basics

QUIZ

3. What from these statements are NOT true for FEG electron sources:
   A. It has a smaller size of emitter tip than Thermionic electron sources
   B. It has a longer operating time than Thermionic electron sources
   C. It has a higher brightness level than Thermionic electron sources
   **D. It has a broader energy spread than Thermionic electron sources**
4. The most important lens in the TEM is:
   A. Condenser lens
   **B. Objective lens**
   C. Intermediate lens
   D. Projective lens

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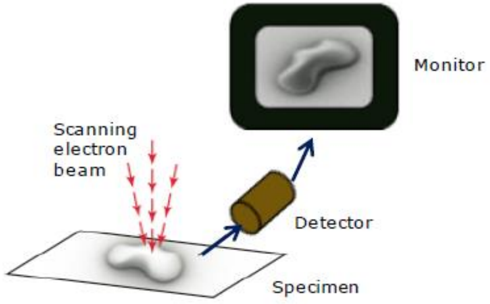
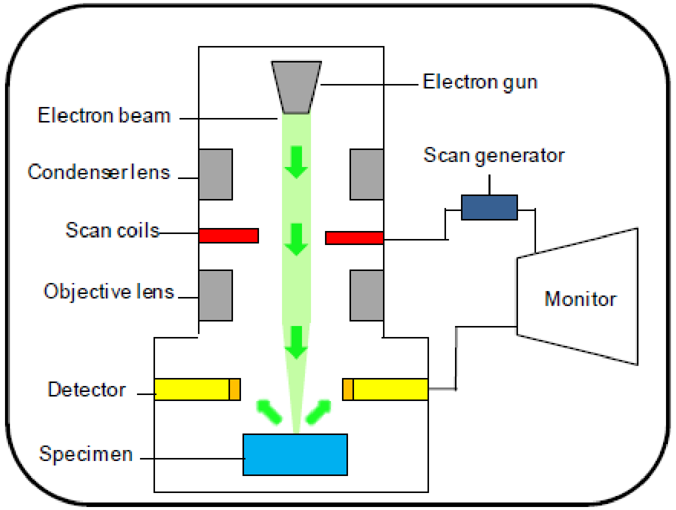
  - Nano Beam Electron Diffraction
- High-resolution TEM

# SEM vs TEM

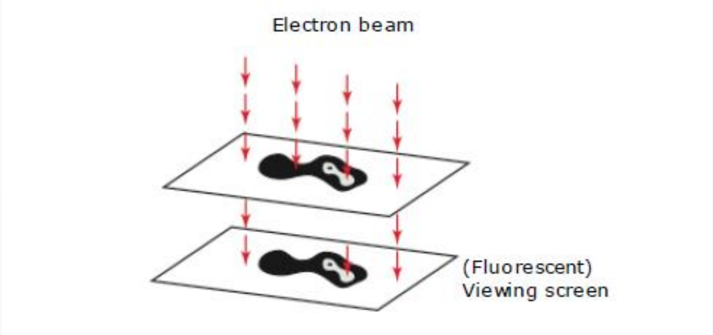
What are the differences?

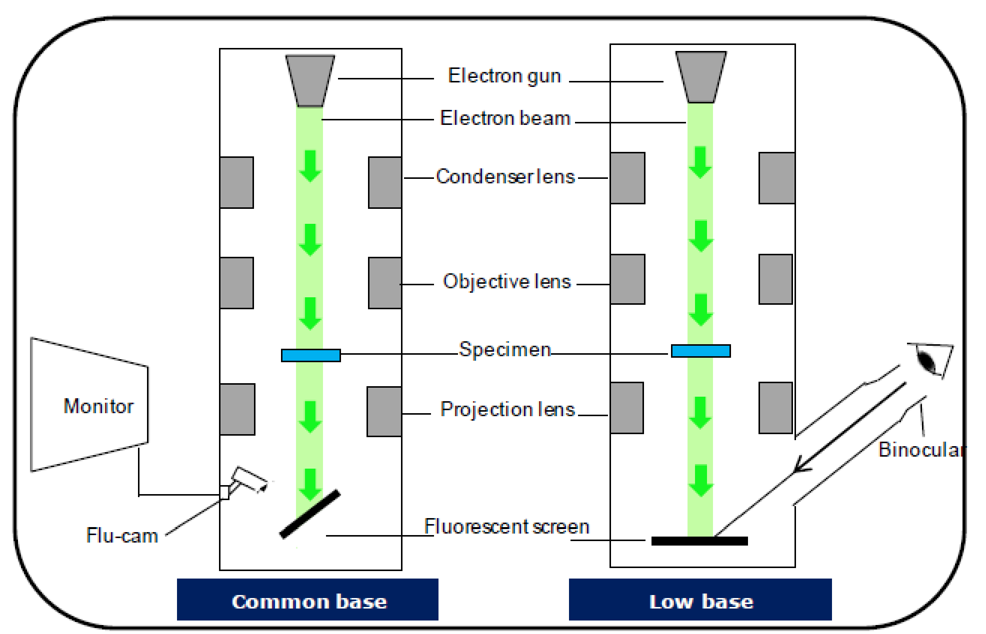
## SEM: Scanning Electron Microscope




## TEM: Transmission Electron Microscope



# SEM vs TEM

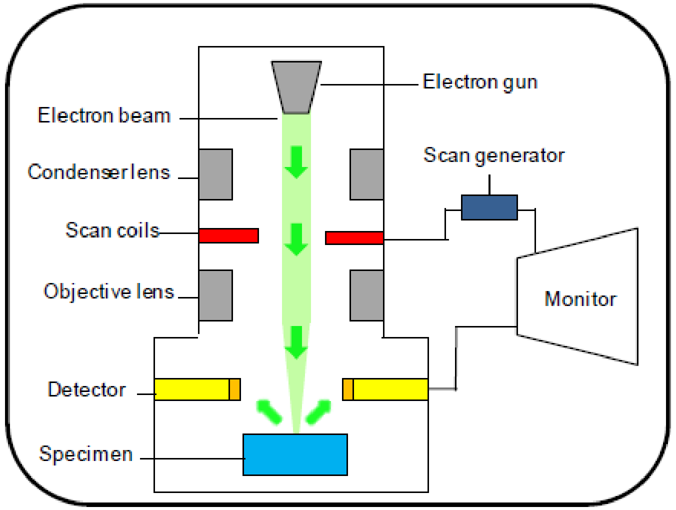
What are the differences?

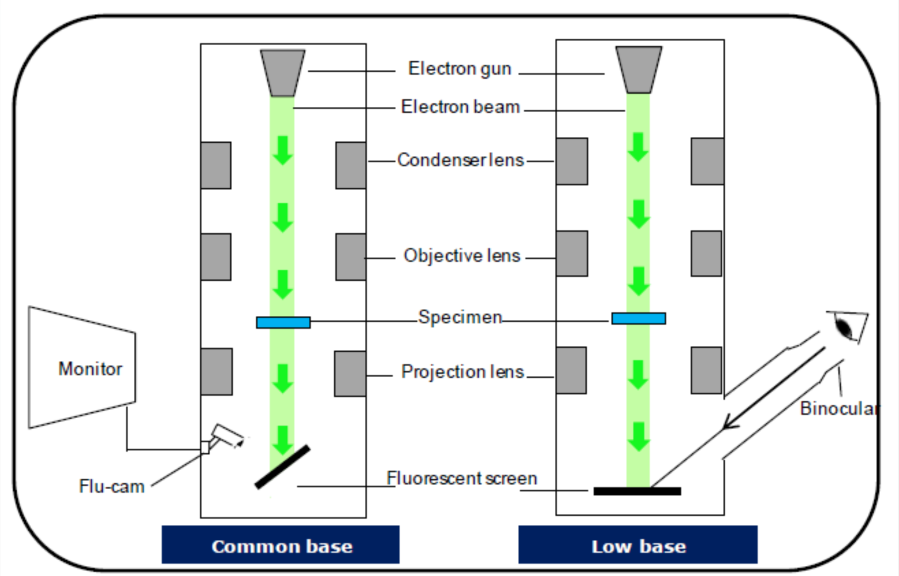
## SEM: Scanning Electron Microscope

- Smaller/shorter
- Acceleration voltage: 5kV – 30 kV
- Resolution ≥ 0.7 nm
- Focused scanning beam
- Larger specimen chamber
- Larger samples

## Transmission Electron Microscope

- Larger/taller
- Acceleration voltage: 60-300kV or *30kV – 1MV**
- Resolution ≤ 0.1 nm
- Broad static beam and *focused scanning beam (STEM)**
- Smaller specimen chamber
- Thin samples of ≈ 100 nm

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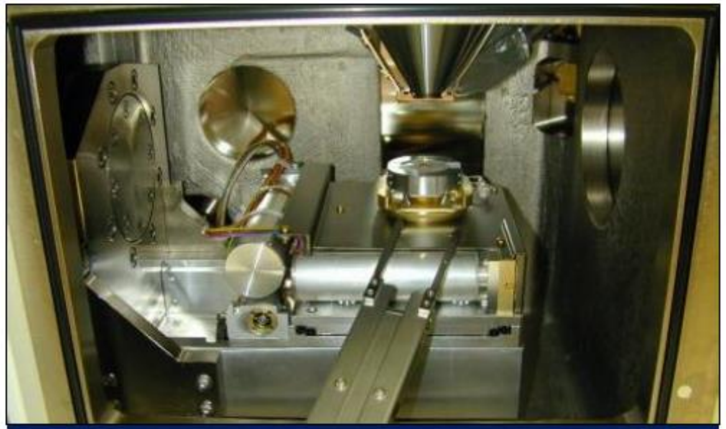
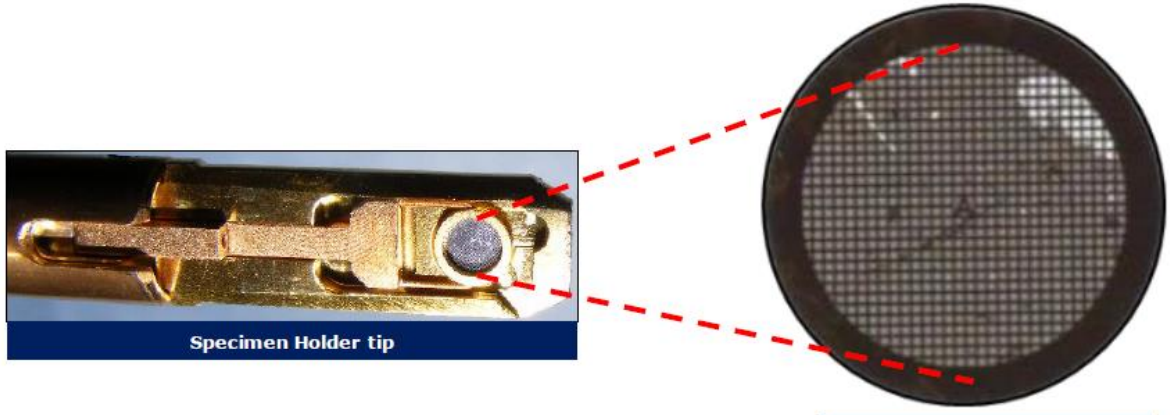
# SEM vs TEM

## Samples


SEM stage

SEM Specimen


Specimen Holder tip

TEM Specimen

- Diameter: 2-3 mm
- Thickness: 100 – 150 nm

# SEM vs TEM

## QUIZ

1. Which statements are true?
   - A. **TEM's sample is more difficult to prepare than SEM's sample**
   - B. Most SEM detectors are located below the sample
   - C. **One can achieve resolution below 1 nm with SEM**
   - D. Scanning mode is only used on SEM
2. Which of these are significant differences between SEM and S/TEM imaging?
   - A. **Sample preparation**
   - B. **Image resolution**
   - C. **Contrast mechanism**
   - D. **Accelerate voltage**

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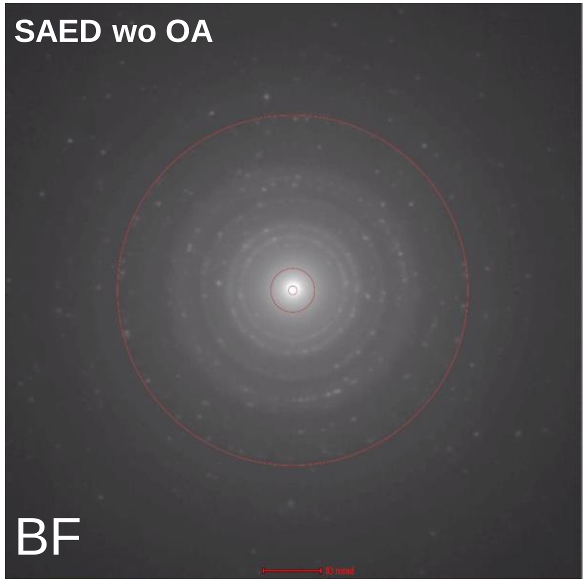
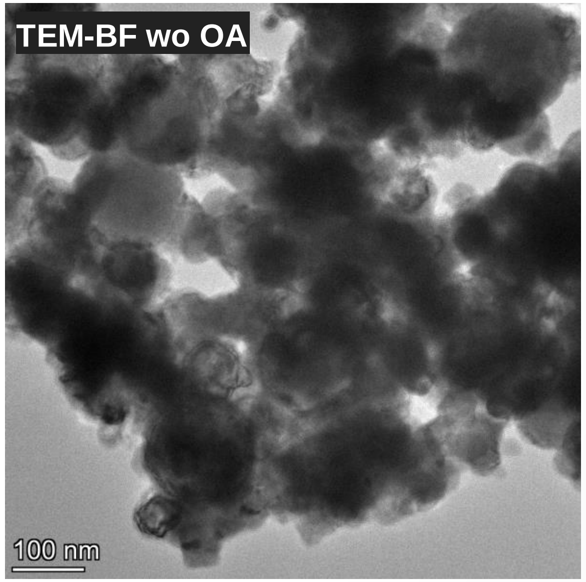
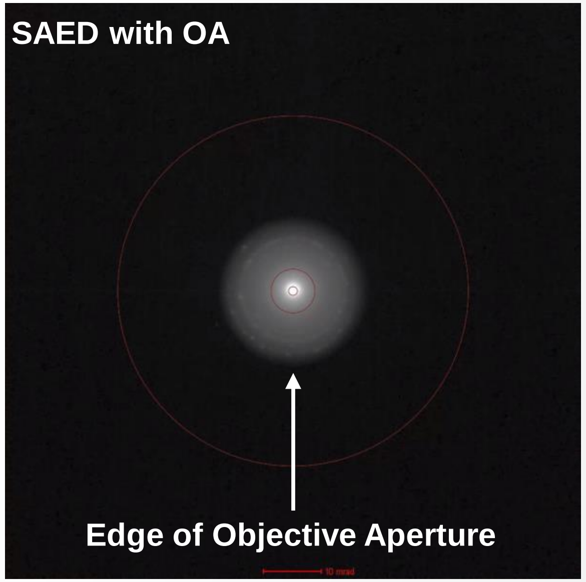
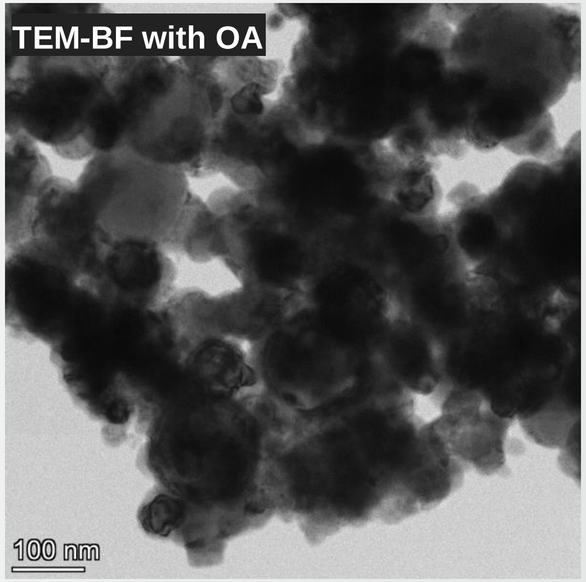
# TEM Bright Field Image

SAED Patterns and respective TEM-BF with and without Objective Aperture (OA)



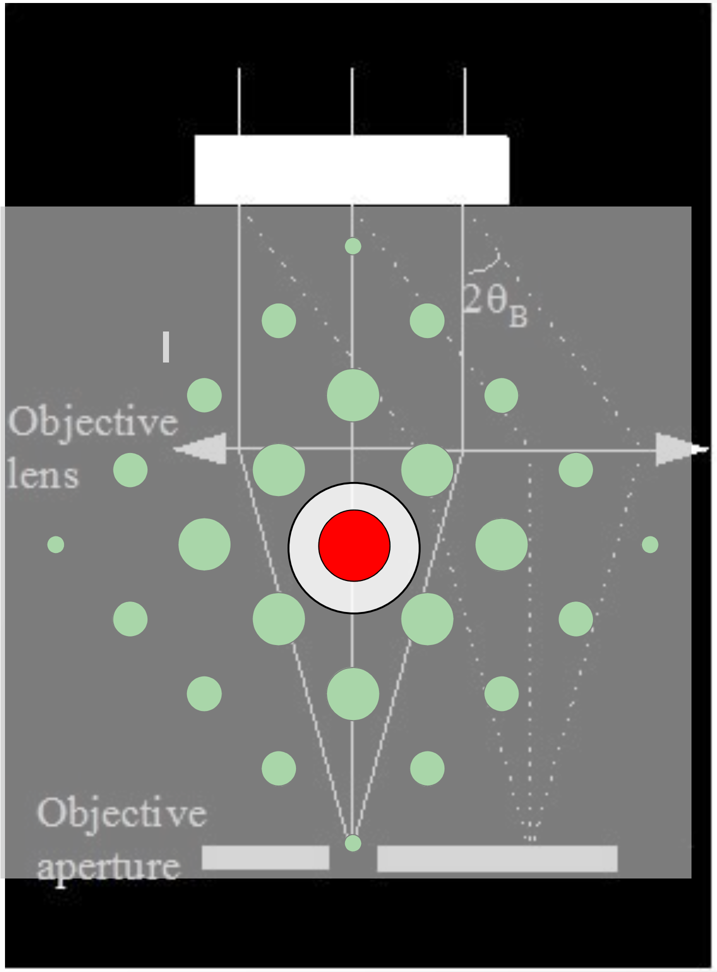

 mohamadriza.iskandar@thermofisher.com 

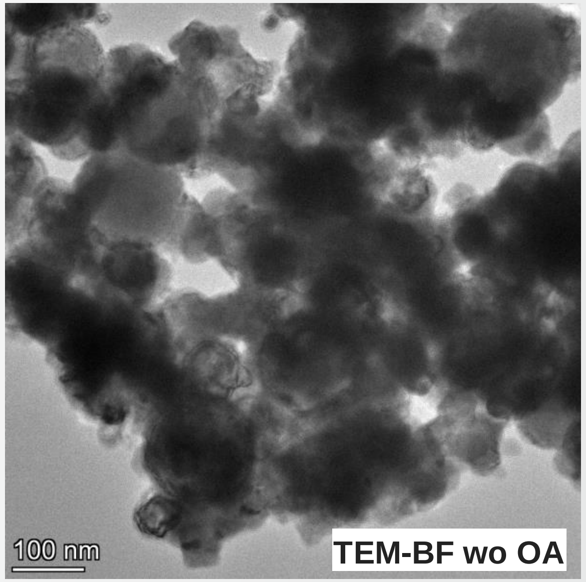
# TEM Bright Field Image

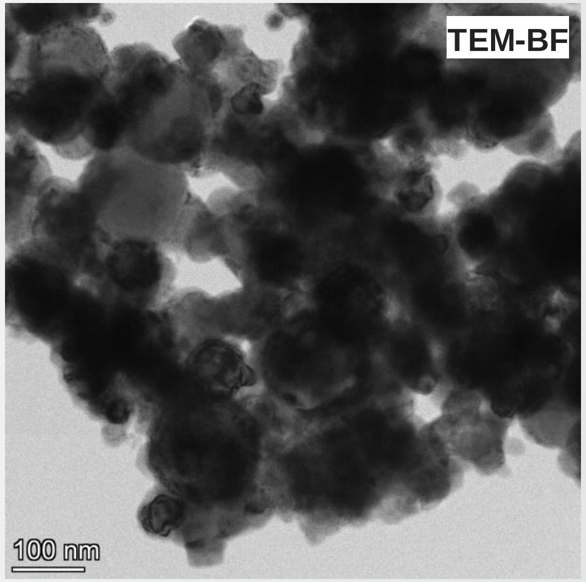
## Bright Field Imaging Formation

- Bright field is the standard mode for imaging
- Selected by the objective aperture (TEM) or the detector (STEM)
- In TEM mode, on-axis dark-field (beam tilting) is better than off-axis dark-field (aperture shifting)

**Objective aperture (OA): an aperture that is used to produce TEM-BF or TEM-DF**



# Bright and Dark Field Imaging

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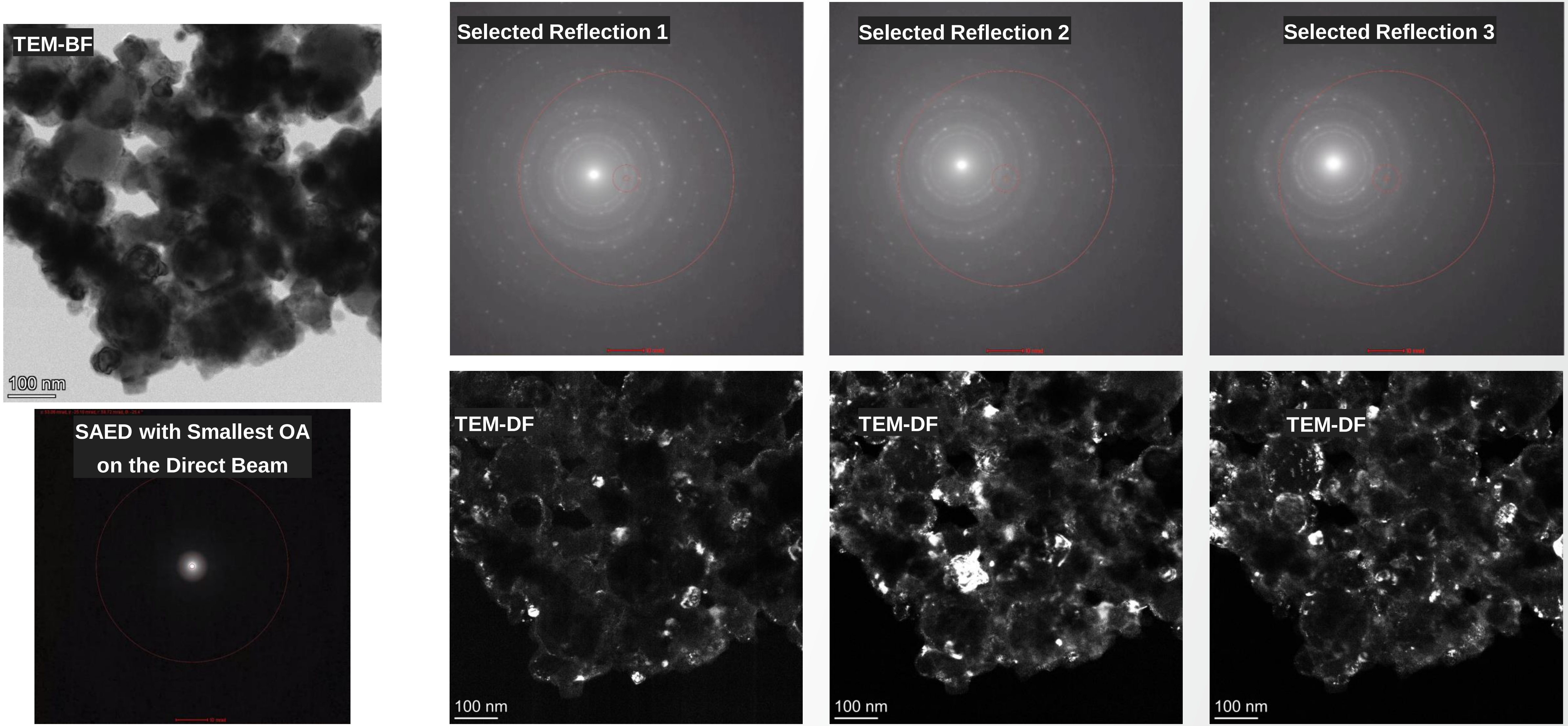
## Bright Field Experiment Important Factors

- Sample is in Eucentric height
- Use the thinner part of the sample
- Refocus the image for fine-tuning (Gaussian Focus)
- Switch to diffraction mode
- If the sample is single crystal, try to orient the sample into a zone axis
- Insert and center the objective aperture
- A smaller objective aperture will give more contrast
- The image can be acquired

- Eucentric height: a sample position where the center of the sample image does not shift during specimen tilt
- Gaussian focus: a focus (imaging) condition for an ideal lens without aberrations
- Zone axis: a direction to a group planes that parallel to certain direction

# TEM-Dark Field Image

## Bright and Dark Field Imaging

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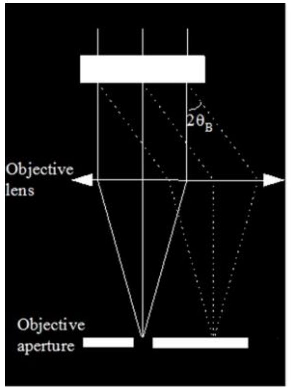
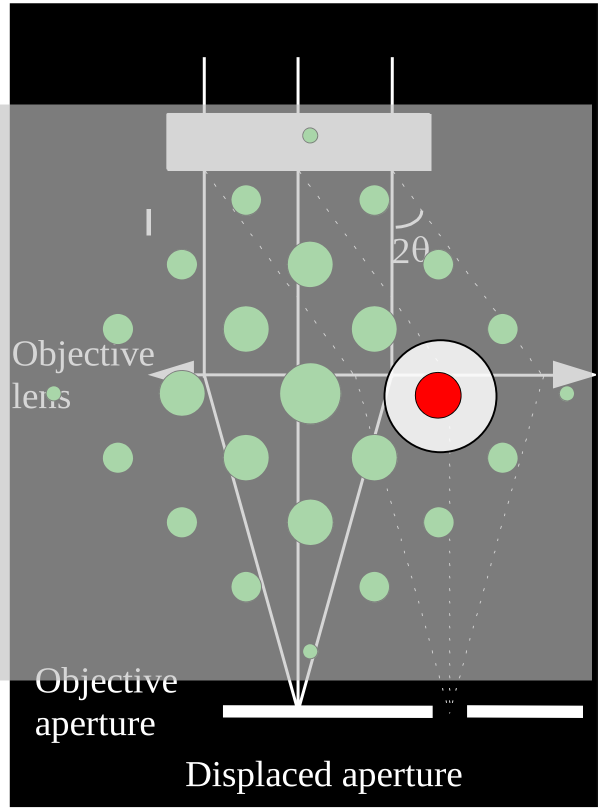
# TEM-Dark Field Image

Dark Field Imaging Formation

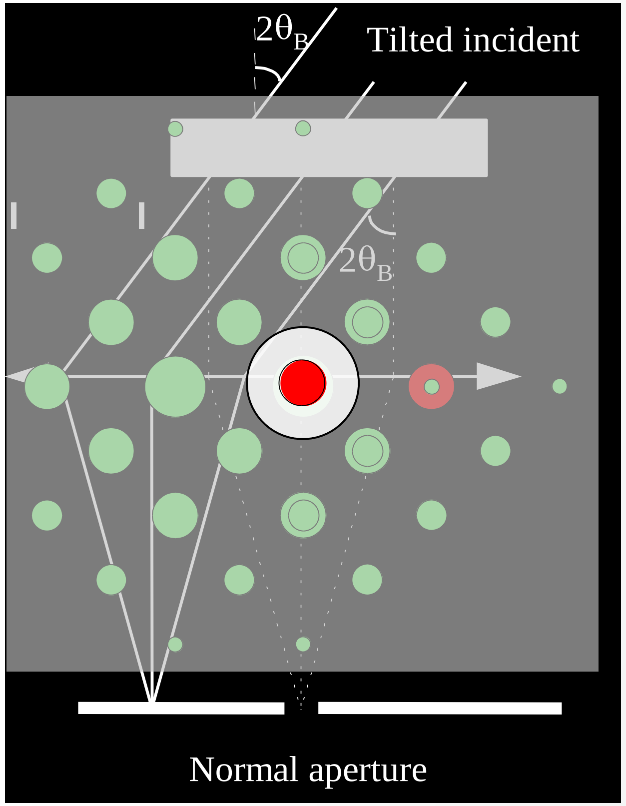
- Standard mode for imaging without the transmitted beam
- Loss of resolution due to higher $C_s$ at off-axis positions
- Two types of DF-Images: Off-axis and On-axis DF



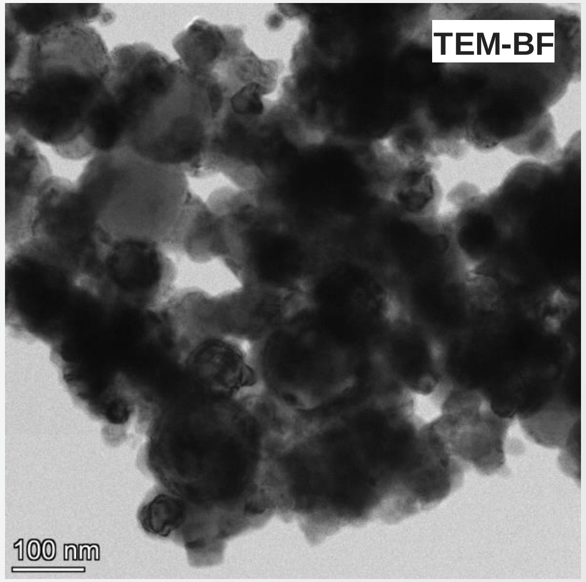

TEM-BF

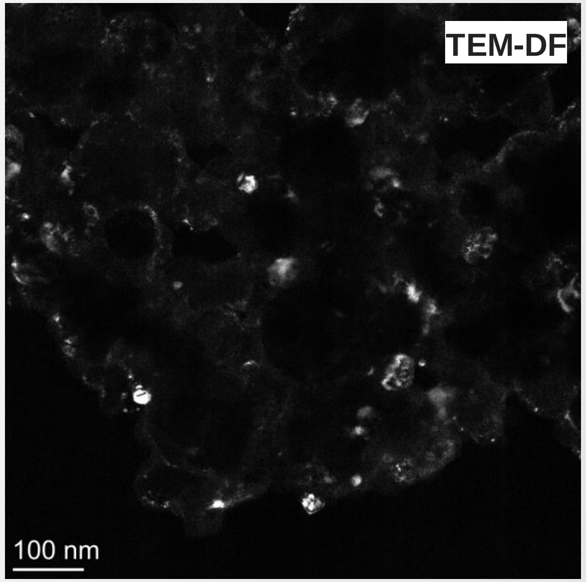



Off-axis DF




On-axis DF

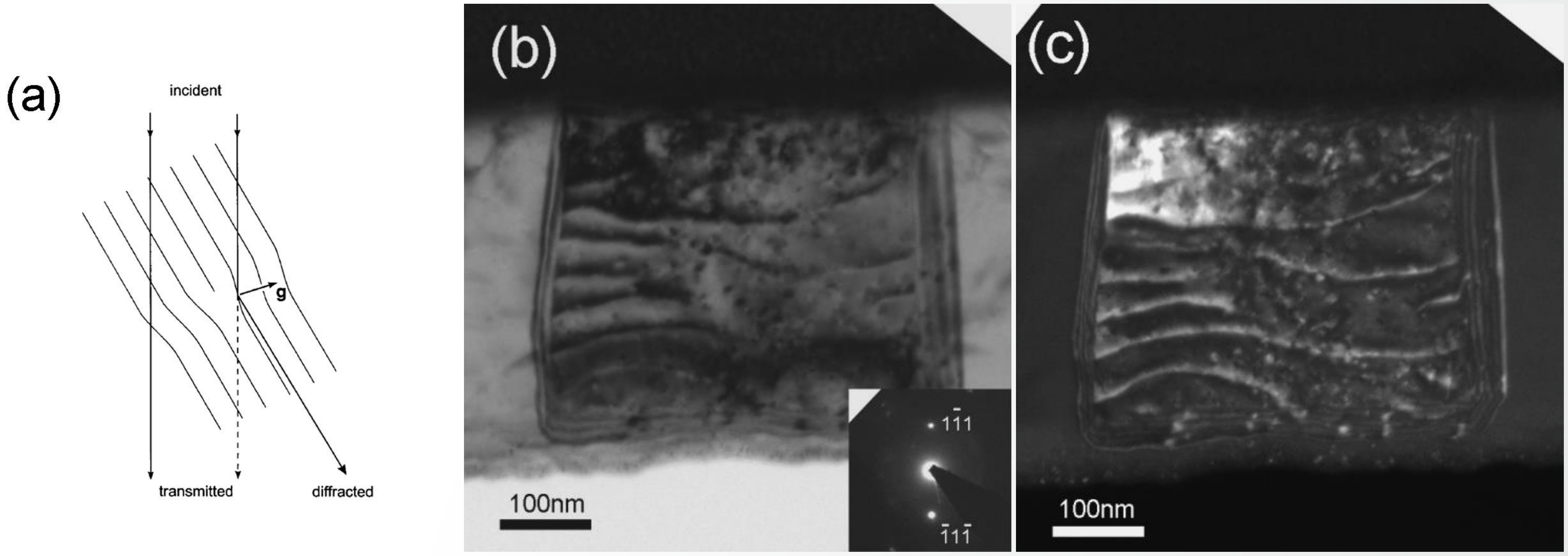


# Why TEM-DF is Important?

## Benefit of Dark Field Image

- Grain Orientation
- Defect Analysis
- Phase formation

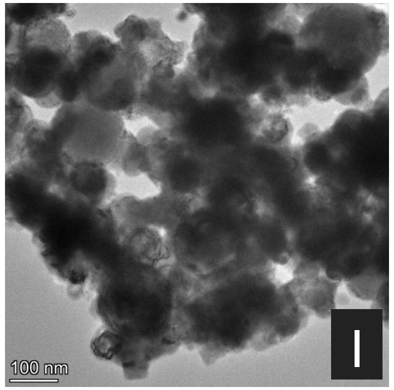


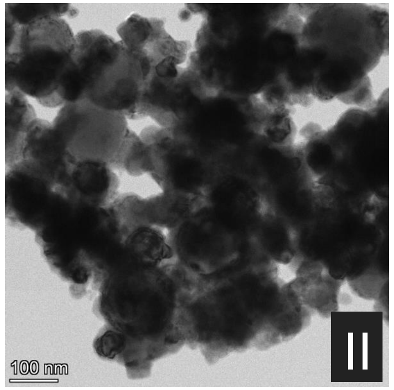

(a) Planes near an edge dislocation bend into the orientation for diffraction (b) BF image and (c) DF image of dislocations under a two-beam condition in an Al thin film. The inset in (b) shows the SAED pattern indicating the orientation condition for BF imaging.

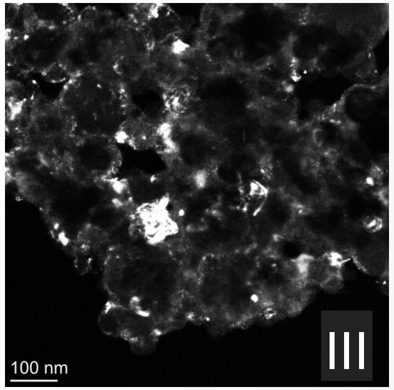
# Conventional TEM Imaging

QUIZ

1. Which one of the following images is the real TEM-BF?




A. I

**B. II**

C. III

D. I,II

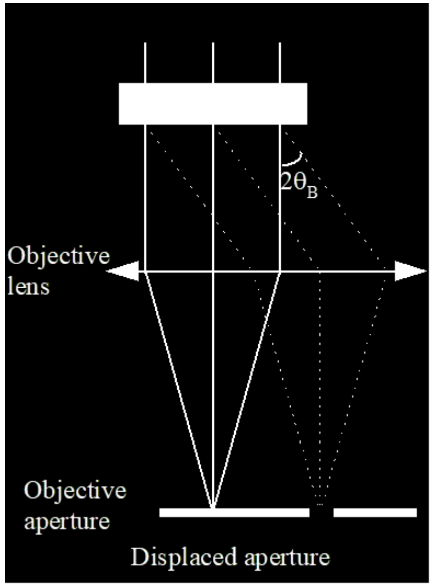
E. II, III

# Conventional TEM Imaging

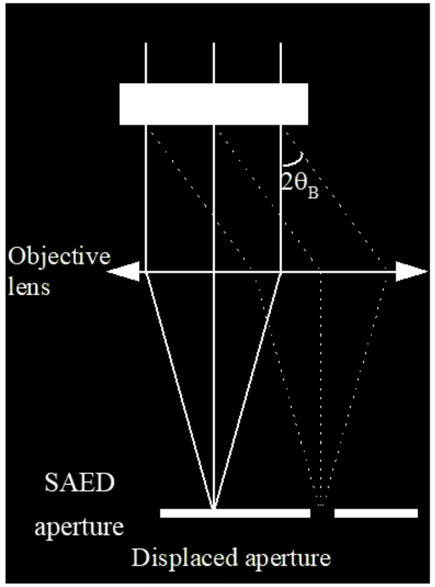
QUIZ

2. A schematic that shows a correct TEM-DF acquisition:

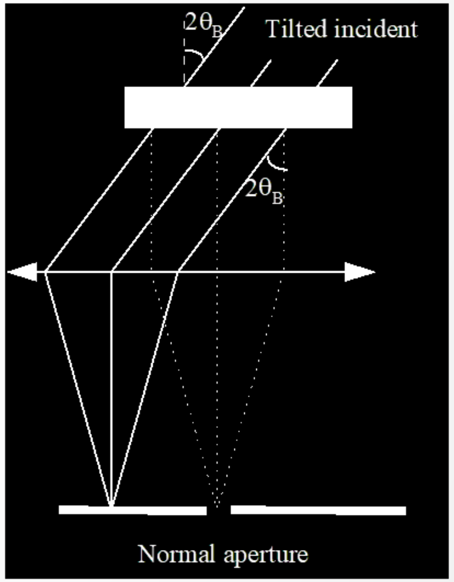
   A. I
   B. II
   C. III
   D. IV
   E. I, II
   F. III, IV



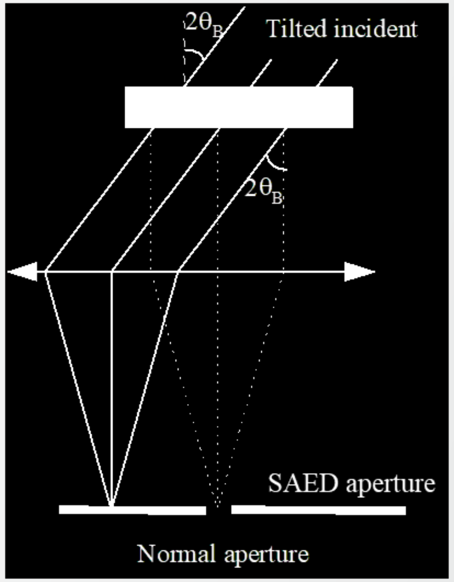

I




II




III

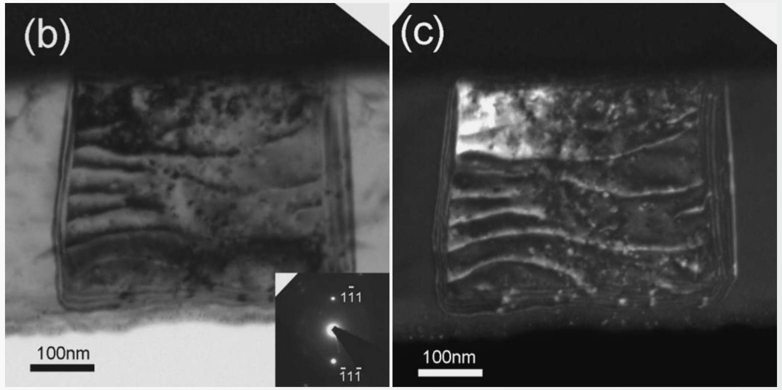



IV

# Conventional TEM Imaging

QUIZ

3. With TEM-DF image, one can not obtain information of:
   A. Crystal Defects
   B. Grain Orientation
   C. **Element Identification**
   D. Phase Distribution




4. Which is valid for acquiring a TEM BF:
   A. **The sample must be in eucentric height**
   B. The sample must be in zone axis
   C. The smallest objective aperture must be used
   D. The camera shortest camera length must be used to increase the contrast

# Introduction to Transmission Electron Microscopy

Thermo Fisher SCIENTIFIC

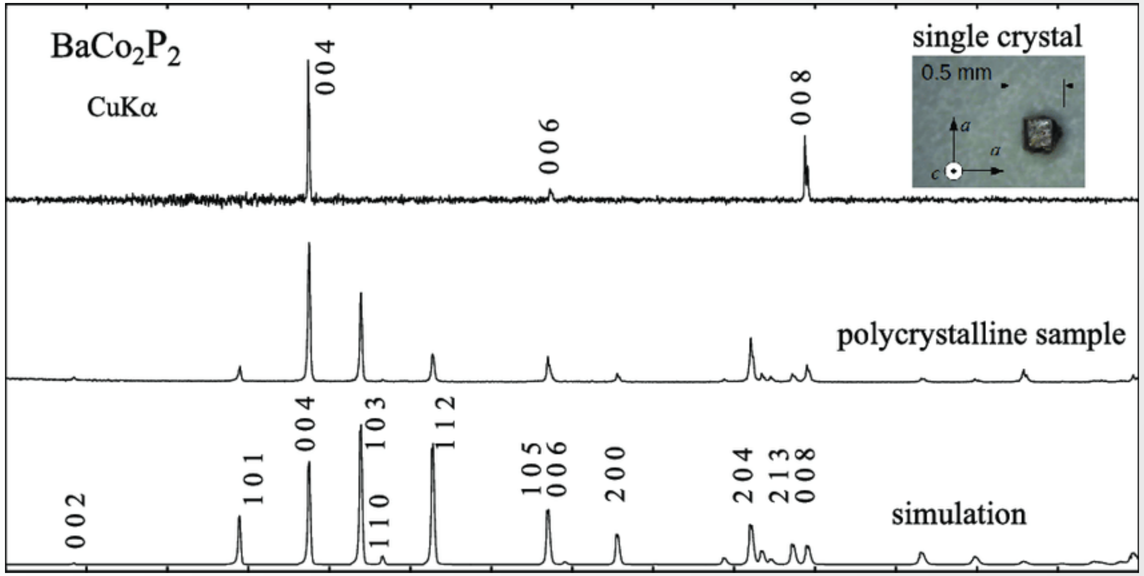
Outline: Part 01

- TEM Basics
- SEM vs TEM: What are the differences?
- Conventional TEM Imaging
  - Bright-Field Imaging
  - Dark-Field Imaging
- Electron Diffraction
  - Selective Area Electron Diffraction
  - Convergence Beam Electron Diffraction
  - Nano Beam Electron Diffraction
- High-resolution TEM

# Electron Diffraction

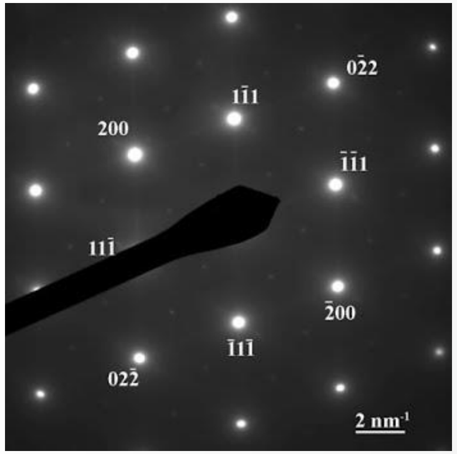
## X-rays vs Electron Diffraction

| X-rays vs Electron Diffraction | | |
|---|---|---|
| **Parameter** | **X-rays** | **Electron** |
| Analysis | Intensity and position | Position |
| Type of wavelength for single crystal | Multi-wavelength | Single-wavelength |
| Acquisition time | Minutes to hours | Less than second |

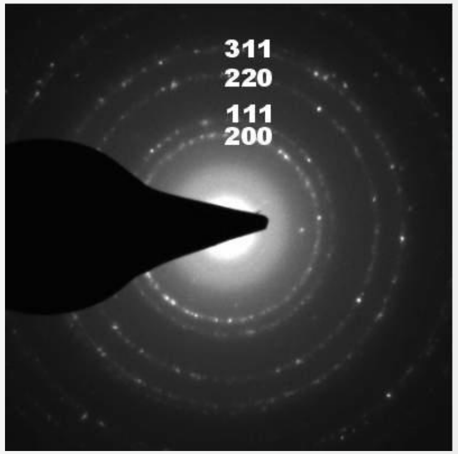



Important facts about electron for diffraction:

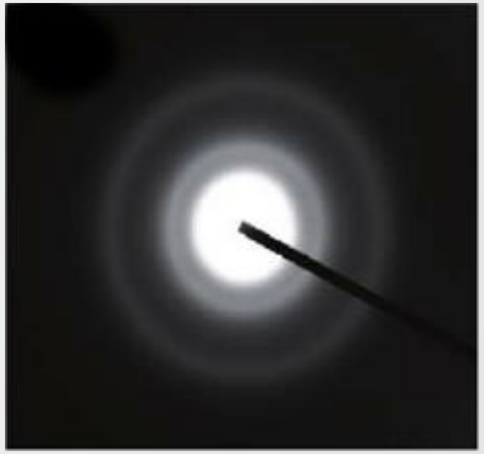
- Electrons have shorter λ than X-rays
- Electron scattered more strongly
- Electron beams are easily directed




Single crystalline SAED pattern



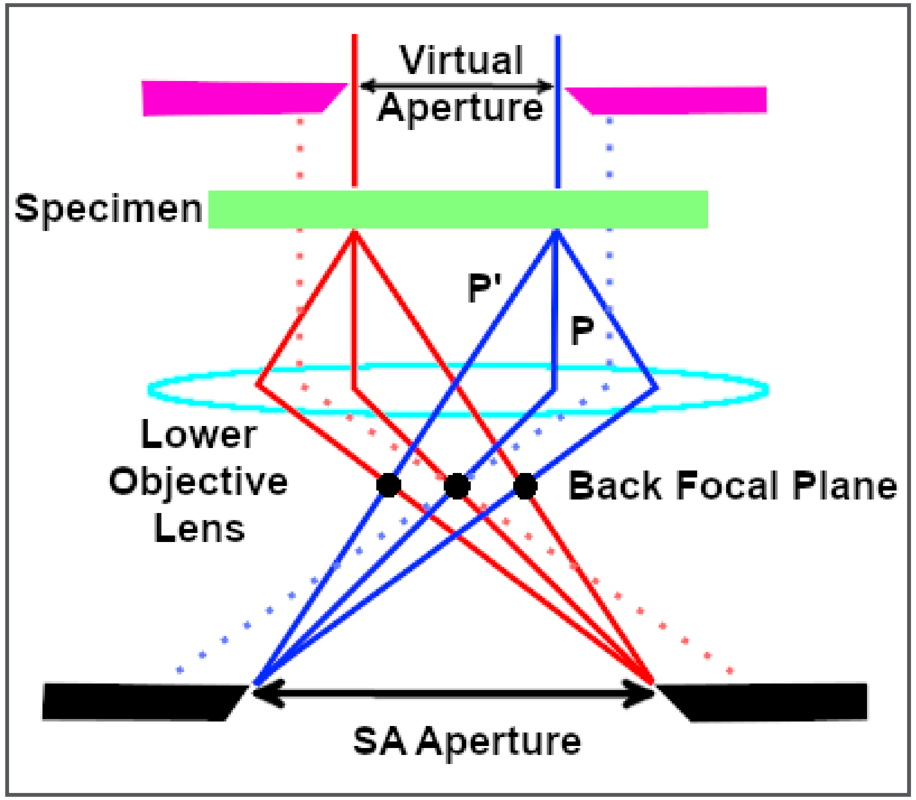

Poly-crystalline SAED pattern



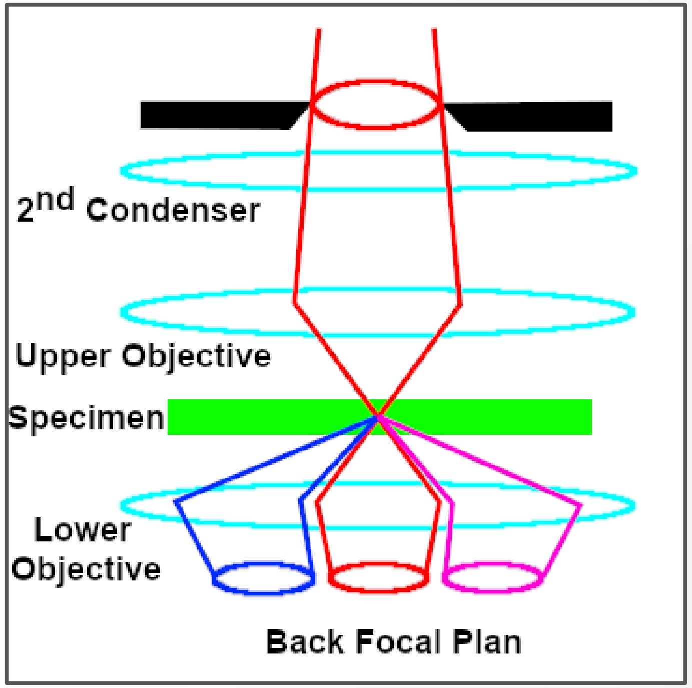
Amorphous SAED pattern

# Type of Electron Diffraction in TEM

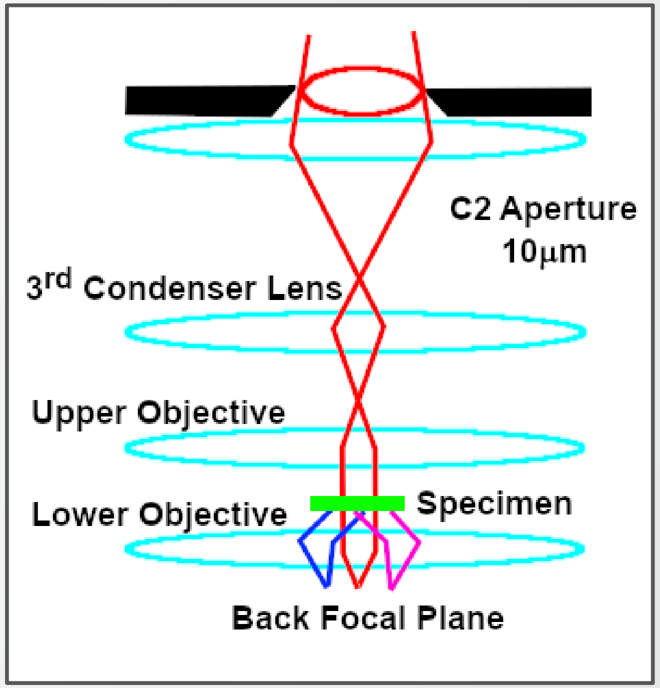
Three Main Electron Diffraction Techniques




Selected Area Electron Diffraction (SAED)



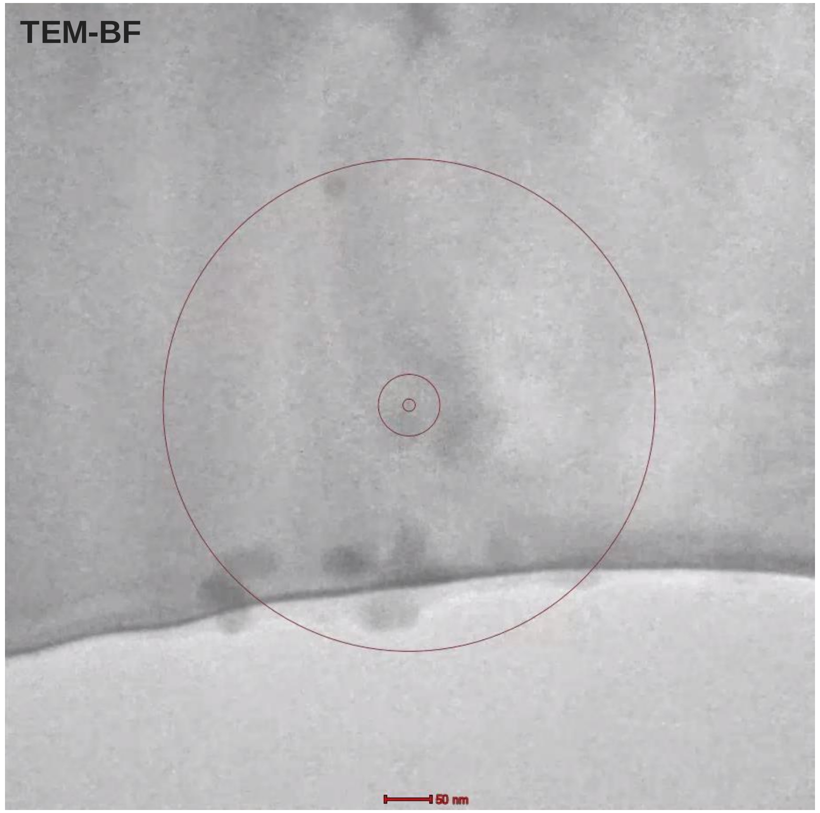

Convergence Beam
Electron Diffraction (CBED)

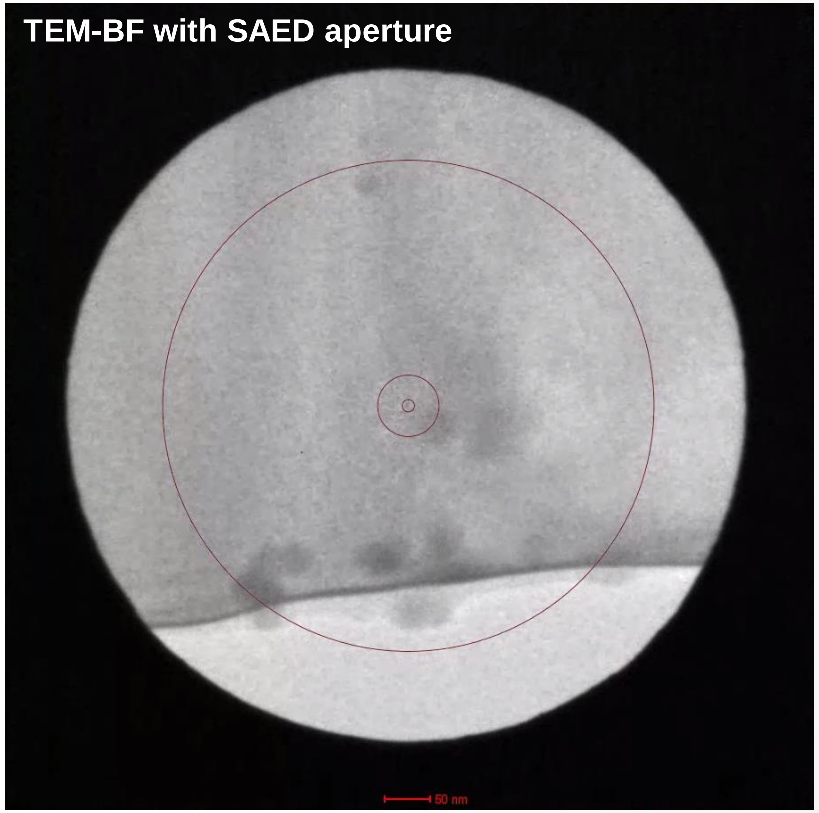


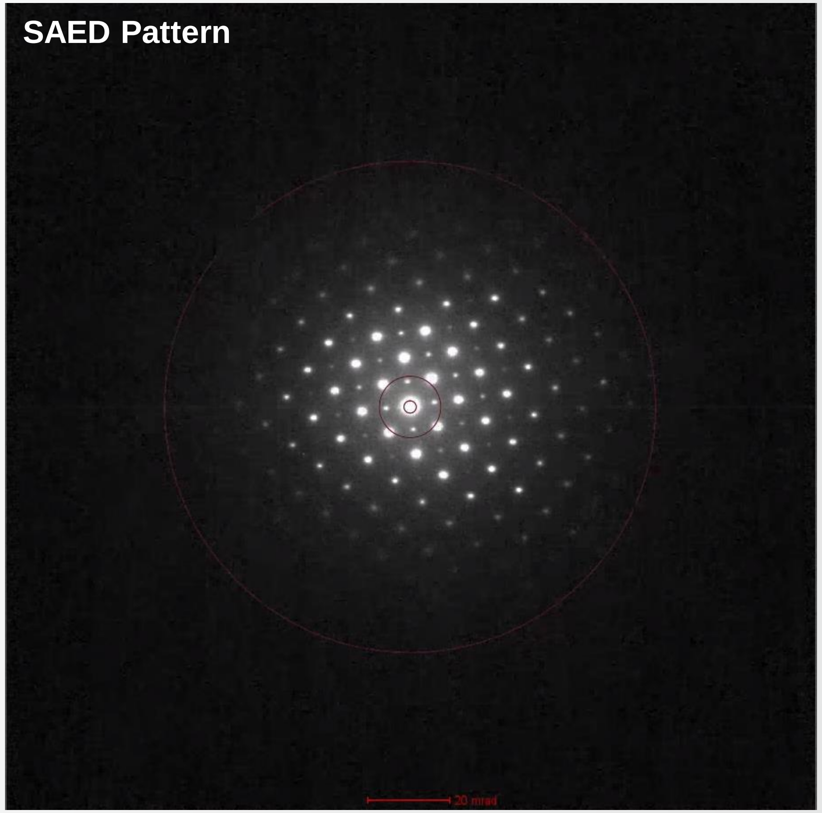

Nano Beam
Electron Diffraction (NBED)

# Selected Area Electron Diffraction (SAED)

## SAED Procedure



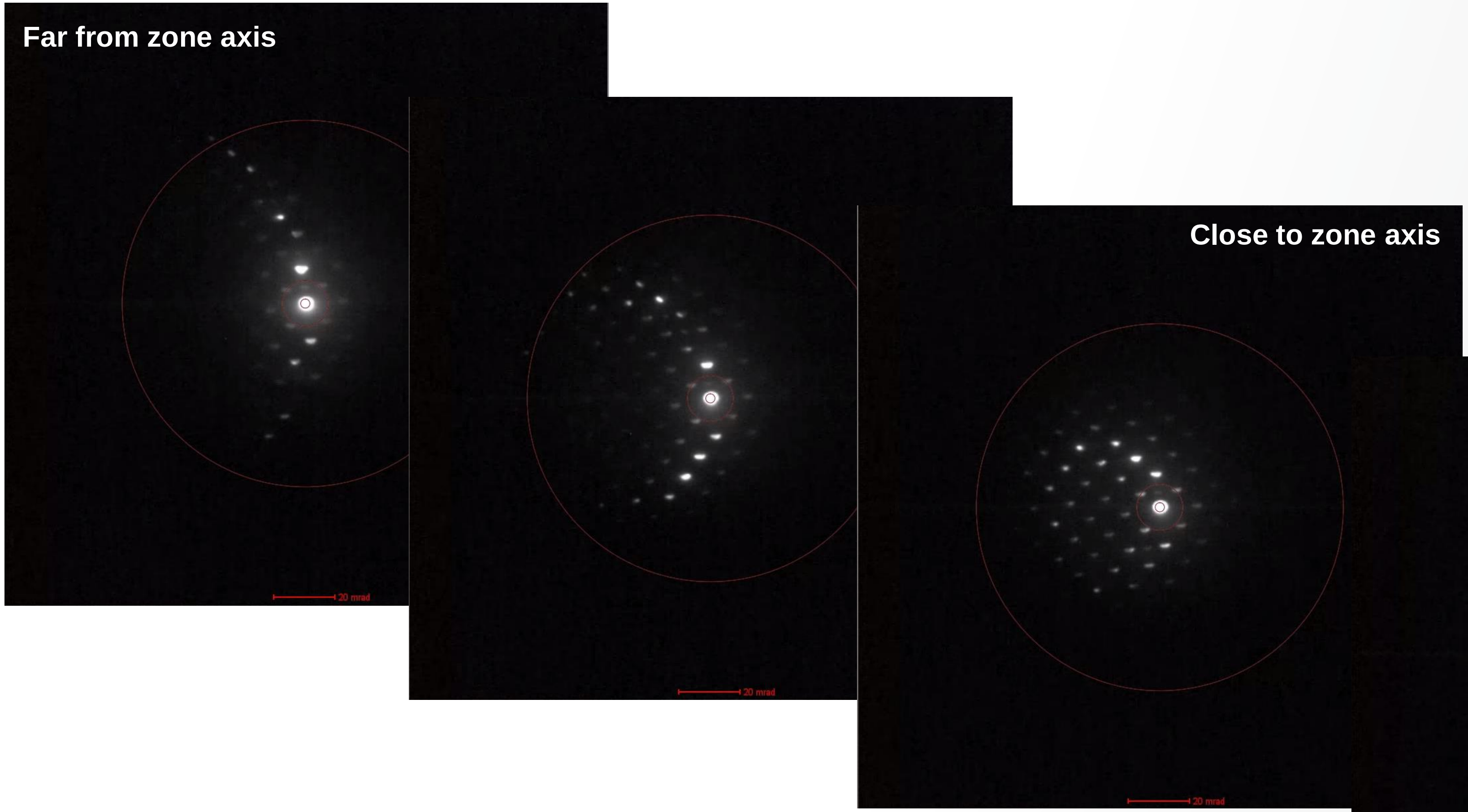



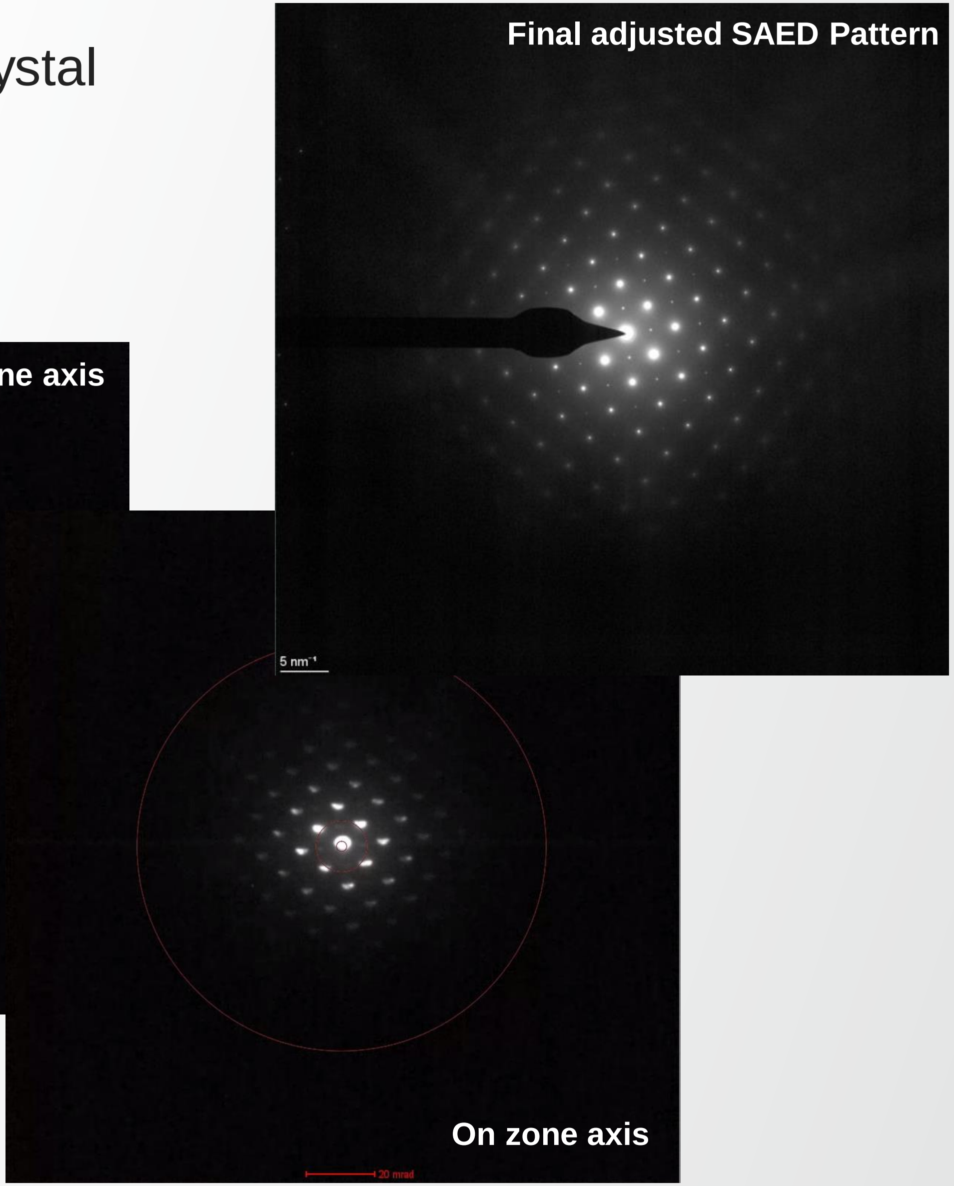

- Make sure sample in eucentric height
- As the name, the aperture is always needed to select an area and produce the Electron Diffraction Pattern
- Always take above three images when acquiring SAED pattern


mohamadriza.iskandar@thermofisher.com

# Selected Area Electron Diffraction (SAED)

Orienting a single crystal using the diffraction pattern: Single Crystal




- The sample tilt is gradually adjusted until the orientation is correct “On zone axis” which is suitable for high resolution lattice imaging.
- A double tilt holder is needed!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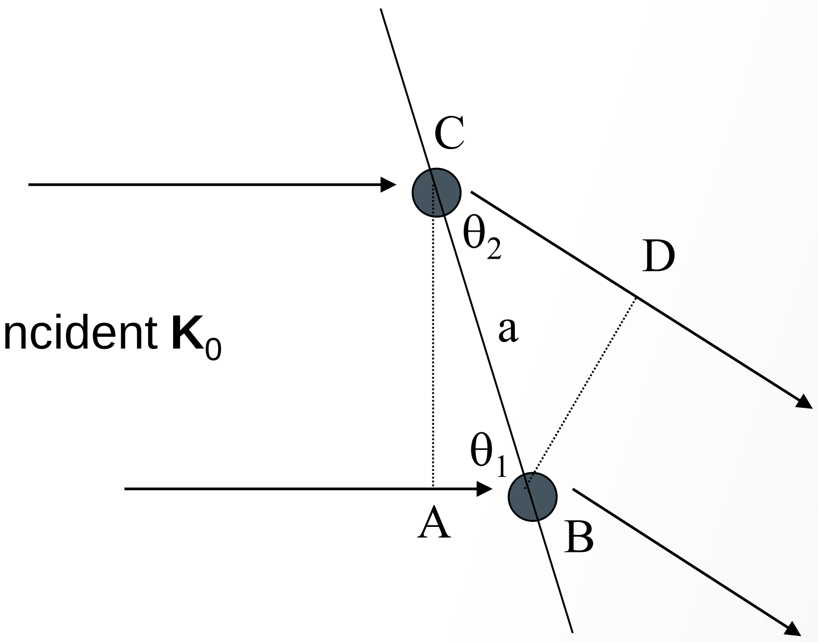
# Selected Area Electron Diffraction (SAED) Analysis

Diffraction and Interference

path difference = $$AB - CD = a(\cos\theta_1 - \cos\theta_2) = n\lambda$$




incident $\mathbf{K}_0$

scattered $\mathbf{K}$

or

$$K - K_0 = B_H$$

= reciprocal vector

## General concept: monochromatic & coherent radiation

- elastic scattering
- constructive interference only in certain directions
- special case: $q_1$ = 90°: high resolution TEM

# Selected Area Electron Diffraction (SAED) Analysis

## Bragg Equation

$$2\, d \,\mathrm{Sin}\, \theta = n\, \lambda$$

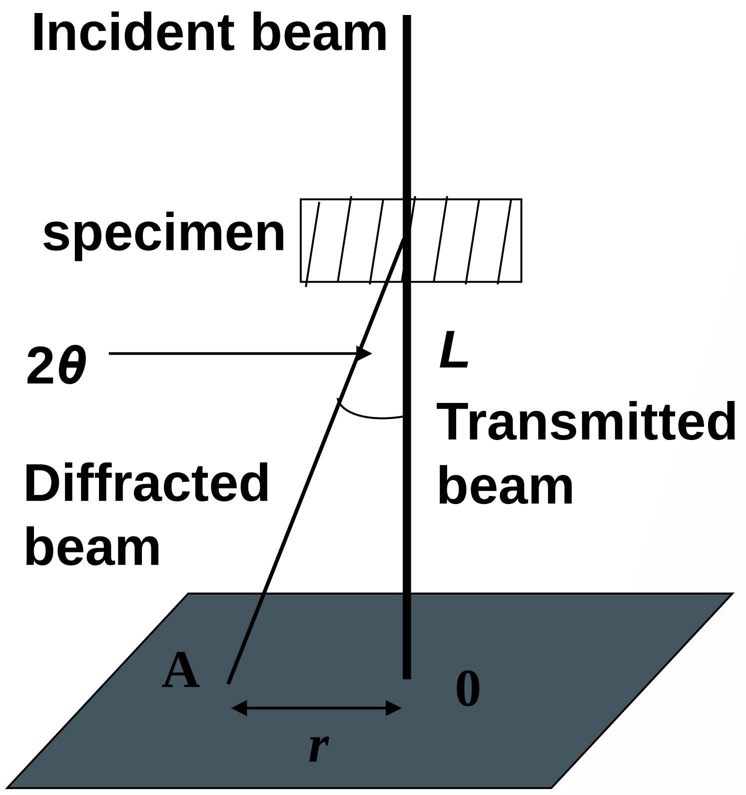
***d*** is the spacing of atoms which scatter the electrons
***n*** is the order of diffraction
***θ*** the Bragg angle

For electron diffraction, electron wavelength is small, $\theta$ is therefore small.

We expect diffraction from planes of atoms almost parallel to the incident electron beam.

# Selected Area Electron Diffraction (SAED) Analysis

(An Overview) SAED Pattern Analysis




**For small angle of diffraction:**

$$r / L = 2\theta$$

But $\lambda = 2\, d\, \theta$

$$r / L = \lambda / d$$

$$\boxed{r\, d = L\, \lambda}$$

***L λ* is the camera constant**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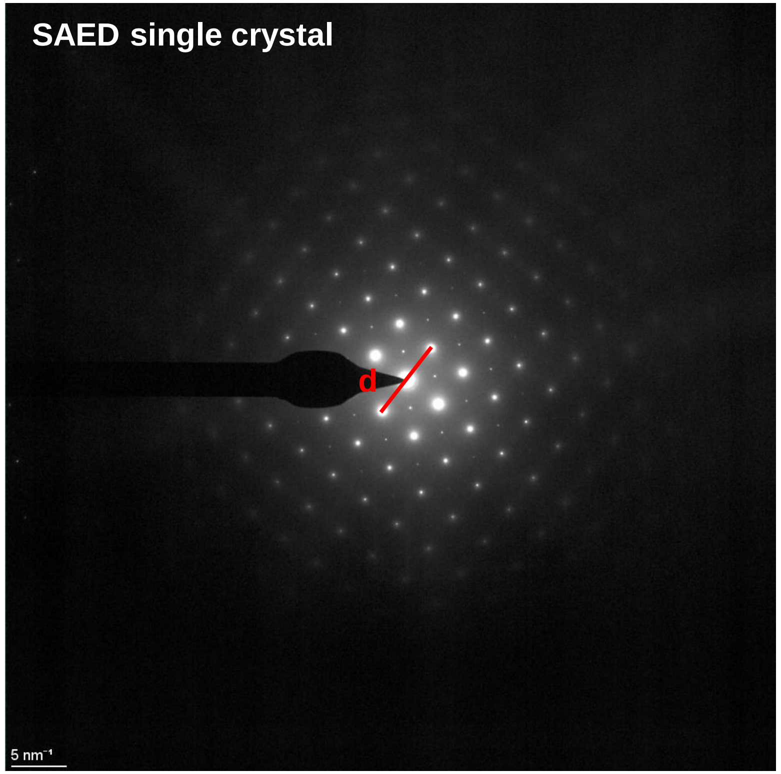
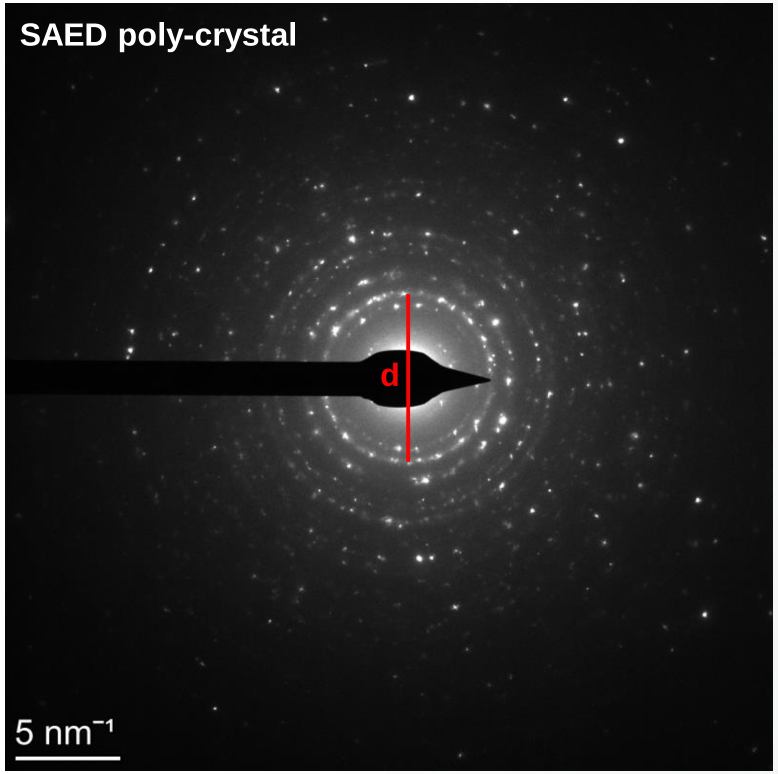
# Selected Area Electron Diffraction (SAED) Analysis

## SAED Pattern Analysis Procedures



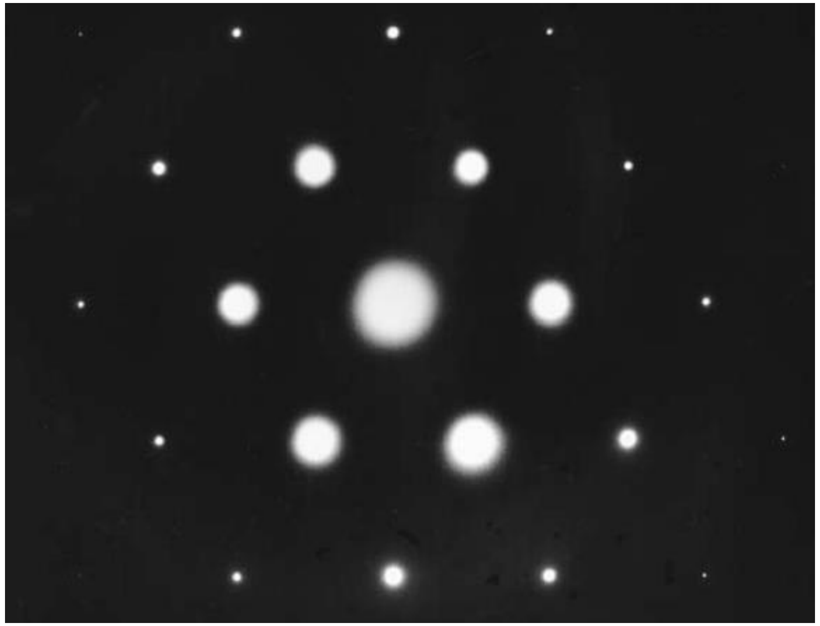




- Make sure that the microscope is calibrated
- Measure the distance from reflections or rings to the main spot (d/2)
- Inversed the measured value
- Compare the data (d-spacing) with database to find the phase presence

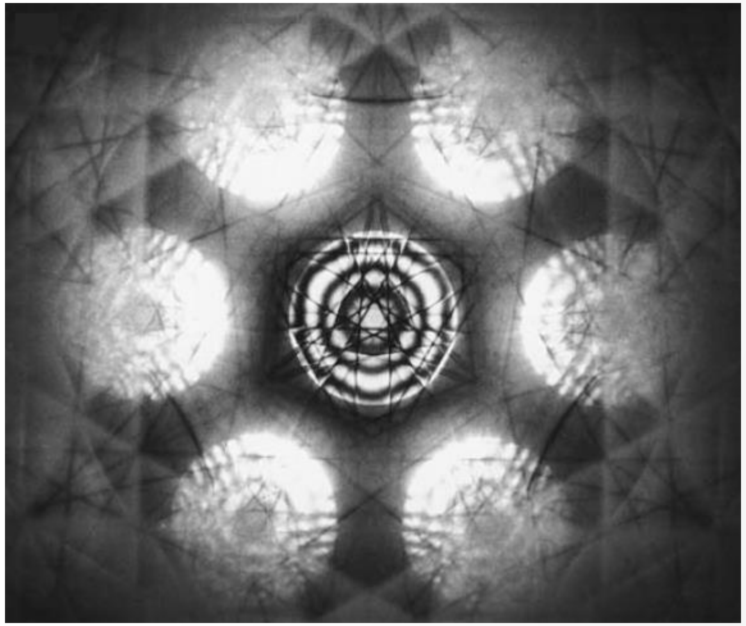
# Convergence beam electron diffraction (CBED)

## More Than Just Electron Diffraction

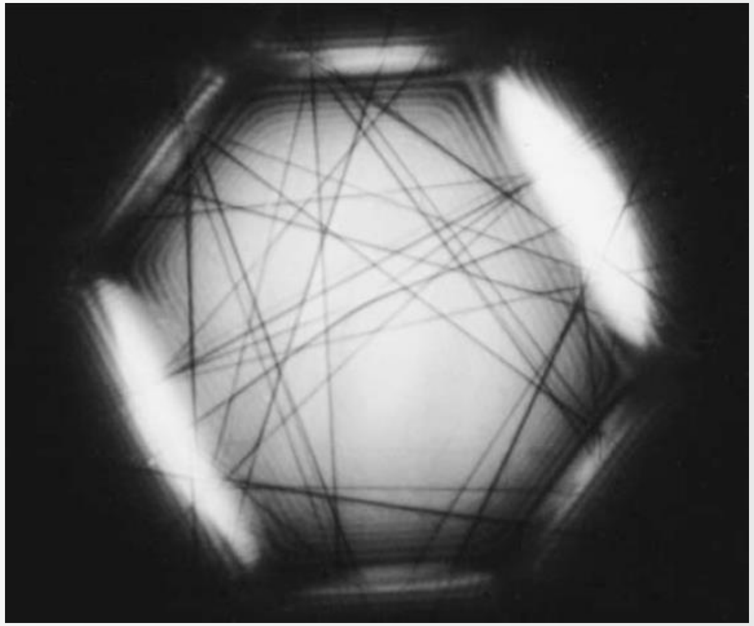
SAED pattern of Si <111>



CBED pattern of Si <111>



Al HOLZ line pattern

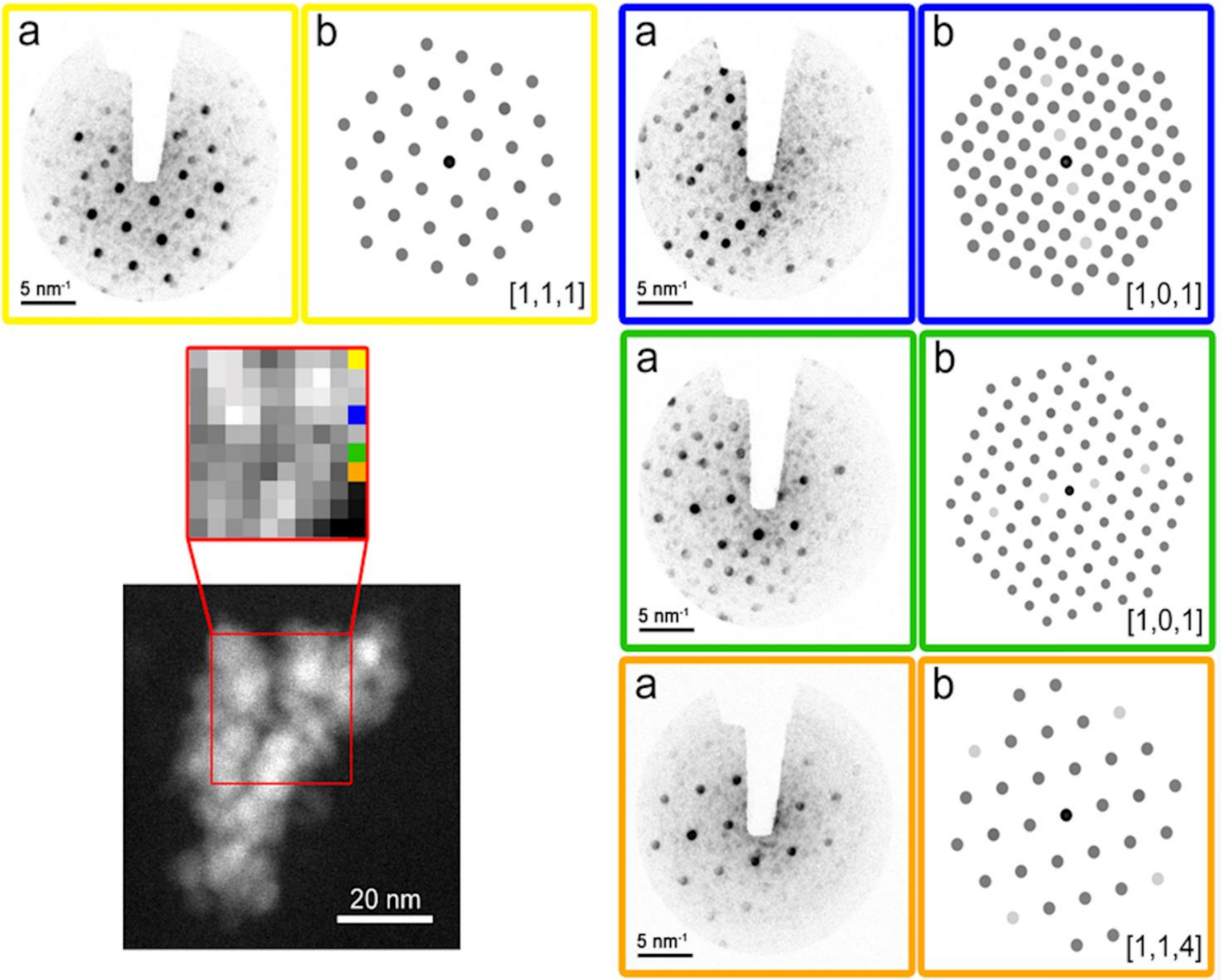


**Ideal for relatively thicker sample**

- Small area ≤ 100 nm
- CBED gives quantitative data on
  - Specimen thickness
  - Crystallographic data such as unit cells, Bravais lattice, crystal system and 3D full symmetry
  - Precise lattice-strain measurements
  - Valence-electron distribution, structure factor, and chemical bonding
  - Characterization online and planar defects

# Nano Beam Electron Diffraction

## Electron Diffraction on Small Volume




- Nanocrystals of $Fe_3O_4$ (magnetite), which have been incorporated in melt spinning polyvinylidene fluoride (PVDF) fibers.
- Experimental NBED in STEM mode (a) with corresponding simulated pattern (b).
- The frame colors refer to the position of electron beam during acquisition of the diffraction patterns.
- Domains or particles can be analyzed at nm-range by collection electron diffraction pattern.

*Data countersy of N.Wirch Central Facility for Electron Microscopy, RWTH Aachen University*

# Electron Diffraction

Thermo Fisher SCIENTIFIC

QUIZ

1. Which is not the properties of electron for diffraction:
   A. Electron has shorter $\lambda$ than X-rays
   **B. Electron is less destructive than X-rays**
   C. Electron scattered more strongly
   D. Electron beams are easily directed
2. The conditions that are needed to analyze SAED patterns:
   **A. The SAED pattern has been calibrated**
   **B. The aperture must be inserted**
   C. The crystal must be oriented in the zone axis
   **D. The sample must be in eucentric height**

# Electron Diffraction

QUIZ

3. Convergence beam electron diffraction (CBED) can be used to produce diffraction on the size of:
   A. 500 nm
   **B. 100 nm**
   C. 1 nm
4. What is true about CBED patterns?
   A. Use an aperture to select an area of interest
   **B. Can give information about specimen thickness**
   **C. Can provide information about chemical bounding**
   **D. It is more suitable for a thicker TEM sample**

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# Introduction to Transmission Electron Microscopy

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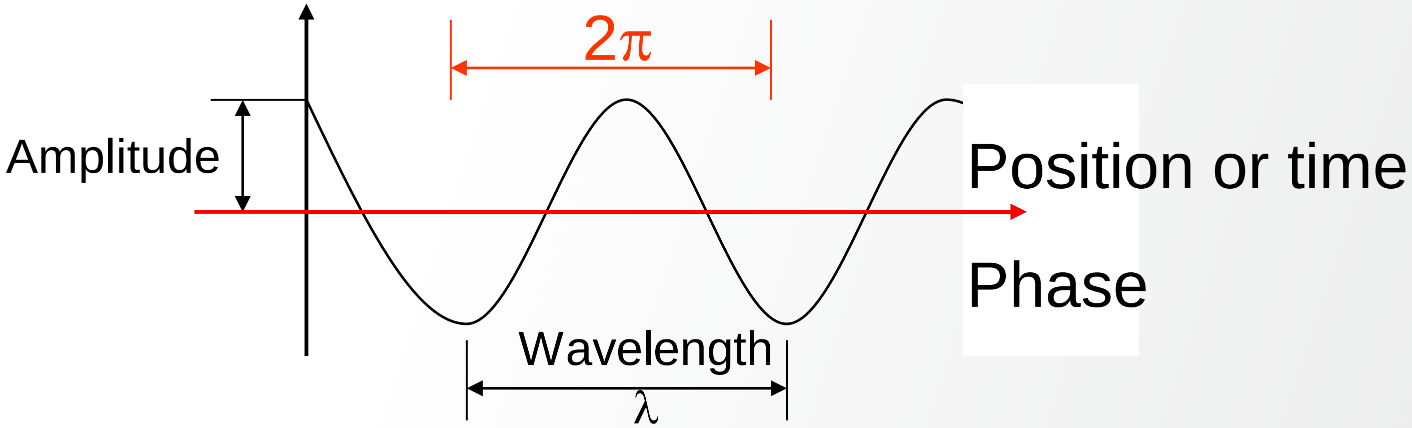
  - Nano Beam Electron Diffraction
- High-resolution TEM

# High-resolution TEM

## Electron as a Wave

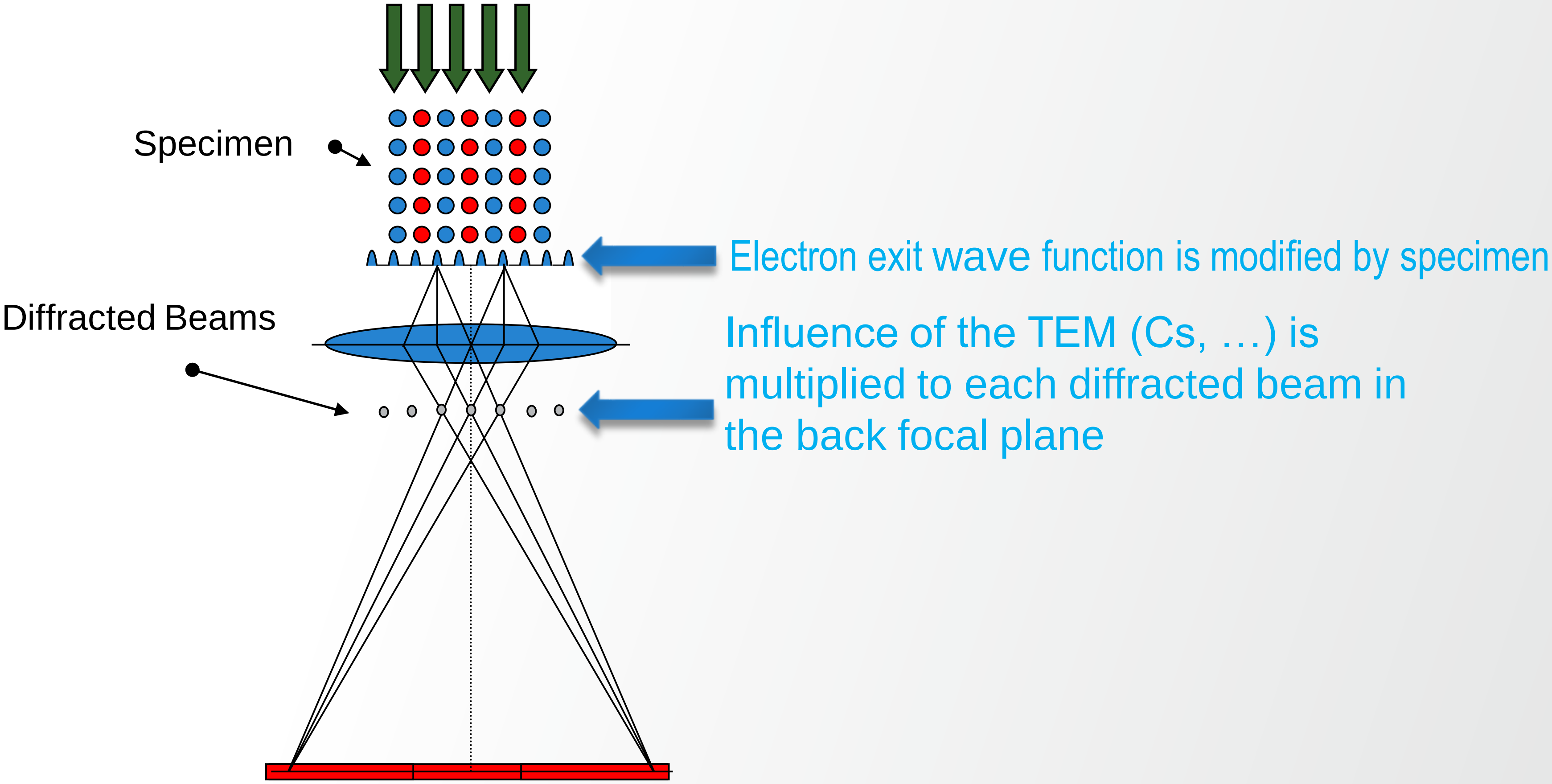
- Electron beam is a wave with *amplitude (A) and phase ($\varphi$)*
- The periodicity is the wave-length (e.g. 0.0025nm at 200kV) or, in terms of phase, $2\pi$



# High-resolution TEM

## Imaging Formation

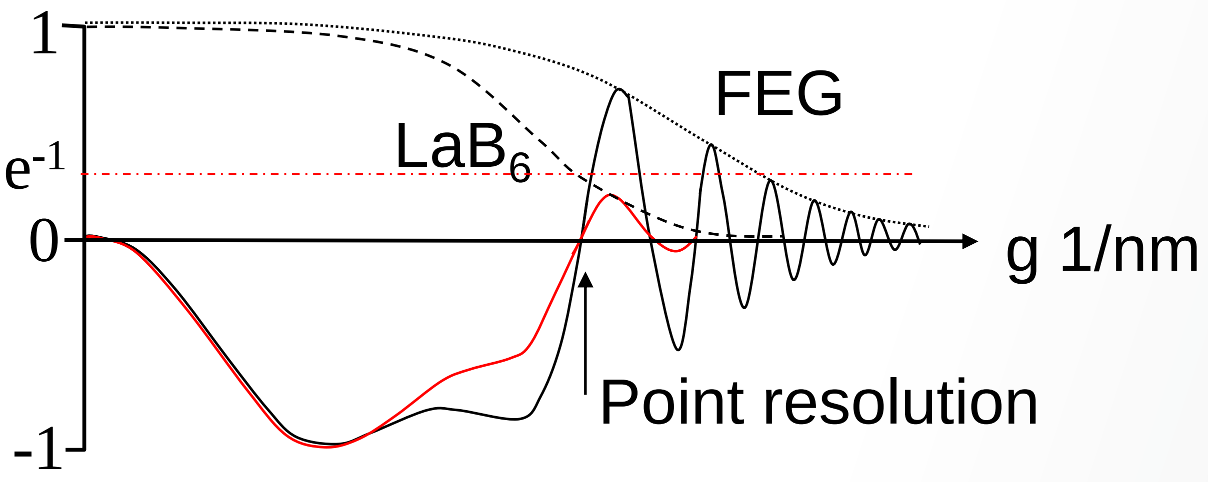
Parallel Incoming electron beam (wave)

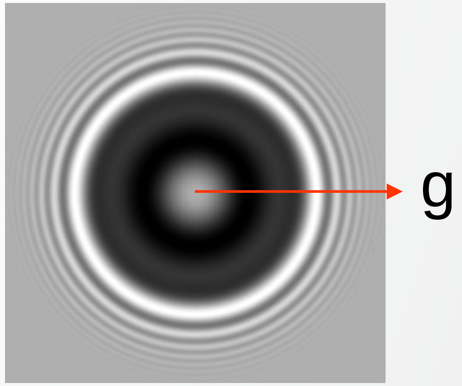


# High-resolution TEM

High-resolution TEM: Contrast Transfer Function (I)

- Point resolution is the function of Cs and λ
- It is the same for W, LaB6 and FEG
- But the information limit is different




- **Point Resolution or Resolution:**
  A wave number at which the phase-constant transfer function at Scherzer focus intersects at phase-zero axis
- **Information limit:**
  Indicates the wave number at which phase information carried by the phase-contrast transfer function disappears.

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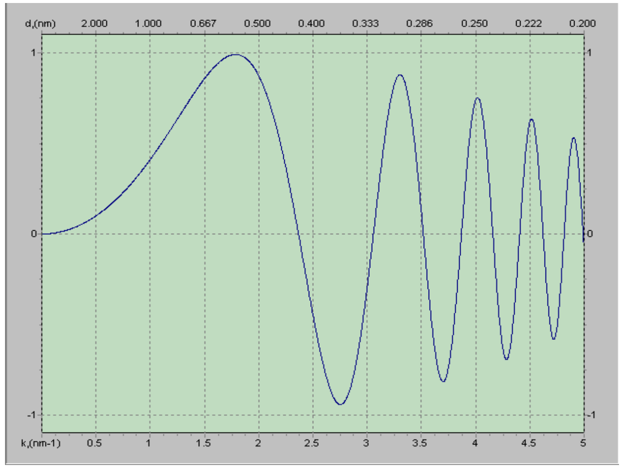
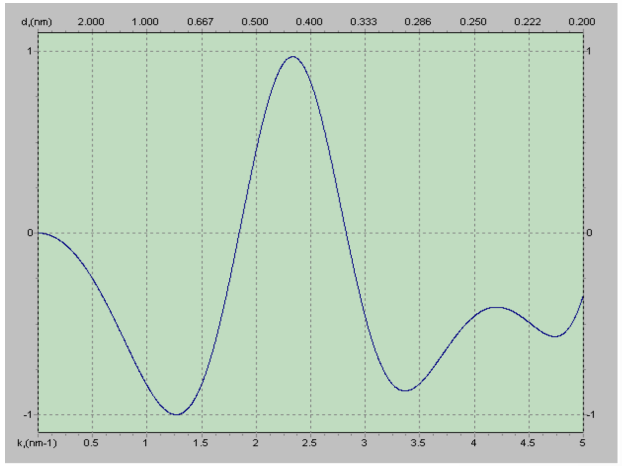
# High-resolution TEM

## High-resolution TEM: Contrast Transfer Function (II)

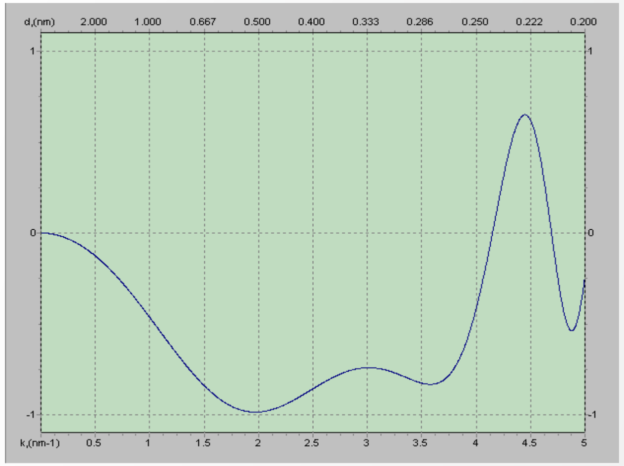
- CTF greatly depends on the focus value (Cs=1.2mm)



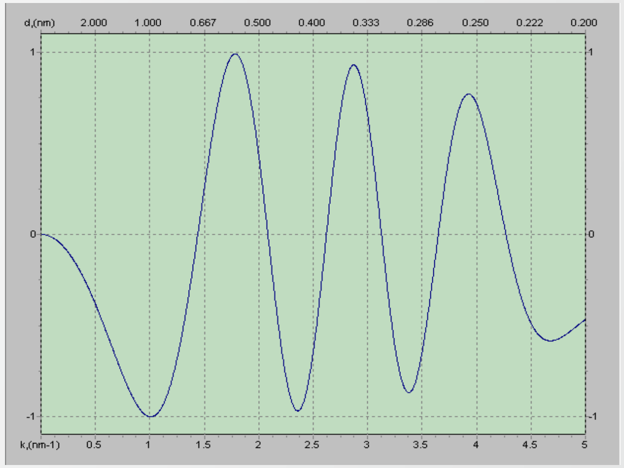
Δf=+50nm



Δf=-130nm



Δf=-65nm

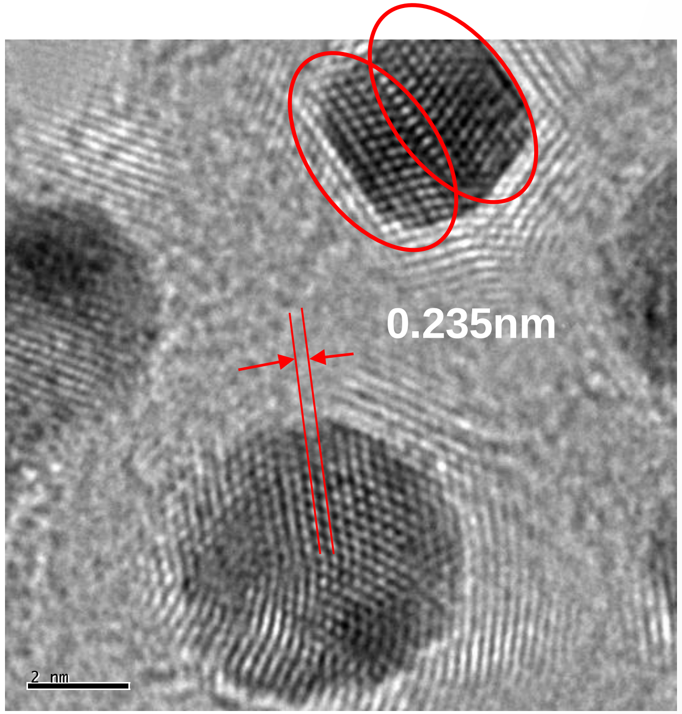


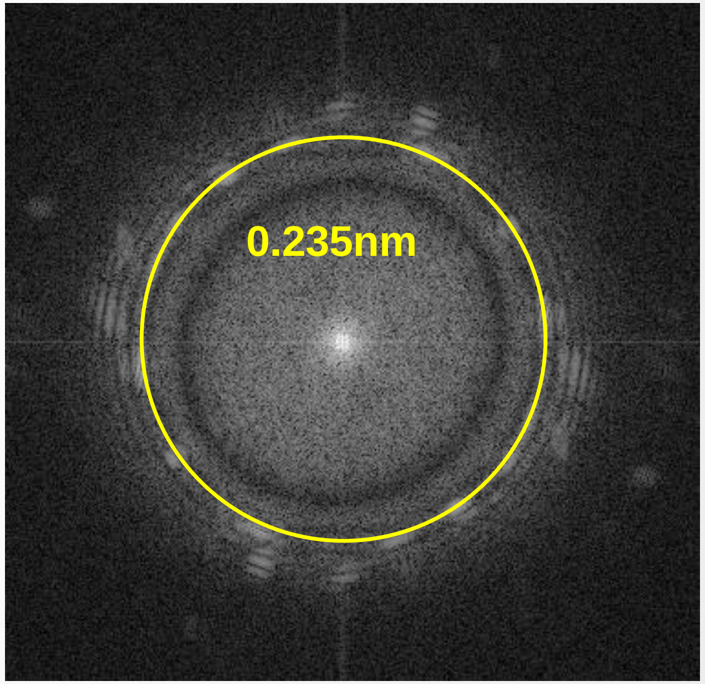
Δf=-200nm

# Advanced TEM Imaging: High-resolution TEM

High-resolution TEM: What do we See on a HRTEM image?

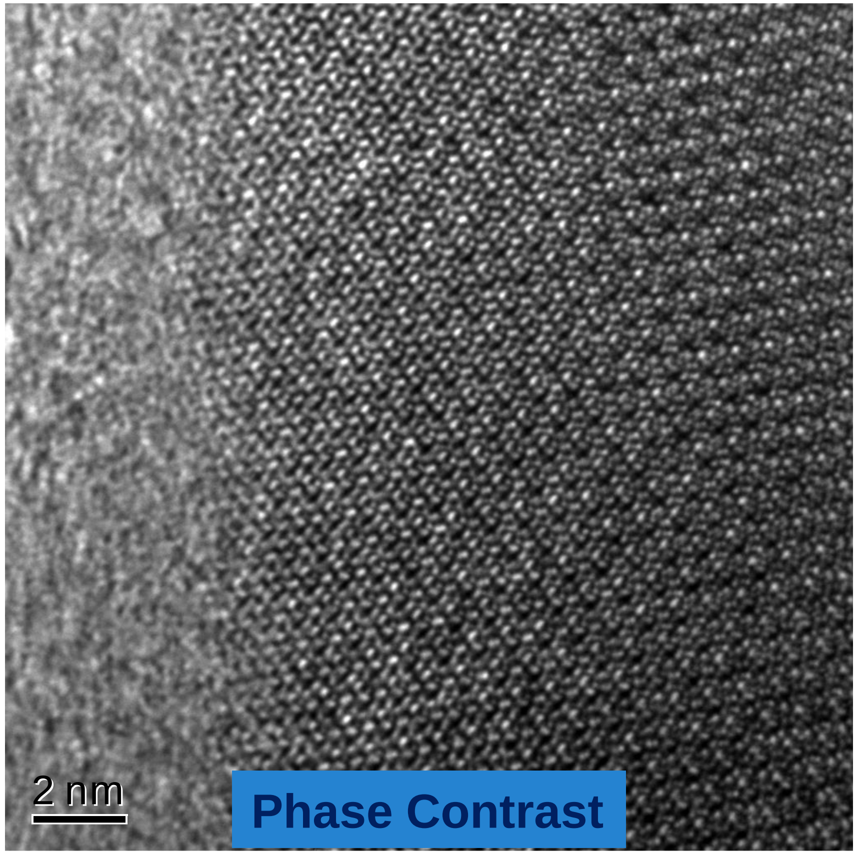
Usually, you can not say where the atom is, but you can tell the distance on atomic scale and crystal defects

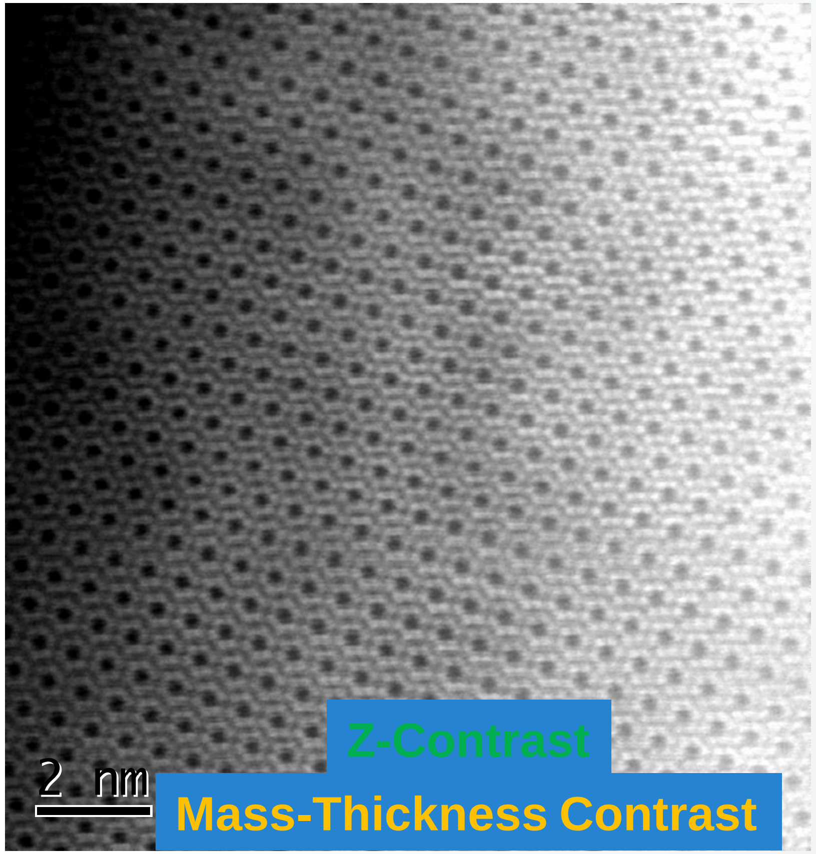


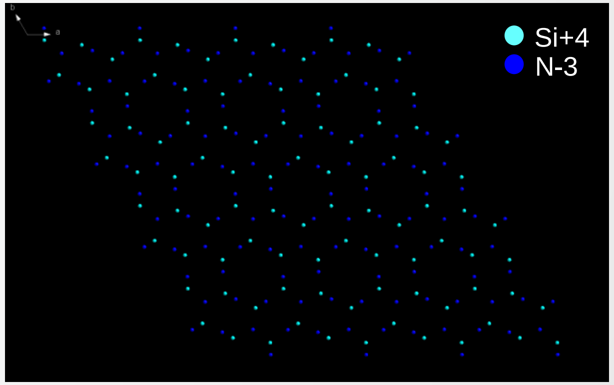



# High-resolution TEM

## HRTEM and HR-STEM Imaging




$Si_3N_4$ structure model

HRTEM and HR-STEM images showing the atomic structure of $Si_3Ni_4$

# High-resolution TEM

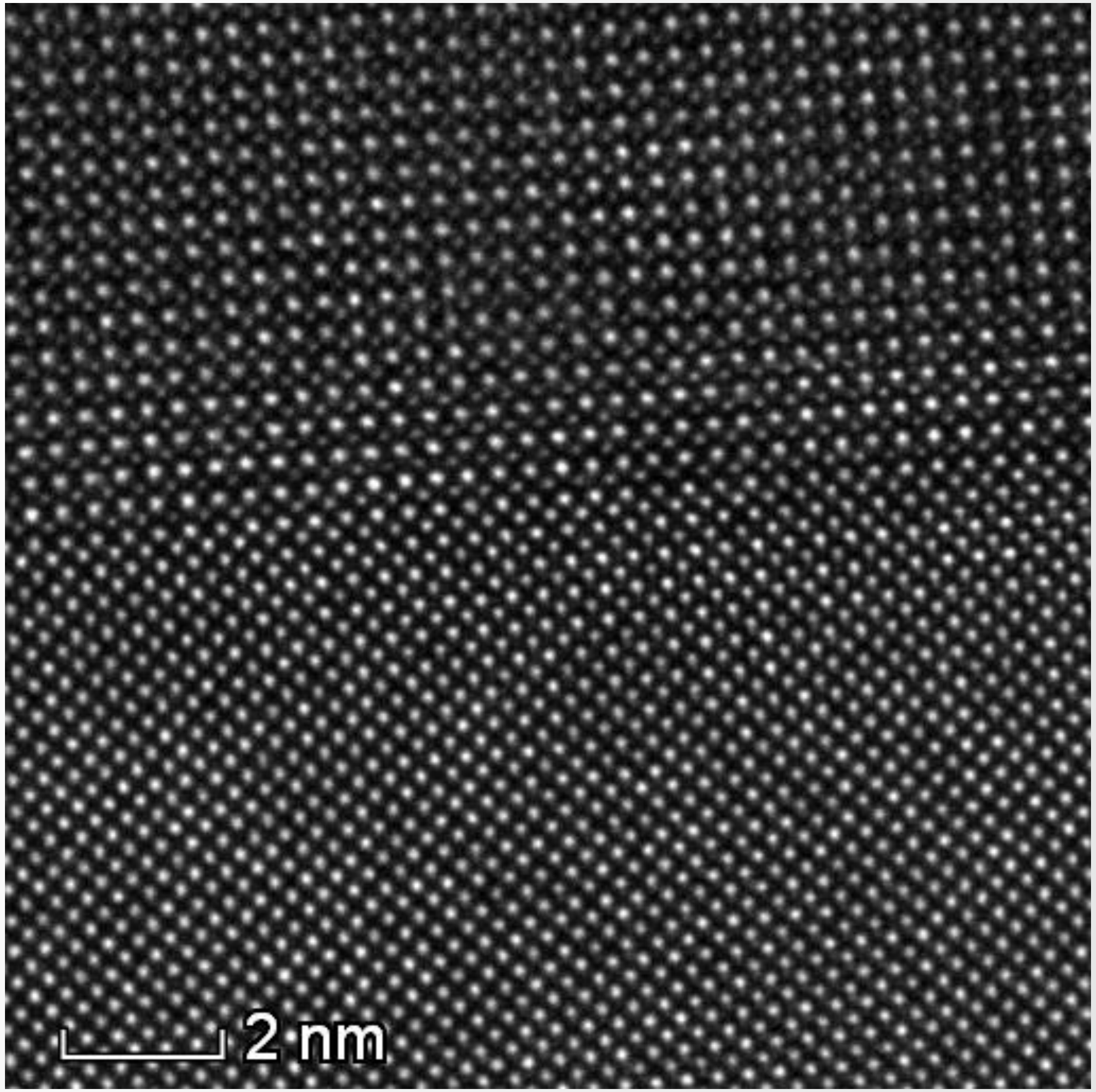
Thermo Fisher SCIENTIFIC

A good and Reliable HRTEM Image

- Sample:
  - Very thin ≤ 50 nm
  - Free from contamination
- Sample position:
  - Eucentric height
  - Oriented in one low zone axis ([001], [120])
- Instrument:
  - Stable enough, i.e. HT on not less than 2 hours
  - No or less sample drift
  - The beam is well aligned
  - Highest magnification as possible*
  - Adequate acquisition time
  - Focus series applied for acquisition

$SrTiO_3$

$La_xSr_{1-x}MnO_3$

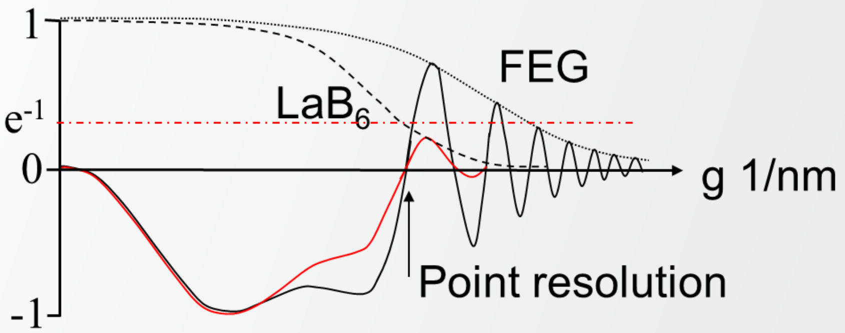


# High-resolution TEM

Thermo Fisher SCIENTIFIC

QUIZ

1. To produce High-resolution TEM images (samples perspective):
   - A. **Very thin**
   - B. **Crystalline**
   - C. **Oriented on a zone axis**
   - D. Cannot work with powder samples
2. Point Resolution depends on the following factors:
   - A. **Spherical Chromatic Aberration (Cs)**
   - B. Electron Sources
   - C. **Wavelength (λ)**
   - D. **Operation Voltage**



# High-resolution TEM

QUIZ

3. The information that we can get from HRTEM images:
   A. The exact atomic position
   B. The structure of the sample
   C. The type of atom
   **D. The distance on an atomic scale and crystal defects**

# Thank you