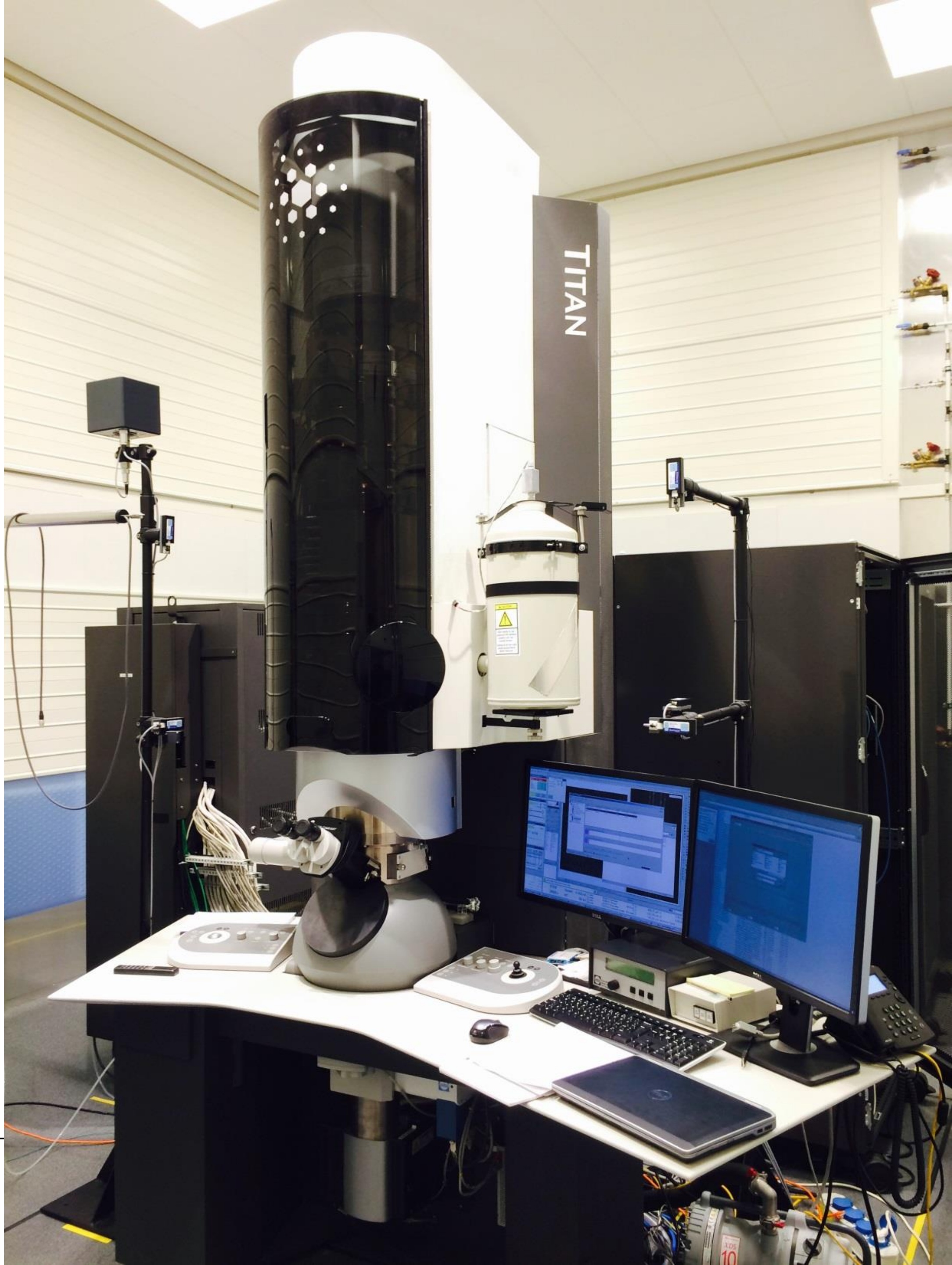
FYS 4340/FYS 9340

Diffraction Methods & Electron Microscopy

Lecture 2

Sandeep Gorantla

UiO University of Oslo
Centre for Materials Science and Nanotechnology (SMN)

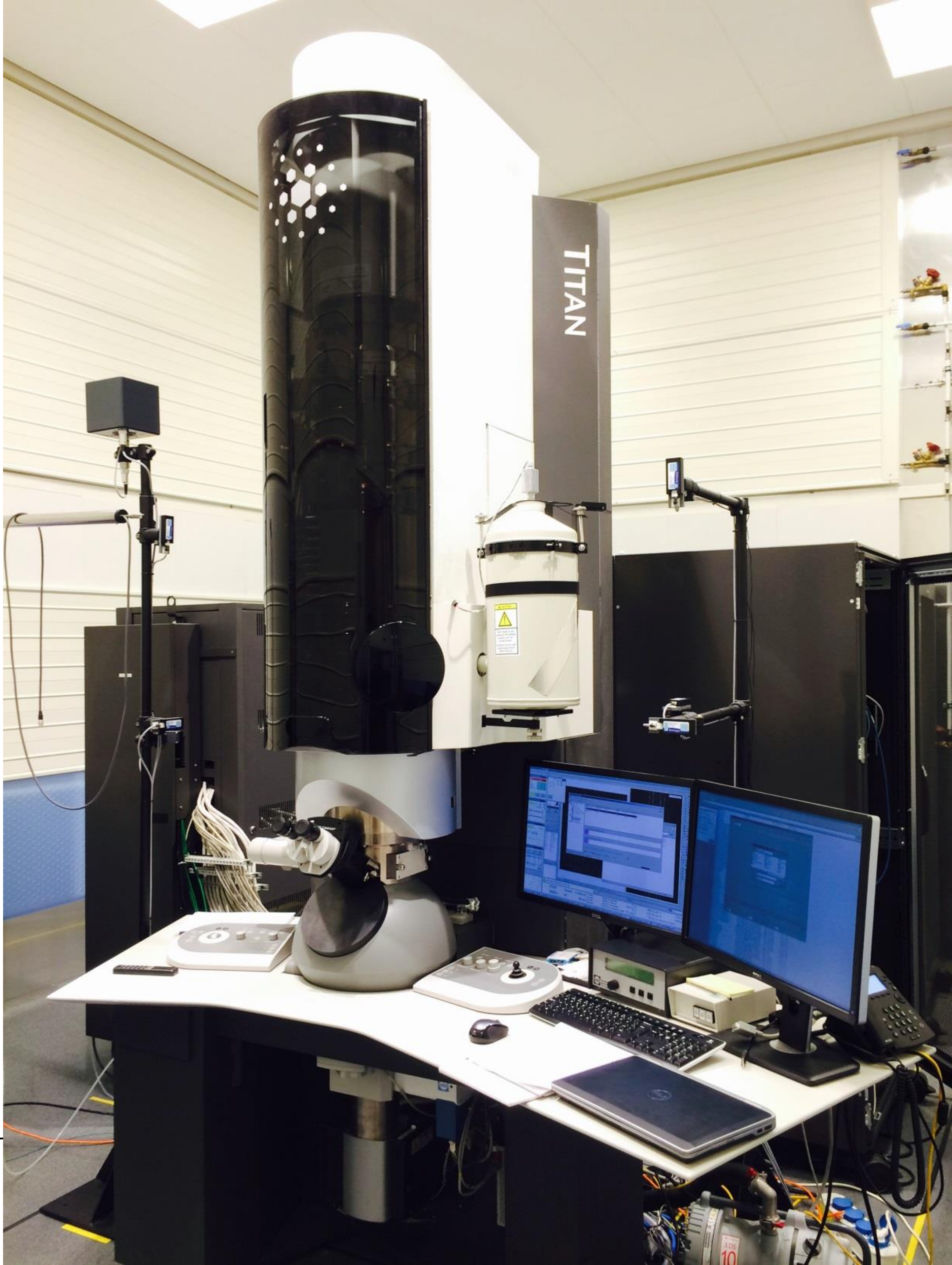


Transmission Electron Microscopy

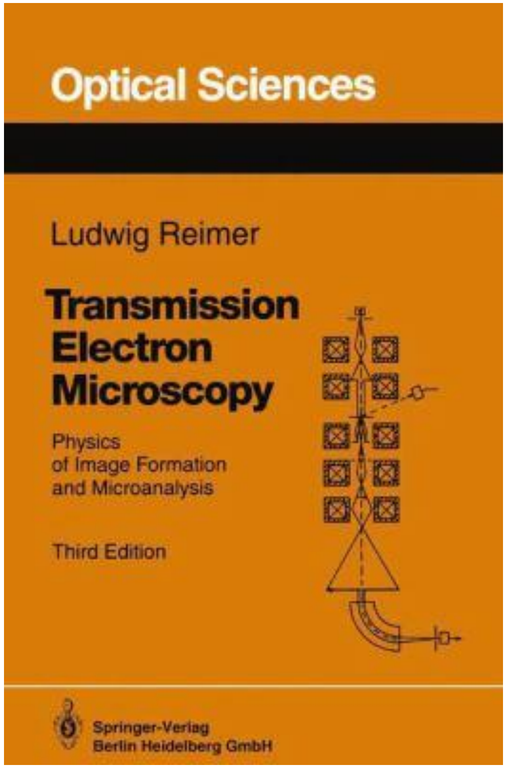
Introduction and Basics
Part- 1

Sandeep Gorantla

UiO : University of Oslo
Centre for Materials Science and Nanotechnology (SMN)



Learning more about TEM!



Optical Sciences

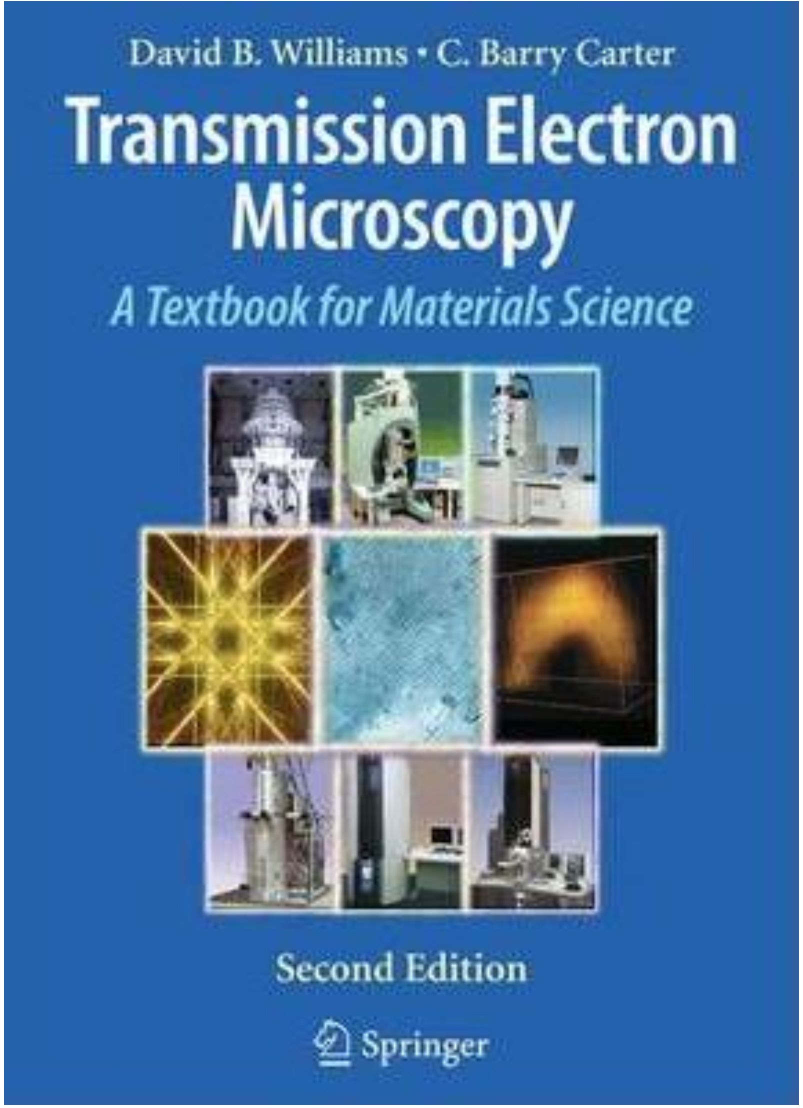
Ludwig Reimer

Transmission Electron Microscopy

Physics
of Image Formation
and Microanalysis

Third Edition

Springer-Verlag
Berlin Heidelberg GmbH



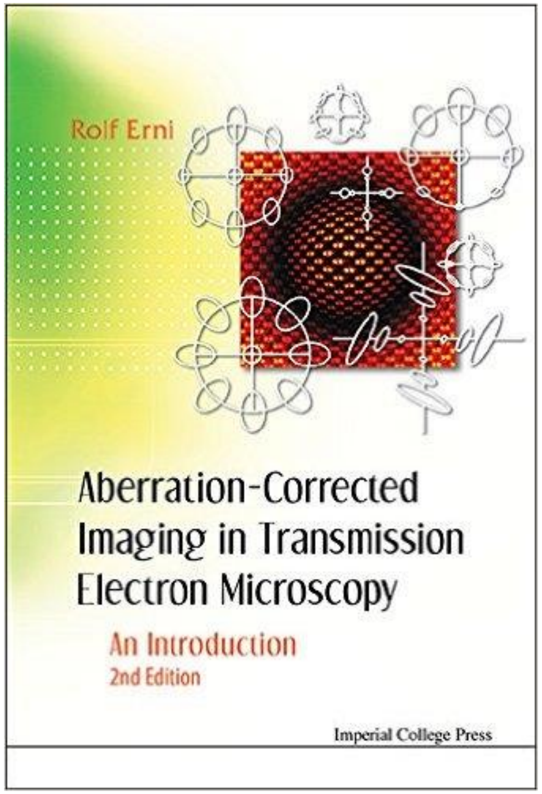
David B. Williams • C. Barry Carter

Transmission Electron Microscopy

A Textbook for Materials Science

Second Edition

Springer



Rolf Erni

Aberration-Corrected Imaging in Transmission Electron Microscopy

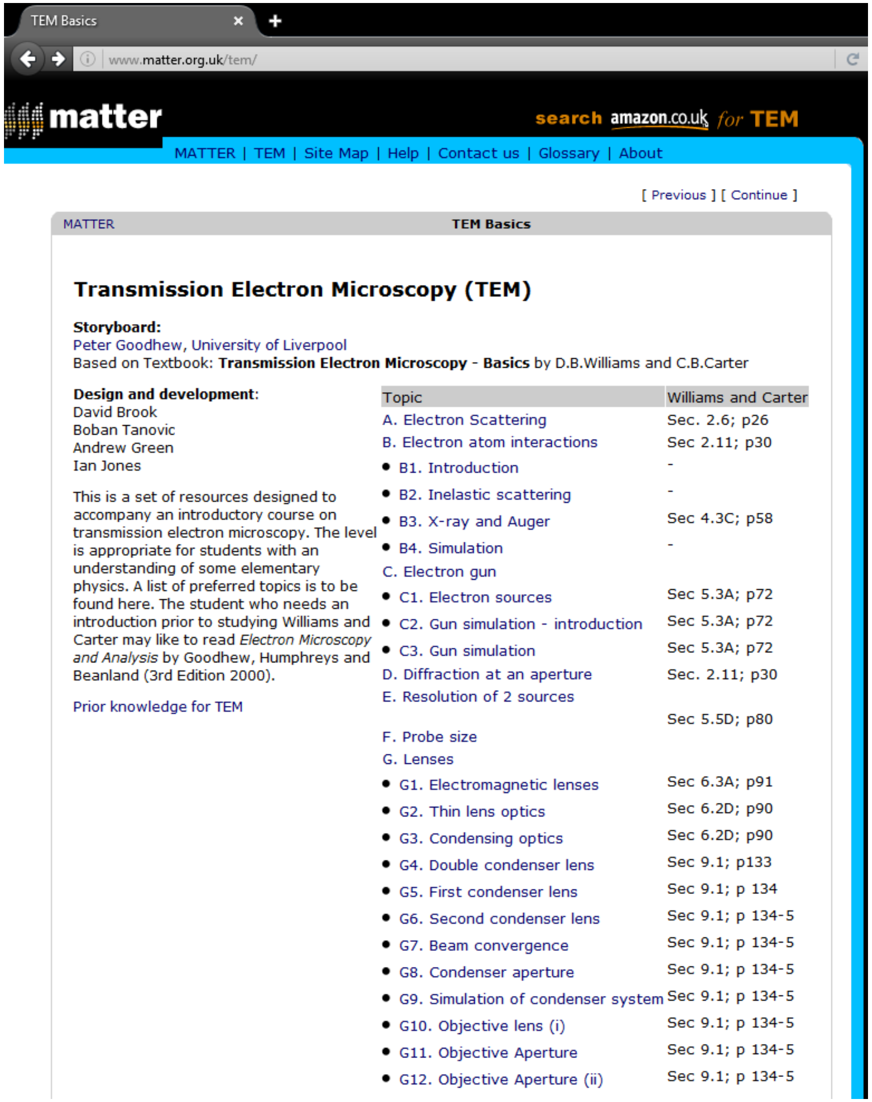
An Introduction

2nd Edition

Imperial College Press

Courtesy: WWW.amazon.com

Learning more about TEM!



Transmission Electron Microscopy (TEM)

Storyboard:
Peter Goodhew, University of Liverpool
Based on Textbook: **Transmission Electron Microscopy - Basics** by D.B.Williams and C.B.Carter

Design and development:
David Brook
Boban Tanovic
Andrew Green
Ian Jones

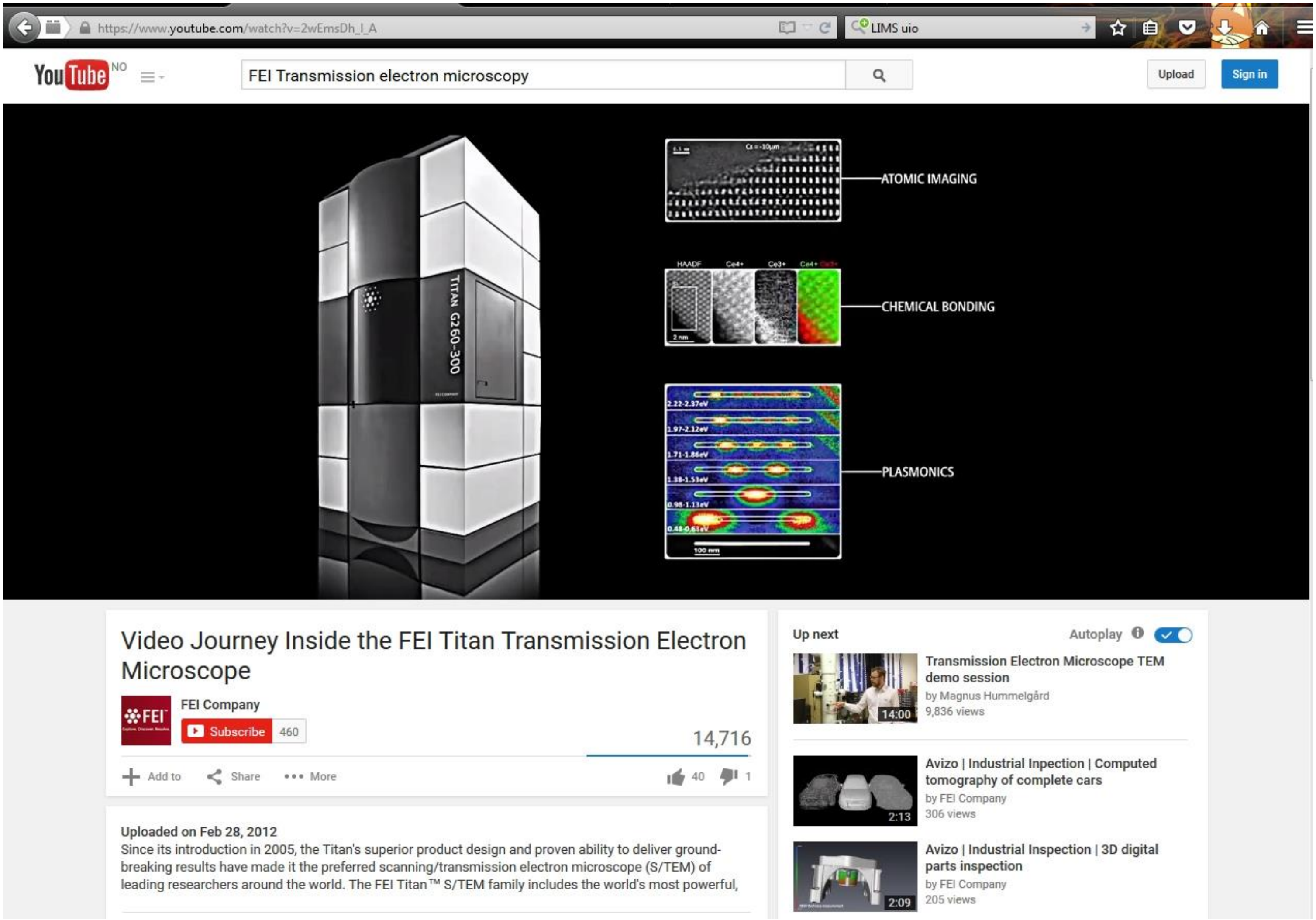
This is a set of resources designed to accompany an introductory course on transmission electron microscopy. The level is appropriate for students with an understanding of some elementary physics. A list of preferred topics is to be found here. The student who needs an introduction prior to studying Williams and Carter may like to read *Electron Microscopy and Analysis* by Goodhew, Humphreys and Beanland (3rd Edition 2000).

Prior knowledge for TEM

Topic	Williams and Carter
A. Electron Scattering	Sec. 2.6; p26
B. Electron atom interactions	Sec 2.11; p30
• B1. Introduction	-
• B2. Inelastic scattering	-
• B3. X-ray and Auger	Sec 4.3C; p58
• B4. Simulation	-
C. Electron gun	
• C1. Electron sources	Sec 5.3A; p72
• C2. Gun simulation - introduction	Sec 5.3A; p72
• C3. Gun simulation	Sec 5.3A; p72
D. Diffraction at an aperture	Sec. 2.11; p30
E. Resolution of 2 sources	Sec 5.5D; p80
F. Probe size	
G. Lenses	
• G1. Electromagnetic lenses	Sec 6.3A; p91
• G2. Thin lens optics	Sec 6.2D; p90
• G3. Condensing optics	Sec 6.2D; p90
• G4. Double condenser lens	Sec 9.1; p133
• G5. First condenser lens	Sec 9.1; p 134
• G6. Second condenser lens	Sec 9.1; p 134-5
• G7. Beam convergence	Sec 9.1; p 134-5
• G8. Condenser aperture	Sec 9.1; p 134-5
• G9. Simulation of condenser system	Sec 9.1; p 134-5
• G10. Objective lens (i)	Sec 9.1; p 134-5
• G11. Objective Aperture	Sec 9.1; p 134-5
• G12. Objective Aperture (ii)	Sec 9.1; p 134-5

http://www.matter.org.uk/tem/

Learning more about TEM!



FEI Transmission electron microscopy

Video Journey Inside the FEI Titan Transmission Electron Microscope

FEI Company

14,716

Uploaded on Feb 28, 2012

Since its introduction in 2005, the Titan's superior product design and proven ability to deliver ground-breaking results have made it the preferred scanning/transmission electron microscope (S/TEM) of leading researchers around the world. The FEI Titan™ S/TEM family includes the world's most powerful,

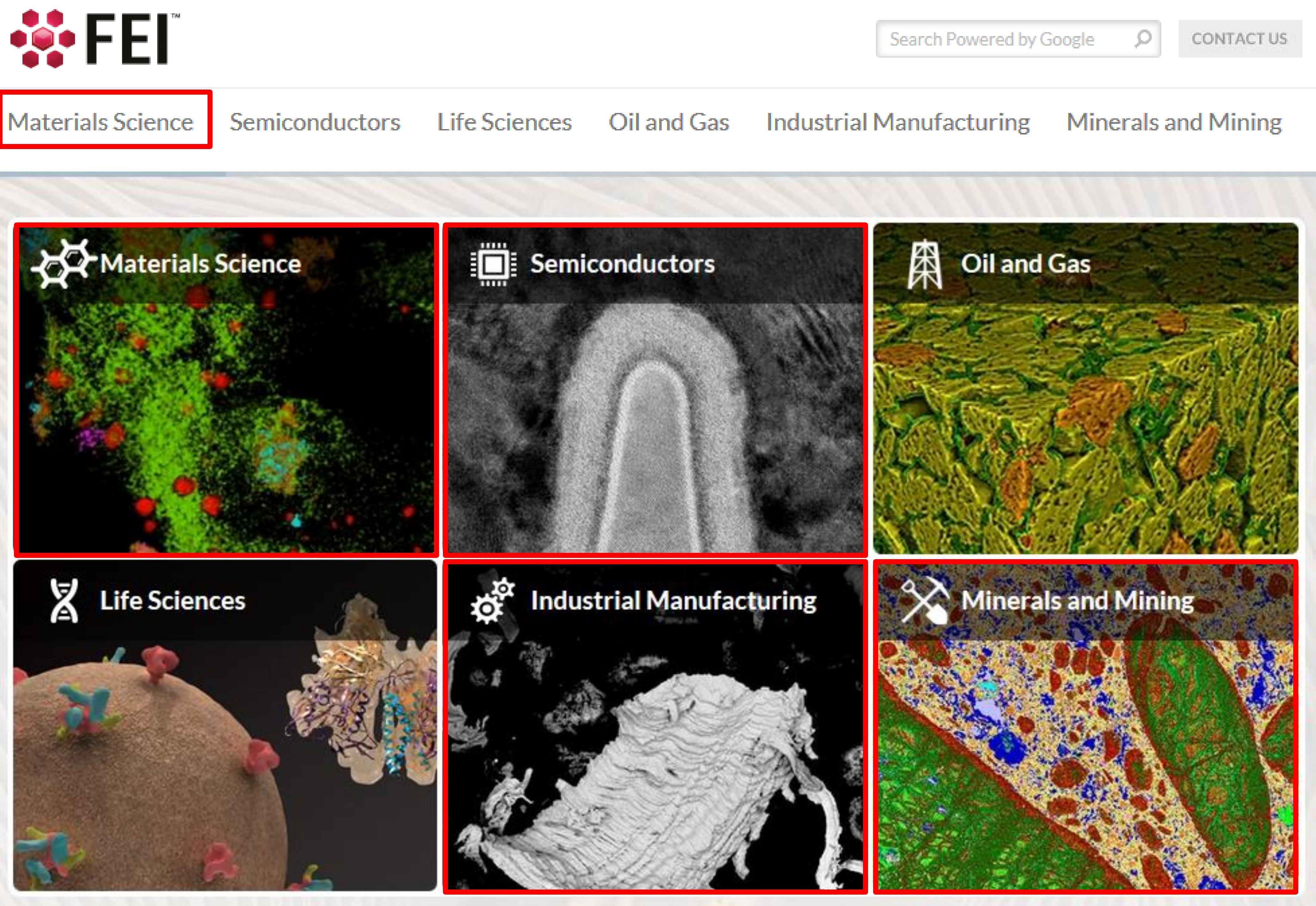
Up next

Transmission Electron Microscope TEM demo session
by Magnus Hummelgård
9,836 views

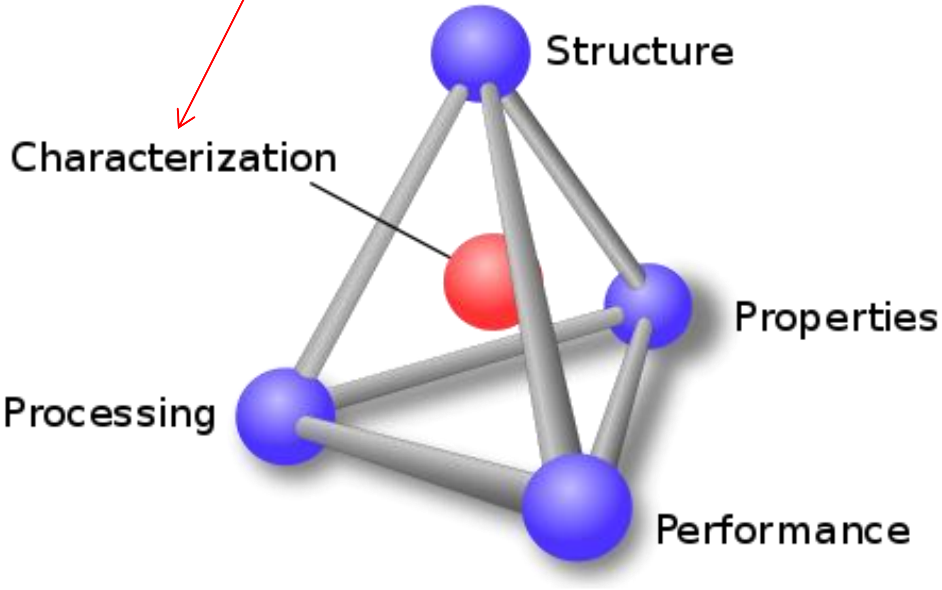
Avizo | Industrial Inpection | Computed tomography of complete cars
by FEI Company
306 views

Avizo | Industrial Inspection | 3D digital parts inspection
by FEI Company
205 views

Why learn about Transmission Electron Microscopy (TEM)?


FEI
Search Powered by Google
CONTACT US
Materials Science
Semiconductors
Life Sciences
Oil and Gas
Industrial Manufacturing
Minerals and Mining
Materials Science
Semiconductors
Oil and Gas
Life Sciences
Industrial Manufacturing
Minerals and Mining

Role of TEM in Materials Science Research and Development

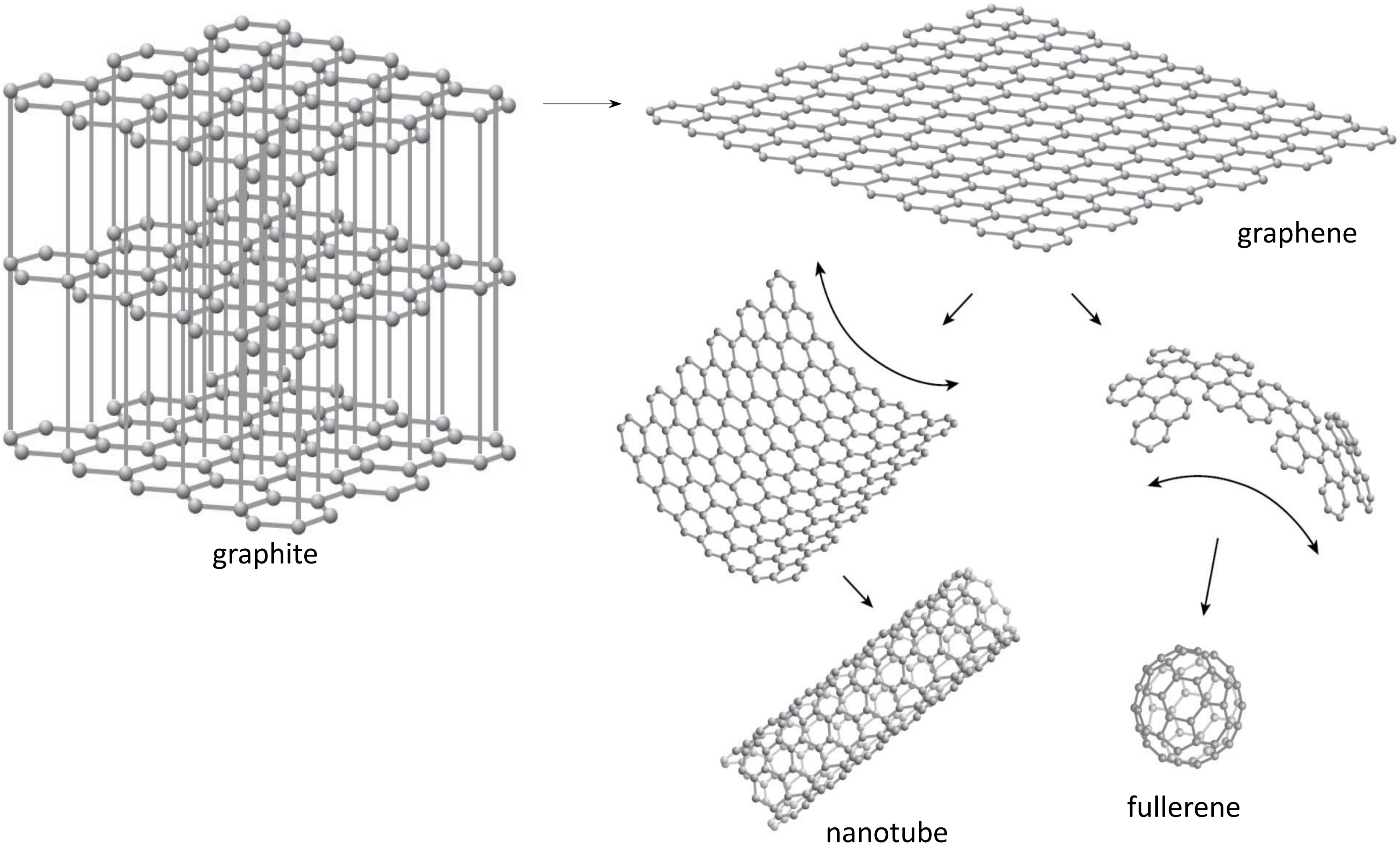


Materials Science Paradigm

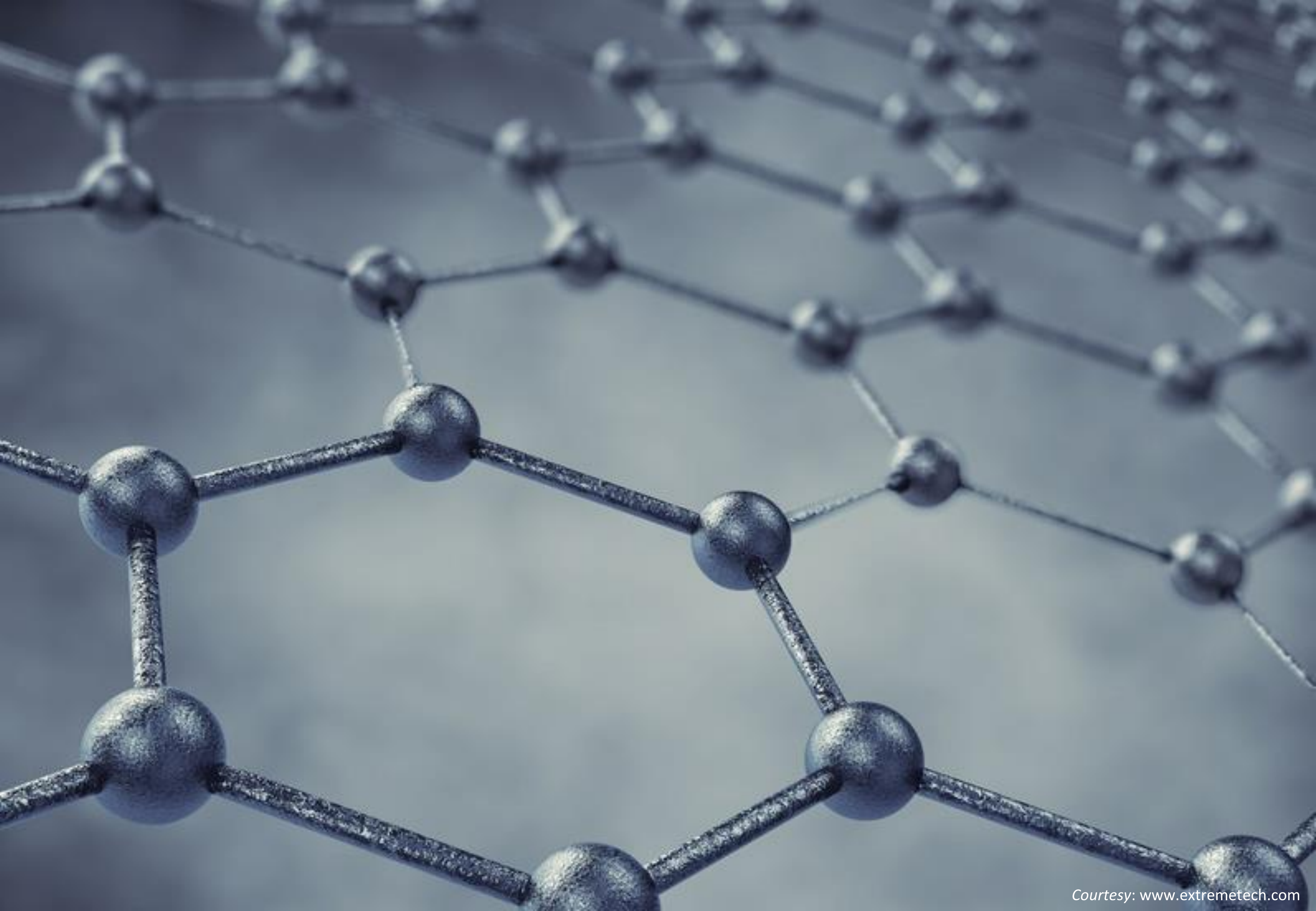
Solving Materials Science problems/mysteries by probing analytically and understanding structure-property relationships at atomic scale level

Courtesy: www.wikipedia.com

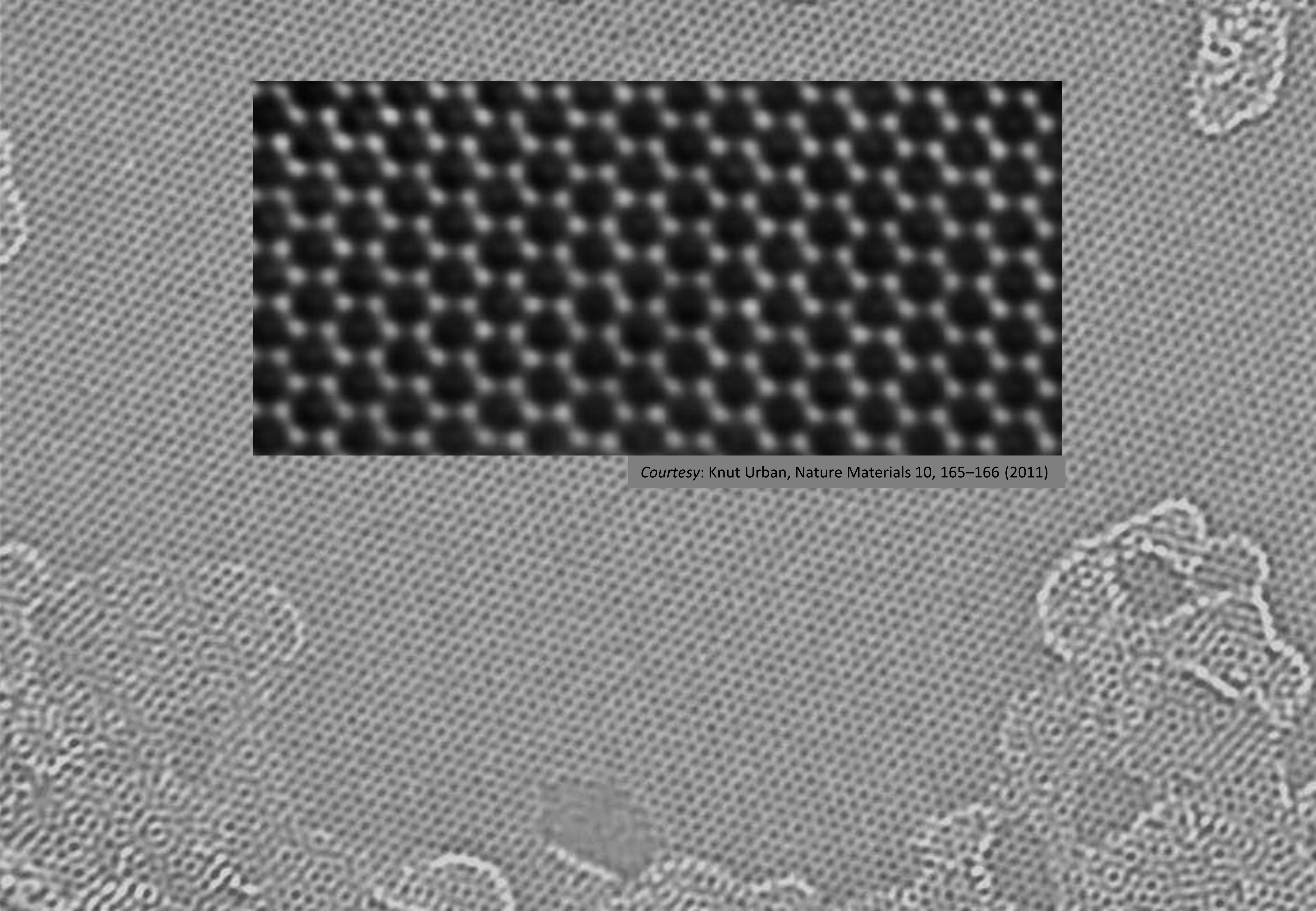
Allotropes of carbon



(Courtesy: The Royal Swedish Academy of Sciences)



Courtesy: www.extremetech.com



Courtesy: Knut Urban, Nature Materials 10, 165–166 (2011)

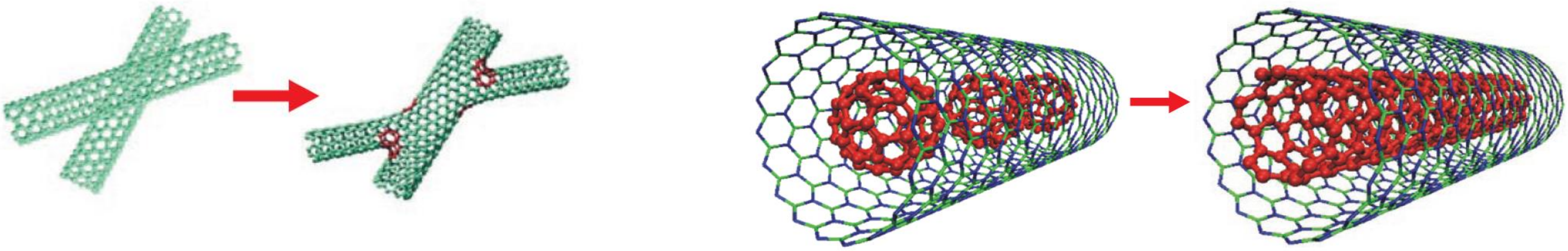
1D nanomaterials modification in TEM

- Irradiation of solids with energetic particles usually leads to damage

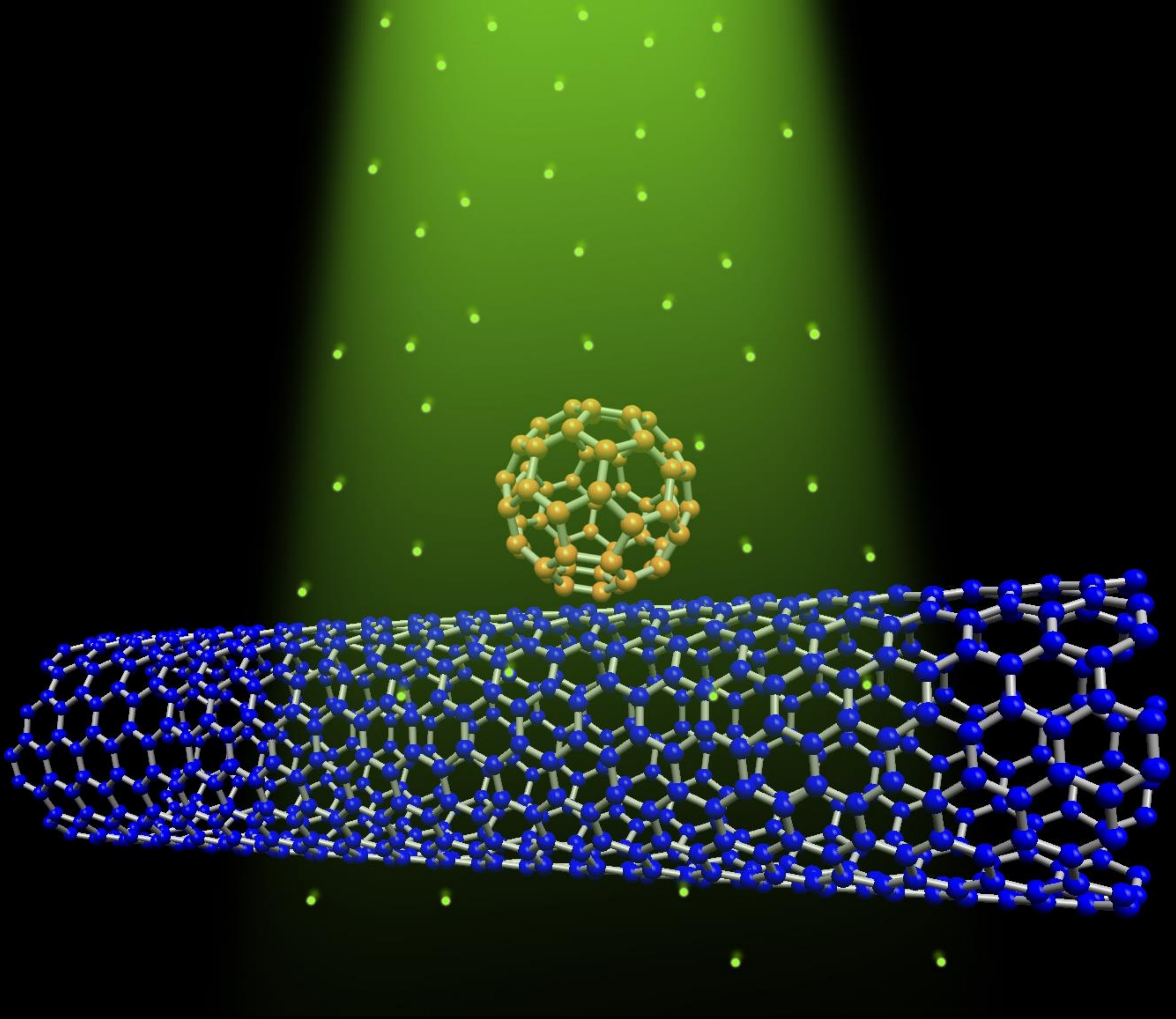
- However, in the case of carbon nanostructures, electron irradiation was observed to have some beneficial effects

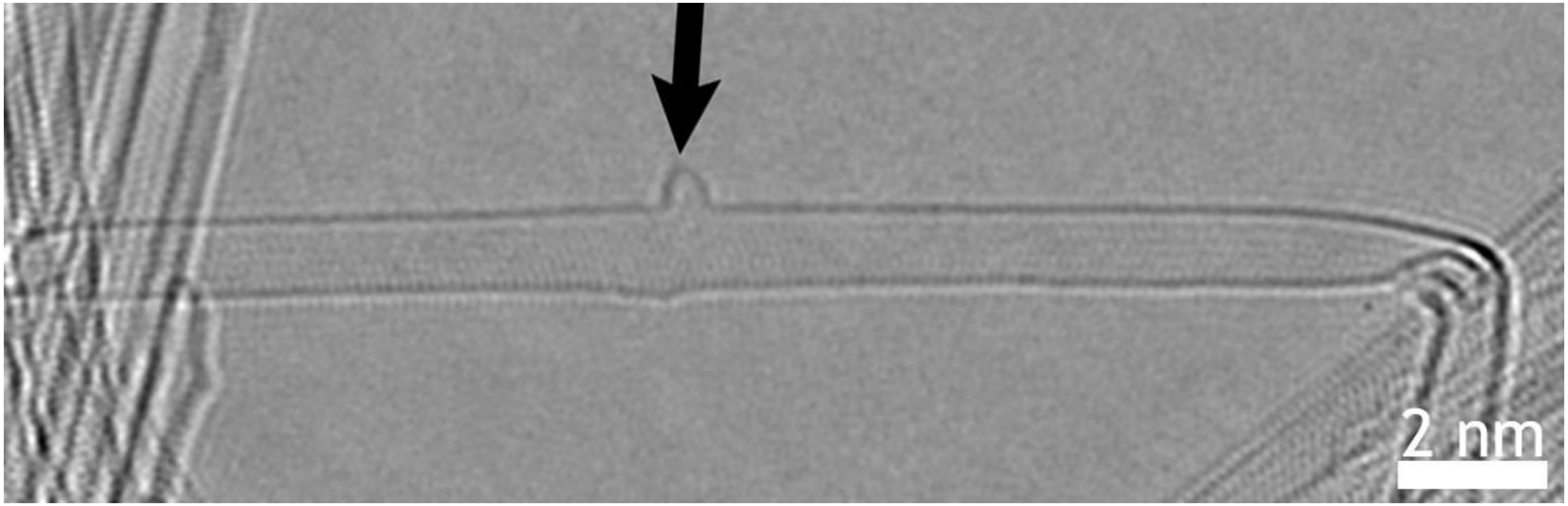
  (a) Irradiation – mediated engineering

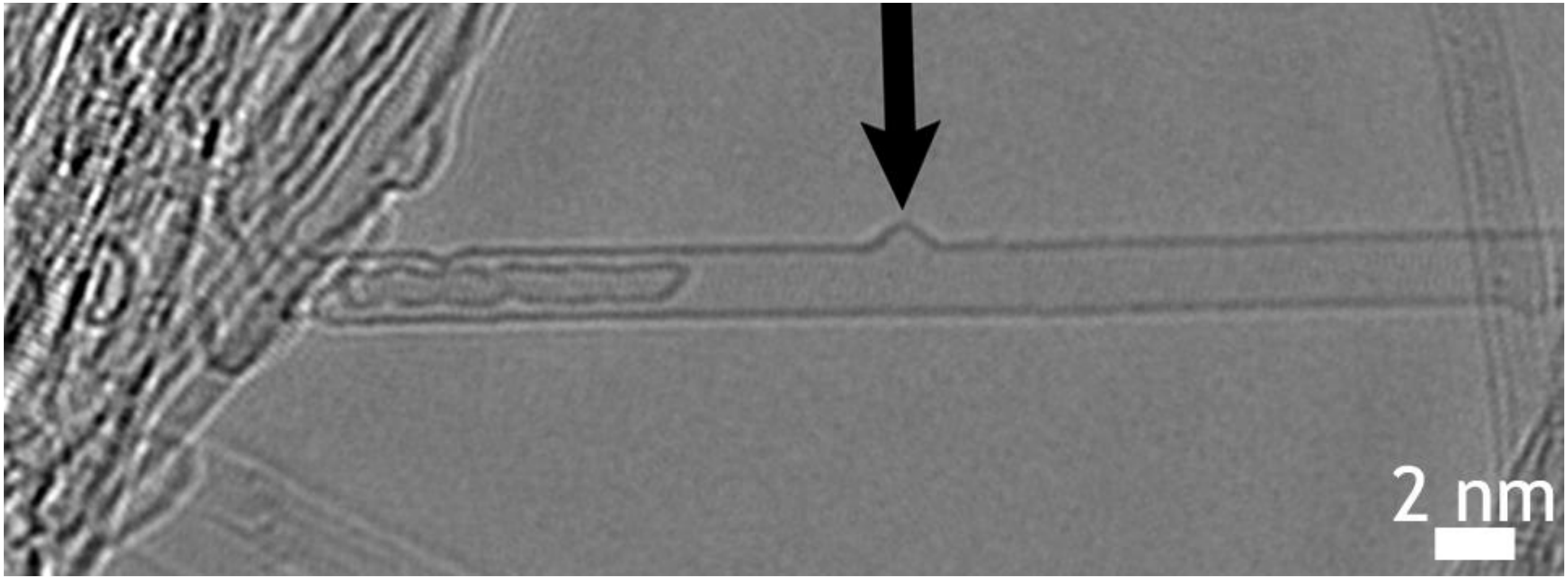
  (b) self-assembly or self-organization



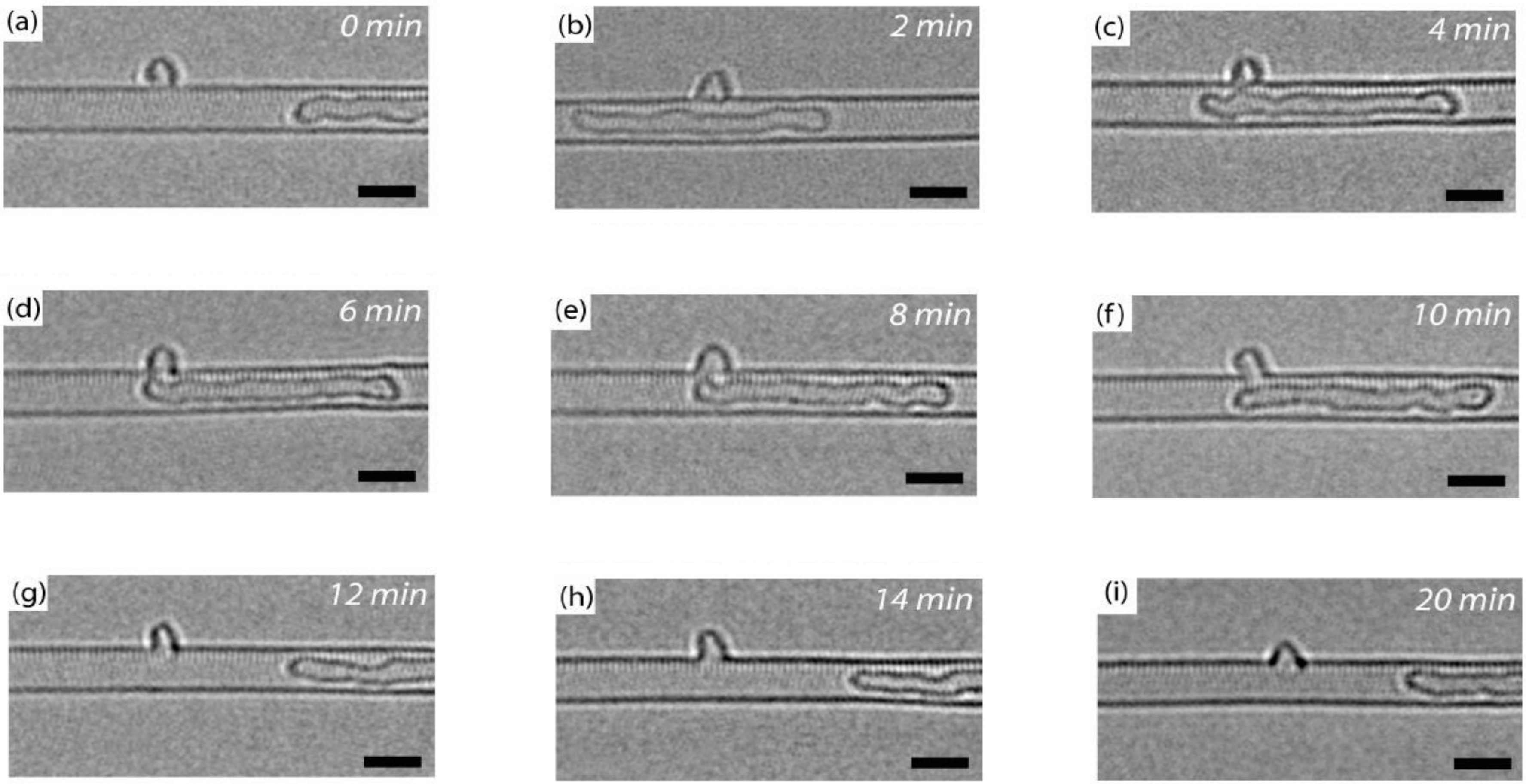
Courtesy: Krasheninnikov, A. V. et al., Nature Mater., 6, 723 (2007)




2 nm


2 nm

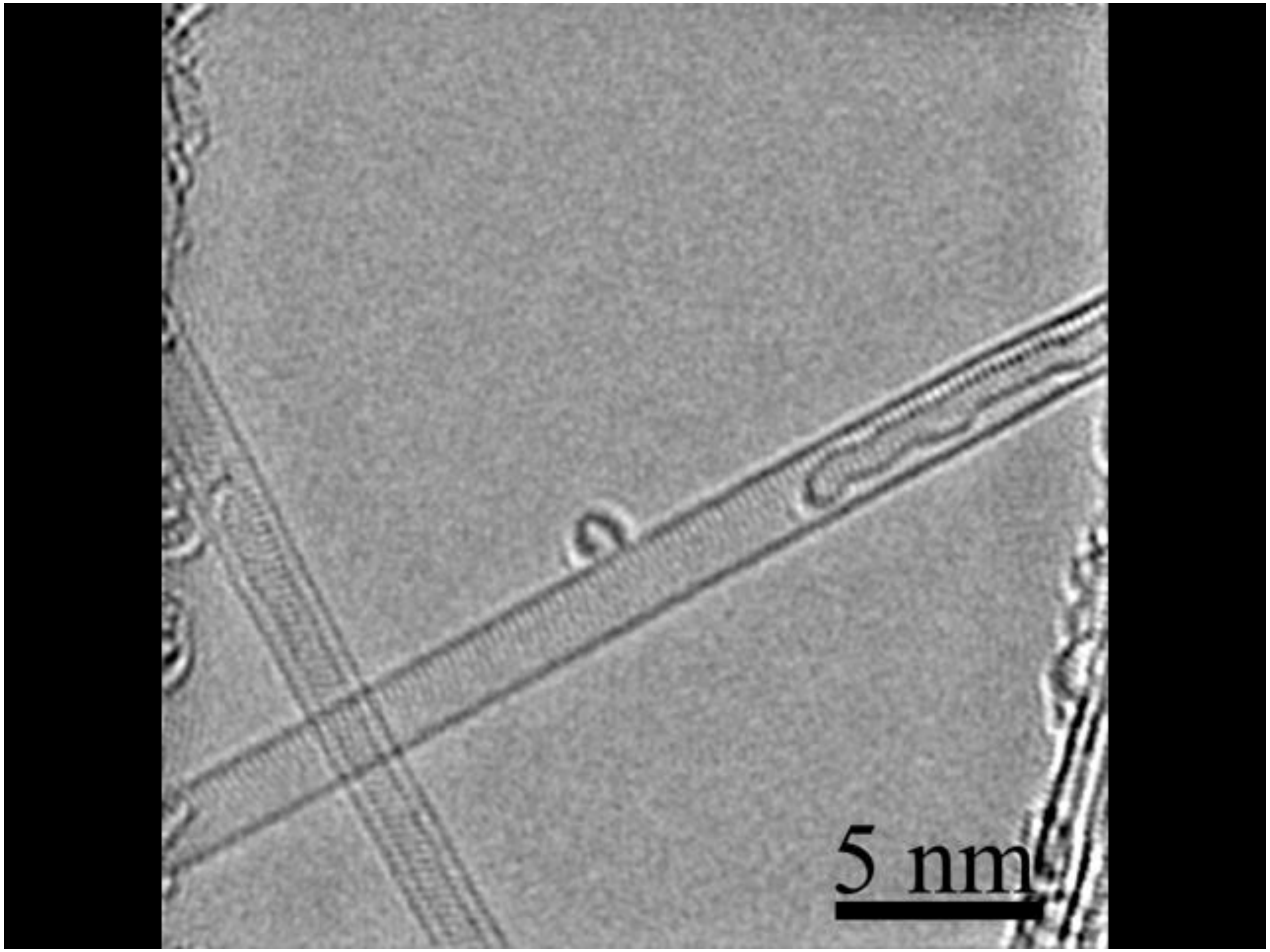
Interface: defects on outer-wall of a nanotube and fullerene



Courtesy: Gorantla, S. et al., Nanoscale, 2, 2077 (2010)

Interface: defects on outer-wall of a nanotube and fullerene

Nanohump formation (Covalent interactions of fullerene fusion)



Movie Settings:

- Frame speed: 0.6 s
- Total Frames: 48

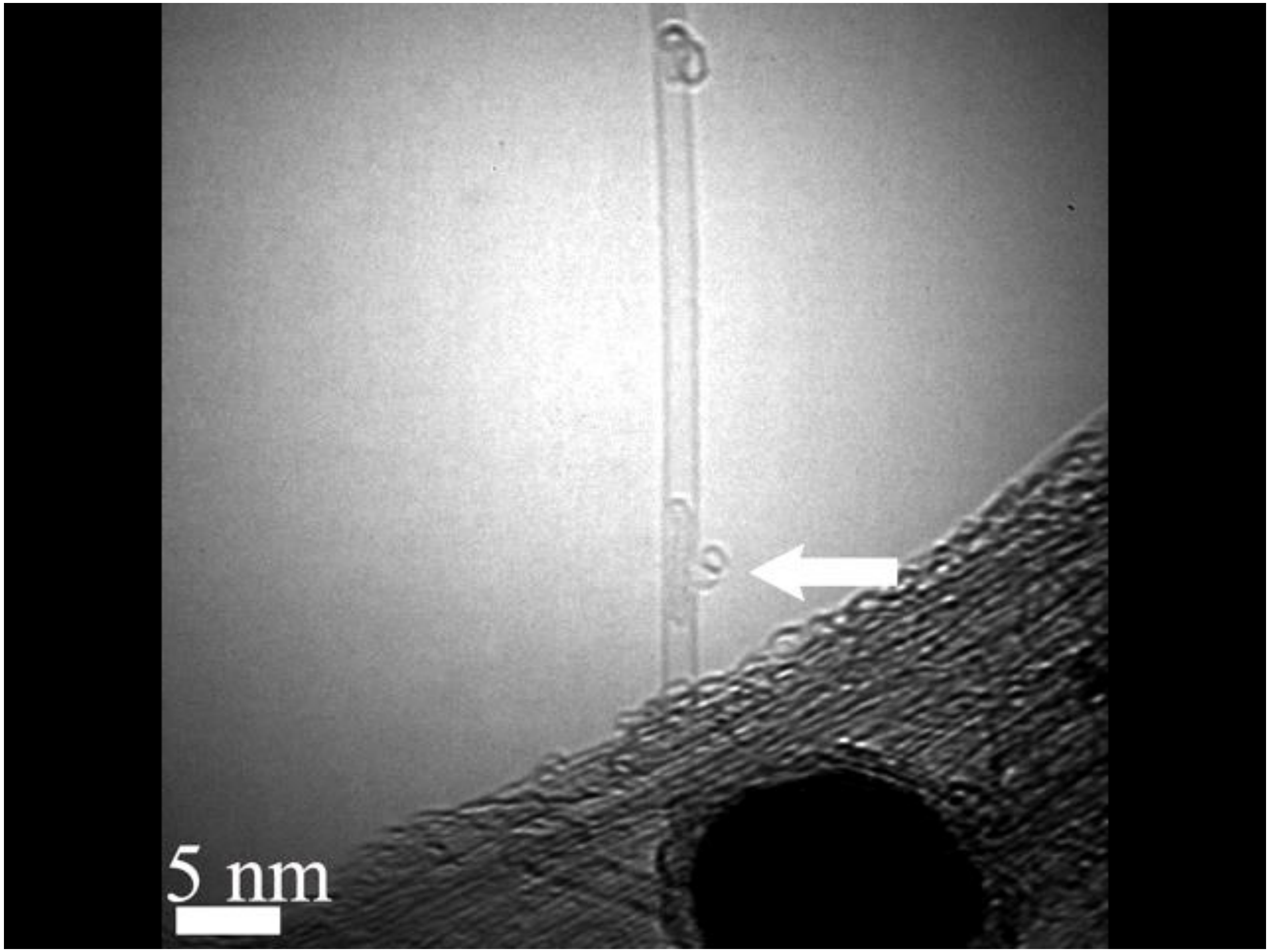
Experimental conditions:

- Acquisition time: 1 s
- Time gap between individual frames: 1s - 30s
- Total time: **14 mins**

Courtesy: Gorantla, S. et al., Nanoscale, 2, 2077 (2010)

Interface: defects on the outer-wall of a SWCNT and fullerene

Fullerene fusion with a nanohump *(Covalent interactions of fullerene fusion)*



Movie Settings:

- Frame speed: 0.6 s
- Total Frames: 48

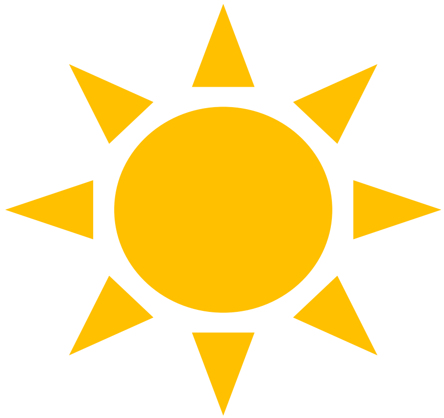
Experimental conditions:

- Acquisition time: 1 s
- Time gap between individual frames: 1 s

Courtesy: Gorantla, S. et al., Nanoscale, 2, 2077 (2010)

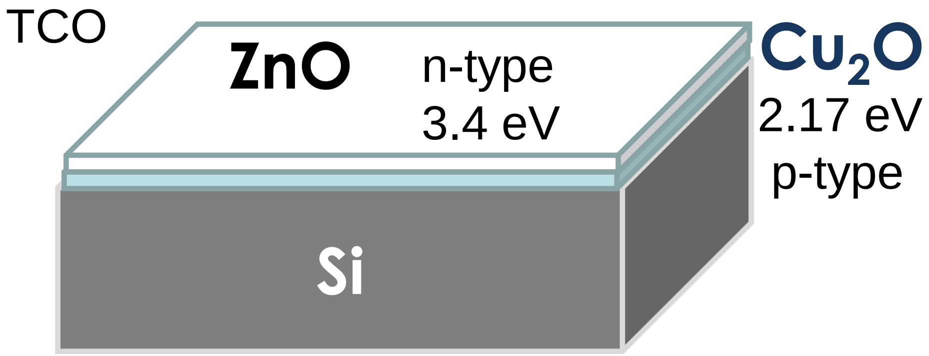
HETEROSOLAR PROJECT

The aim of the work



Develop new solar cell devices base on ZnO/Cu_2O heterojunctions coupled with convetional Si based solar cells

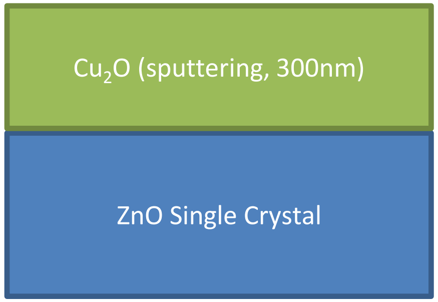
Properties determined by the structures, faults and interfaces.



* Theoretical eficiency ~20 %

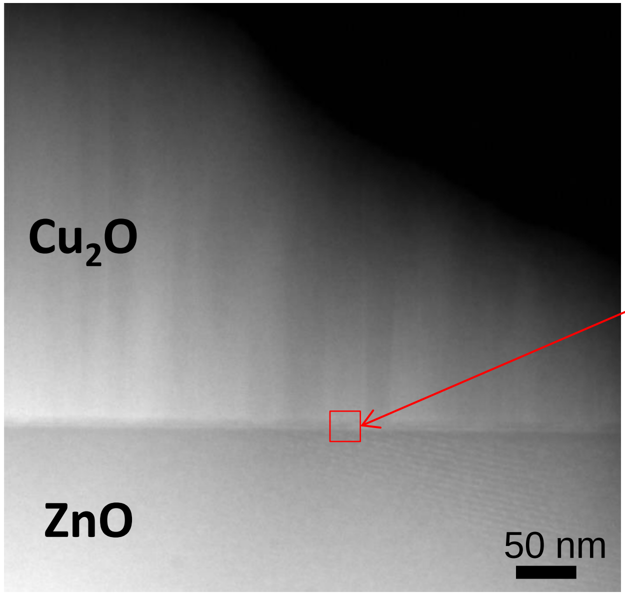
* Highest exp. eficiency 1-4 %

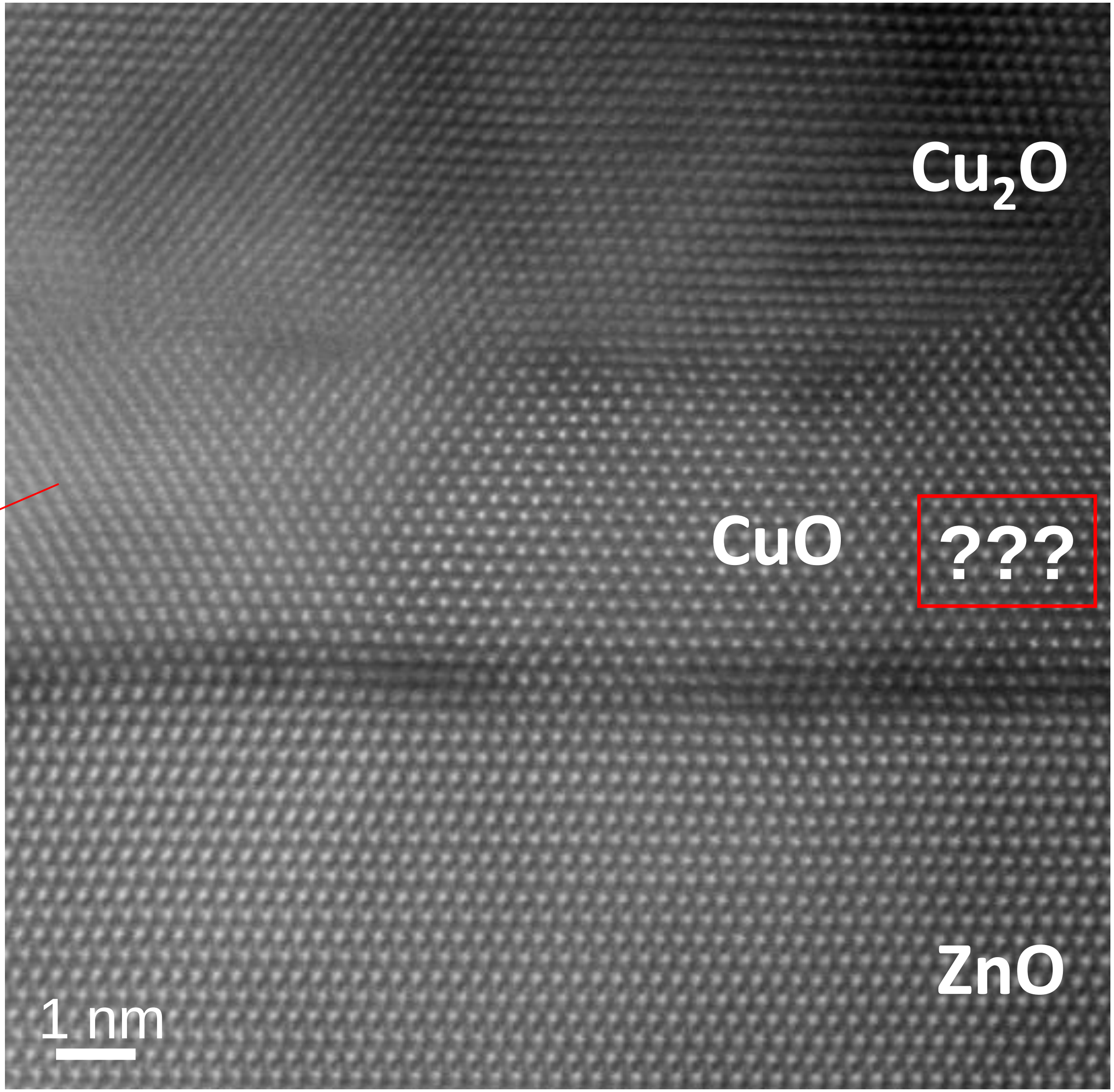
Sub project : (S)TEM to characterize the thin films and their interfaces.

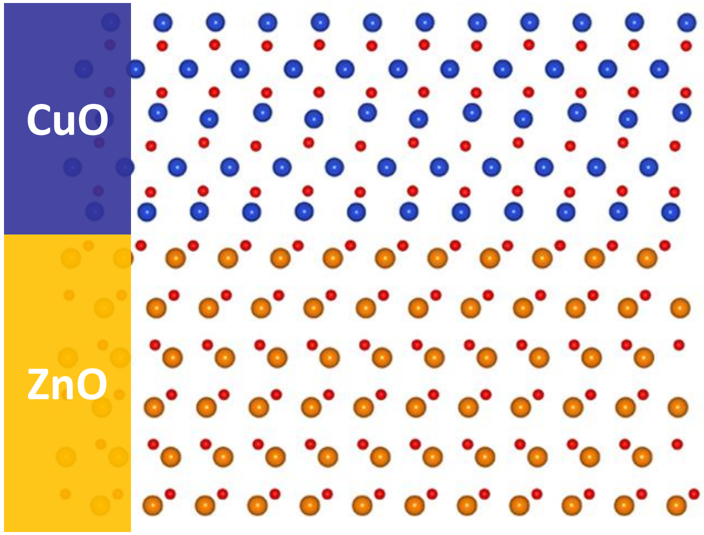


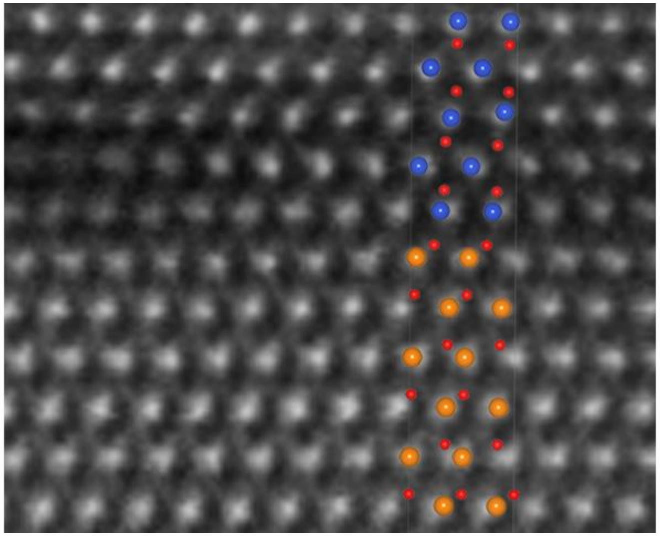
Cu$_2$O (sputtering, 300nm)

ZnO Single Crystal






CuO
ZnO



Transmission Electron Microscope

Brief History

Brief History: The first electron microscope

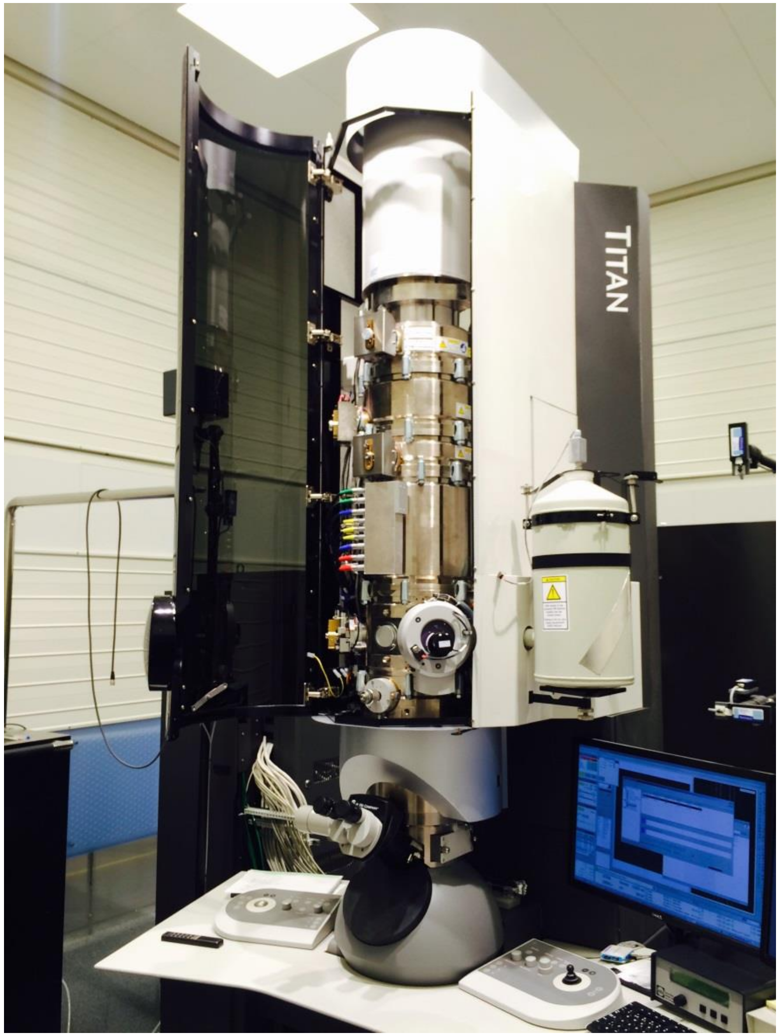
Ernst Ruska:
Nobel Prize in physics 1986

Electron Microscope Deutsches Museum, 1933 model

- Knoll and Ruska, first TEM in 1931
- Idea and first images published in 1932
- By 1933 they had produced a TEM with two magnetic lenses which gave 12 000 times magnification.

Brief History: The state-of-art TEM

Electron Microscope Deutsches Museum, 1933 model



FEI Titan 60-300 TEM, NORTEM facility- UiO Installed: 2014

Brief History: The state-of-art TEM

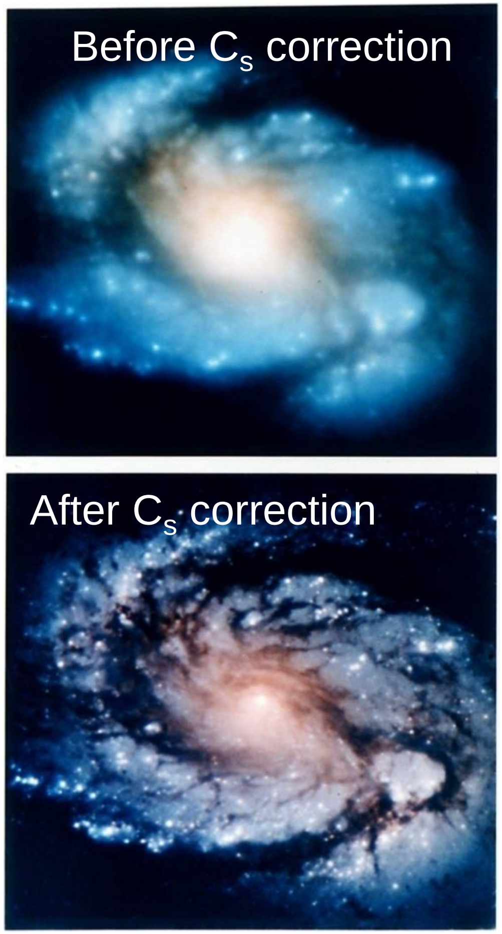
BIG LEAP: Introduction of **Lens Aberration Correctors** allowing atomic resolution at low accelerating voltages.

Resolution limit

Year	**Resolution**
1940s	**~10nm**
1950s	~0.5-2nm
1960s	0.3nm (transmission) ~15-20nm (scanning)
1970s	0.2nm (transmission) 7nm (standard scanning)
1980s	0.15nm (transmission) 5nm (scanning at 1kV)
1990s	0.1nm (transmission) 3nm (scanning at 1kV)
2000s	**<0.1 nm (Cs correctors)**

Typical TEM operating voltages
in Materials Science Research

300 kV
200 kV
80 kV
60 kV



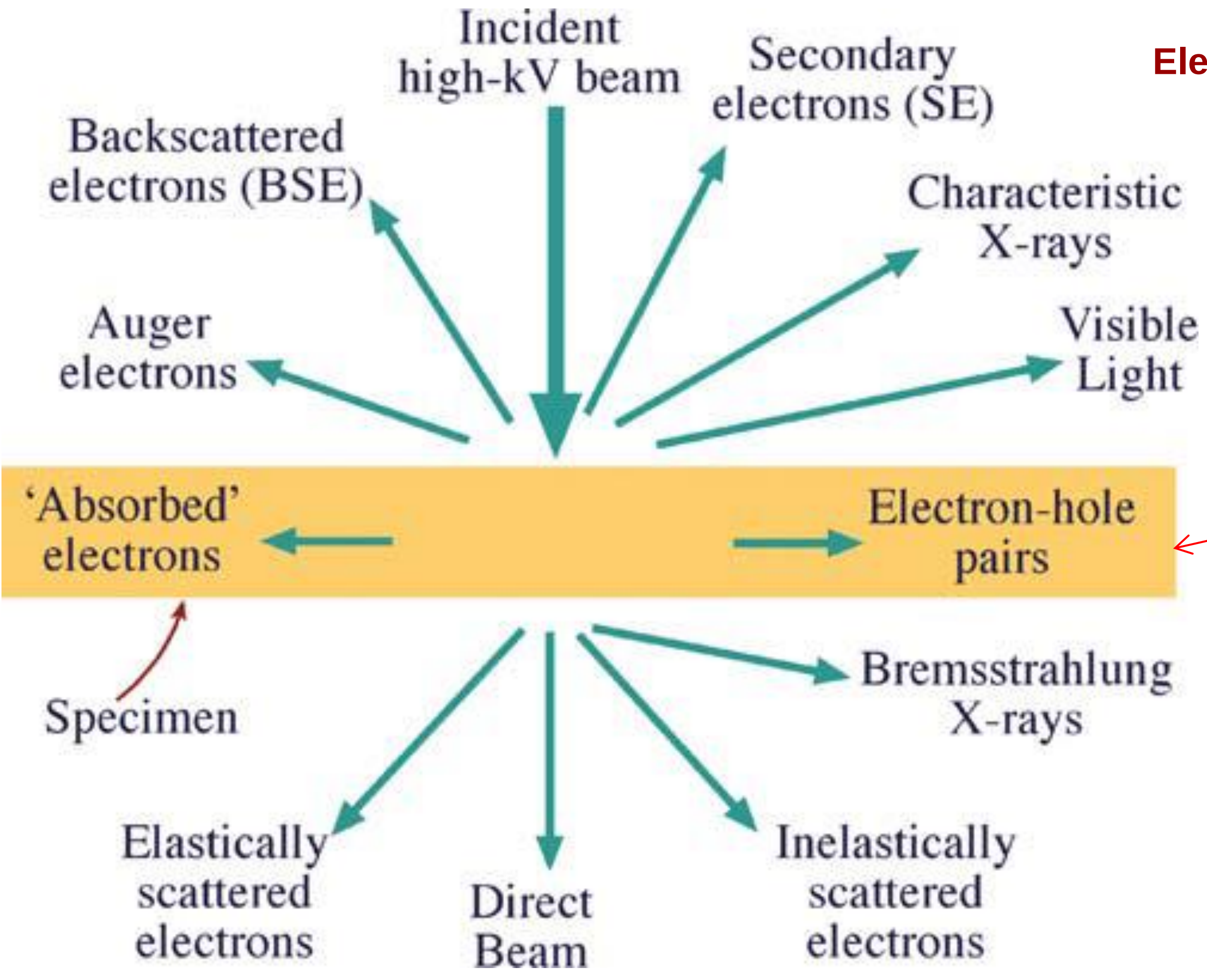
Core of the M100 galaxy seen through Hubble (source: NASA)

Courtesy: http://www.sfc.fr/Material/hrst.mit.edu/hrs/materials/public/ElecMicr.htm

Transmission Electron Microscope

Fundamentals

Electrons interaction with the specimen



Electrons have both wave and particle nature

Typical specimen thickness

~ 100 nm or less

Typical TEM operating voltages in Materials Science Research

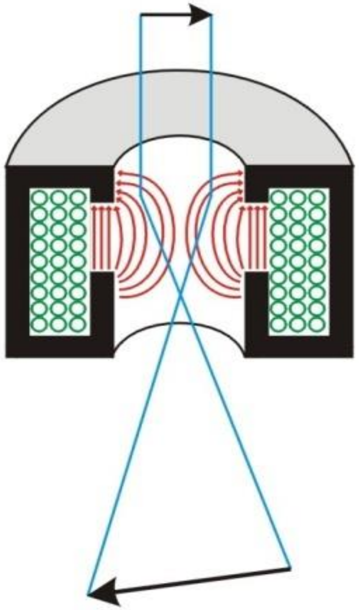
300 kV
200 kV
80 kV
60 kV

Courtesy: D.B. Williams & C.B. Carter, Transmission electron microscopy

Electron lenses

Any axially symmetrical electric or magnetic field have the properties of an ideal lens for paraxial rays of charged particles.

- Electrostatic **F= -eE**
  - Not used as imaging lenses, but are used in modern monochromators
- ElectroMagnetic **F= -e(v x B)**
  - Can be made more accurately
  - Shorter focal length



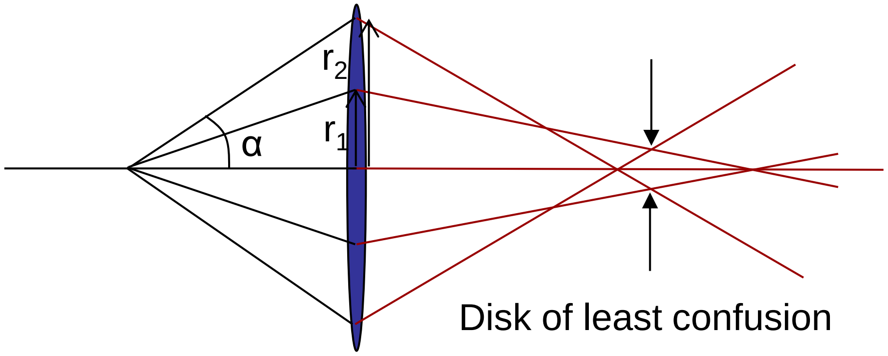
Courtesy: http://www.matter.org.uk/tem/lenses/electromagnetic_lenses.htm

TEM Lens Aberrations

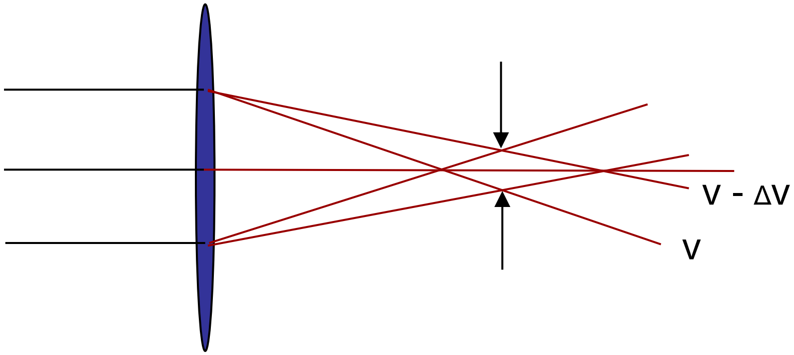
- **Spherical aberration coefficient**

  $d_s = 0.5MC_s\alpha^3$

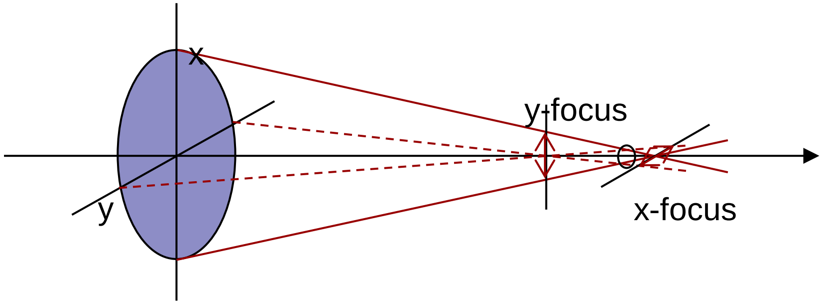
  M: magnification
  C_s :Spherical aberration coefficient
  α: angular aperture/
  angular deviation from optical axis



Spherical aberration



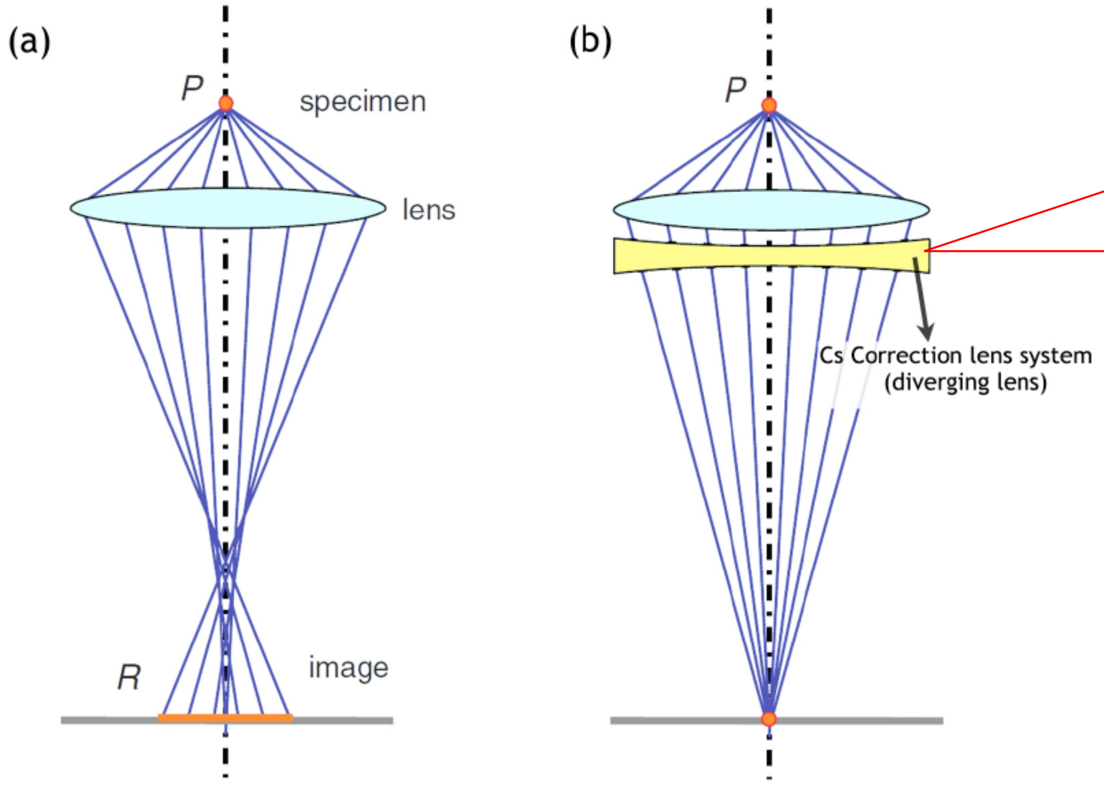
Chromatic aberration



Astigmatism

TEM Lens Aberrations

Schematic of spherical aberration correction



Probe-forming

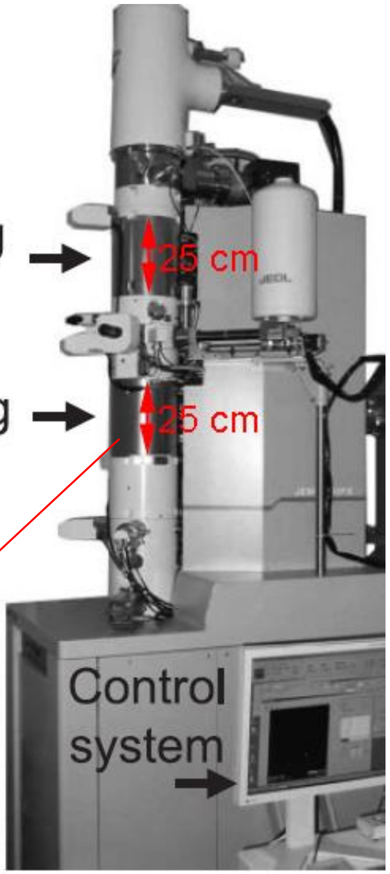
Cs corrector

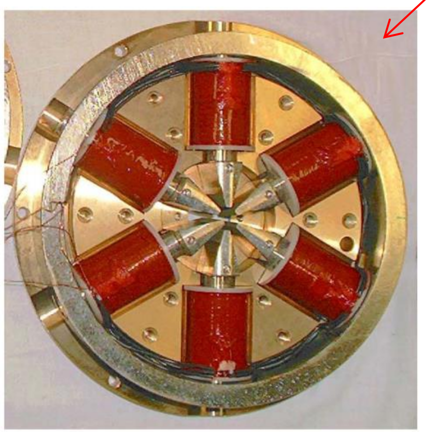
(CEOS GmbH)

Image-forming

Cs corrector

(CEOS GmbH)





Courtesy: Knut W. Urban, Science 321, 506, 2008; CEOS gmbh, Germany; www.globalsino.com

TEM Lens Aberrations


(*Courtesy*: NASA)

Why we need an aberration-corrected TEM at 80kV???

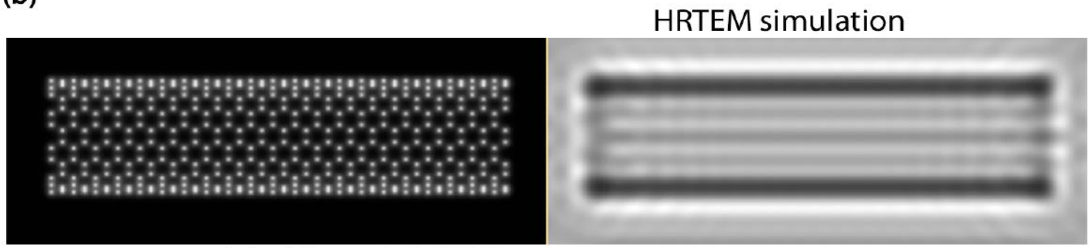
-Correcting aberrations improves the TEM resolution at 80 kV

Uncorrected 80 kV → ~ 0.3 nm

Corrected 80 kV → ~ 0.14 nm

- Improved resolution enables the possibility of imaging carbon nanostructures at atomic level

(b)

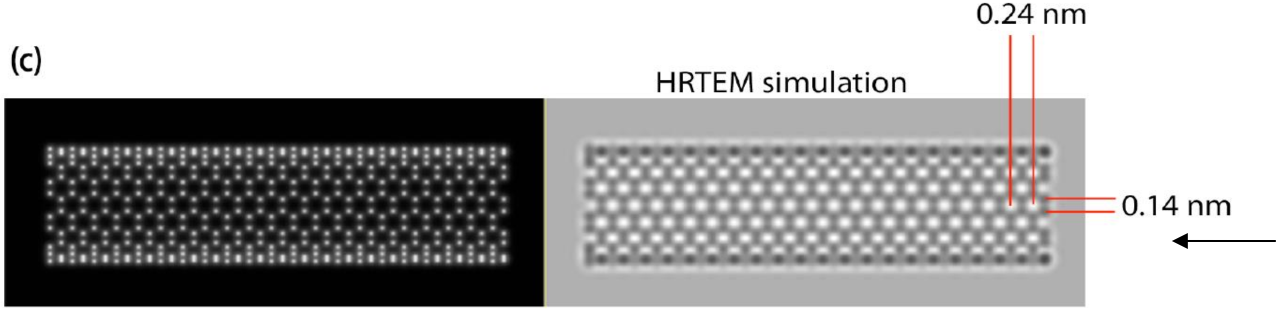


structure model of [10,10] SWCNT

C_S = 0.5 mm, Scherzer Defocus

← Uncorrected 80 kV

(c)



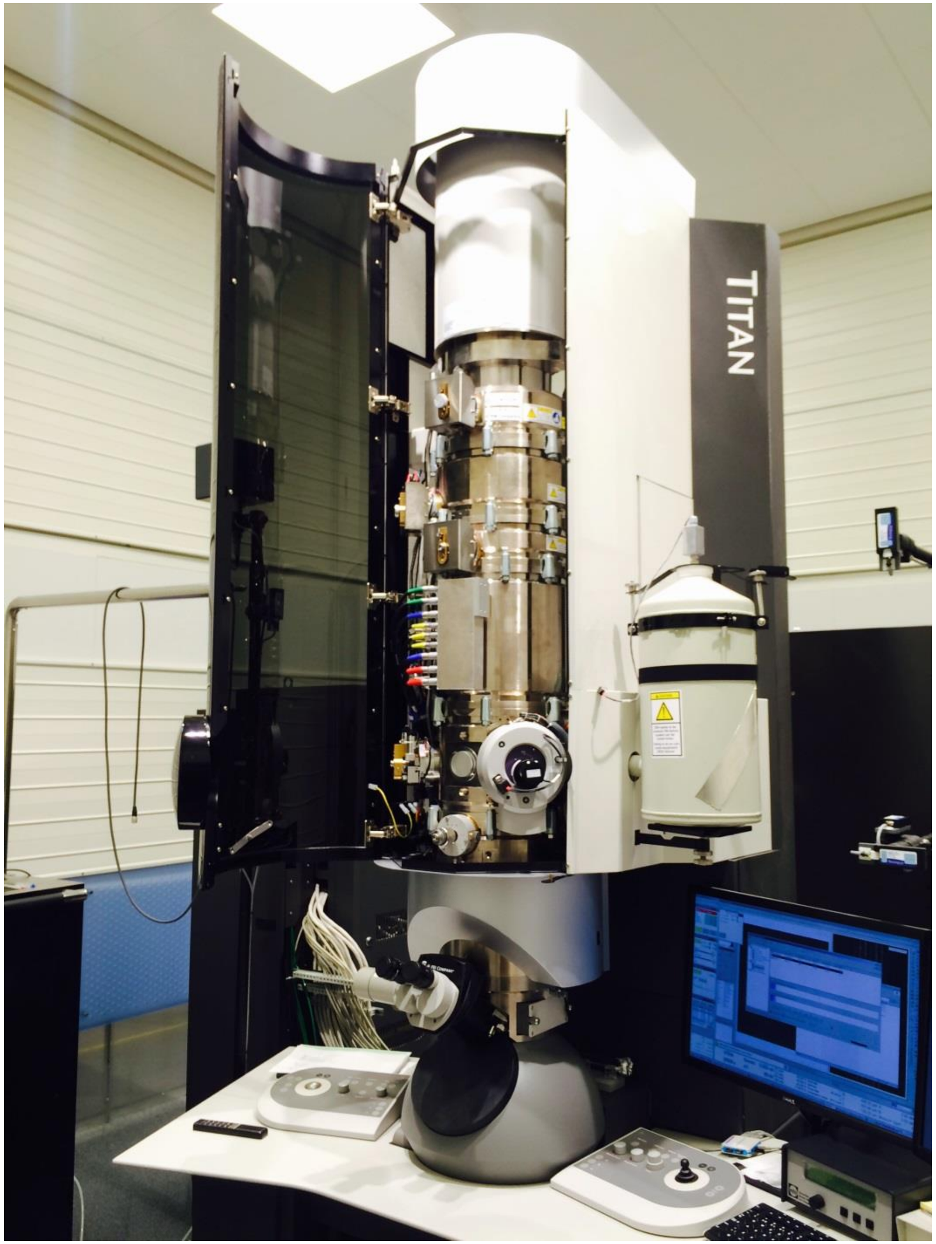
structure model of [10,10] SWCNT

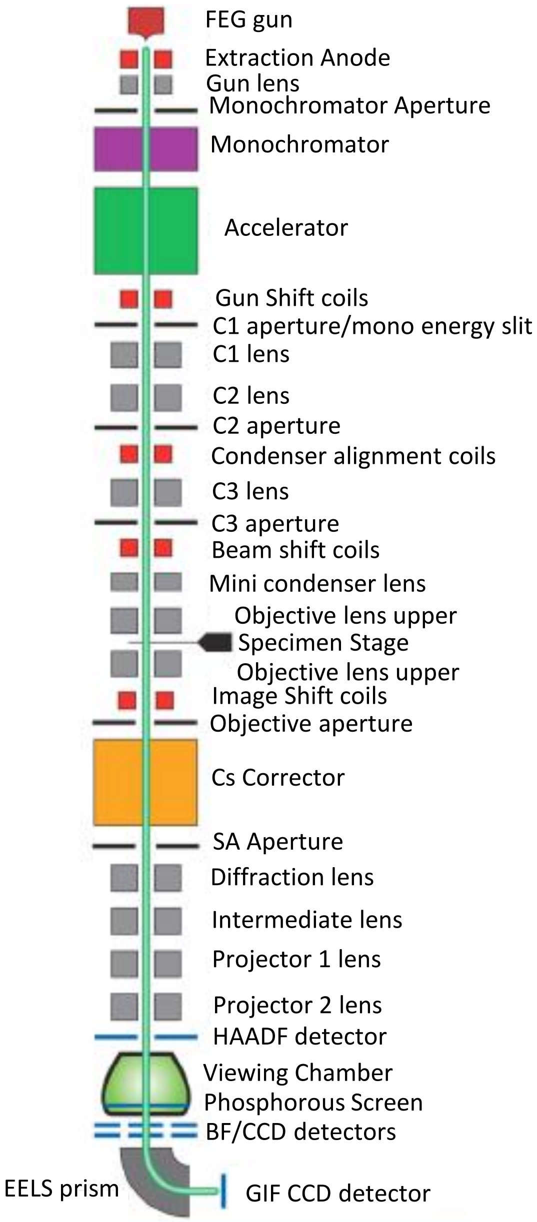
C_S = 0.001 mm, Scherzer Defocus

← Aberr. corrected 80 kV

Transmission Electron Microscope

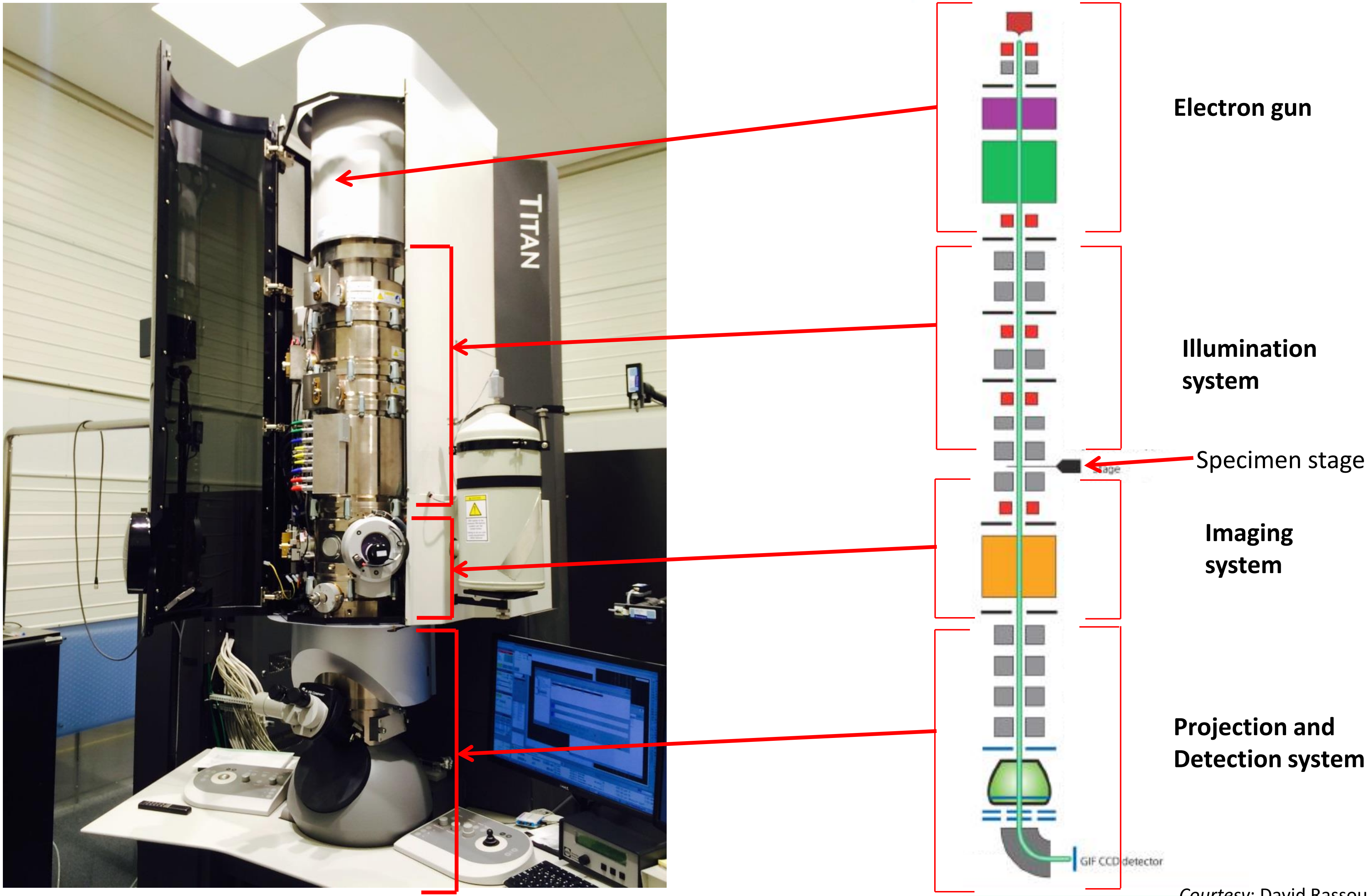
Instrumentation – Part 1





FEG gun
Extraction Anode
Gun lens
Monochromator Aperture
Monochromator
Accelerator
Gun Shift coils
C1 aperture/mono energy slit
C1 lens
C2 lens
C2 aperture
Condenser alignment coils
C3 lens
C3 aperture
Beam shift coils
Mini condenser lens
Objective lens upper
Specimen Stage
Objective lens upper
Image Shift coils
Objective aperture
Cs Corrector
SA Aperture
Diffraction lens
Intermediate lens
Projector 1 lens
Projector 2 lens
HAADF detector
Viewing Chamber
Phosphorous Screen
BF/CCD detectors
EELS prism
GIF CCD detector

Courtesy: David Rassouw, CCEM, Canada



Electron gun

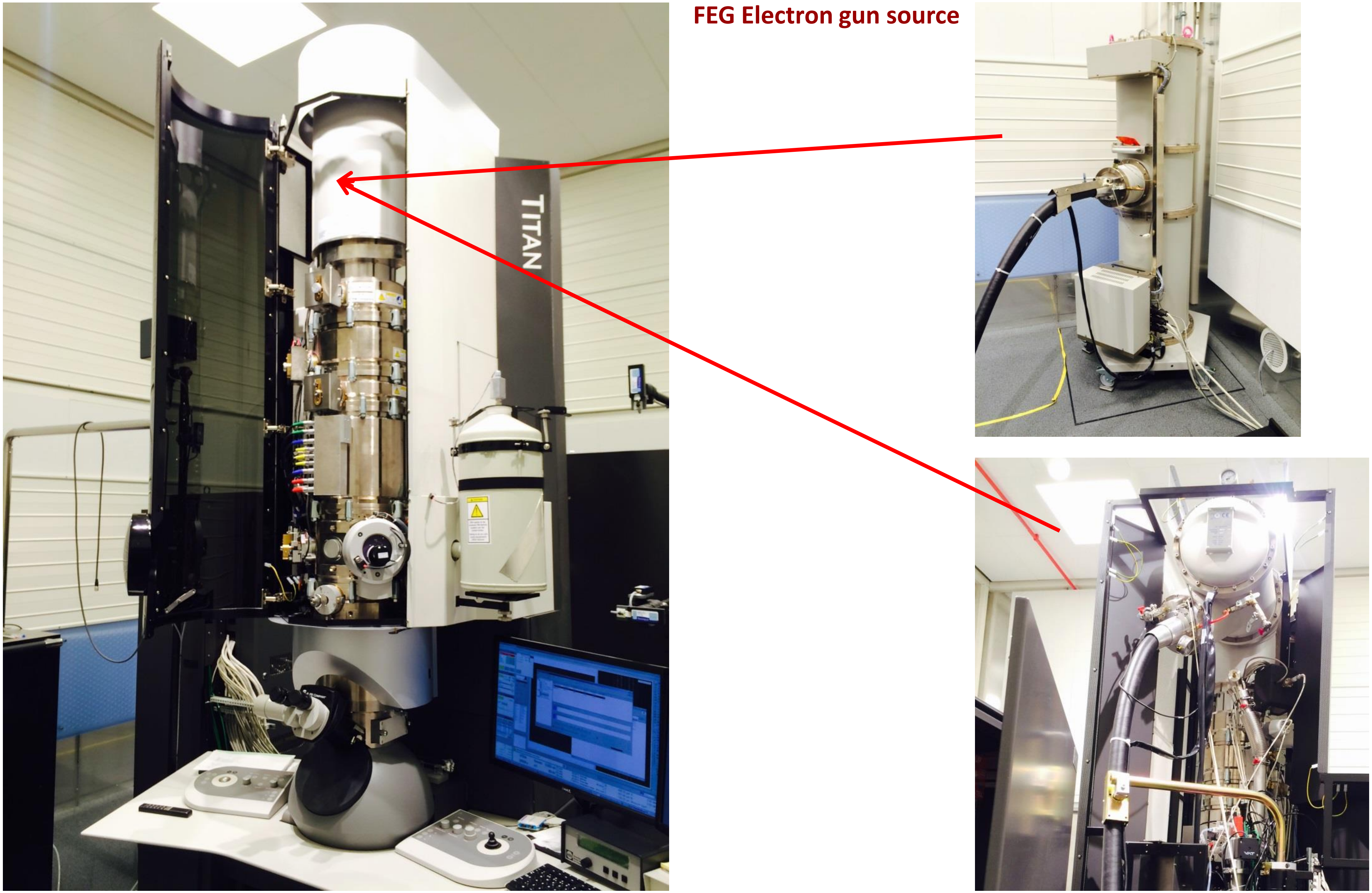
Illumination system

Specimen stage

Imaging system

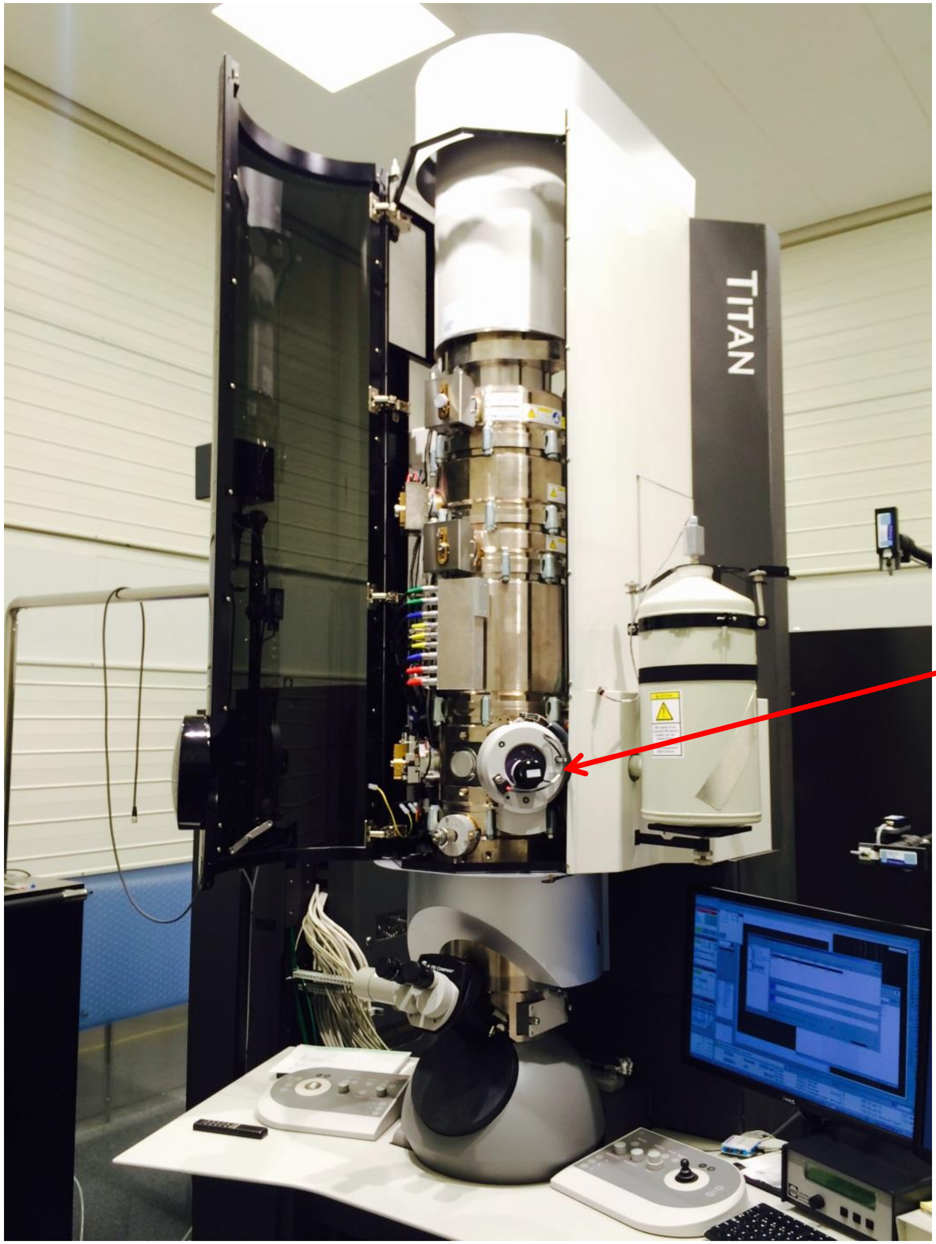
Projection and Detection system

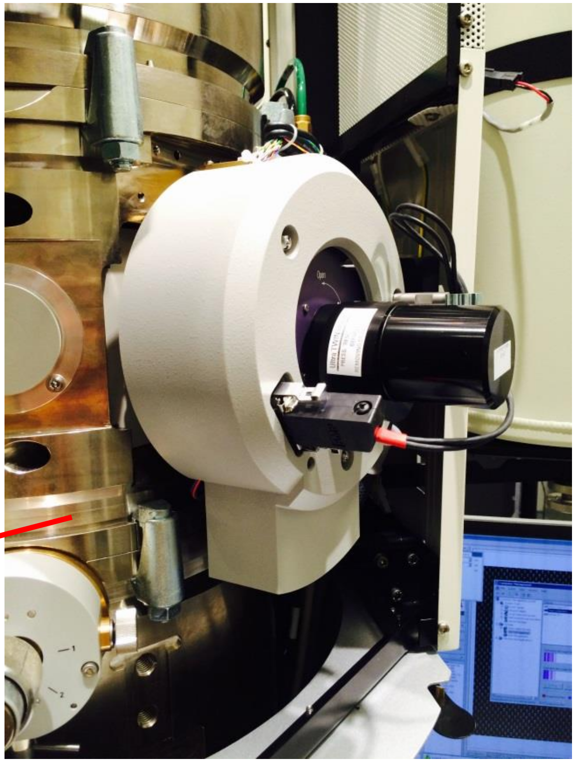
Courtesy: David Rassouw



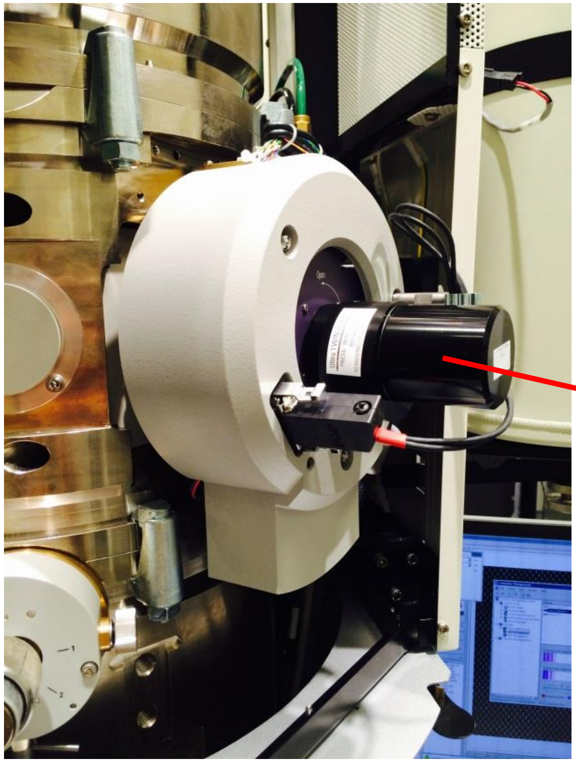
FEG Electron gun source

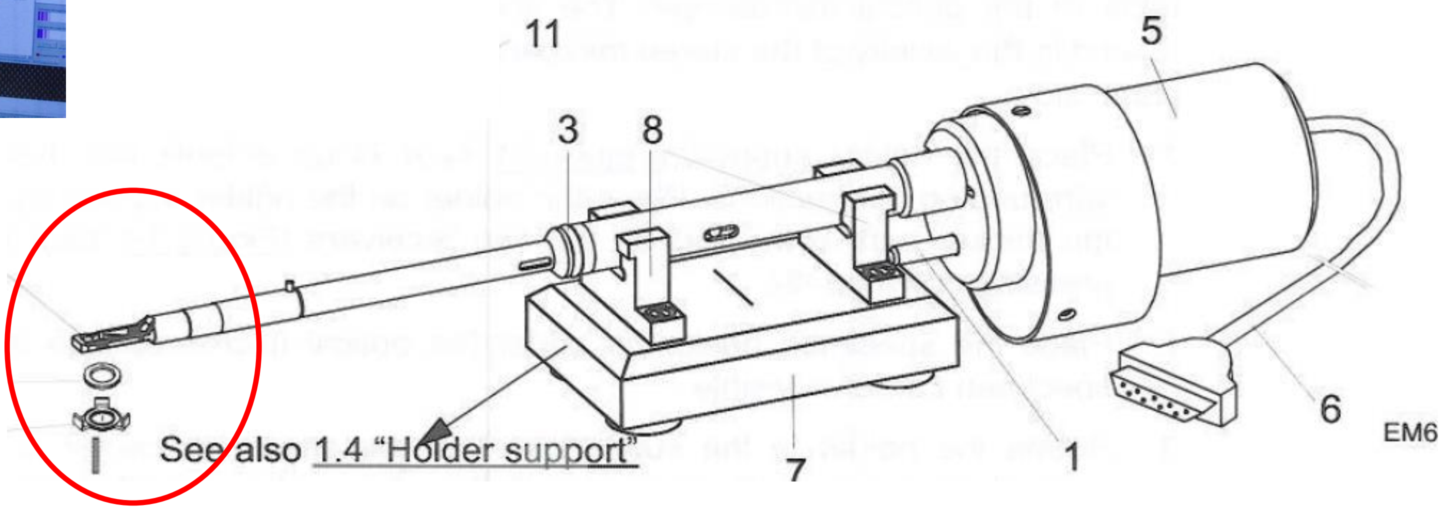
Specimen Stage





TEM Specimen Holder



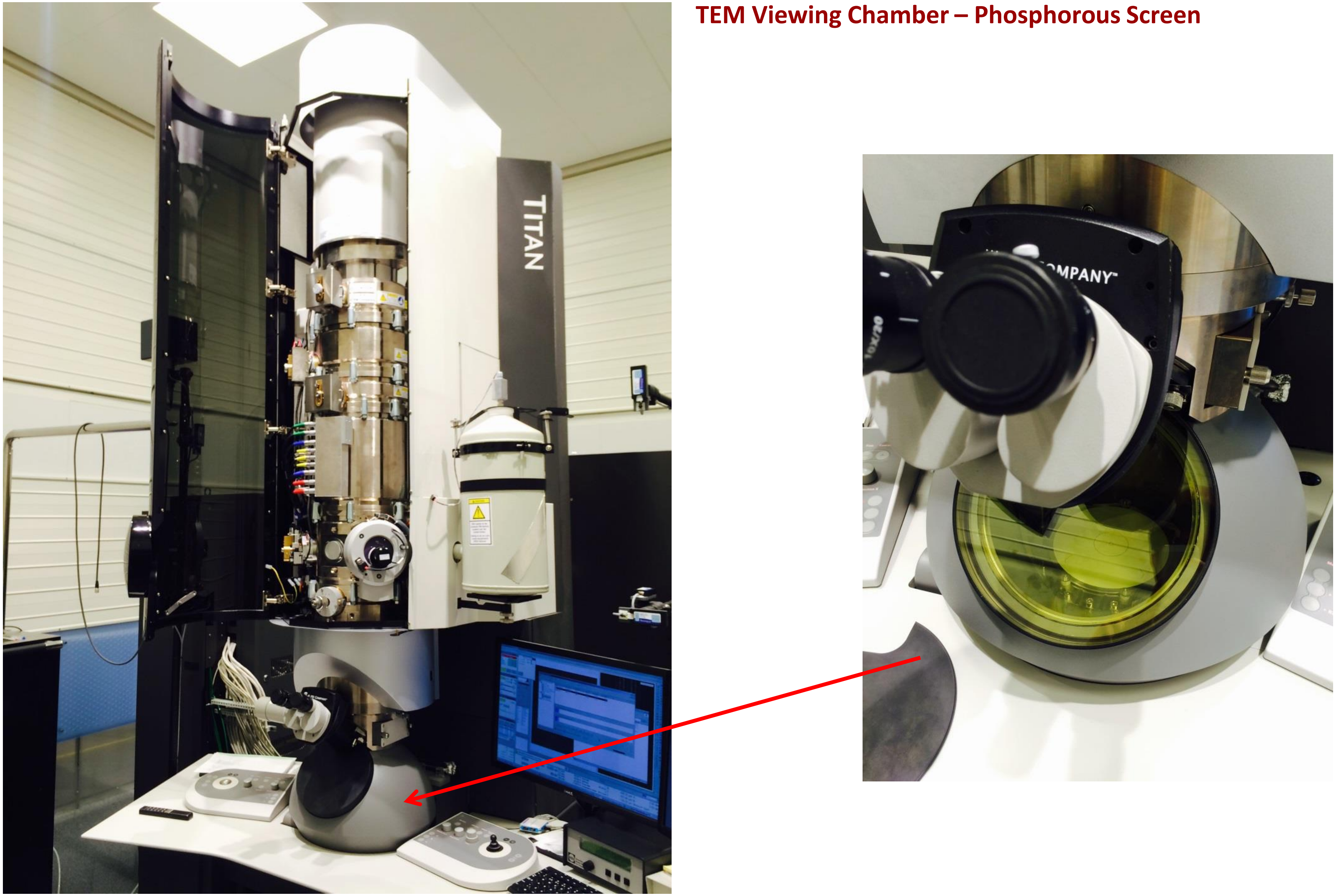


TEM Specimens

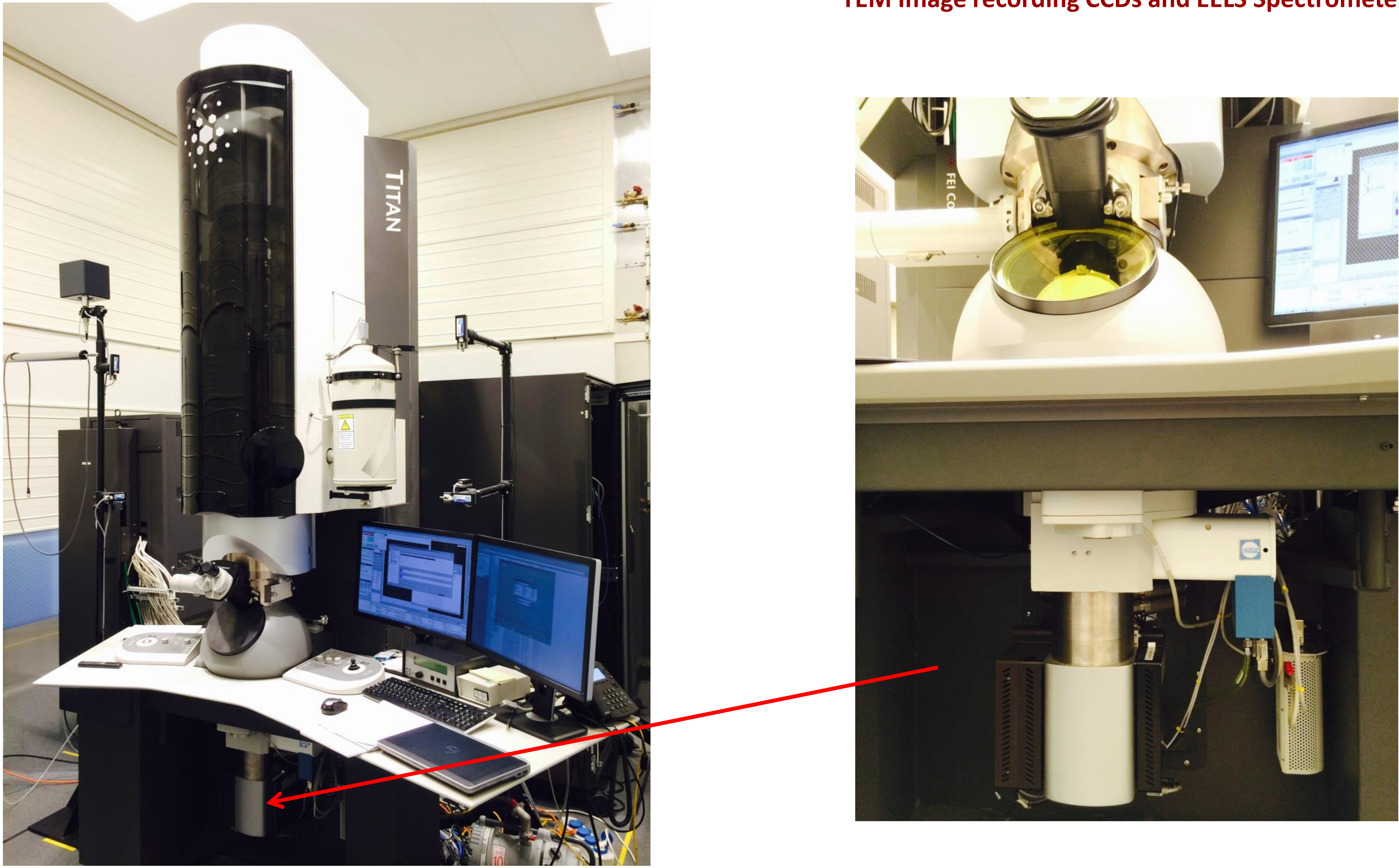
- Typically 3 mm in diameter

Courtesy: http://asummerinscience.blogspot.no

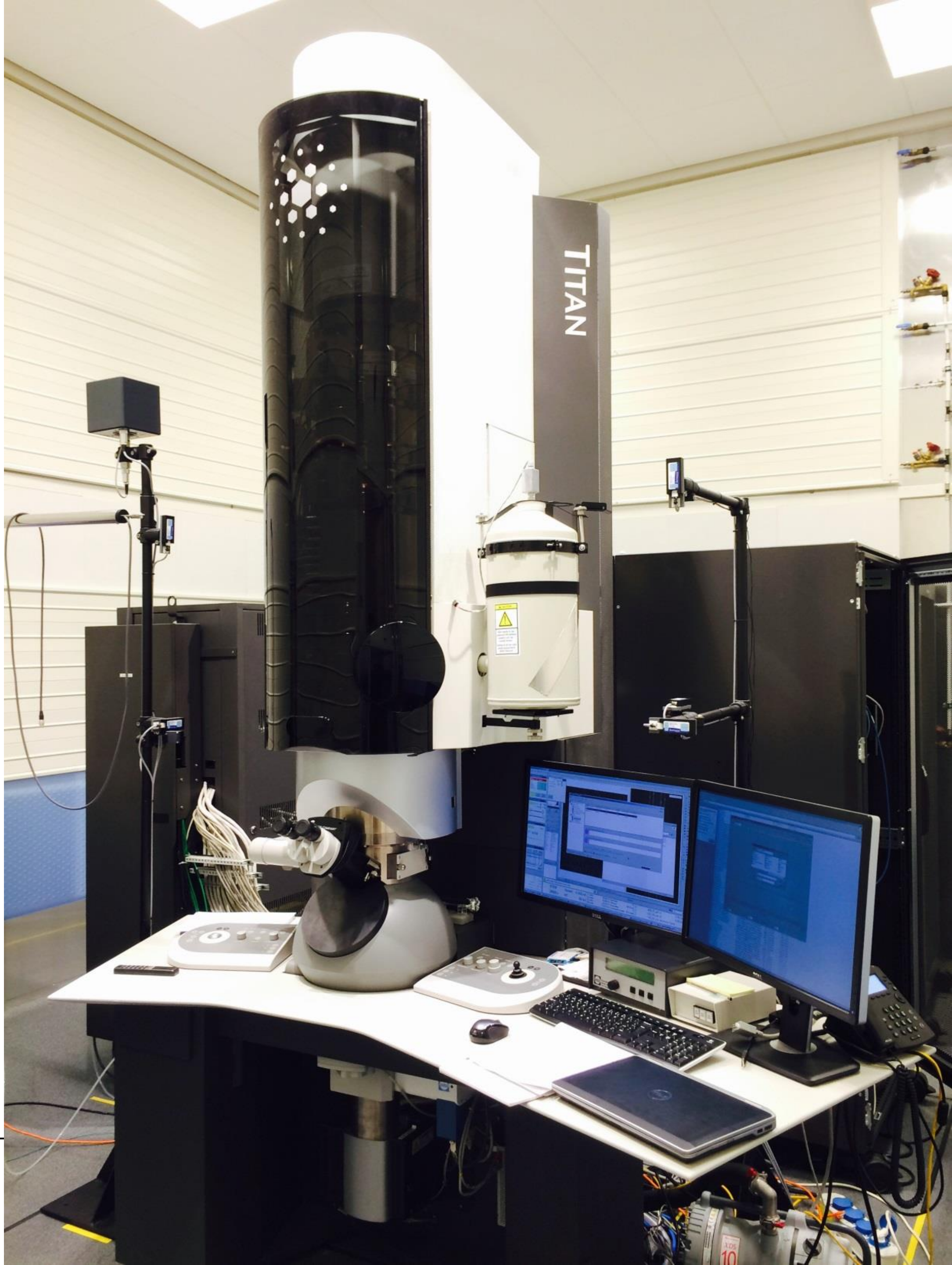
TEM Viewing Chamber – Phosphorous Screen



TEM Image recording CCDs and EELS Spectrometer



Transmission Electron Microscopy



Introduction and Basics
Part-2

UiO : University of Oslo
Centre for Materials Science and Nanotechnology (SMN)

TEM in Materials Science

The interesting objects for TEM is not the average structure or homogenous materials but

local structure and inhomogeneities

Defects

Interfaces

Chemical composition

Precipitates

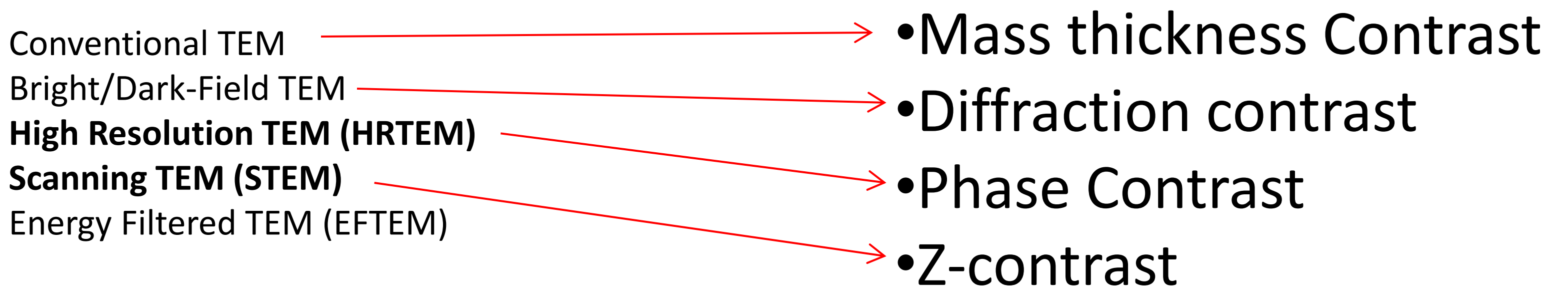
Chemical bonding

Atomic Structure

Electronic Structure

TEM techniques

Imaging



Main Constrast phenomena in TEM

Conventional TEM
Bright/Dark-Field TEM
High Resolution TEM (HRTEM)
Scanning TEM (STEM)
Energy Filtered TEM (EFTEM)

- Mass thickness Contrast
- Diffraction contrast
- Phase Contrast
- Z-contrast

Diffraction

Selected Area Electron Diffraction
Convergent Beam Electron Diffraction

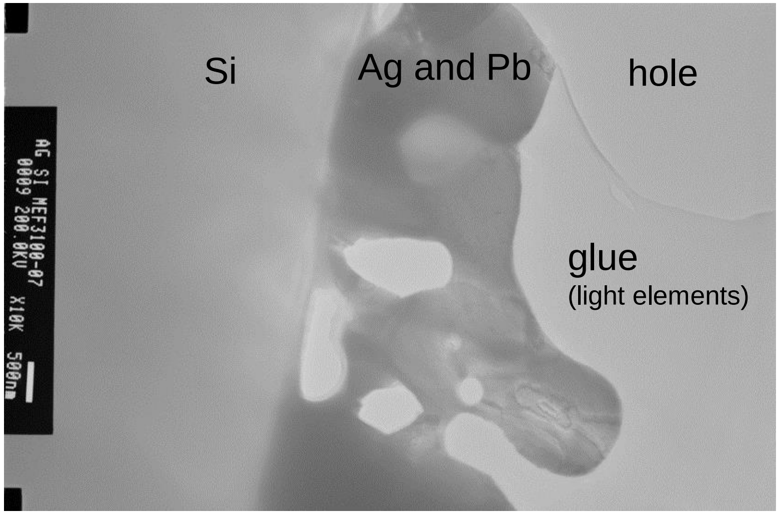
Phase identification, defects, orientation relationship between different phases, nature of crystal structure (amorphous, polycrystalline, single crystal)

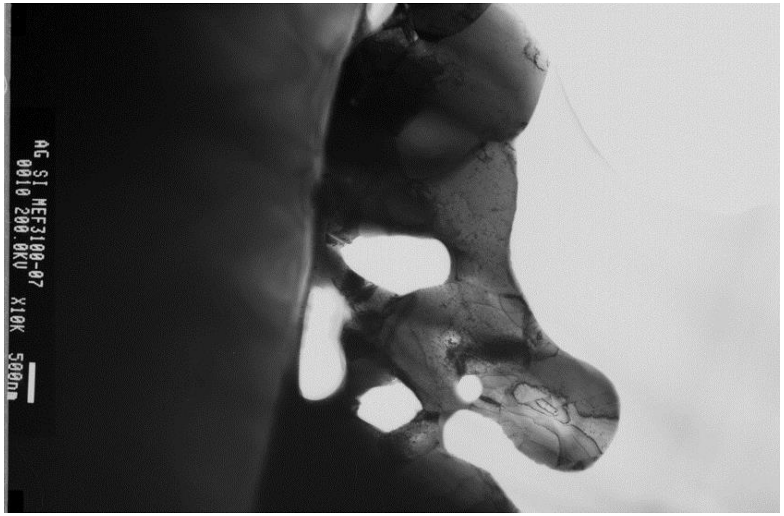
Spectroscopy

Electron Dispersive X-ray Spectroscopy (EDS)
Electron Energy Loss Spectroscopy (EELS)

Chemical composition, electronic states, nature of chemical bonding (EDS and EELS).
Spatial and energy resolution down to the atomic level and ~0.1 eV.

Objective aperture: Contrast enhancement



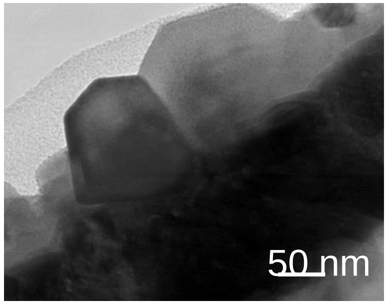


All electrons contributes to the image.

Intensity: Thickness and density dependence

Mass-thickness contrast

A small aperture allows only electrons in the central beam in the back focal plane to contribute to the image.



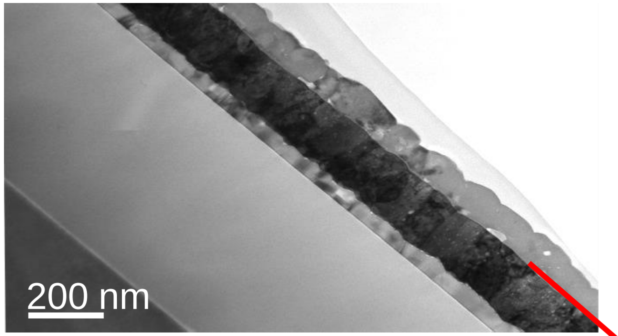
Diffraction contrast
(Amplitude contrast)

One grain seen along a low index zone axis.

TEM techniques

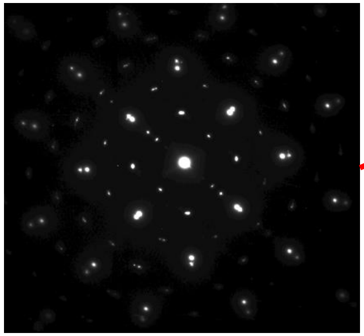
Imaging

Conventional TEM
Bright/Dark-Field TEM
High Resolution TEM (HRTEM)
Scanning TEM (STEM)
Energy Filtered TEM (EFTEM)



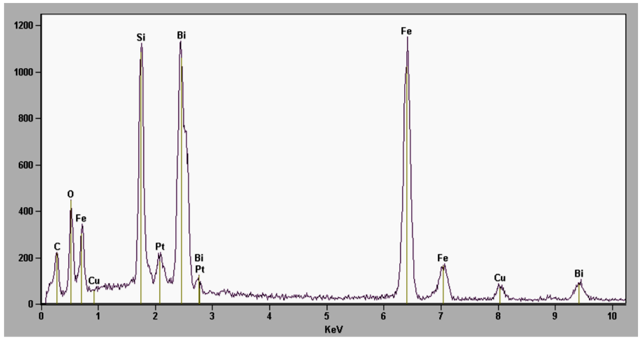
Diffraction

Selected Area Electron Diffraction
Convergent Beam Electron Diffraction

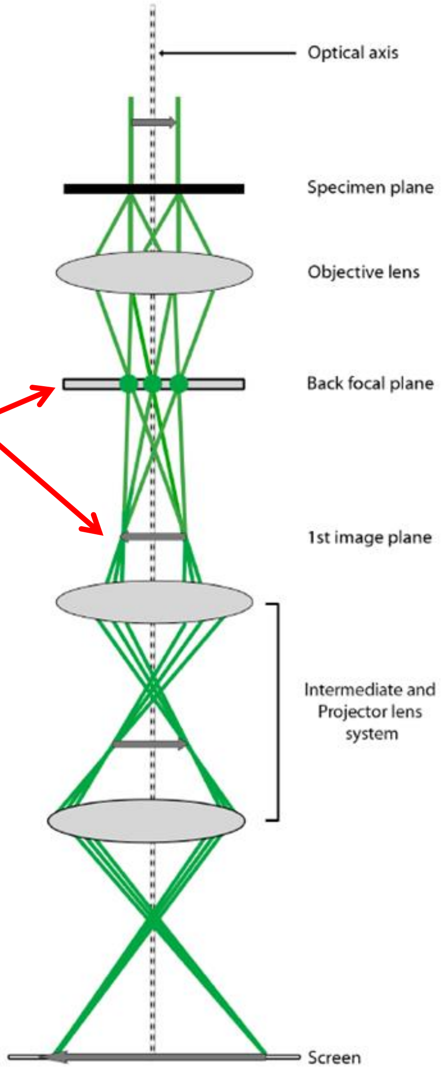


Spectroscopy

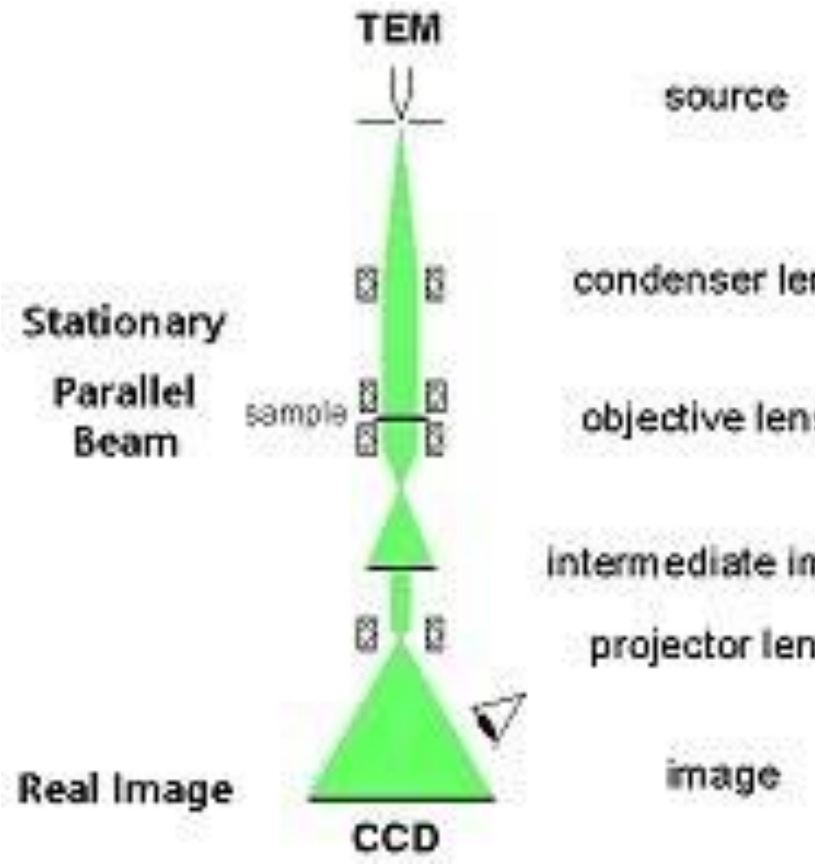
Electron Dispersive Spectroscopy (EDS)
Electron Energy Loss Spectroscopy (EELS)

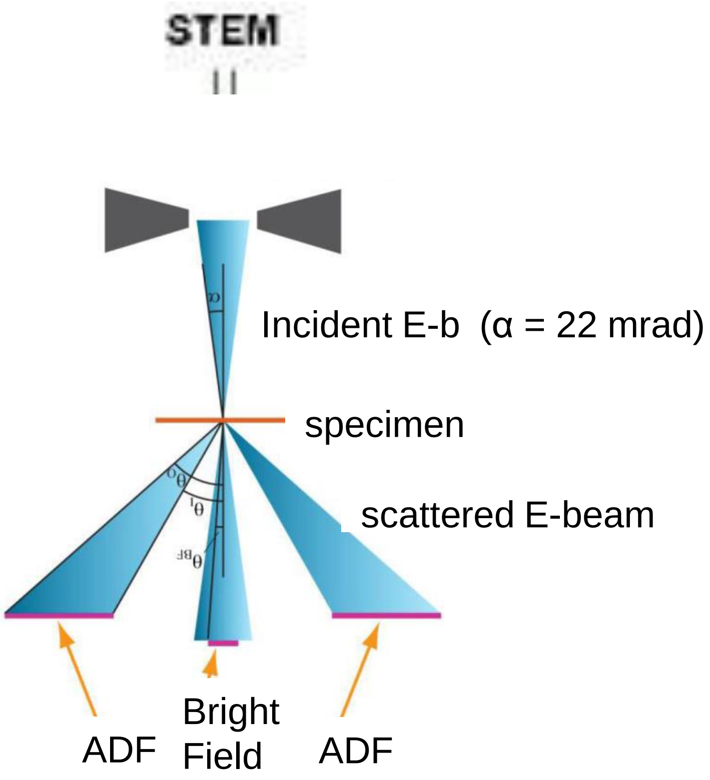


Simplified ray diagram of conventional TEM



Imaging

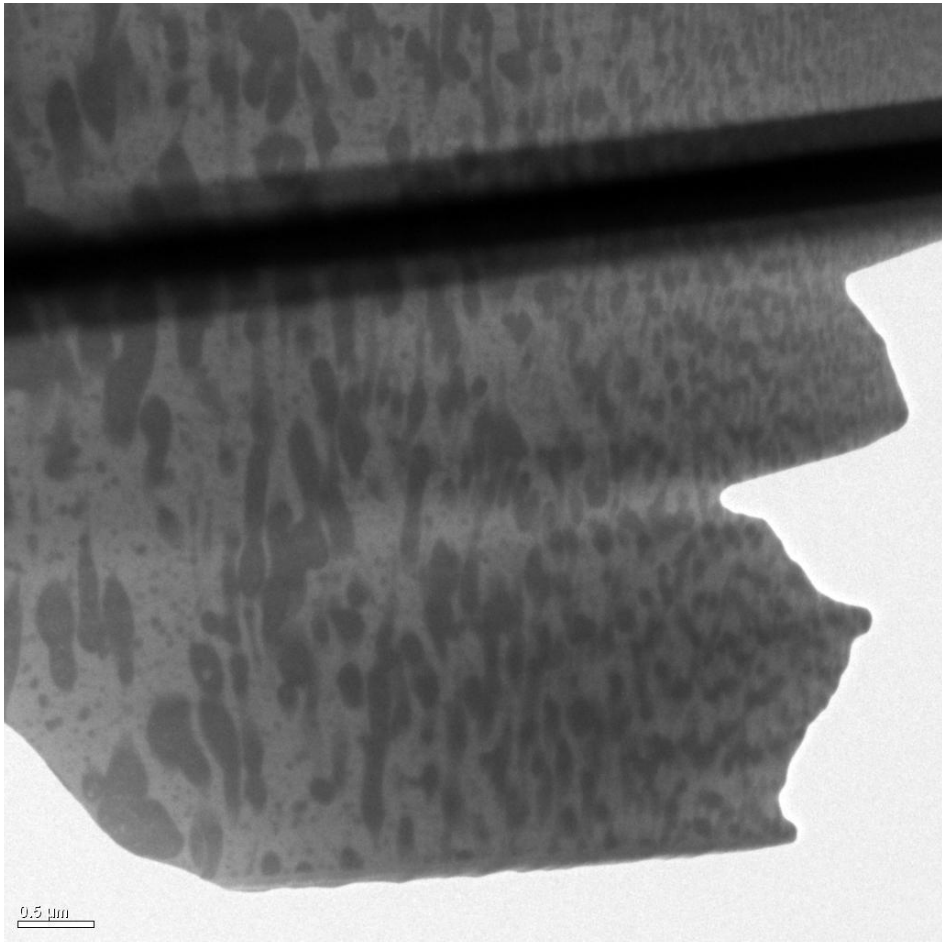




Courtesy: http://www.ifam.fraunhofer.de; I.MacLauren et al, International Materials Review, 59, 115 (2004)

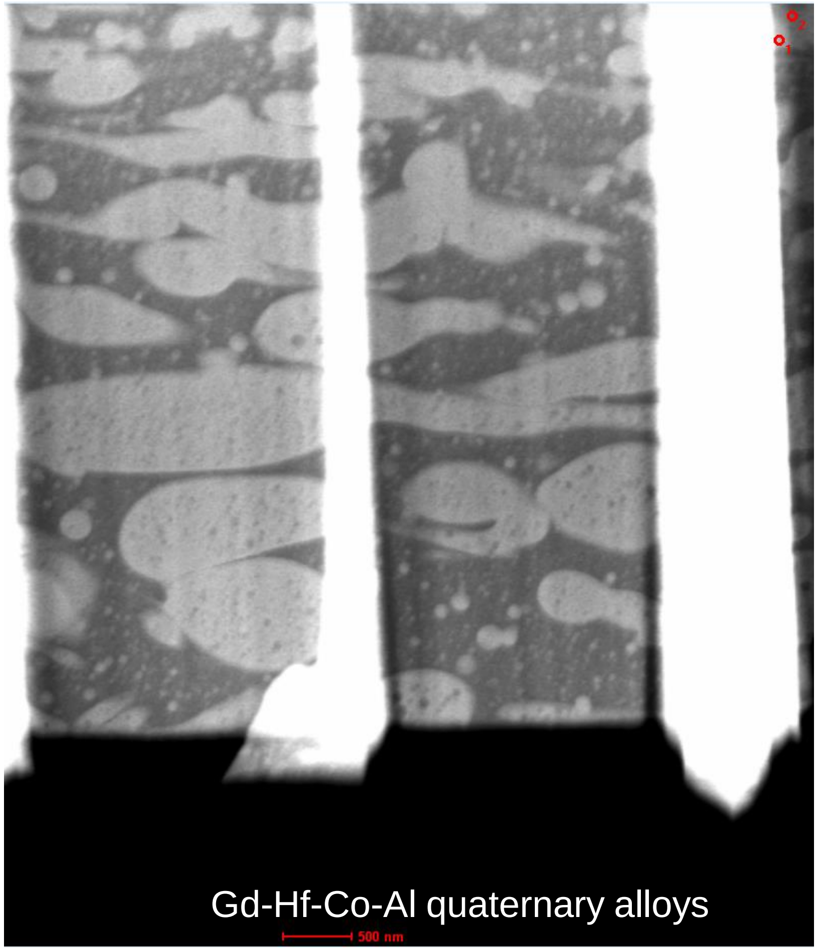
Imaging

TEM



Mass thickness and diffraction contrast

STEM

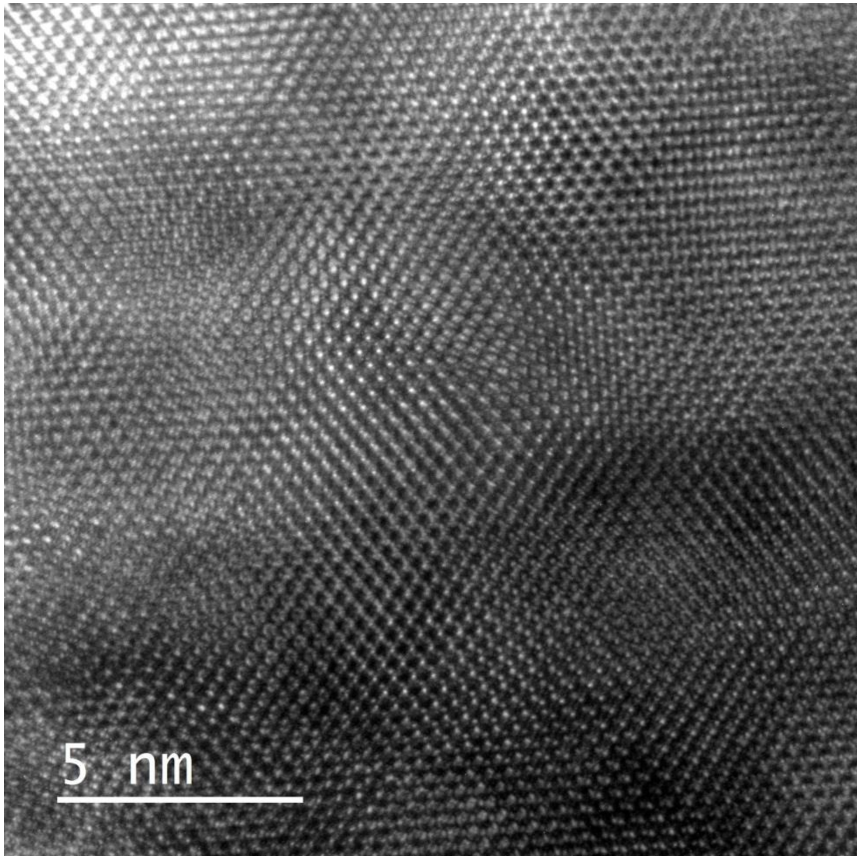

Gd-Hf-Co-Al quaternary alloys

Mass thickness and Z- contrast

	Z
Gd	64
Hf	72
Co	27
Al	13

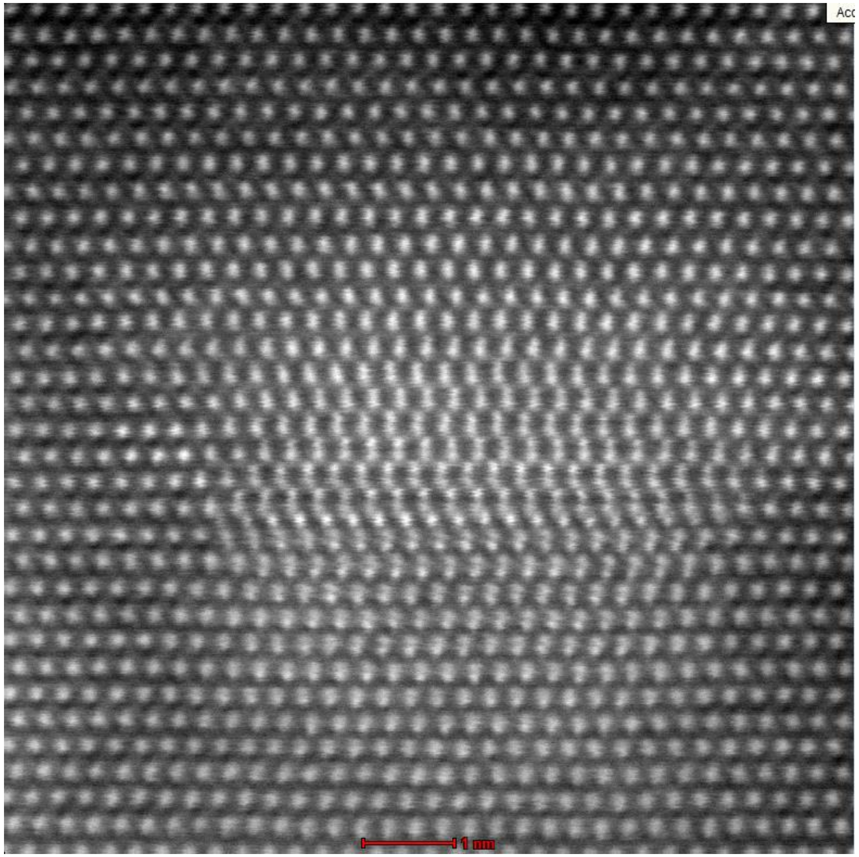
Imaging

HRTEM

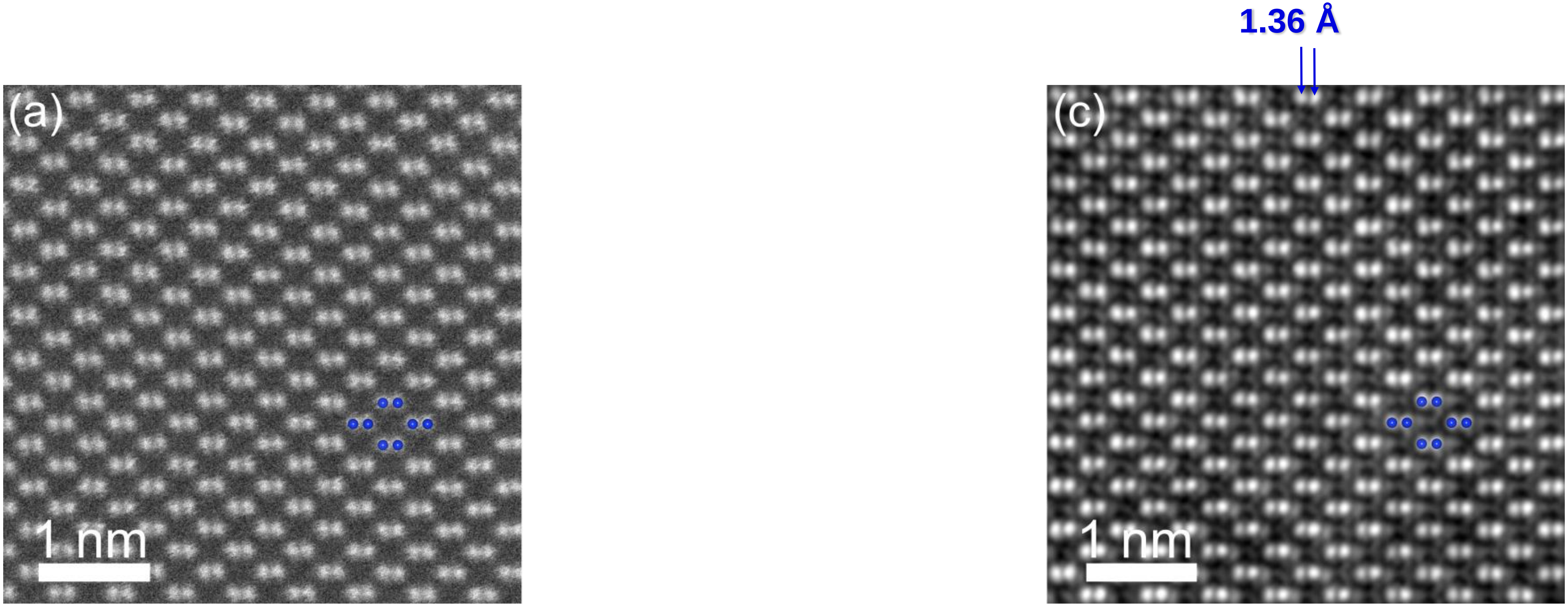


Phase contrast

STEM



Z- contrast



HAADF-STEM

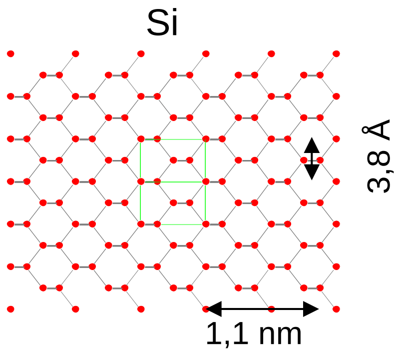
HRTEM

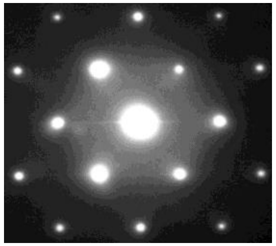
Raw HAADF-STEM, ABF-STEM and HRTEM image of Si in the [110] zone axis by FEI Titan 60-300 with spatial resolutions of 0.8 Å for STEM and 2.0 Å for TEM.

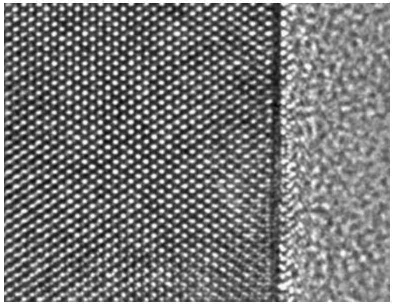
Courtesy: Wei Zhan, Øystein Prytz, *et al.* (2015), SMN, UiO

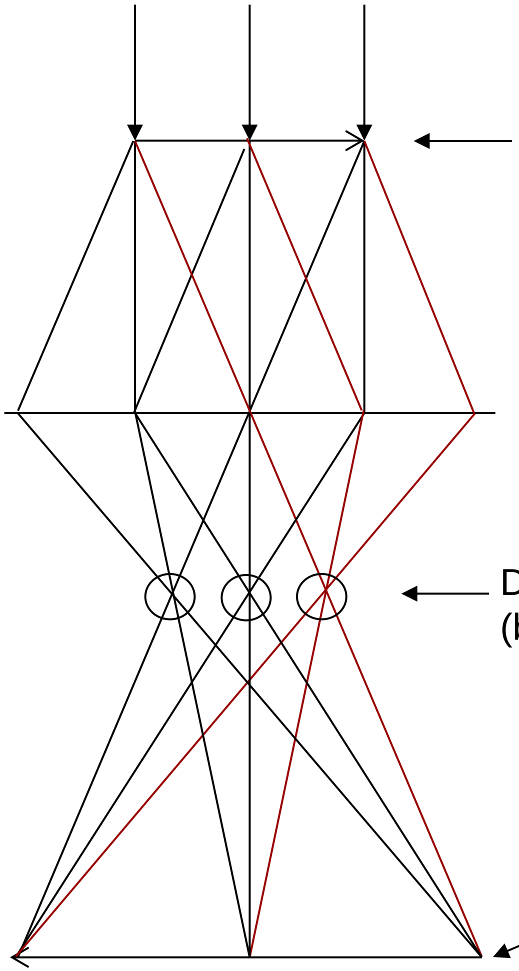
Electron Diffraction in TEM

Simplified ray diagram









Parallel incoming electron beam

Sample

Objective lense

Diffraction plane
(back focal plane)

Objective aperture

Image plane

Selected area
aperture

Electron Diffraction in TEM

Elastic scattered electrons

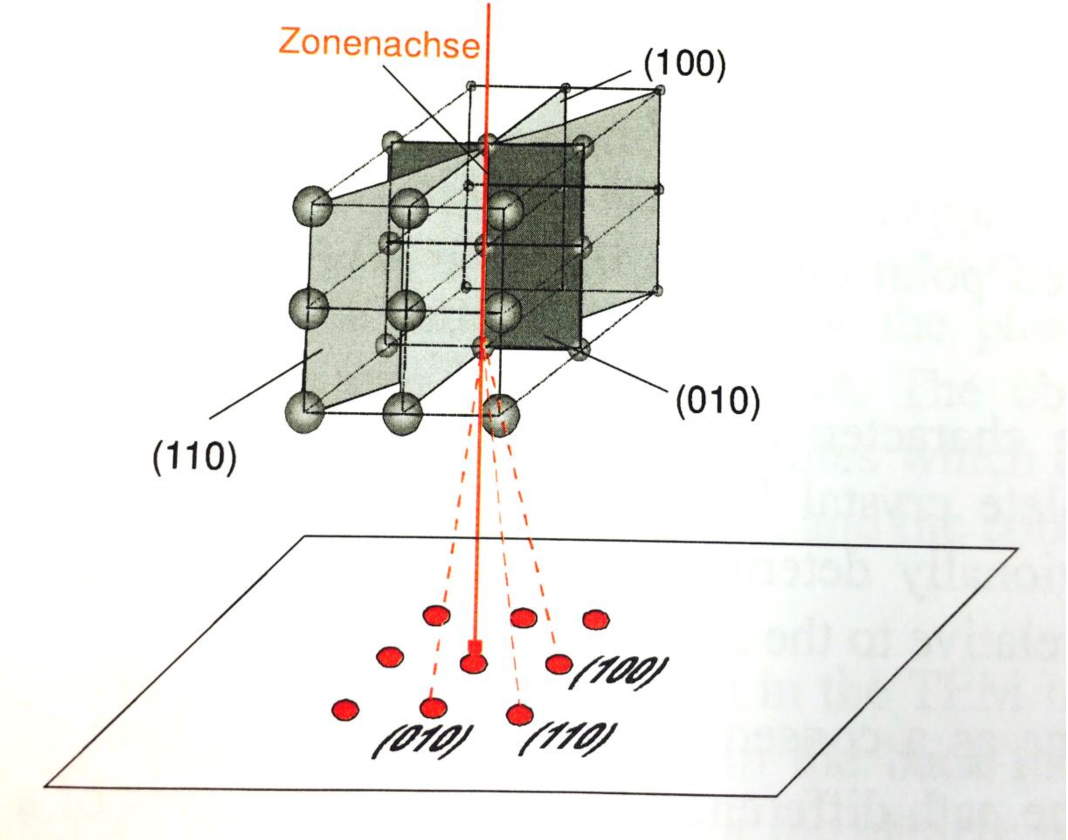
Only the direction of **v** is changing.
(Bragg scattering)

Elastic scattering is due to Coulomb interaction between the incident electrons and the electric charge of the electron clouds and the nucleus. (Rutherford scattering).

The elastic scattering is due to the average position of the atoms in the lattice.

Reflections satisfying Braggs law:

$$2d\sin\theta = n\lambda$$

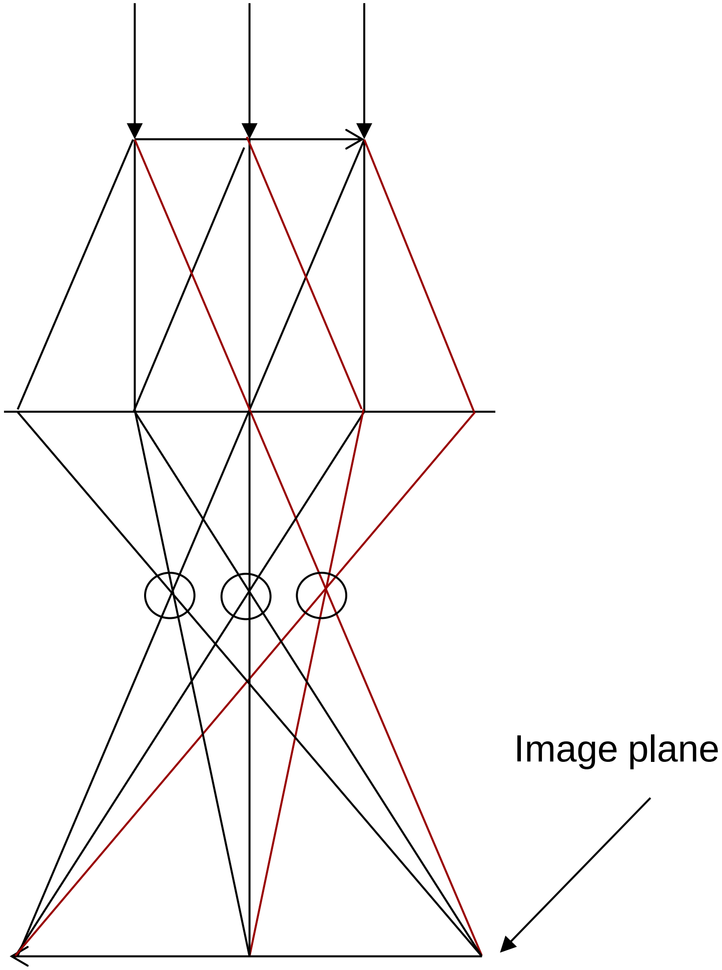


Electrons interacts 100-1000 times stronger with matter than X-rays
-more absorption (need thin samples)
-can detect weak reflections not observed with XRD technique

Courtesy: Dr. Jürgen Thomas, IFW-Dresden, Germany

Selected area diffraction(SAD)

- Parallel incoming electron beam and a selection aperture in the image plane.
- Diffraction from a single crystal in a polycrystalline sample if the SAD aperture is small enough/crystal large enough.
- Orientation relationships between grains or different phases can be determined.
- ~2% accuracy of lattice parameters
  - Convergent electron beam better



Camera constant

$R = L \tan 2\theta_B \sim 2L\sin\theta_B$

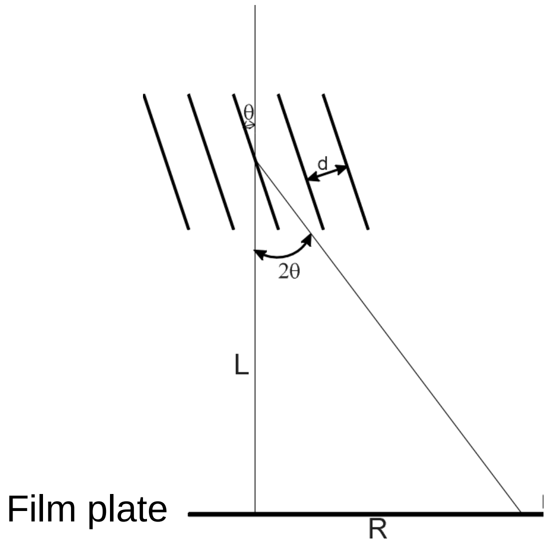
$2d\sin\theta_B = \lambda$

$\downarrow$

$\mathbf{R = L\lambda/d}$

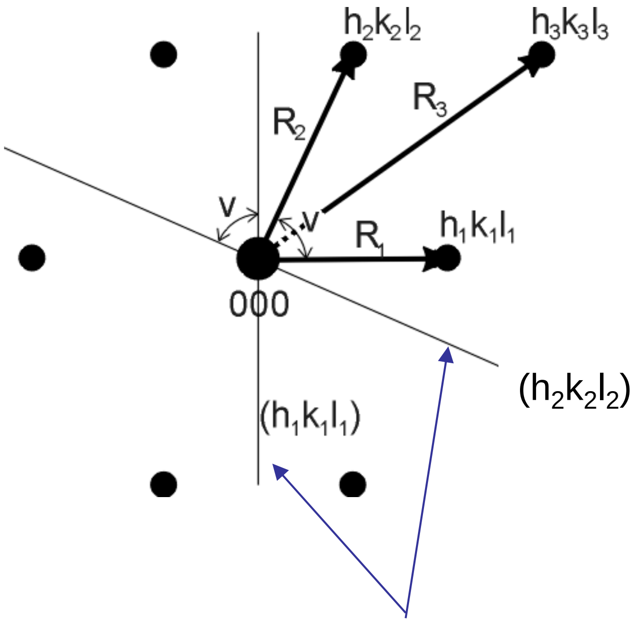
Camera constant:

$\mathbf{K = \lambda L}$



Indexing diffraction patterns

The **g** vector to a reflection is normal to the corresponding (h k l) plane and IgI=1/$d_{nh\ nk\ nl}$

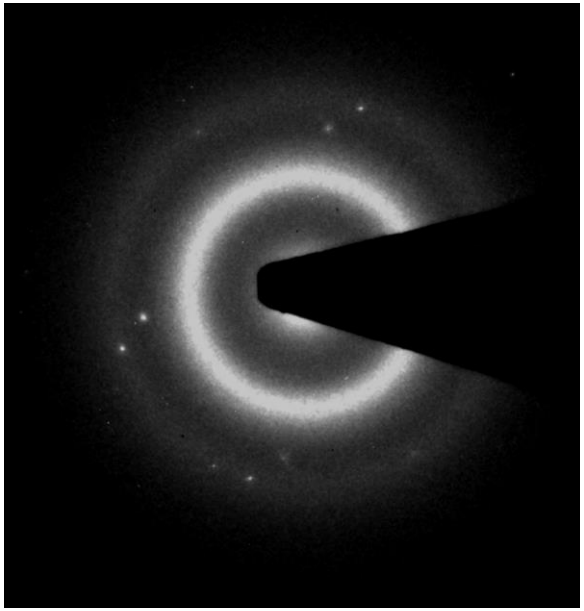


Orientations of corresponding planes in the real space

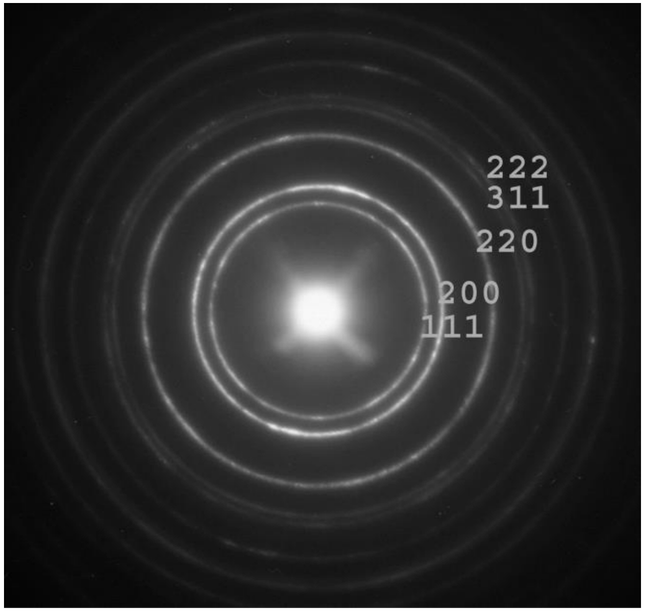
- Measure R_i and the angles between the reflections
- Calculate d_i , i=1,2,3 (=K/R_i)
- Compare with tabulated/theoretical calculated d-values of possible phases
- Compare R_i/R_j with tabulated values for cubic structure.
- $\mathbf{g}_{1,hkl}+\mathbf{g}_{2,hkl}=\mathbf{g}_{3,hkl}$ (vector sum must be ok)
- Perpendicular vectors: $\mathbf{g}_i \bullet \mathbf{g}_j = 0$
- Zone axis: $\mathbf{g}_i \times \mathbf{g}_j = [HKL]_z$
- All indexed **g** must satisfy: $\mathbf{g} \bullet [HKL]_z=0$

Electron Diffraction in TEM

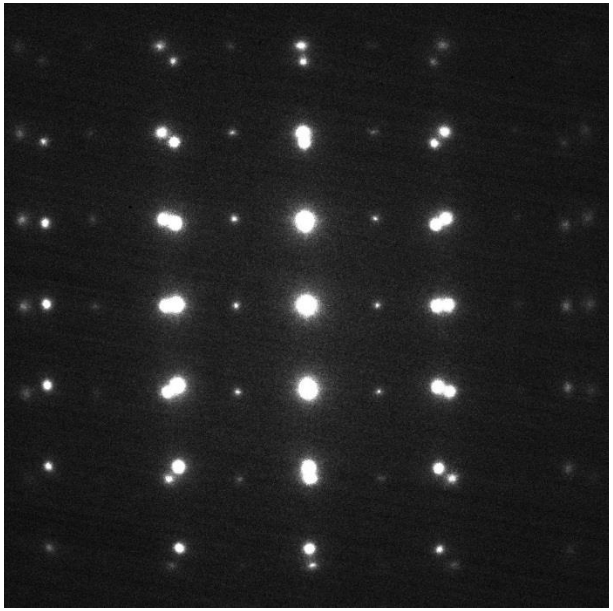
Amorphous phase



Poly crystalline sample

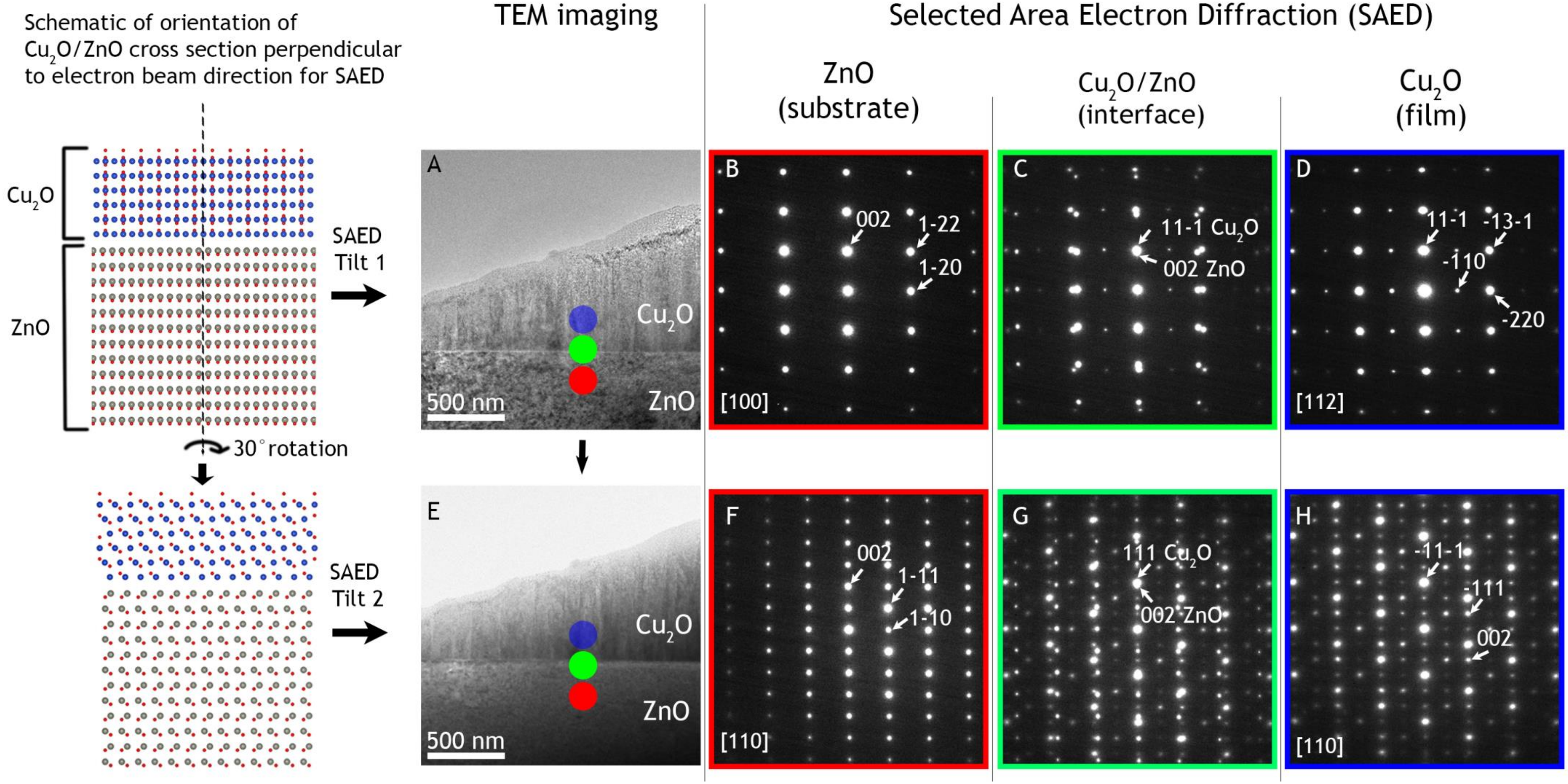


Single Crystals
Interface between two different phases
epitaxially grown



The orientation relationship between the phases can be determined with ED.

Schematic of orientation of Cu_2O/ZnO cross section perpendicular to electron beam direction for SAED



TEM imaging

Selected Area Electron Diffraction (SAED)

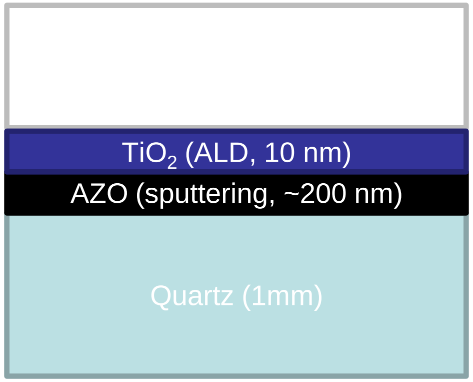
ZnO (substrate)

Cu_2O/ZnO (interface)

Cu_2O (film)

Spectroscopy

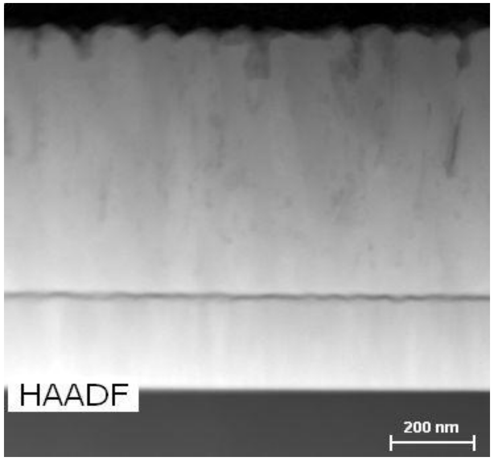
X-ray Energy Dispersive Spectroscopy

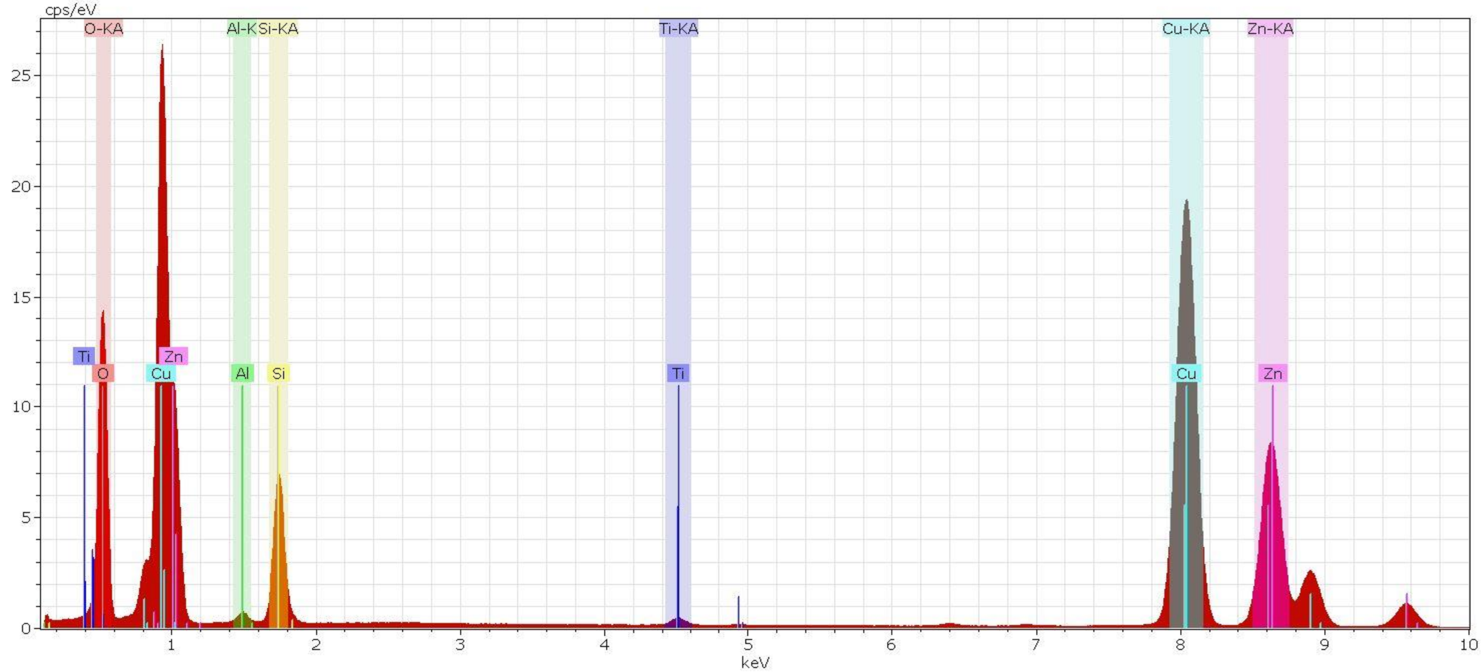


TiO_2 (ALD, 10 nm)

AZO (sputtering, ~200 nm)

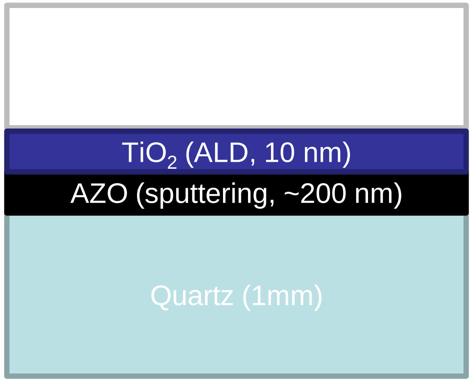
Quartz (1mm)





We detect the X-rays generated by the sample on a spectrometer
Each element has a unique atomic structure and hence a characteristic X-ray energy

Energy Dispersive X-ray Spectroscopy



TiO_2 (ALD, 10 nm)

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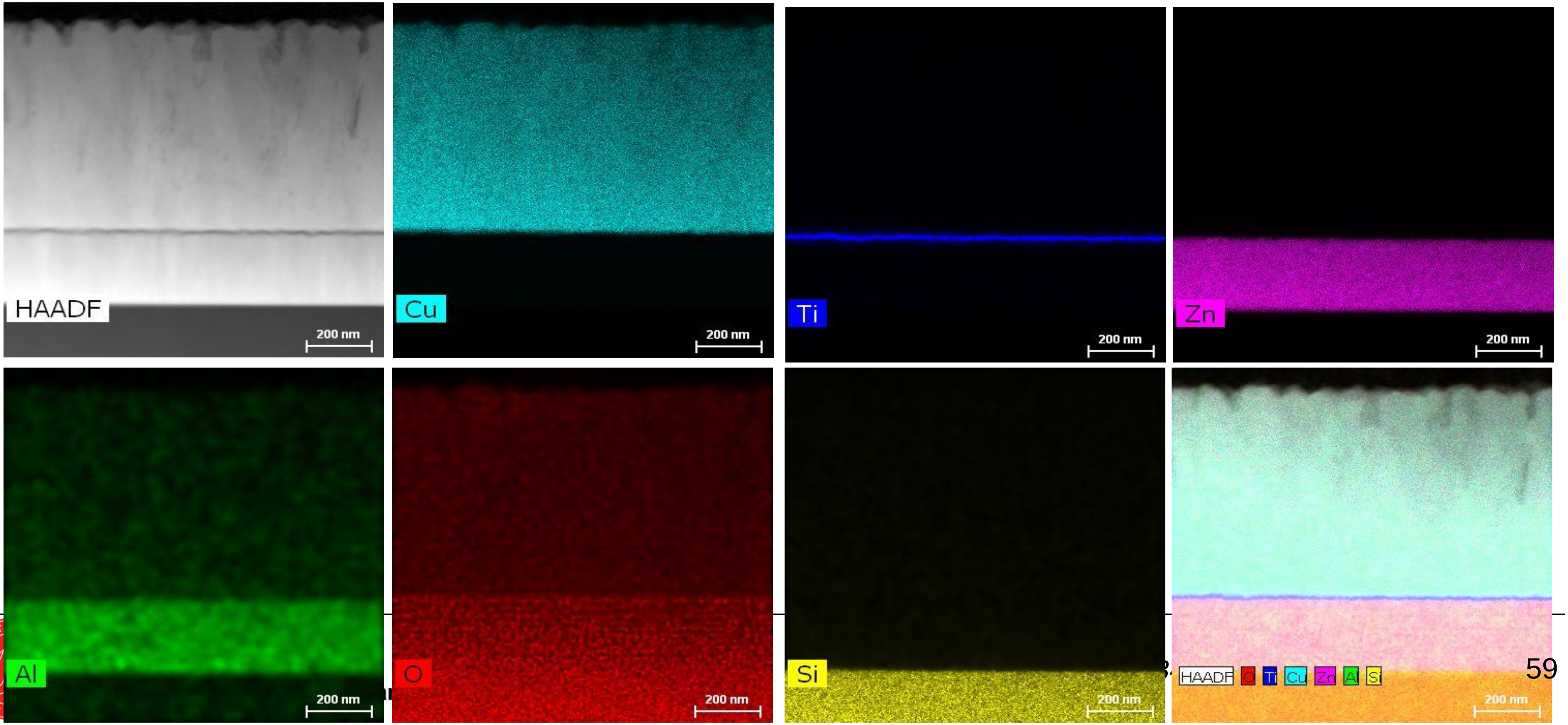
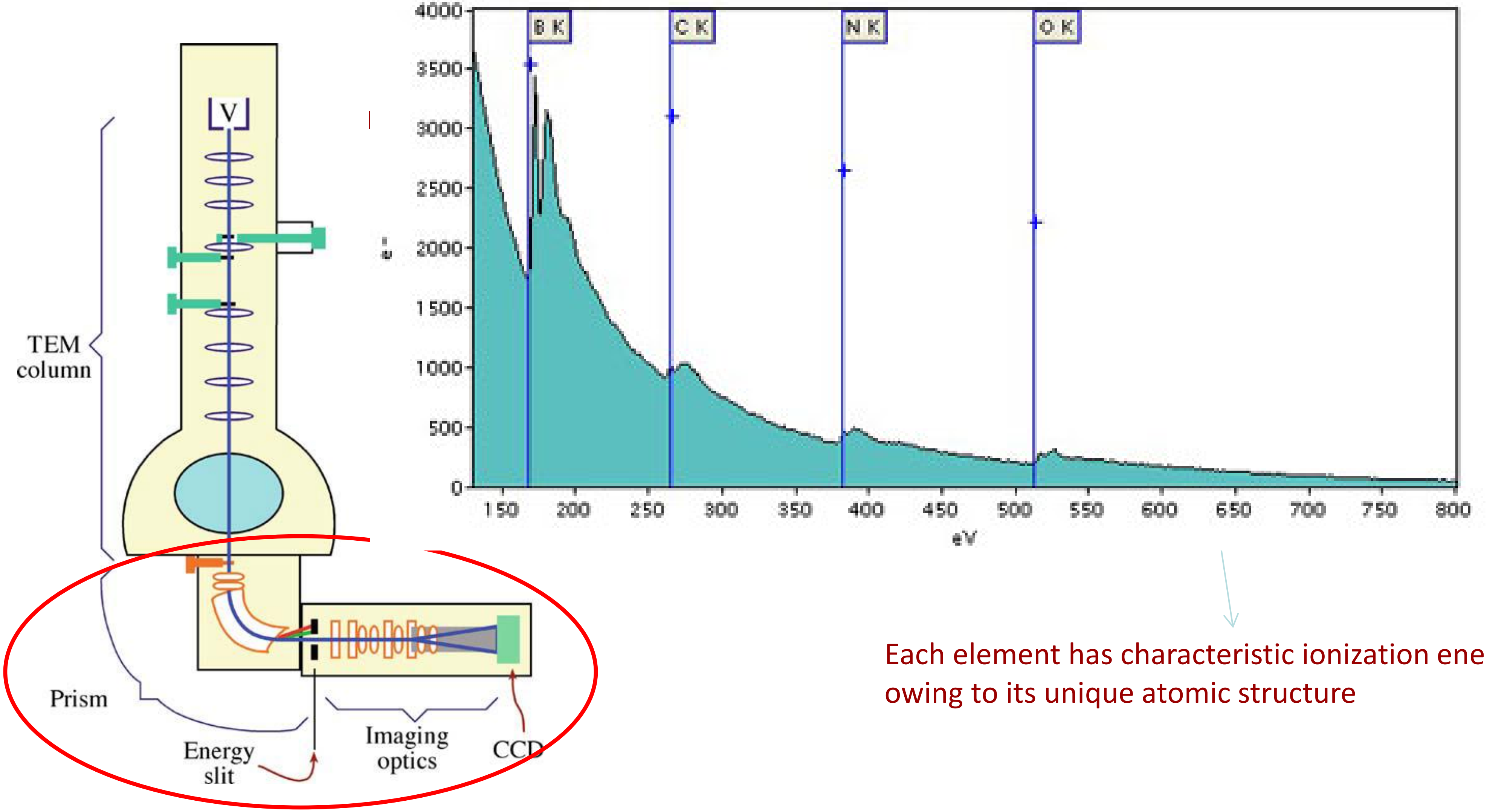


Electron Energy Loss Spectroscopy (EELS)

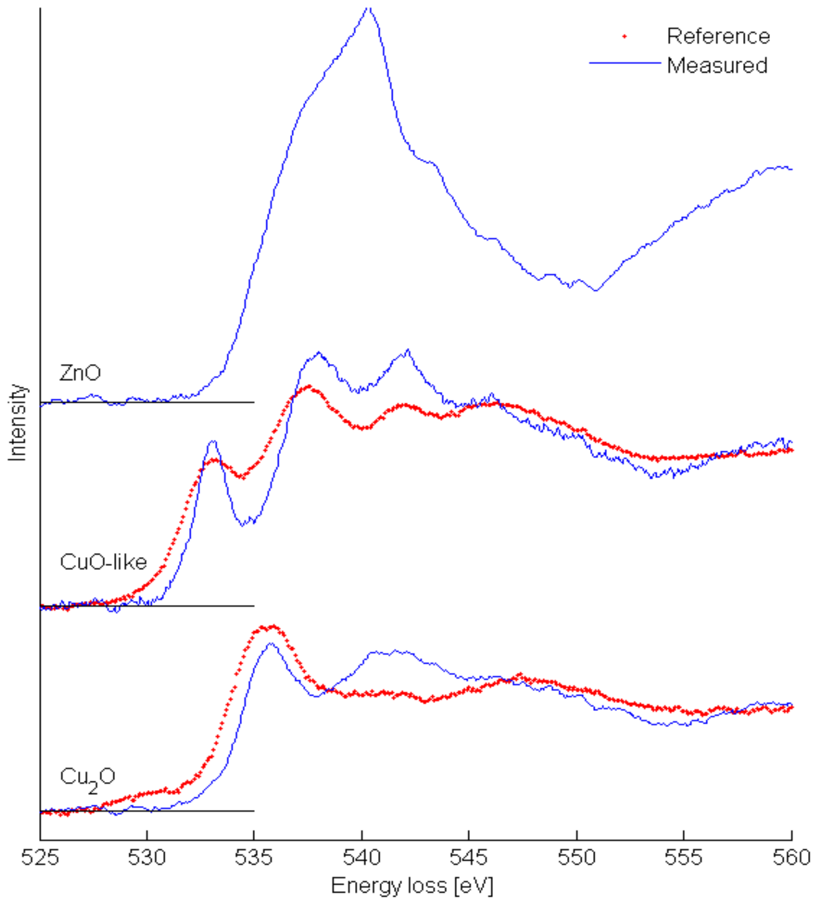


Each element has characteristic ionization energy owing to its unique atomic structure

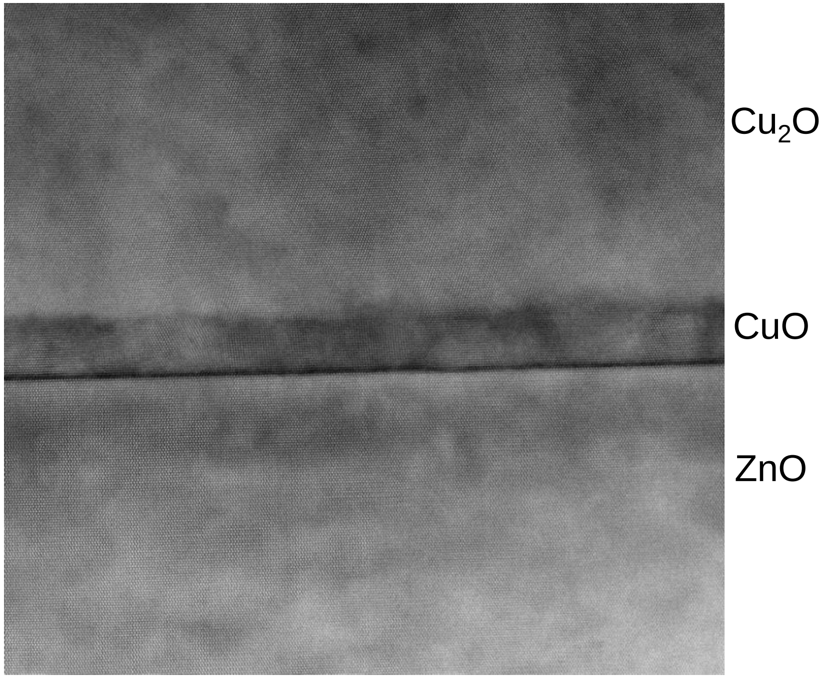
Courtesy: William & Carter, Transmission Electron Microscopy; EM group, Univ. of Nevada, Reno.

Electron Energy Loss Spectroscopy (EELS)

EELS of the Oxygen K edge



The reference spectra of Cu_2O and CuO are from online EELS database[1]. The reference spectra were shifted in energy to match the first O K peak in our experimental, and scaled by the total counts in the energy-loss 560-590 eV.



Courtesy: Cecilie Granerod, SMN, UiO

[1] Ngantcha, Gerland, Kihn & Riviere, *Eur. Phys. J. Appl. Phys*. **29**, (2005) 83.

Next Lecture

- TEM Instrumentation – Part 2

  (Text book Chapters: 5 – 9)

- TEM Specimen Preparation

  (Text book Chapters: 10)