



# Transmission Electron Microscopy (TEM)

CH-633

Introduction to transmission electron microscopy (TEM)

TEM imaging and diffraction

High-resolution TEM

Scanning TEM (STEM)

Analytical techniques

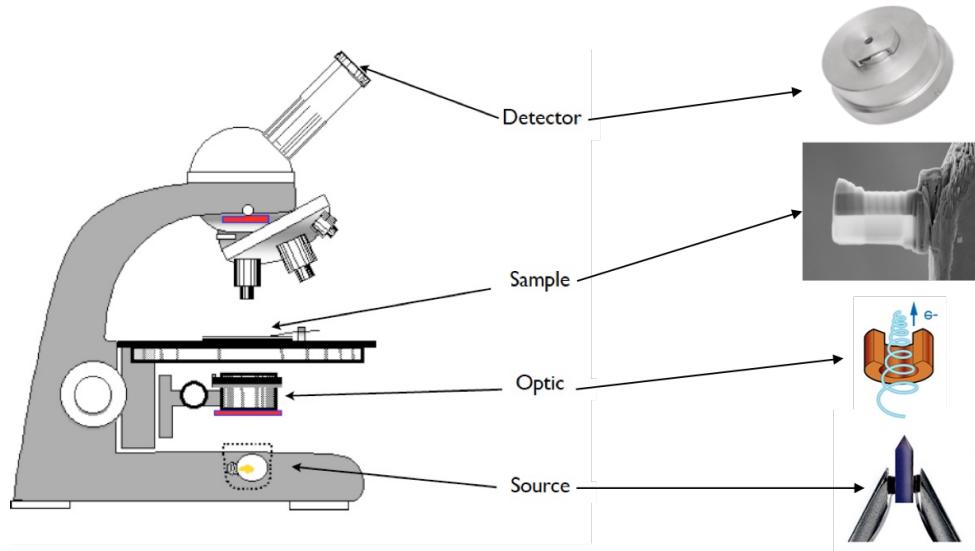
Energy-dispersive X-ray (EDX) analysis in STEM

Electron energy-loss spectroscopy (EELS)

*In situ* TEM

Some examples and Summary

# Introduction to TEM

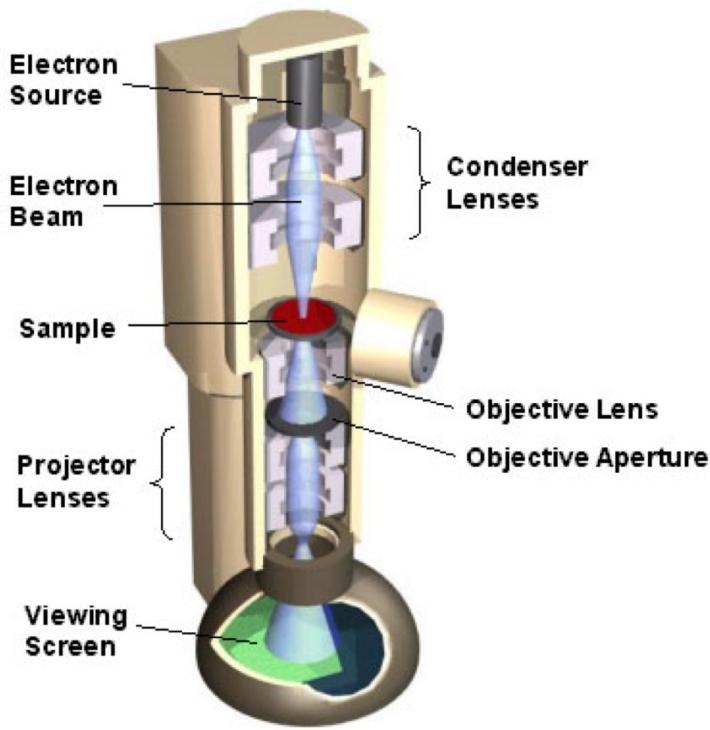


Visible light

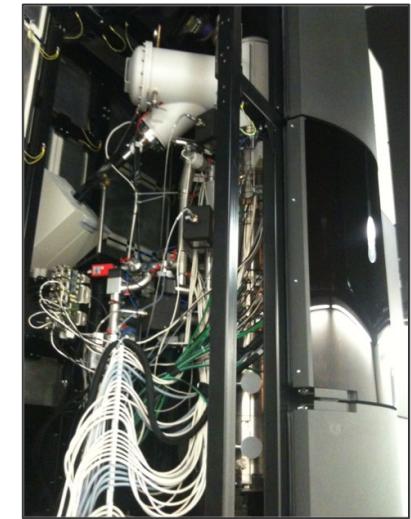


Fast electrons  
100–300 keV  
( $0.5$ – $0.8 c$  !)

# Introduction to TEM



Double Cs-corrected FEI Titan-Themis @ CIME-EPFL



Specimen should be electron transparent:  
several nanometers thick

# Why fast electrons?

	Advantageous	Disadvantageous
Visible light	Not very damaging Easily focused Eye detector	Long wavelengths (400 nm)
X-rays	Small wavelength (Angstrom) Good penetration	Hard to focus Damage sample
Neutrons	Low sample damage Small wavelength (pm)	How to produce? How to focus?
Electrons	Small wavelength (pm) Can be focused to a sub-Å size probe Wave-particle duality	Damage sample Poor penetration (< x00 nm)

High energy electrons have a short wavelength (e.g. 2.5 pm @ 200 keV)

Easy to produce high brightness electron beams

Easy to manipulate: focused

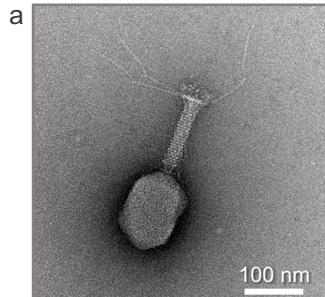
**Interact strongly with matter**

Electron microscopes are used not only for obtaining good resolution images but also:

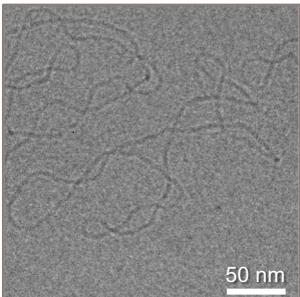
- **can be used as a diffractometer**
- **for chemical analyses (EDX and EELS)**
- **for imaging/measuring electric-, magnetic-, or strain-field in the sample**
- **for measuring optoelectronic properties (e.g. cathodoluminescence), ...**

**\*Aberrations & instabilities → Actual resolution around 25-50 Å, but atomic/sub-Å resolution possible in TEM**

## Biology and life science

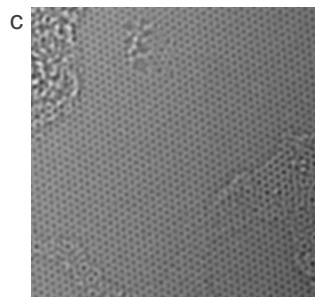


T4 virus <sup>a</sup>  
A bacteriophage that infects Escherichia coli bacteria

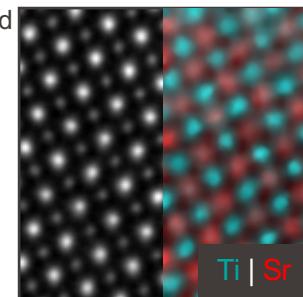


DNA molecule <sup>a</sup>

## Chemistry &amp; materials research

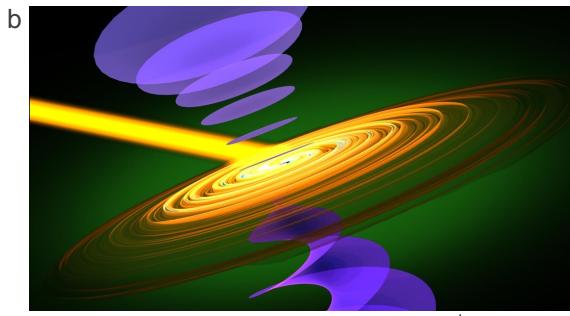


Imaging and spectroscopy at atomic resolution <sup>c,d</sup>



Ti | Sr

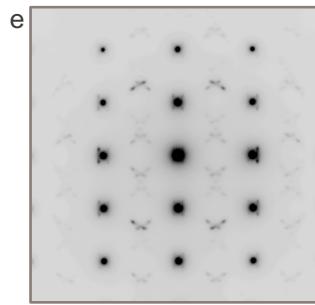
## Fundamental physics



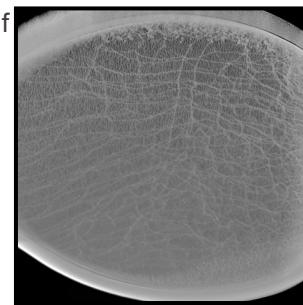
Ultrafast electron vortex beams <sup>b</sup>

<sup>a</sup> Images courtesy of Dr. D. Demurtas, CIME-EPFL.

<sup>b</sup> Vanacore et al., Nature Materials (2019).



Crystallographic studies <sup>e</sup>

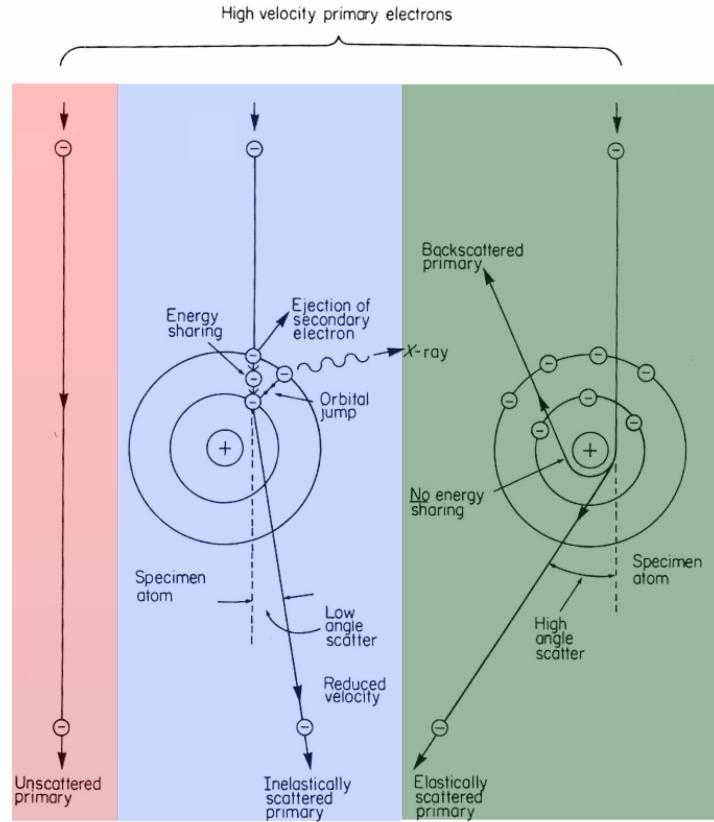


Analyzing crystal defects <sup>f</sup>

<sup>c</sup> Huang et al., Nat. Comm. 2018.

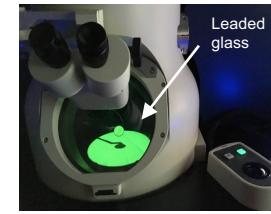
<sup>f</sup> Oveisi et al., Scripta Mat. (2013).

<sup>d</sup> Bencan et al., Nat. Comm. (2021).



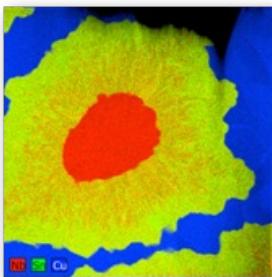
**Inelastic events:** The result is a **transfer of beam energy** to the specimen atom (**energy loss**) and a potential expulsion of an electron from that atom as a **secondary electron (SE)**.

If the vacancy due to the creation of a secondary electron is filled from a higher level orbital, an X-Ray or Auger characteristic of that energy transition is produced.



That's why TEMs are shielded!

Characteristic X-rays



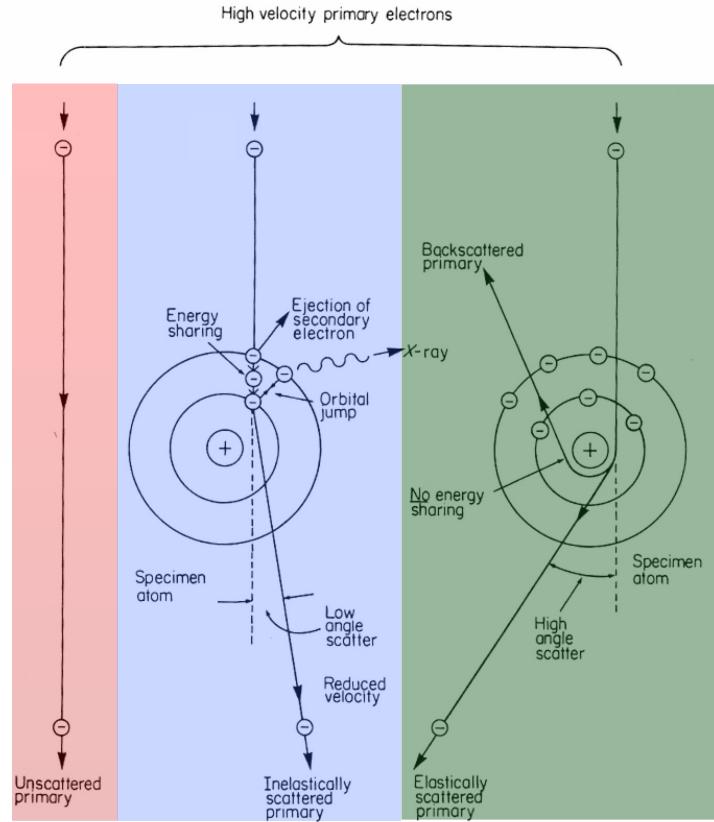
Chemical composition

Electron energy-loss spectrum



Chemical composition, band-gap & optical properties

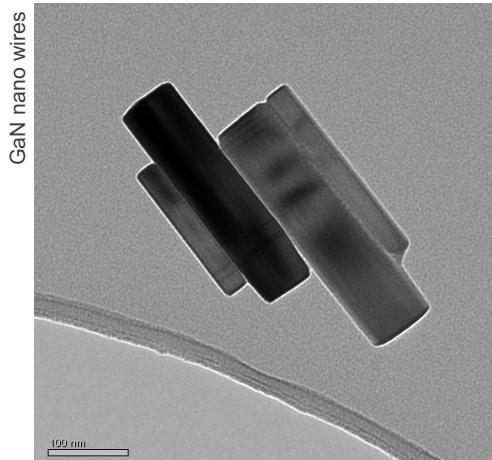
# e- matter interactions



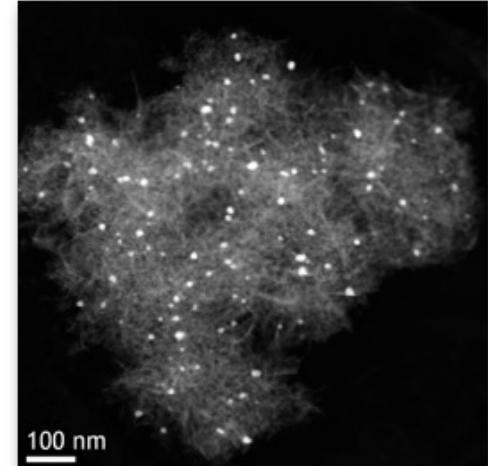
**Elastic events** occur when a beam electron interacts with the electric field of the nucleus or electron cloud of a specimen atom (Coulomb forces), resulting in a change in the direction of the beam electron **without a significant change in the energy** of the beam electron (< 1 eV).

Coulombic interaction within the electron cloud, Low-angle scattering  
Coulombic attraction by the nucleus, Higher-angle scattering

Thicker specimen or larger nucleus → More scattering



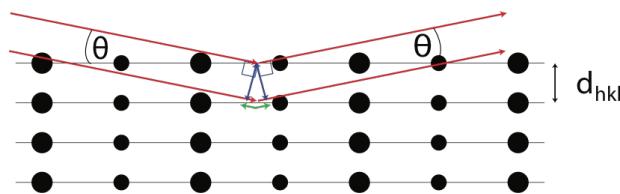
Mass/thickness contrast  
Bright-field TEM image



Mass/thickness contrast  
HAADF-STEM image

# e<sup>-</sup> as wave & Bragg scattering

For X-ray diffractometer



### X-ray scattering:

Path difference between reflection from planes distance  $d_{hkl}$  apart  
 $= 2d_{hkl} \sin\theta$

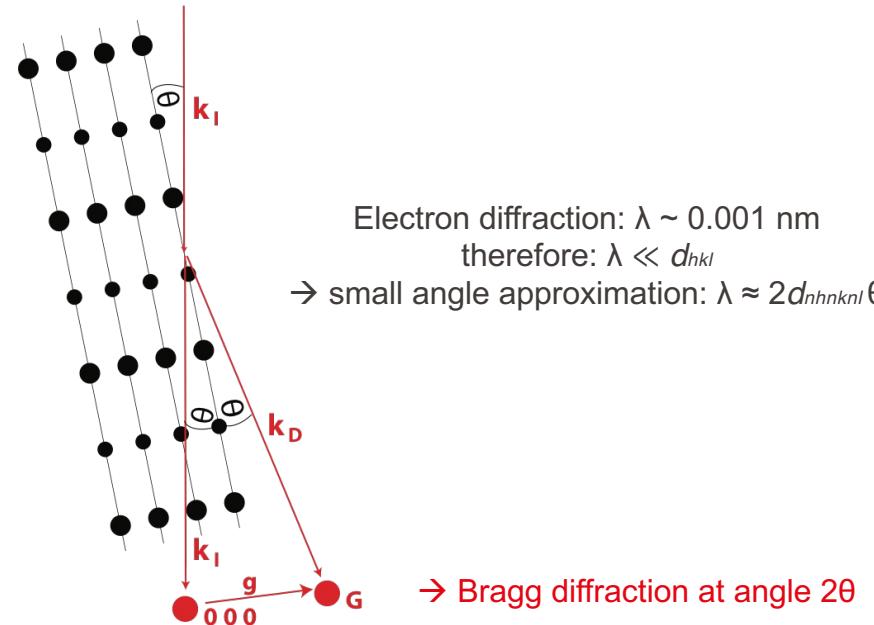
### Bragg law

$$n\lambda = 2d_{hkl} \sin\theta$$

or

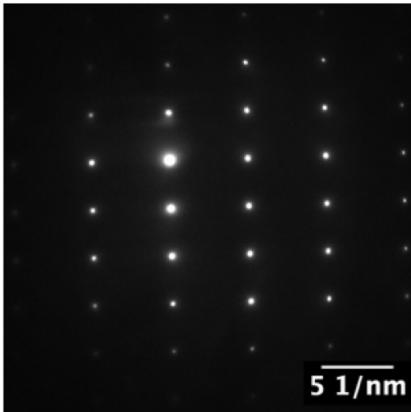
$$\lambda = 2d_{hnl} \sin\theta$$

For TEM electrons come from top, but otherwise geometrically the same

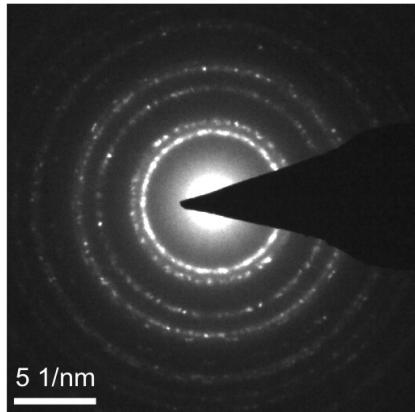


# e<sup>-</sup> as wave & Bragg scattering

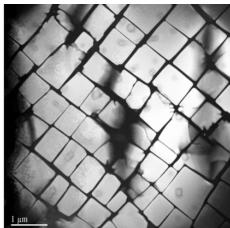
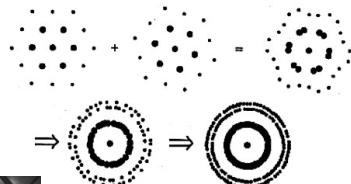
Electron diffraction pattern



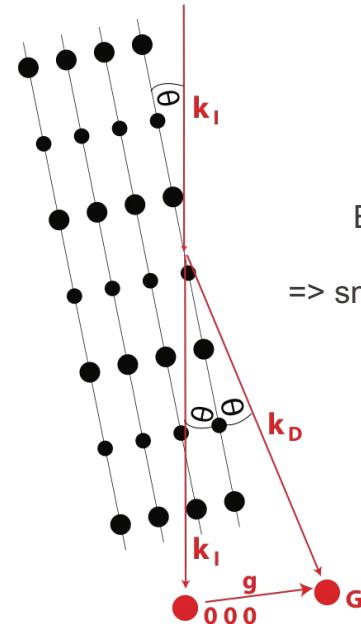
Spot pattern from a single crystal  
Spots represent different  $(hkl)$  planes



Ring pattern as many crystallites oriented  
differently in diffraction conditions



Therefore TEM gives image & diffraction!



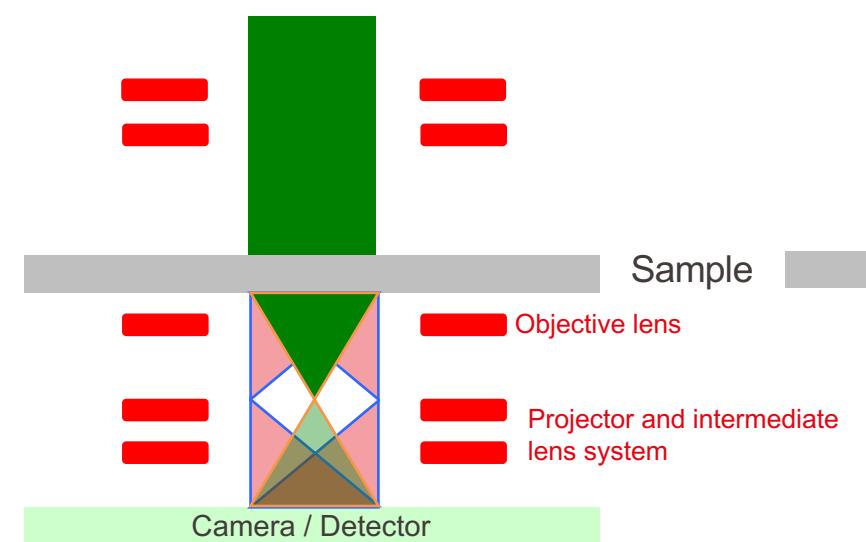
Electron diffraction:  $\lambda \sim 0.001$  nm  
therefore:  $\lambda \ll d_{hkl}$

=> small angle approximation:  $\lambda \approx 2d_{hkhkn}\theta$

=> Bragg diffraction at angle  $2\theta$

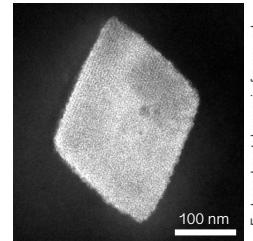
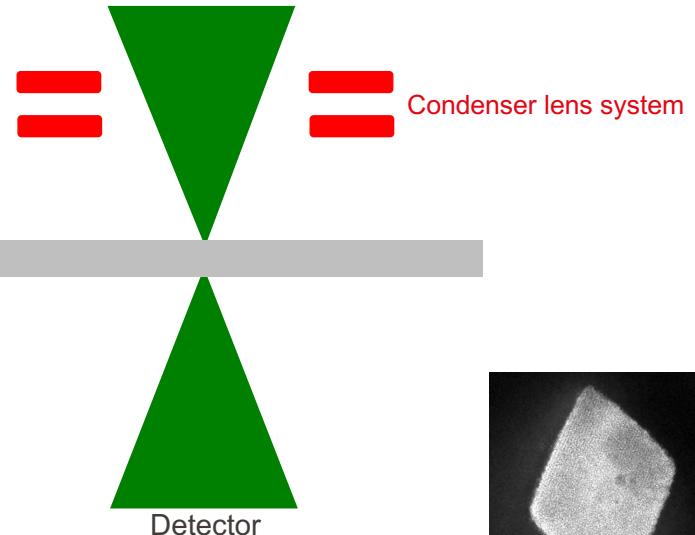
# TEM operation modes

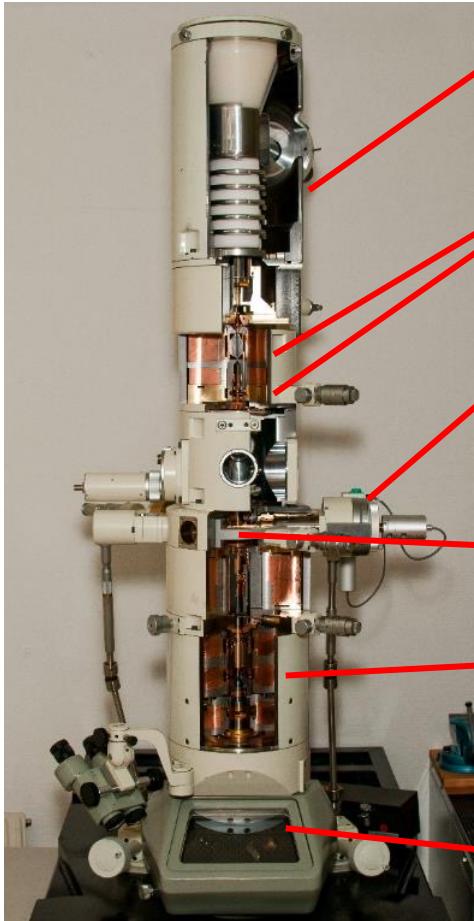
## Conventional TEM



## Scanning mode (STEM)

Electron beam (30-300 keV)



**Electron Source**

Produces high energy, large current, and high coherence electron beams necessary for generating diffraction patterns and high spatial resolution images

**Condenser system and condenser aperture**

Controls spot size and illumination area on sample (beam intensity)

**Specimen holder and goniometer****Objective lens and objective aperture**

Images sample and is the strongest lens in the system

**Intermediate and projector lenses**

Changes modes from diffraction to imaging

Controls magnification

**Detectors / Camera**

Various different configurations designed to collect signals produced by the high-energy electron beam

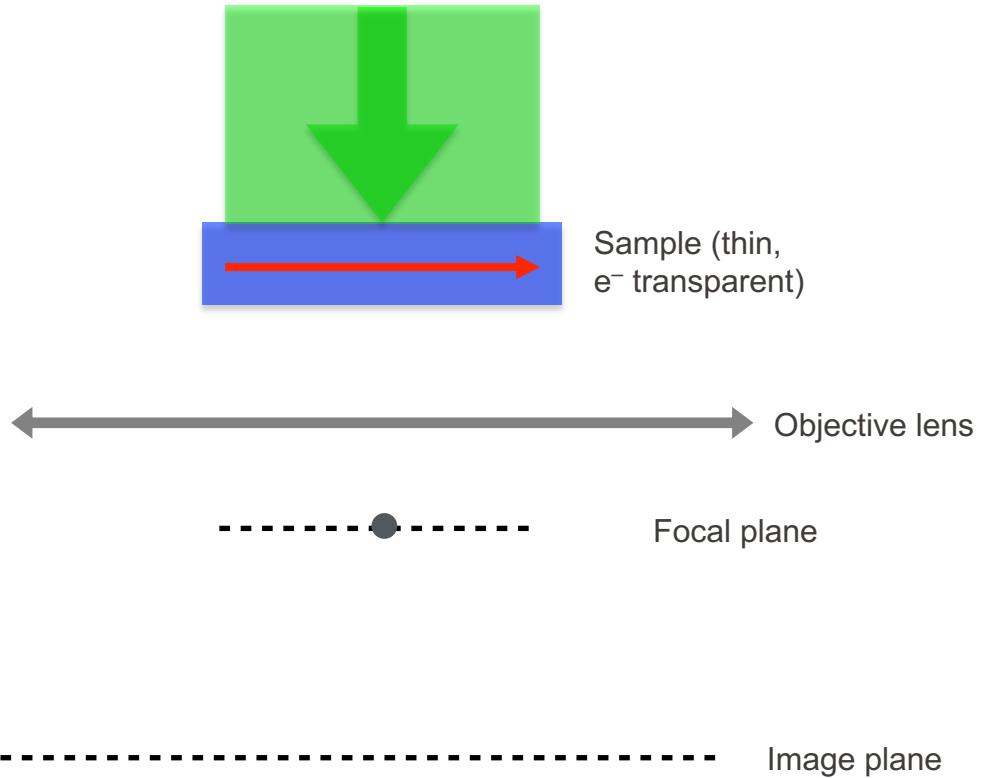
## ▪ Image formation in TEM

- Image and diffraction modes
- Bright- and dark-field modes
- High-resolution TEM

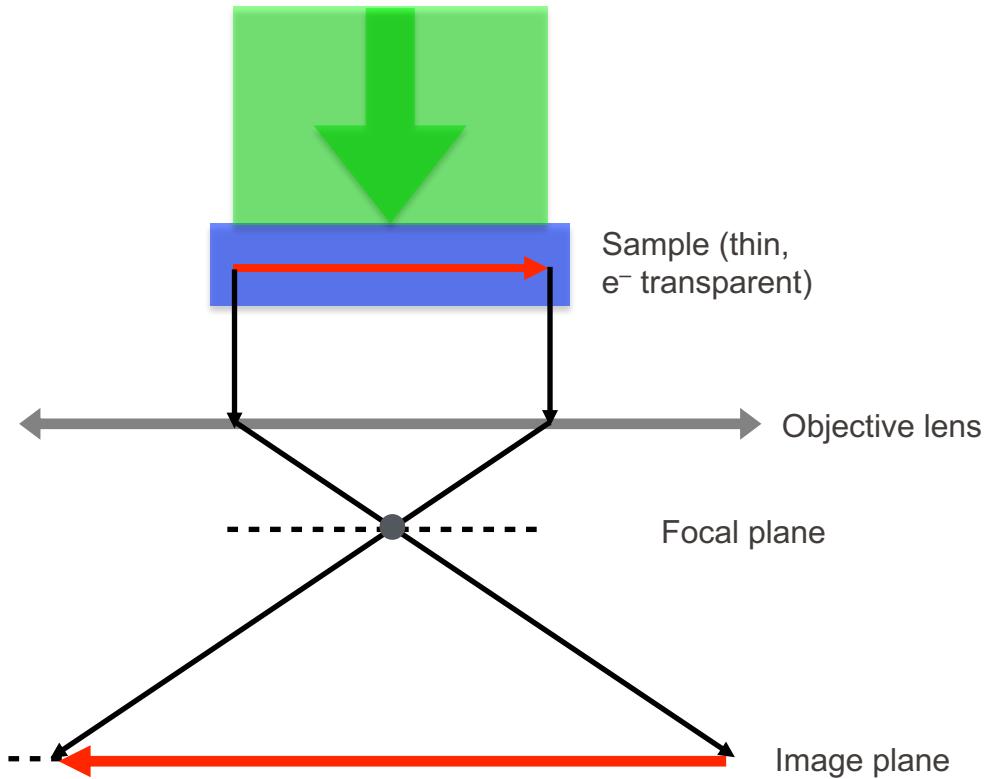
## ▪ Image contrast in TEM

- Mass-thickness contrast
- Diffraction contrast
- Phase contrast

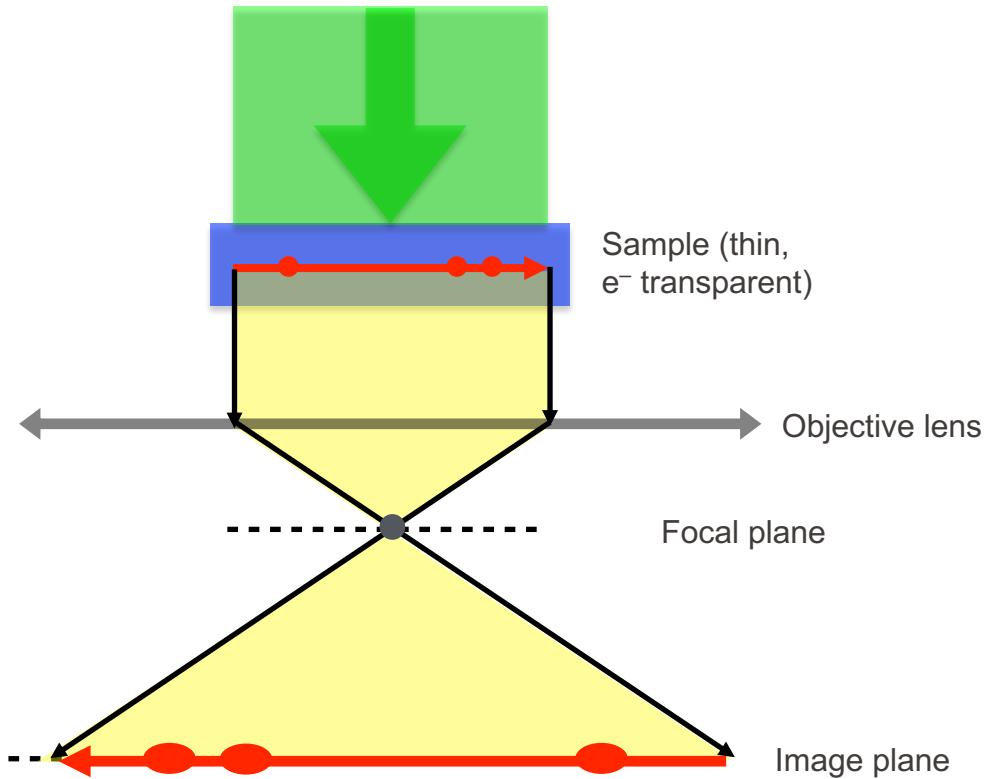
Parallel incident  $e^-$  beam;  $\lambda \approx 0.02\text{--}0.03 \text{ \AA}$



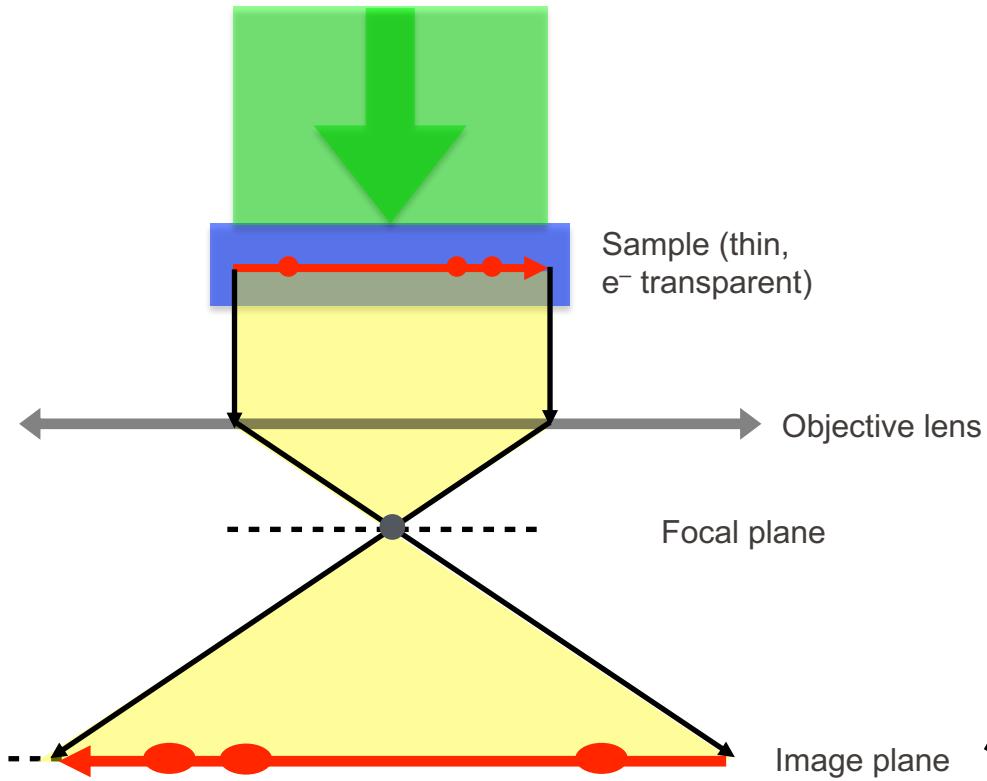
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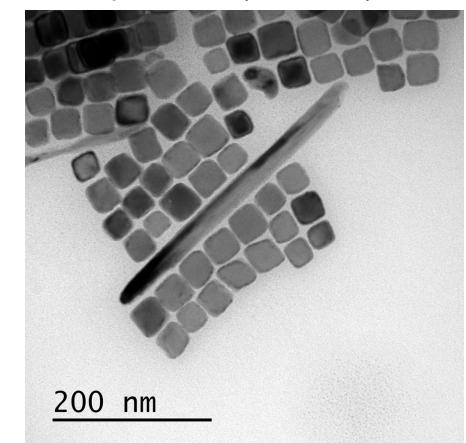


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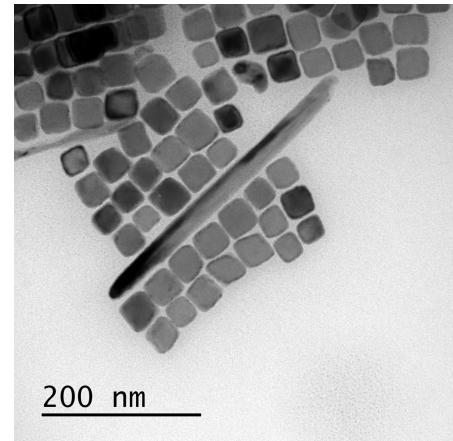


Why some cubic particles appear darker than others?

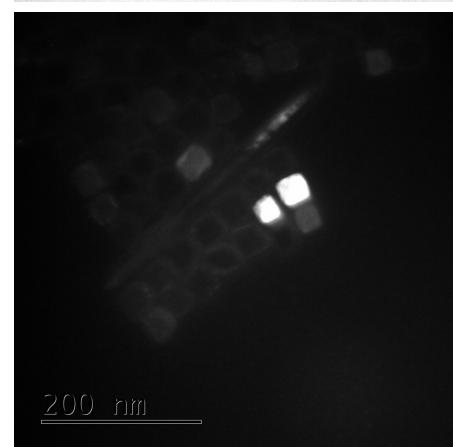
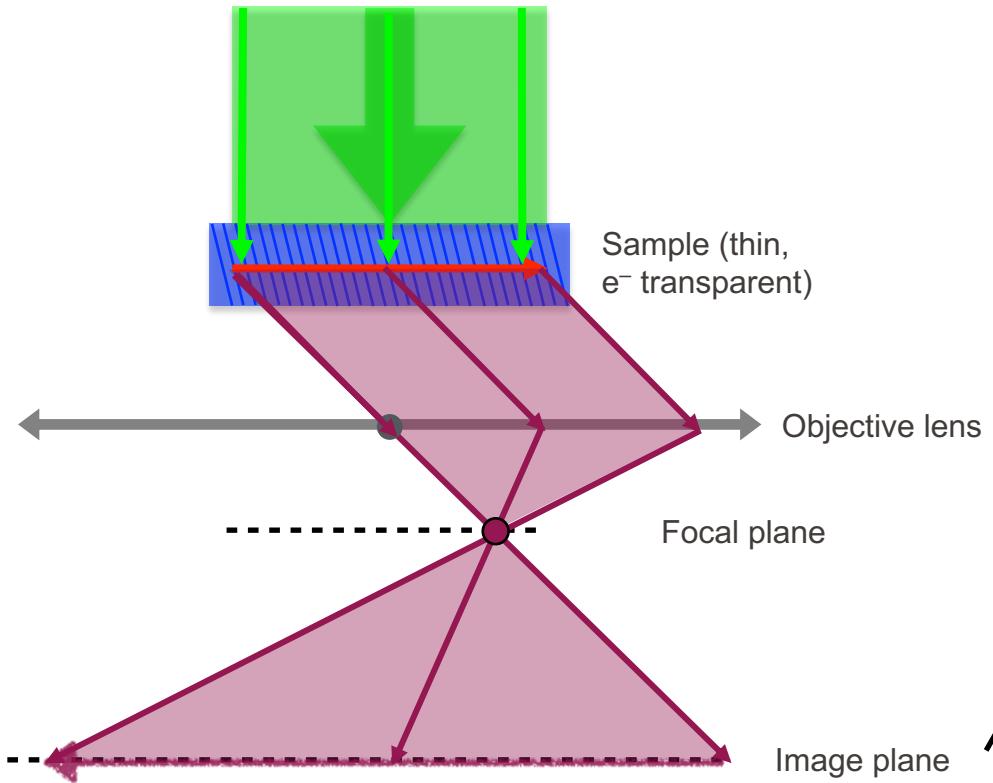
Cubic particles ( $\approx 40 \text{ nm}$ ) of Cu



Bright-field image

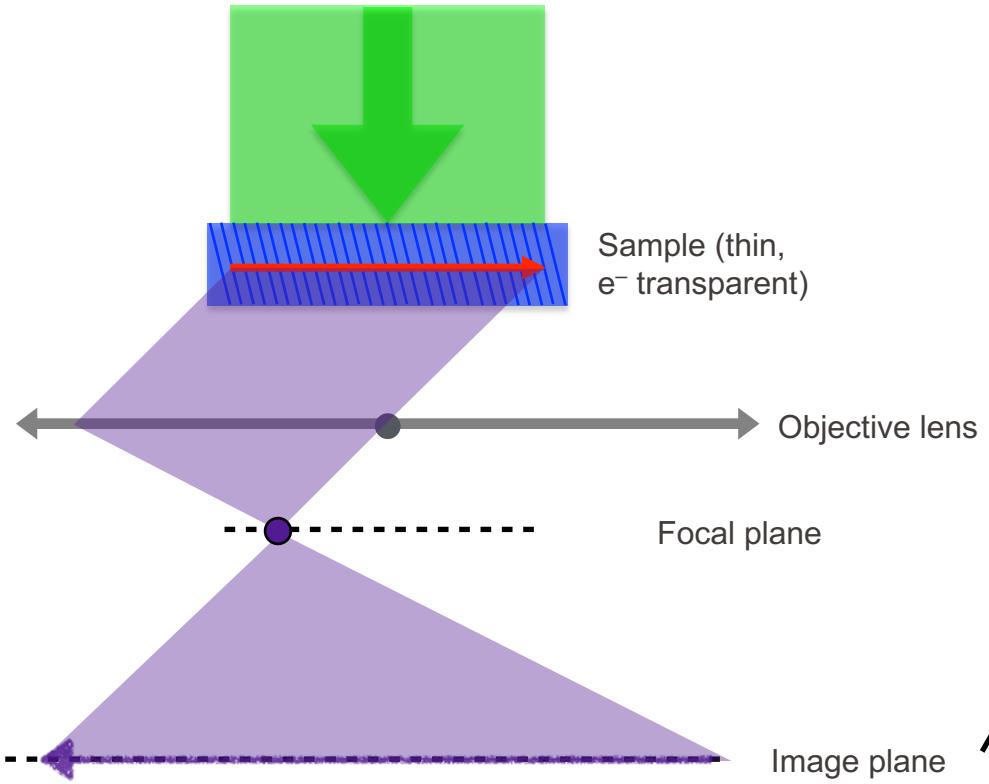


Parallel incident  $e^-$  beam;  $\lambda \approx 0.02\text{--}0.03 \text{ \AA}$

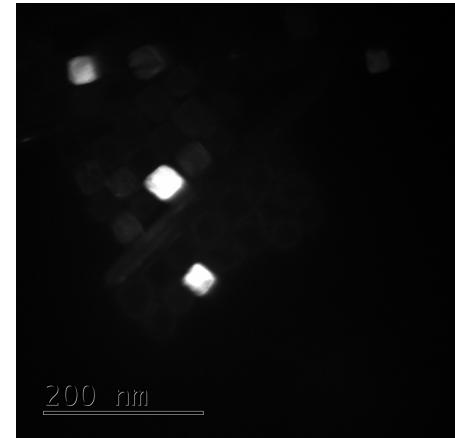
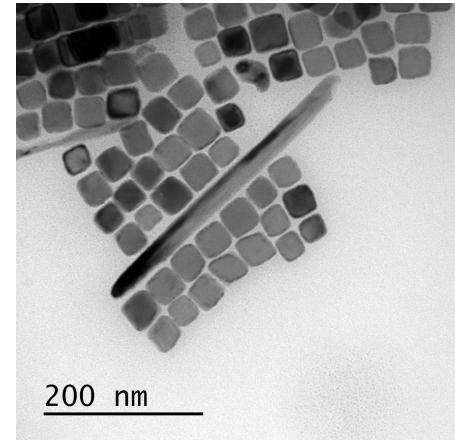


Dark-field image:  
made by selected diffracted electrons

Parallel incident  $e^-$  beam;  $\lambda \approx 0.02\text{--}0.03 \text{ \AA}$

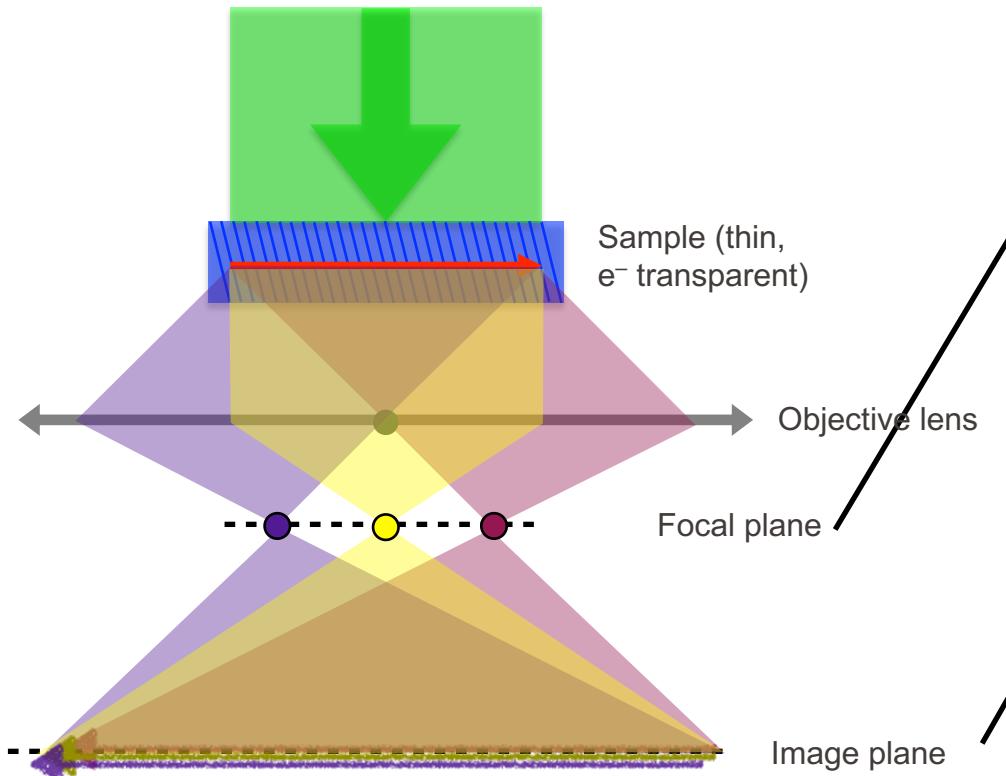


Bright-field image

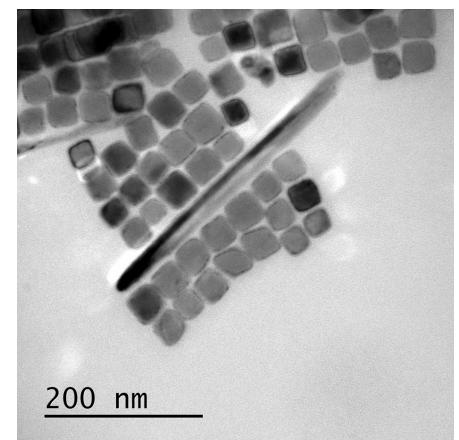
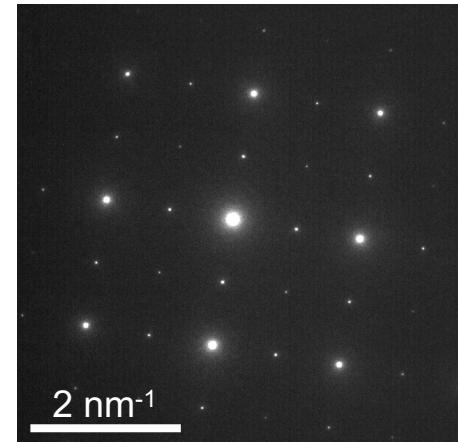


Dark-field image:  
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Parallel incident  $e^-$  beam;  $\lambda \approx 0.02\text{--}0.03 \text{ \AA}$

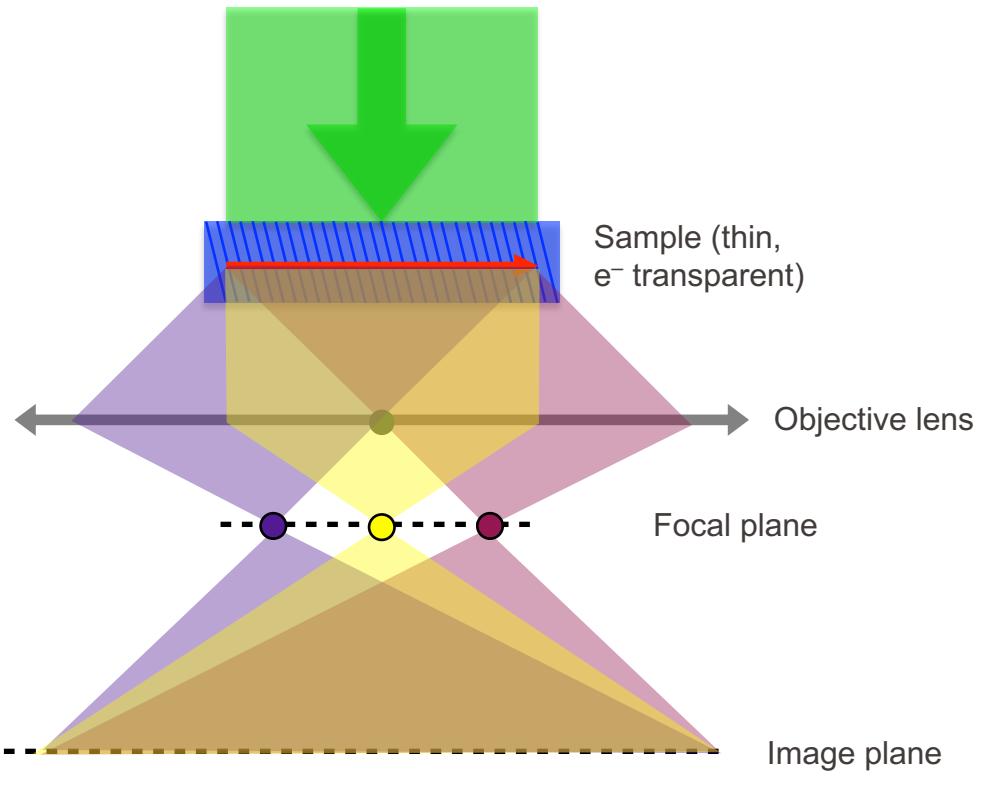


Electron diffraction pattern

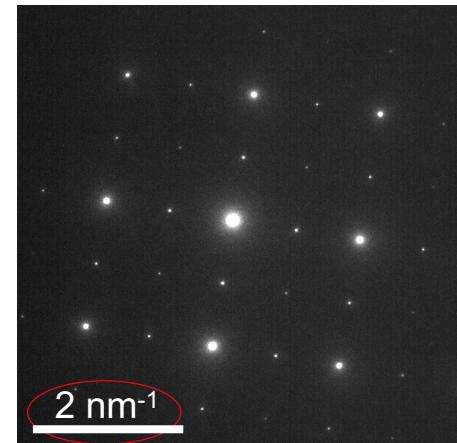


Ghost image:  
Superposition of images made  
by direct and diffracted electrons

Parallel incident  $e^-$  beam;  $\lambda \approx 0.02\text{--}0.03 \text{ \AA}$

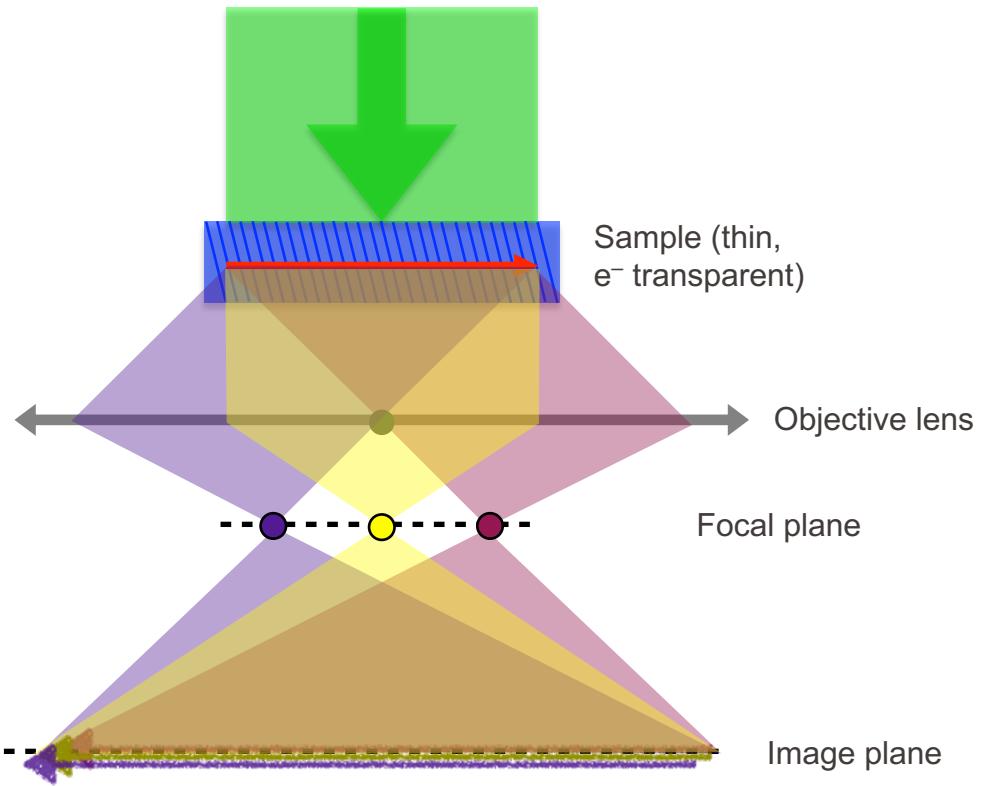


Electron diffraction pattern

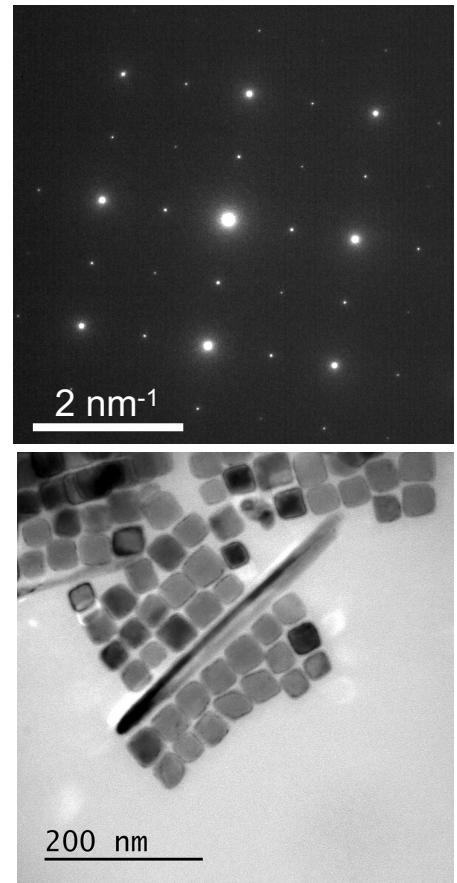


- *In back focal plane of objective lens parallel rays focused to point*
- *Diffraction – coherent scattering – creates sets of parallel rays from different crystal planes*
- *Focusing of these parallel rays in back focal plane creates spots of strong intensity: the diffraction pattern*

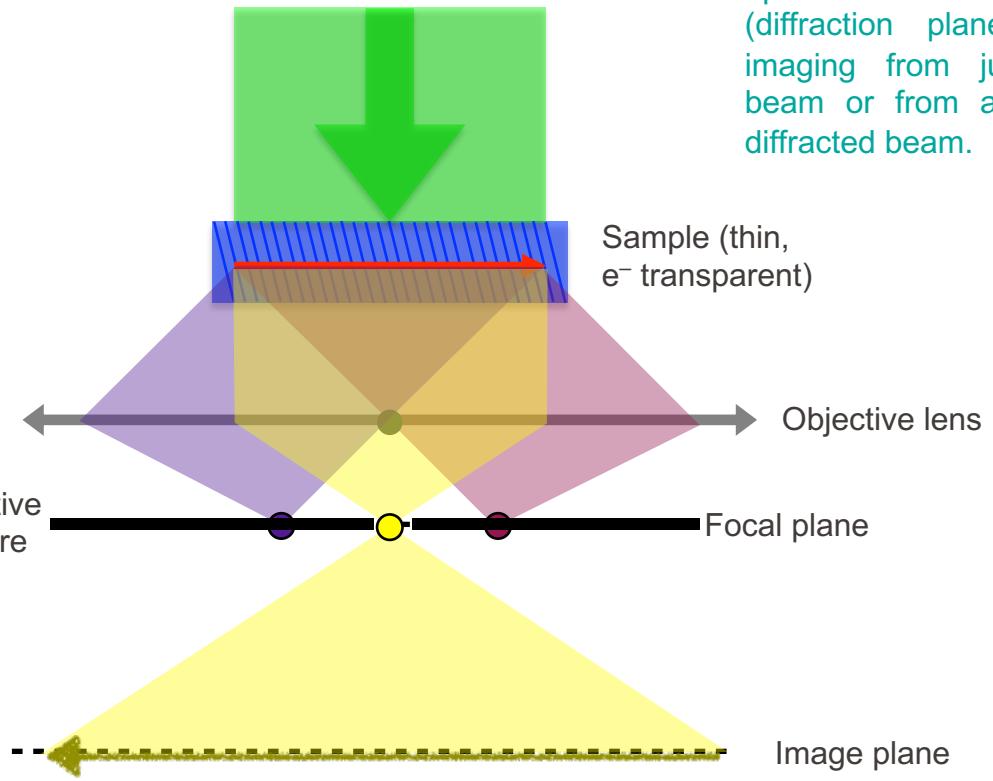
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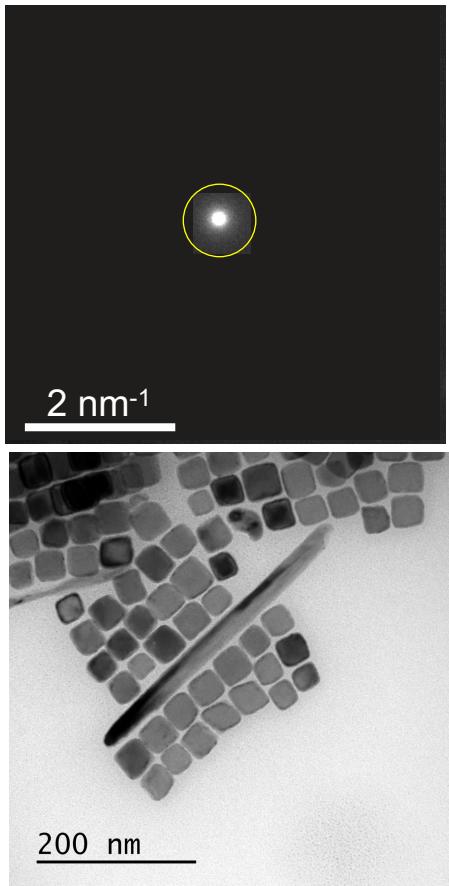
Electron diffraction pattern



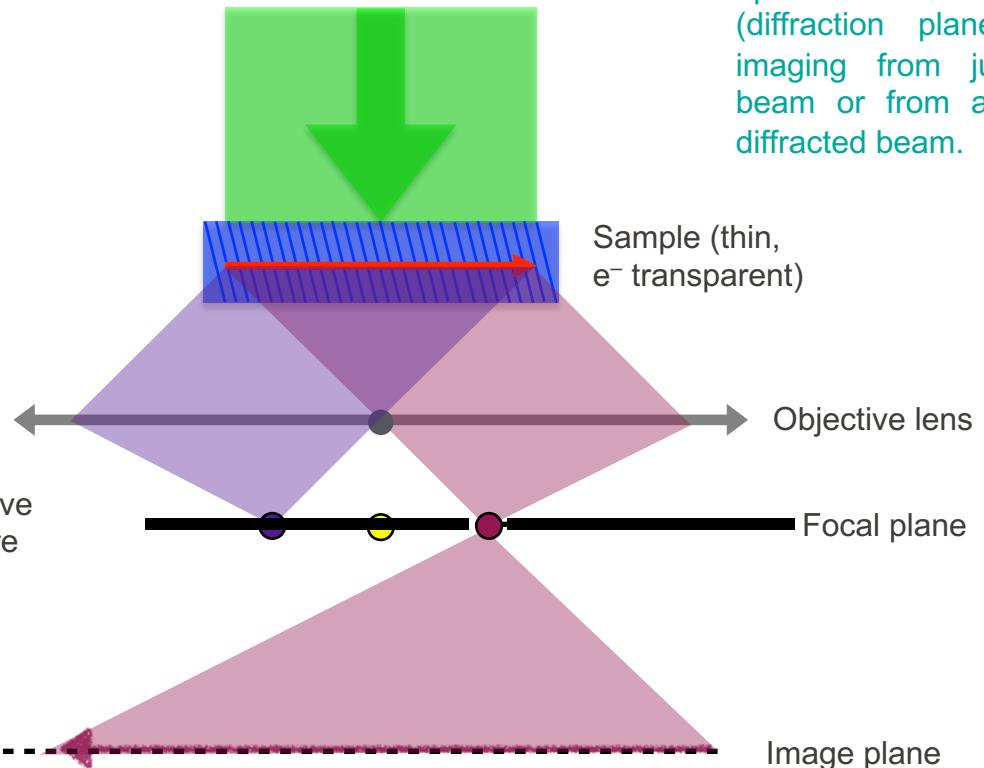
Parallel incident  $e^-$  beam;  $\lambda \approx 0.02\text{--}0.03 \text{ \AA}$



Insertion of the “objective aperture” in focal plane i.e. (diffraction plane) allows imaging from just direct beam or from a selected diffracted beam.

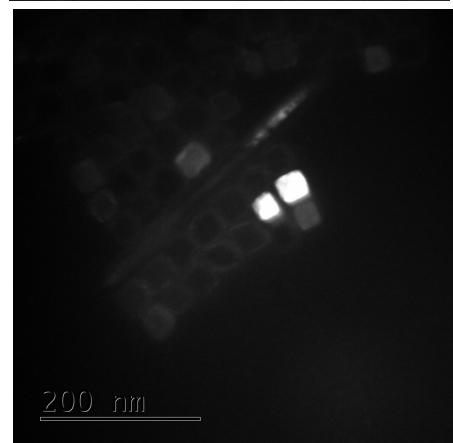
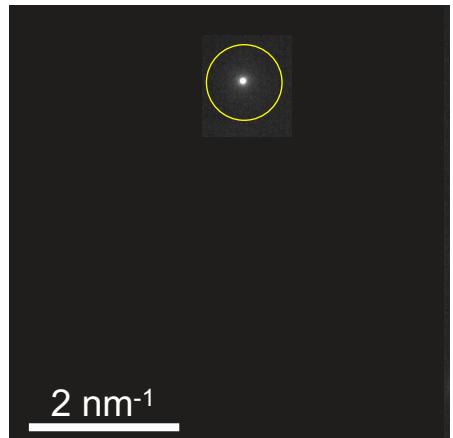


Parallel incident  $e^-$  beam;  $\lambda \approx 0.02\text{--}0.03 \text{ \AA}$

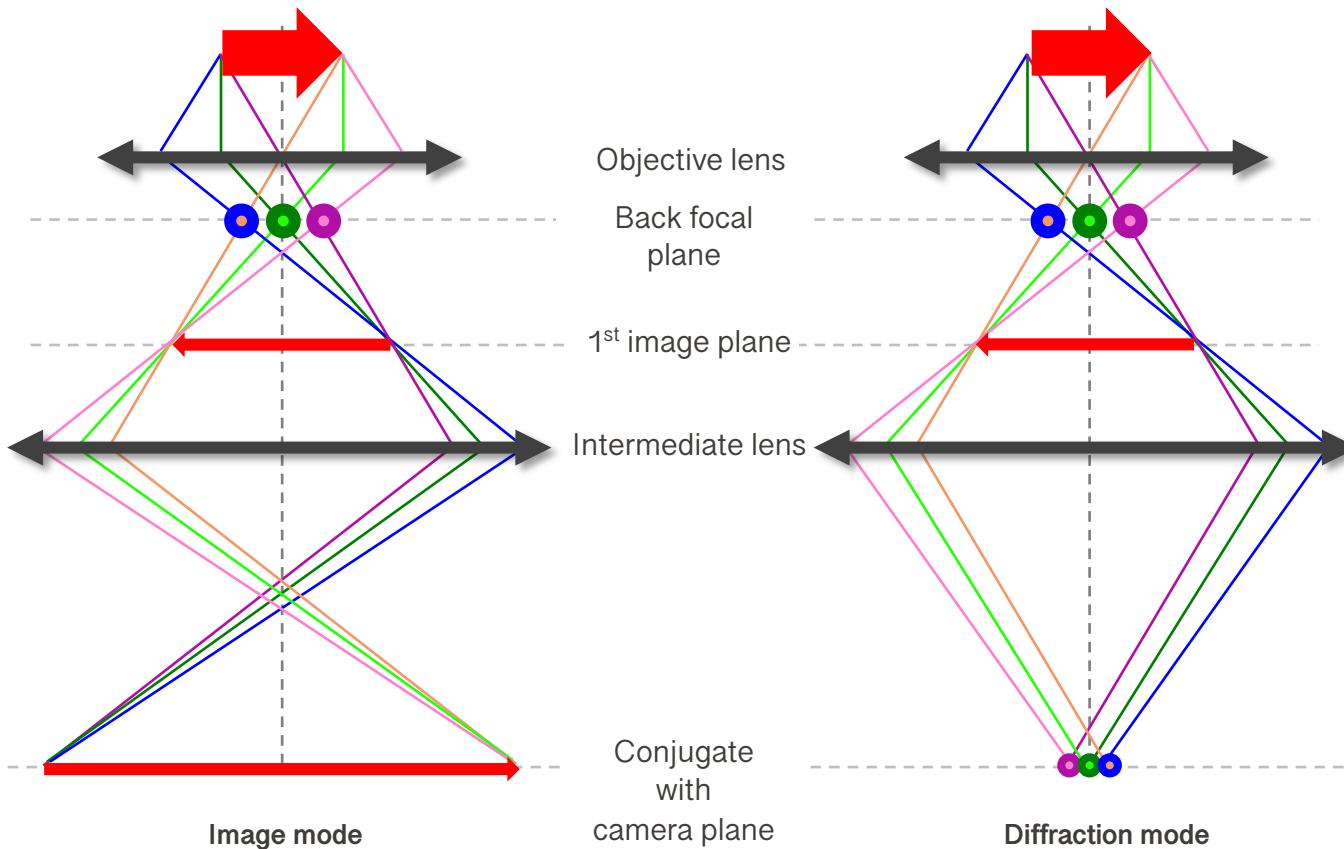


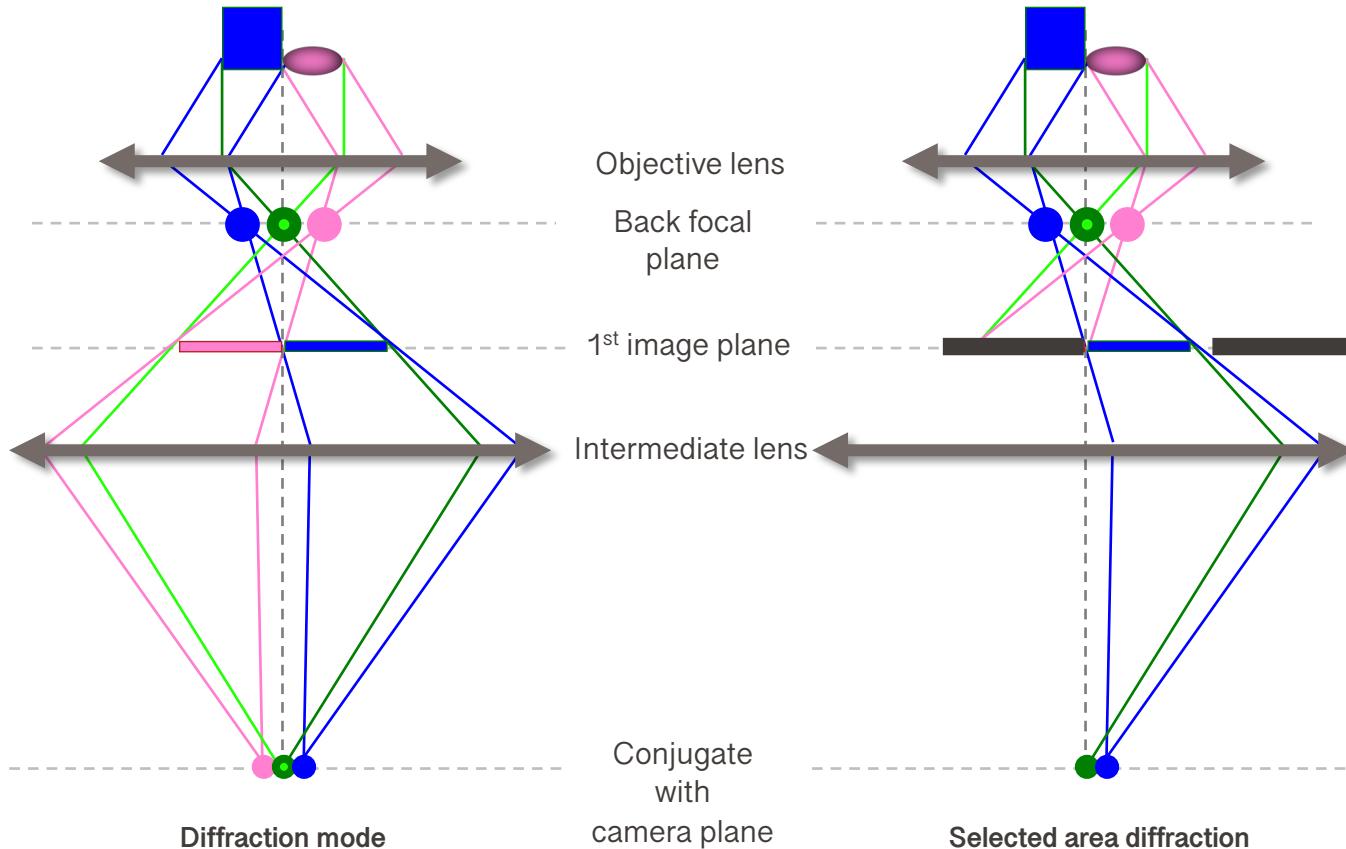
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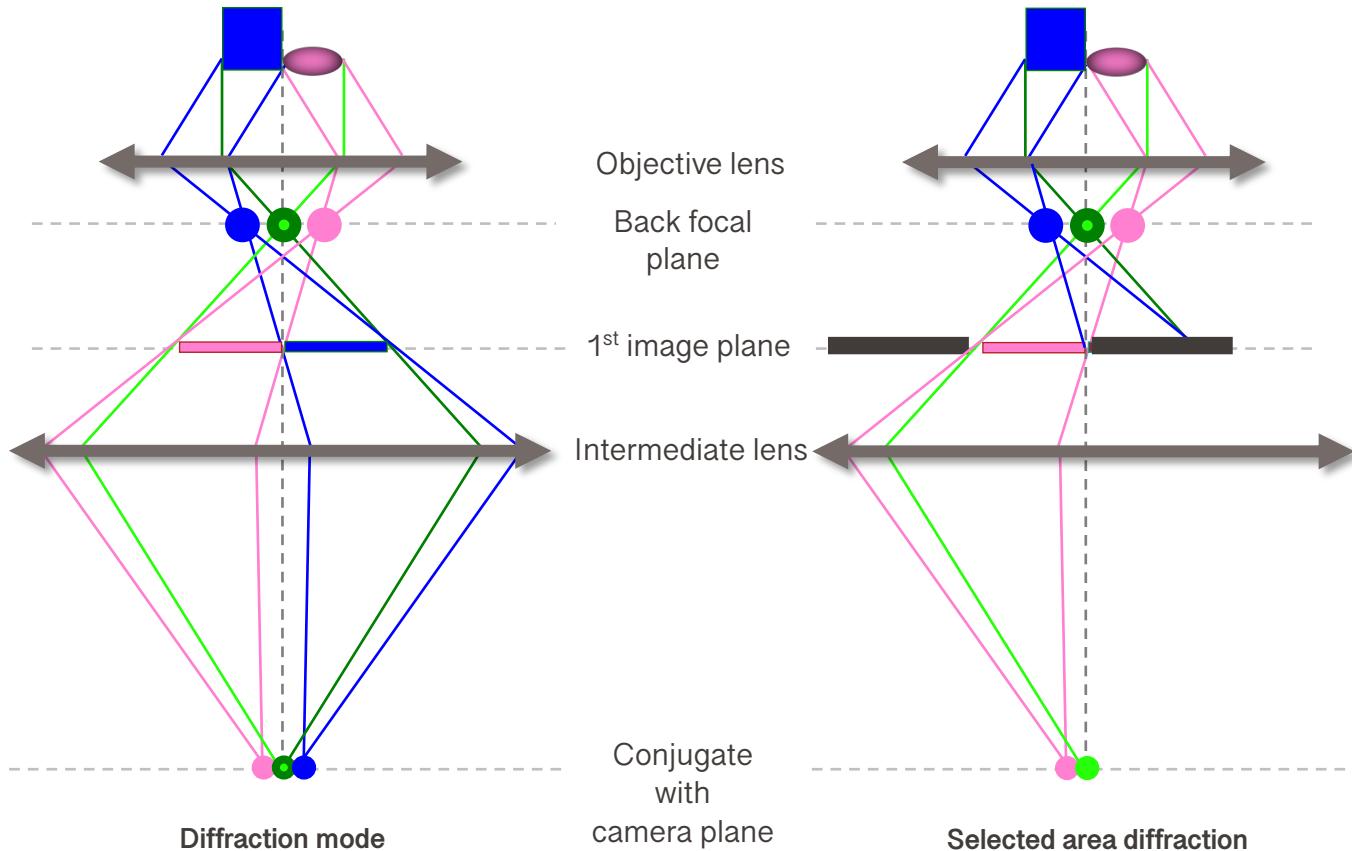
Electron diffraction pattern

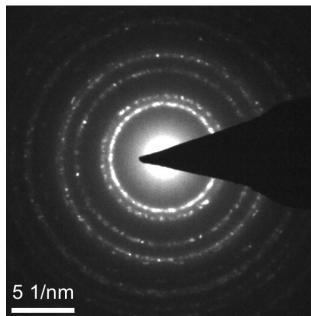
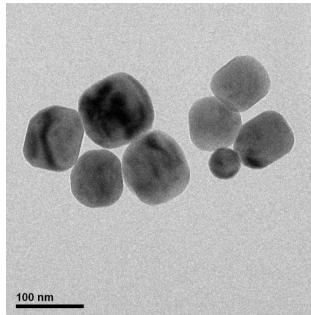
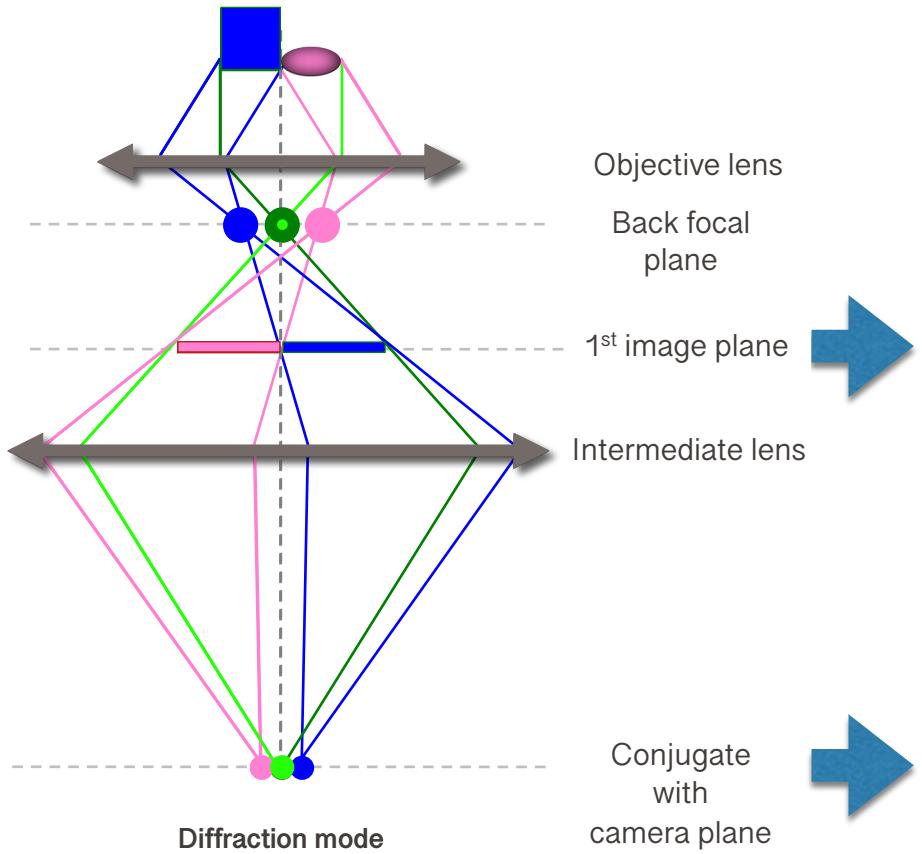


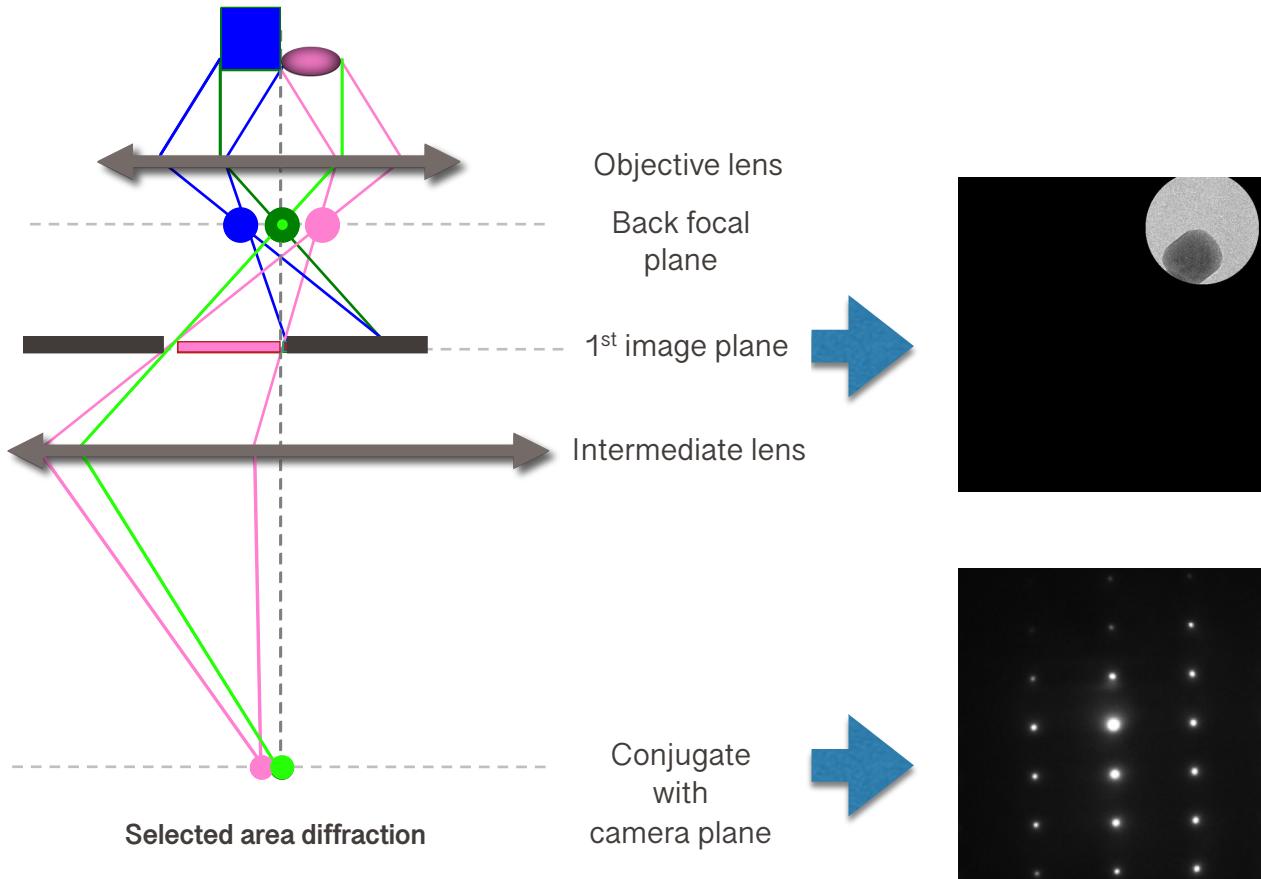
Dark-field image:  
made by selected diffracted electrons





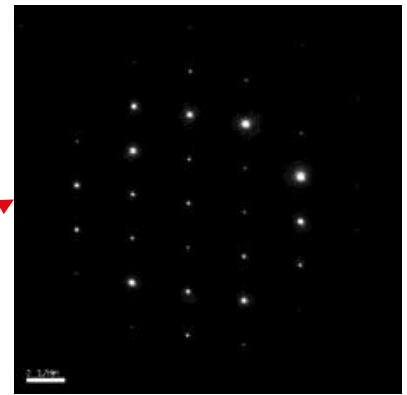
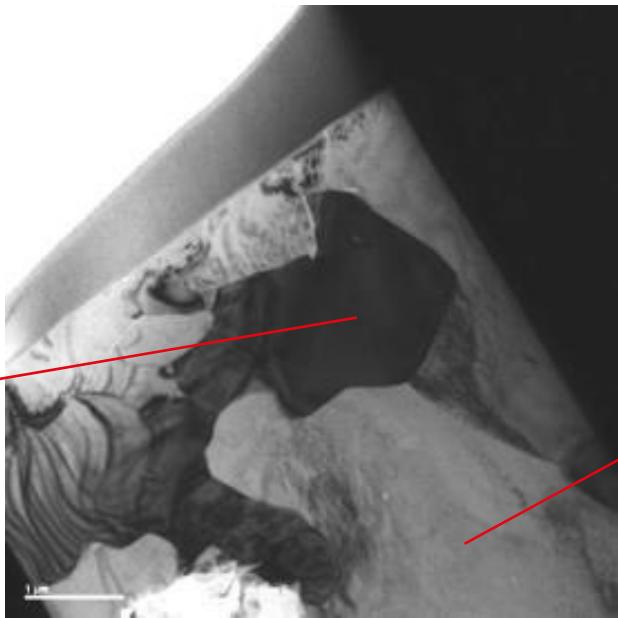






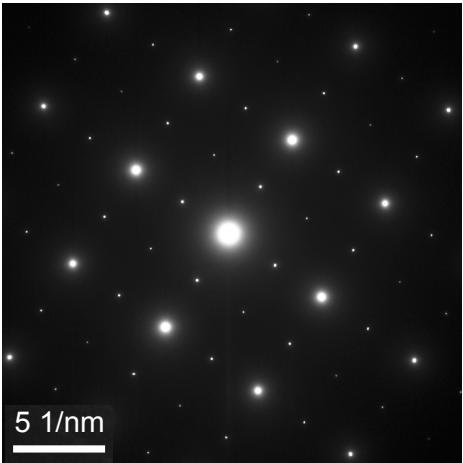


Monoclinic  $\text{Al}_3\text{Fe}$



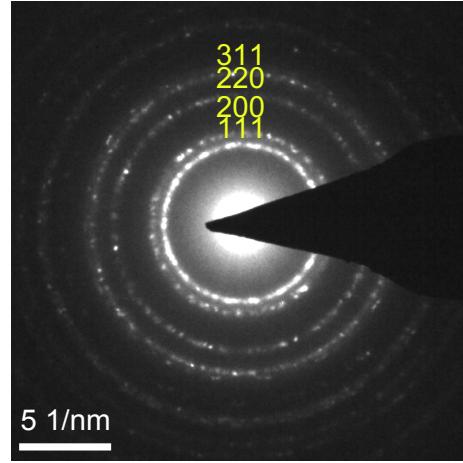
Hexagonal  $\gamma$ -(AlFeSi)

Monocrystal



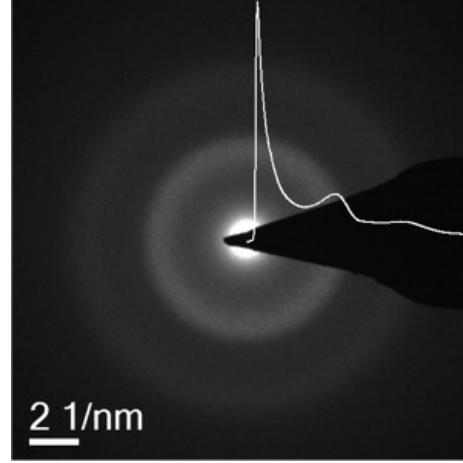
Oriented along a zone axis many  $\langle hkl \rangle$  plans in diffraction conditions. Spot represent different  $\langle hkl \rangle$  planes.

Polycrystalline

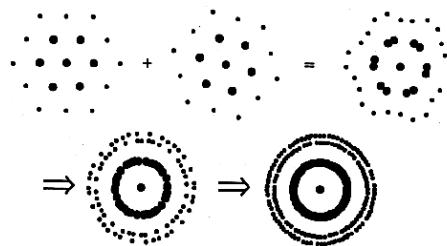


Ring pattern as many crystallites oriented differently in diffraction conditions. Each ring represents one family of  $\langle hkl \rangle$  planes

Amorphous



Diffuse ring due to short-range order as interatomic distance  $\sim$  constant  
Radial distribution function

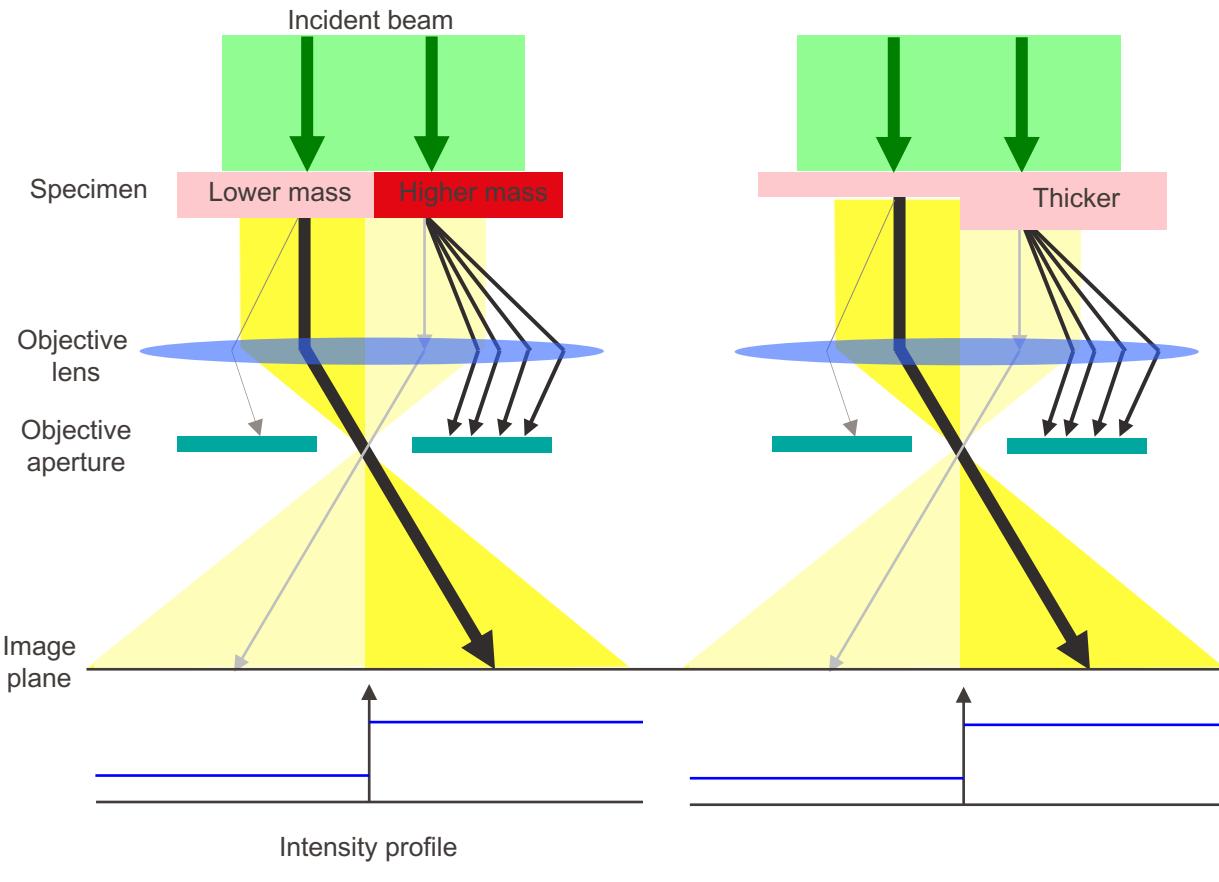


## ▪ Image formation in TEM

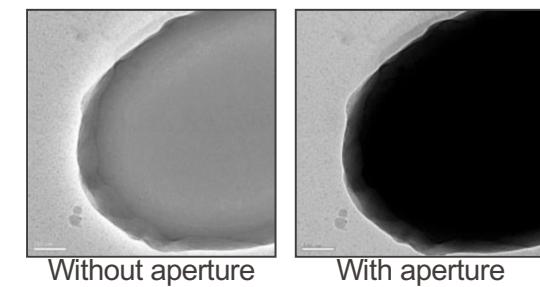
- Image and diffraction modes
- Bright- and dark-field modes
- High-resolution TEM

## ▪ Image contrast in TEM

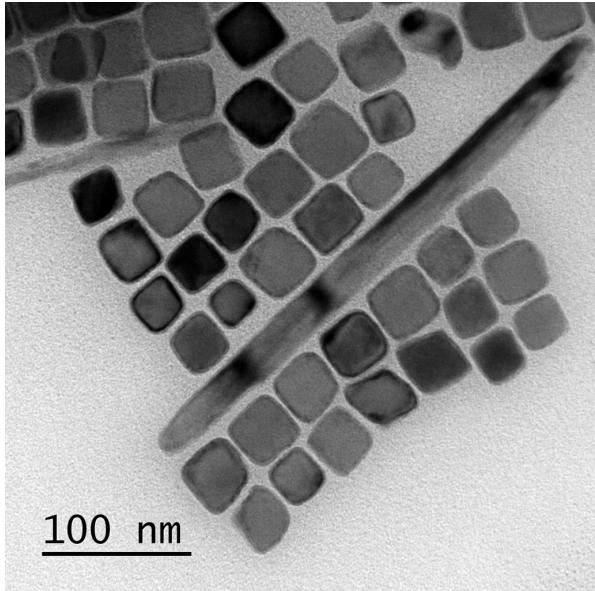
- Mass-thickness contrast
- Diffraction contrast
- Phase contrast



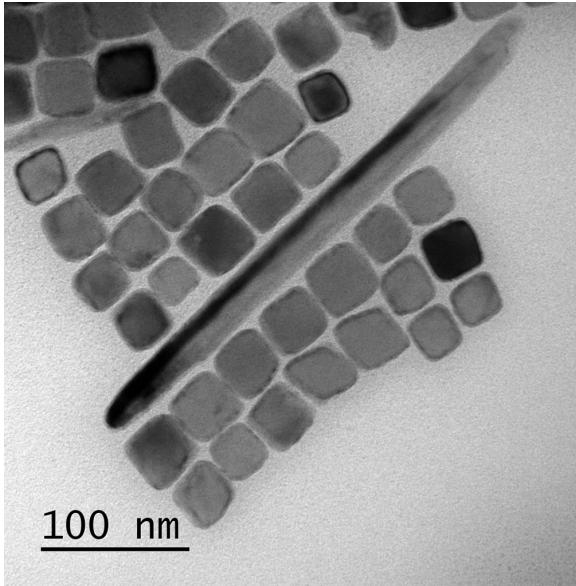
- Areas of higher mass/thickness scatter electrons more than others.
- Electrons are captured by the aperture.
- Areas of higher mass/thickness will therefore appear dark in the image formed by the direct beam.
- This is known as:
  - **mass thickness contrast**,
  - **scattering contrast**,
  - **aperture contrast** or
  - **amplitude contrast!**
- Applies to both Crystalline and Amorphous materials.



Example: Copper nano particles

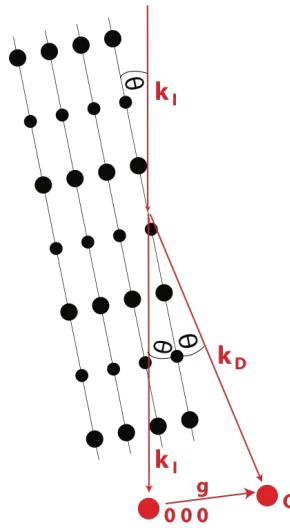


Tilting the sample



Why some of cubic particles appear darker than others?

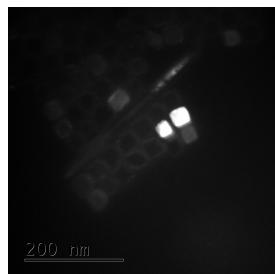
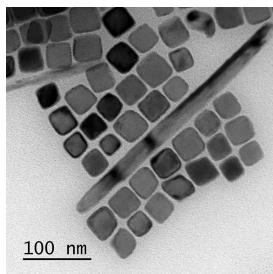
Note that contrast changes when tilting the specimen



Path difference between reflection from planes distance  $d_{hkl}$  apart  
 $= 2d_{hkl} \sin\theta$

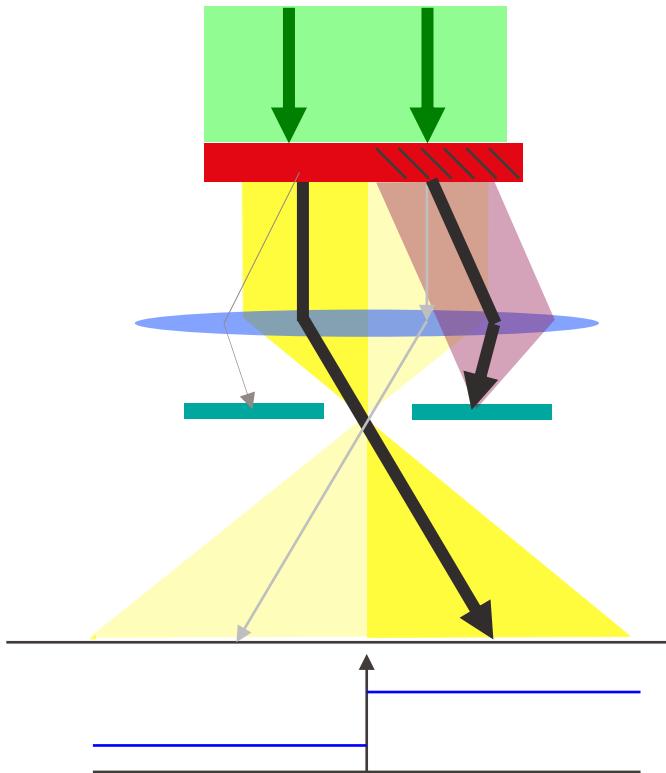
Bragg law:  $\lambda = 2d_{nhnkl} \sin\theta$

→ Bragg diffraction at angle  $2\theta$



**TEM gives image & diffraction information!**

## Diffraction contrast



### Principle of diffraction contrast imaging:

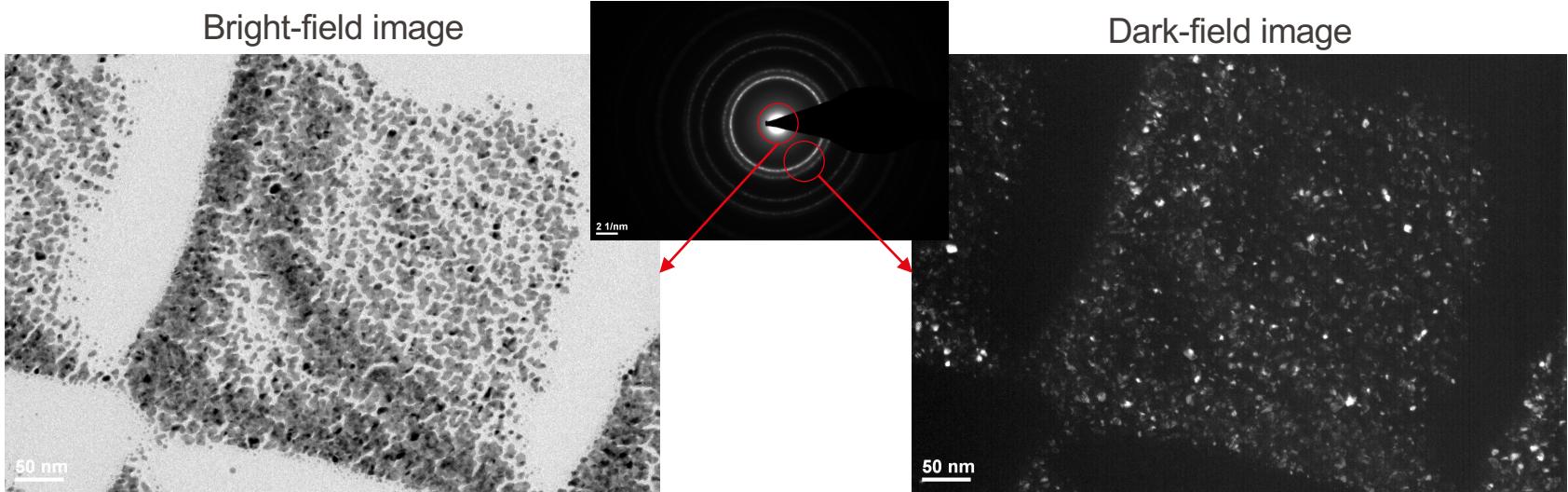
Typically we use an objective aperture to select either the direct beam or a specific diffracted beam in the back-focal plane.

If the diffraction condition changes across the sample the intensity in the selected beam changes; the intensity in the image changes correspondingly.

In other words we make a spatial map of the intensity distribution across the sample in the selected beam: *it is a mapping technique.*

In this way we can image changes in crystal phase and structural defects such as dislocations

As an example such TEM imaging was a key piece of evidence proving the existence of dislocations

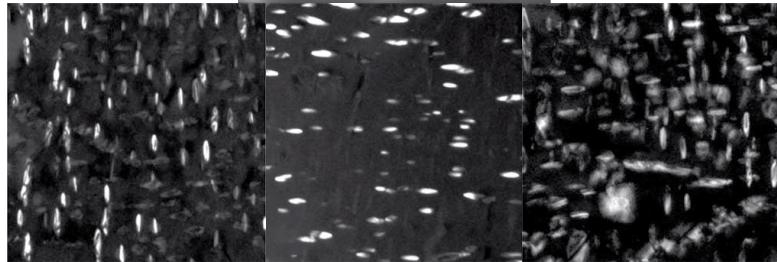
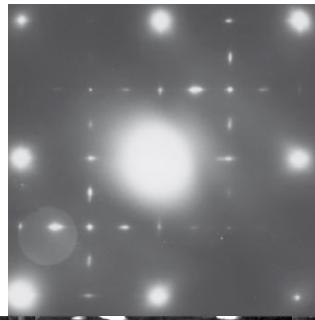


- Strongly diffracting crystals: dark
- Removes ghost image

- Only crystals strongly diffracting into objective aperture: bright
- Possibility of crystal phase/orientation discrimination

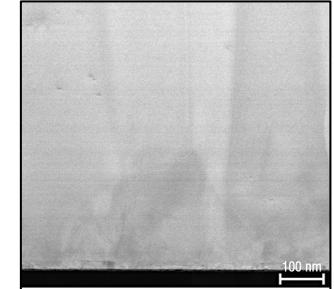
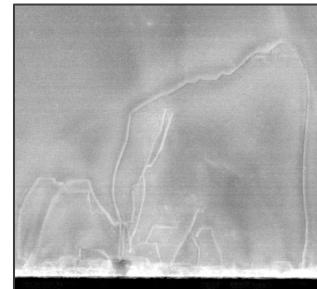
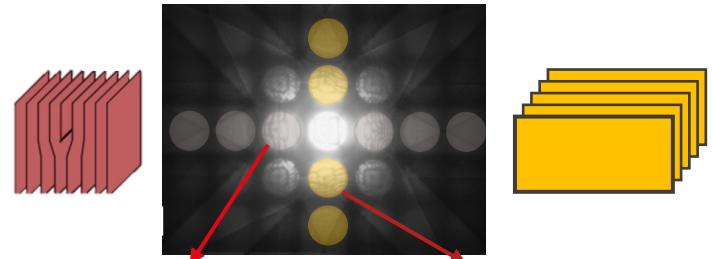
## Map intensity in the diffracted beam

Crystal phase/orientation discrimination



Al-alloy containing precipitates with preferential growth direction

Crystal's defect imaging/analysis



Dark-field images of dislocations in GaN thin film on sapphire

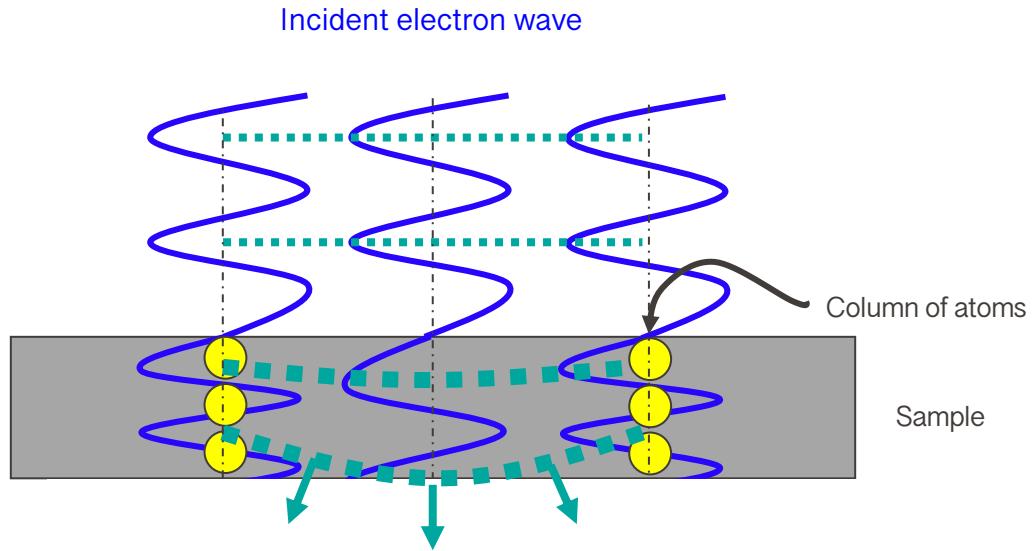
## ■ **Image formation in TEM**

- Image and diffraction modes
- Bright- and dark-field modes
- High-resolution TEM

## ■ **Image contrast in TEM**

- Mass-thickness contrast
- Diffraction contrast
- Phase contrast

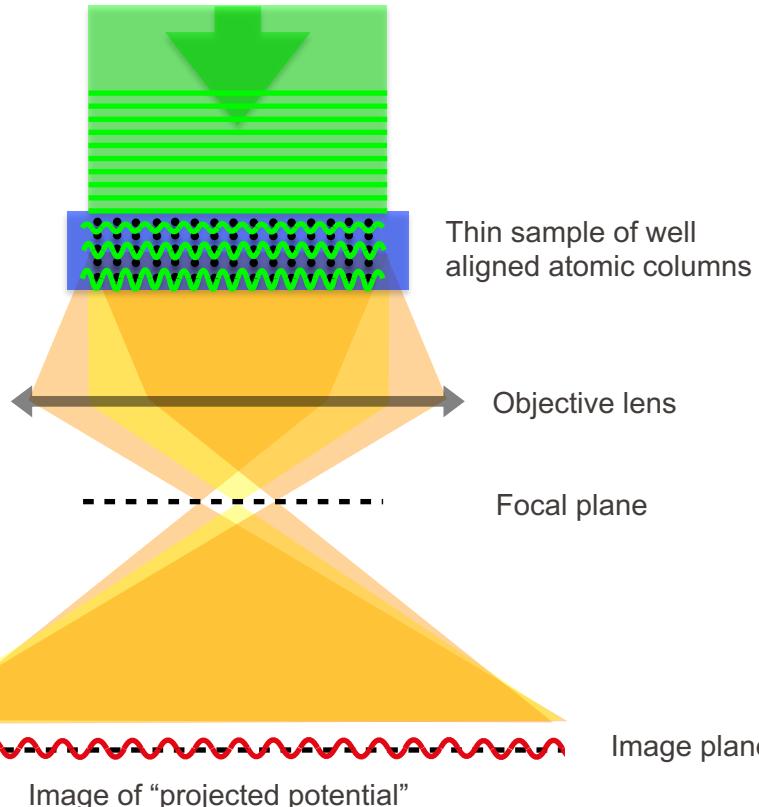
What happens when an electron wave passes through the sample?



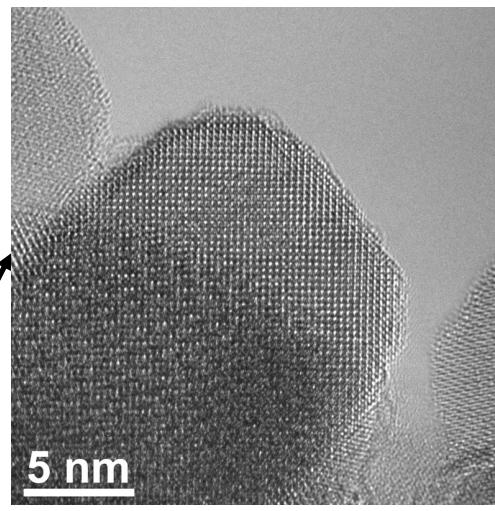
Variations in the projected potential produce local relative phase shifts of the electron wave. The wave front therefore bends as wave travels through medium.

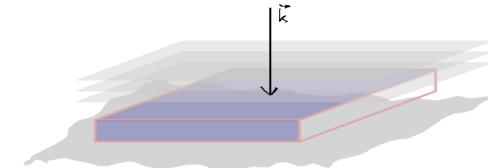
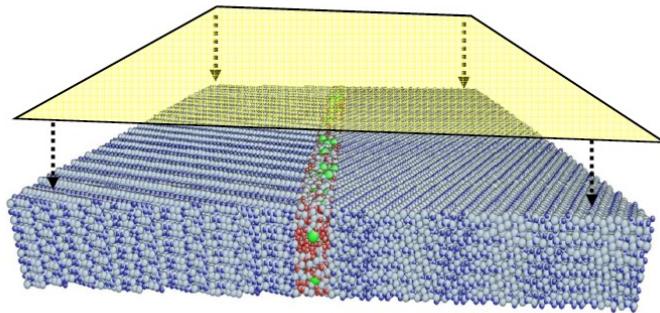
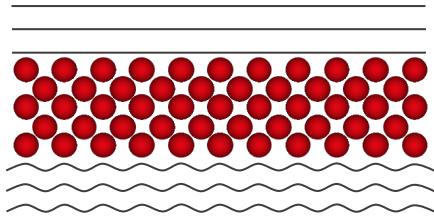
The direction of propagation of the electron may change!  $\Rightarrow$  Diffraction!

Coherent incident plane wave front



Due to changes in sample thickness and orientation, as well as lens imperfections, image interpretation is complex.



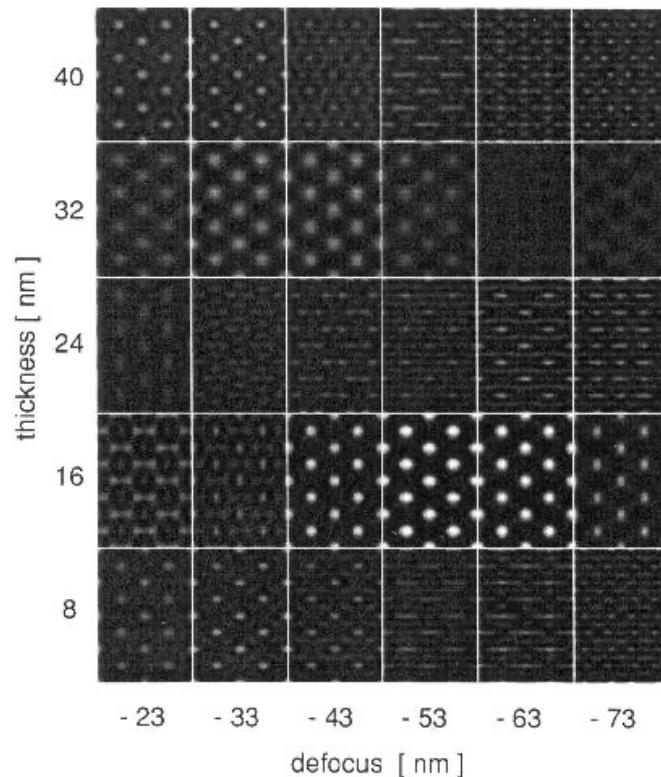


But ...  
not a perfect lens!

HR-TEM can give you local structural information of your specimen. However, do not expect straightforward interpretation.

**Resist the temptation of interpreting the spots as atoms!**

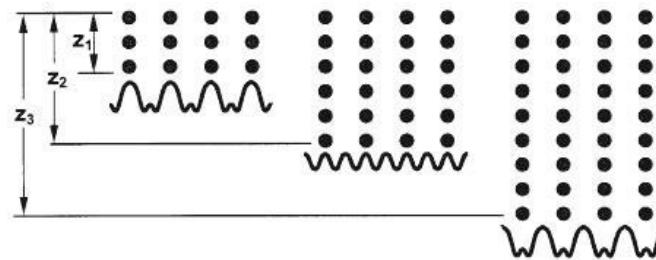
Phase contrast images can be difficult to interpret, because many factors contribute to phase shift:  
Thickness, orientation, scattering factor, focus, spherical aberration



Resist the temptation of interpreting the spots as atoms!

The crystal potential serves to phase shift the electron wave locally; and crystal thickness affects phase shifts.

Thus at the crystal exit surface, the electron wave carries information about the crystal potential, projected in the direction of the incident beam.



The objective lens (CTF) determines how much phase signal gets transmitted to the real space wave-function in the image plane.

### Mass-Thickness contrast

- Applies for both crystalline and amorphous phase

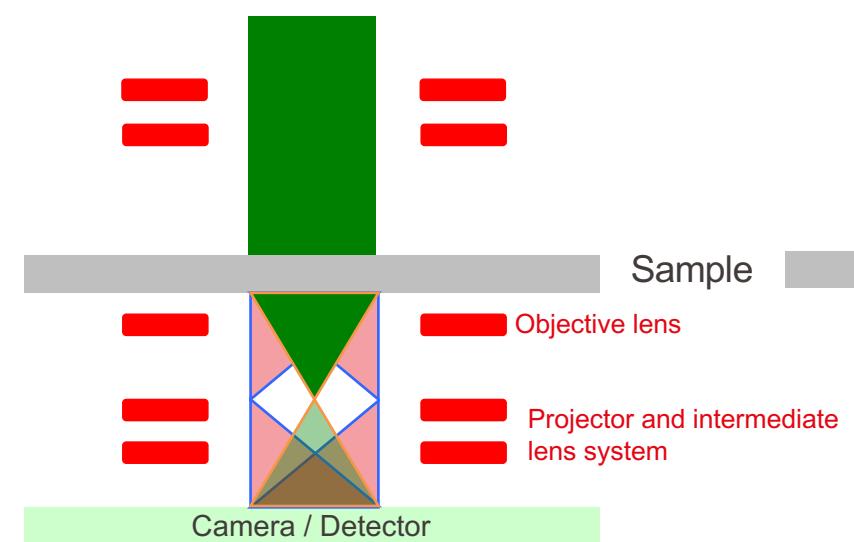
### Diffraction contrast

- Bright-field: objective aperture centered on “unscattered” beam
- Dark-field: objective aperture centered on “diffracted beams”

### HR-TEM Phase contrast

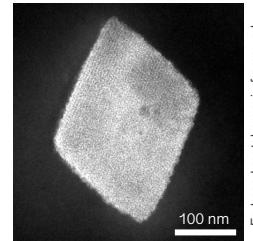
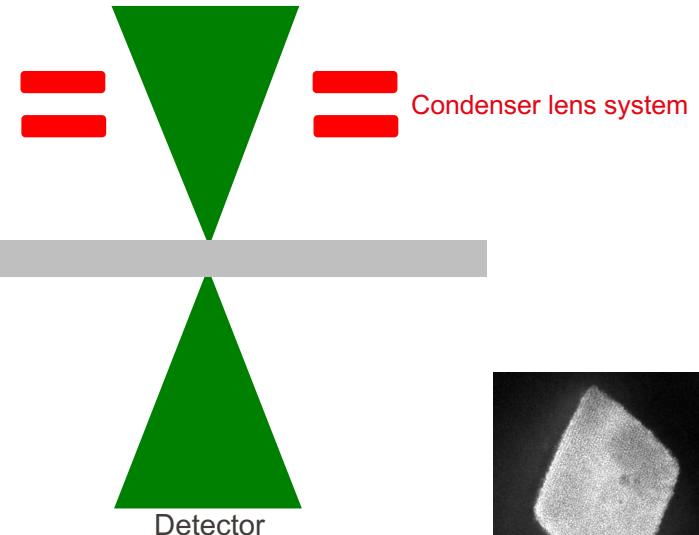
- Developed by the interference of two waves, one which undergoes a phase shift due to the interactions with the sample crystal structure of a given thickness and is altered by the microscope aberrations
- Simulations are needed to interpret phase contrast images properly

## Conventional TEM

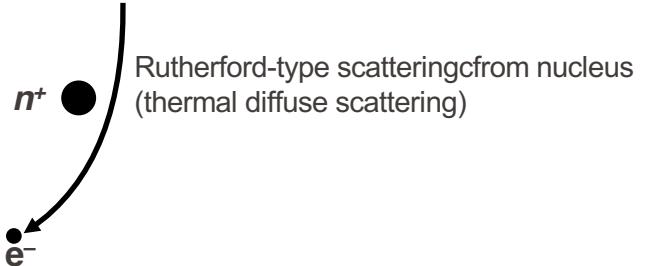
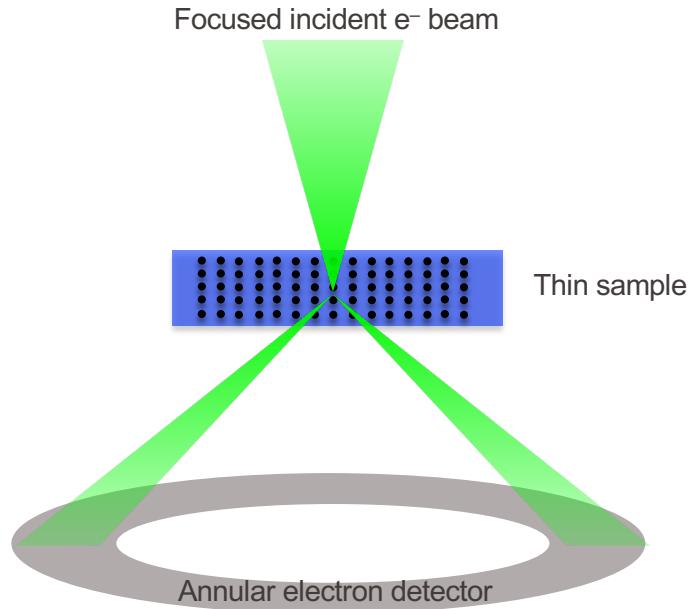


## Scanning mode (STEM)

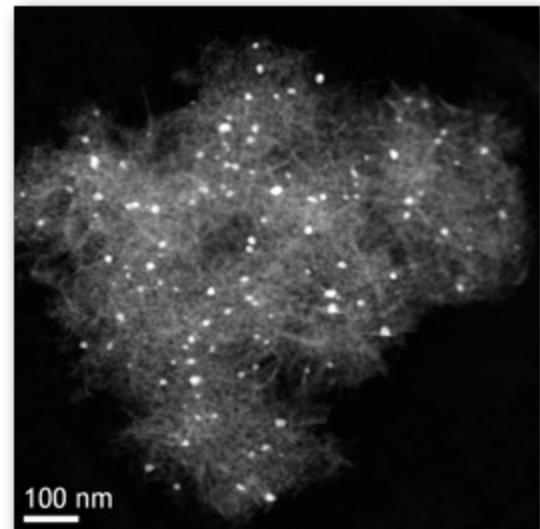
Electron beam (30-300 keV)



# Scanning TEM (STEM) with focused probe



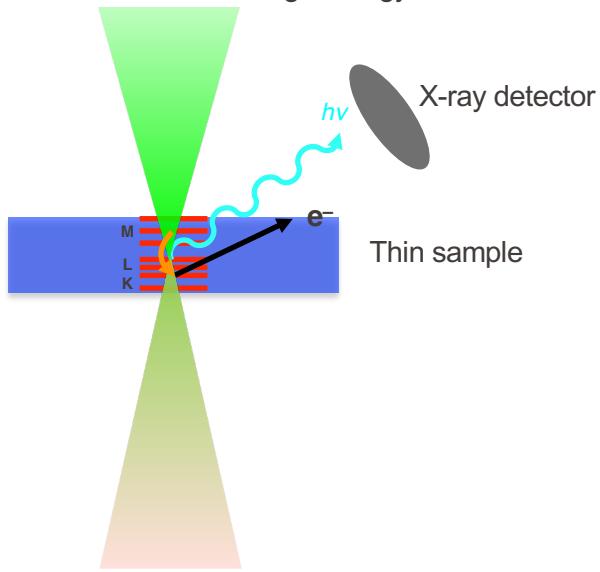
High-angle annular dark-field (HAADF) image



Mass/thickness contrast image

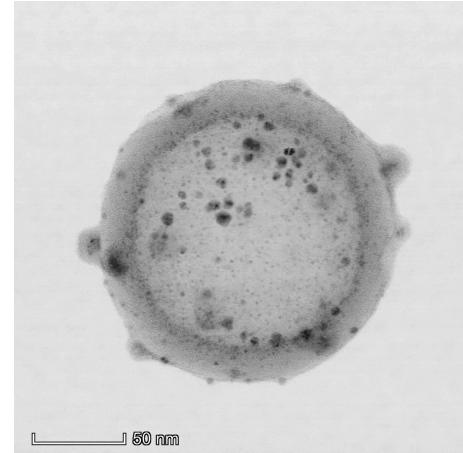
- Incoherent Rutherford-type scattering deflects transmitted  $e^-$  to high angle of scattering
- Larger nucleus / thicker the specimen → More scattering
- Map image intensity as function of probe position ( $x, y$ )
- Image intensity:  $I(x, y) \propto t_* Z^{1.6-2}$

Focused incident  $e^-$  beam: high energy!

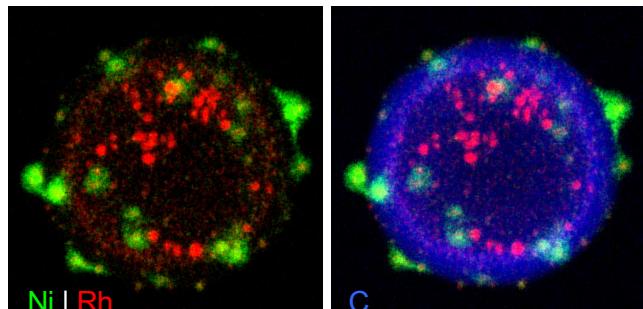


Transmitted beam suffers energy loss → Used in EELS

- High energy incident electrons (ballistic) → Ejection of inner shell  $e^-$
- Upper shell  $e^-$  descends
- Transition energy emitted as X-ray;  
Characteristic of element |  $E_{X\text{-ray}} = E_{\text{Upper shell}} - E_{\text{Inner shell}}$



Ni-Rh catalytic nanoparticles in carbon shell



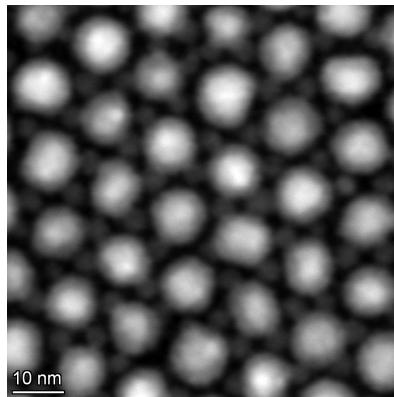
Map X-ray intensity as function of probe position (x,y)

With fast mapping STEM-EDX has become a regular feedback tool for materials synthesis

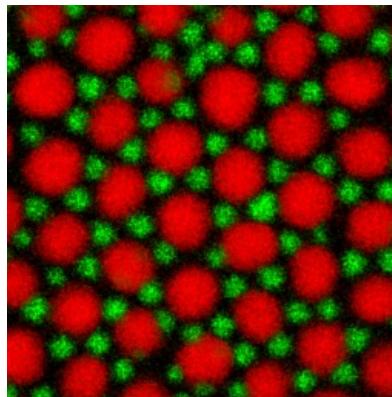
Sample characterized in a couple of minutes/hours

With aberration-corrected STEM, atomic resolution EDX became possible

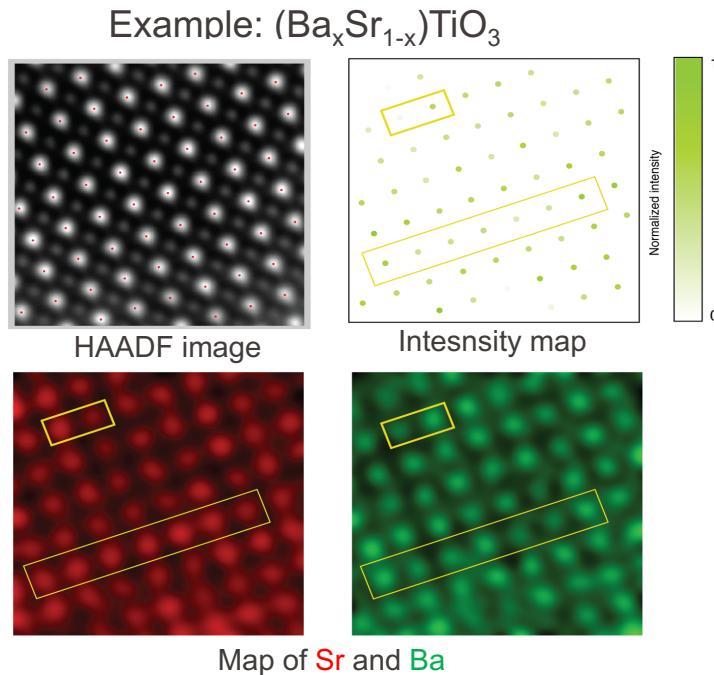
Example : Assembly of  $\text{Fe}_3\text{O}_4$  and Cu particles



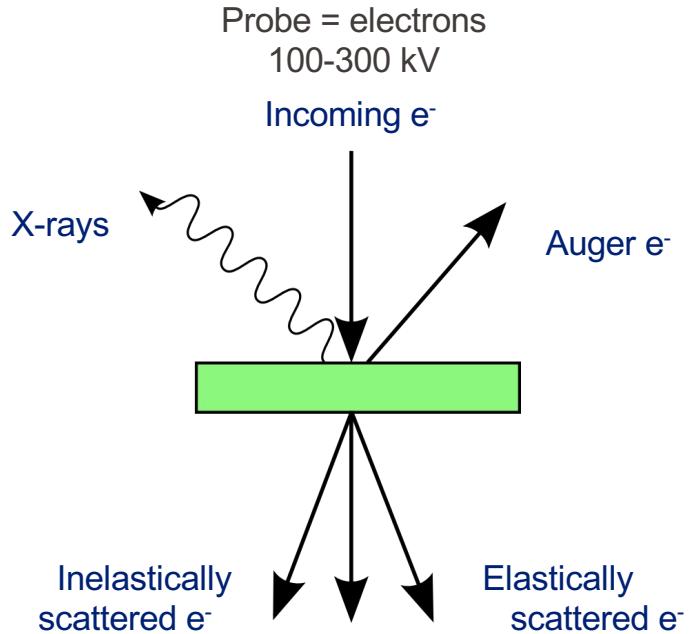
HAADF image



Map of Cu and Fe

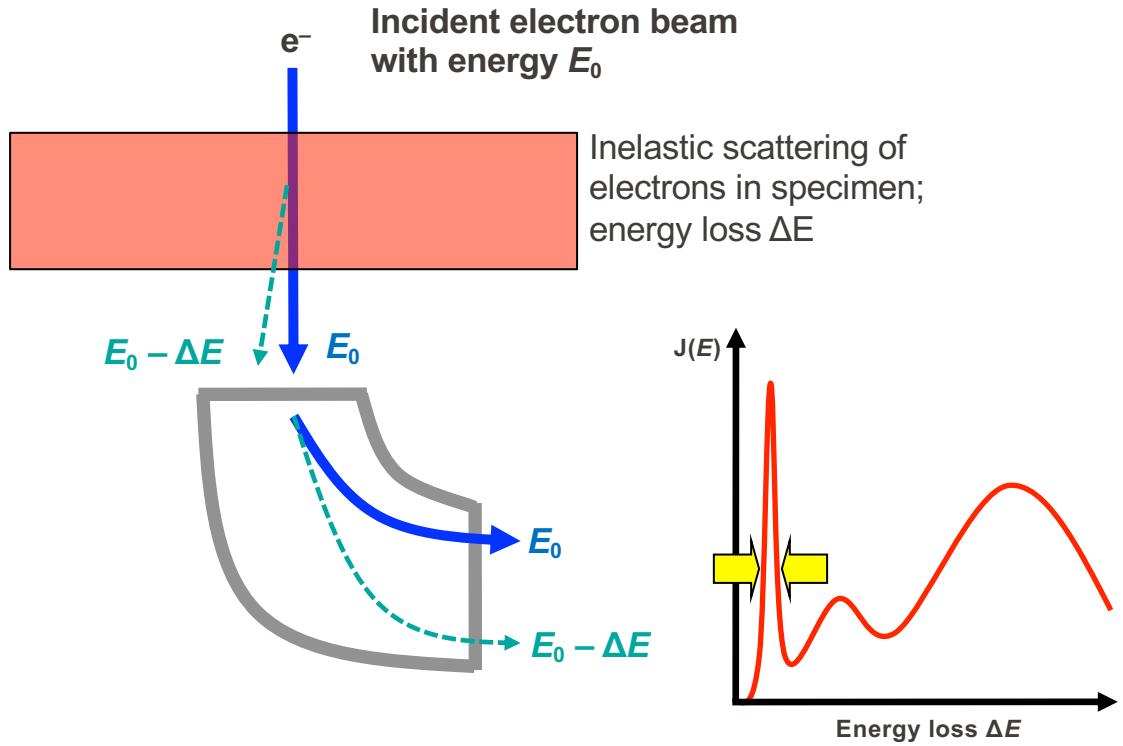


The TEM specific microanalysis technique!

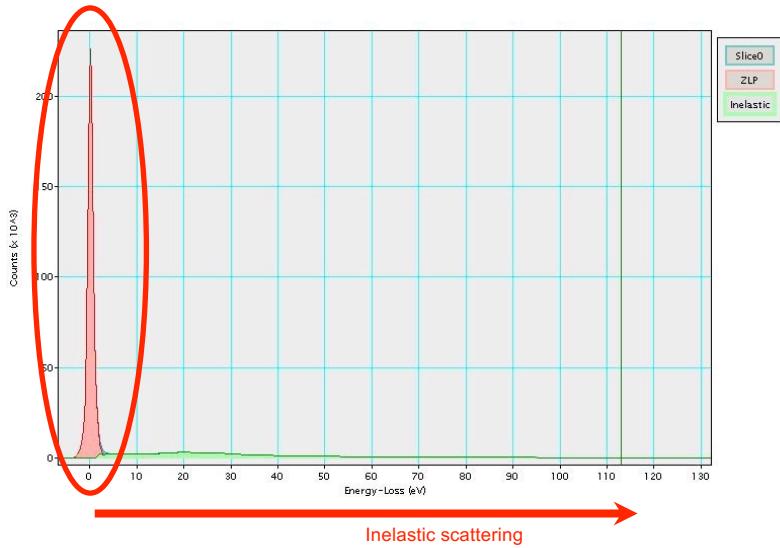




e.g. Gatan Enfina



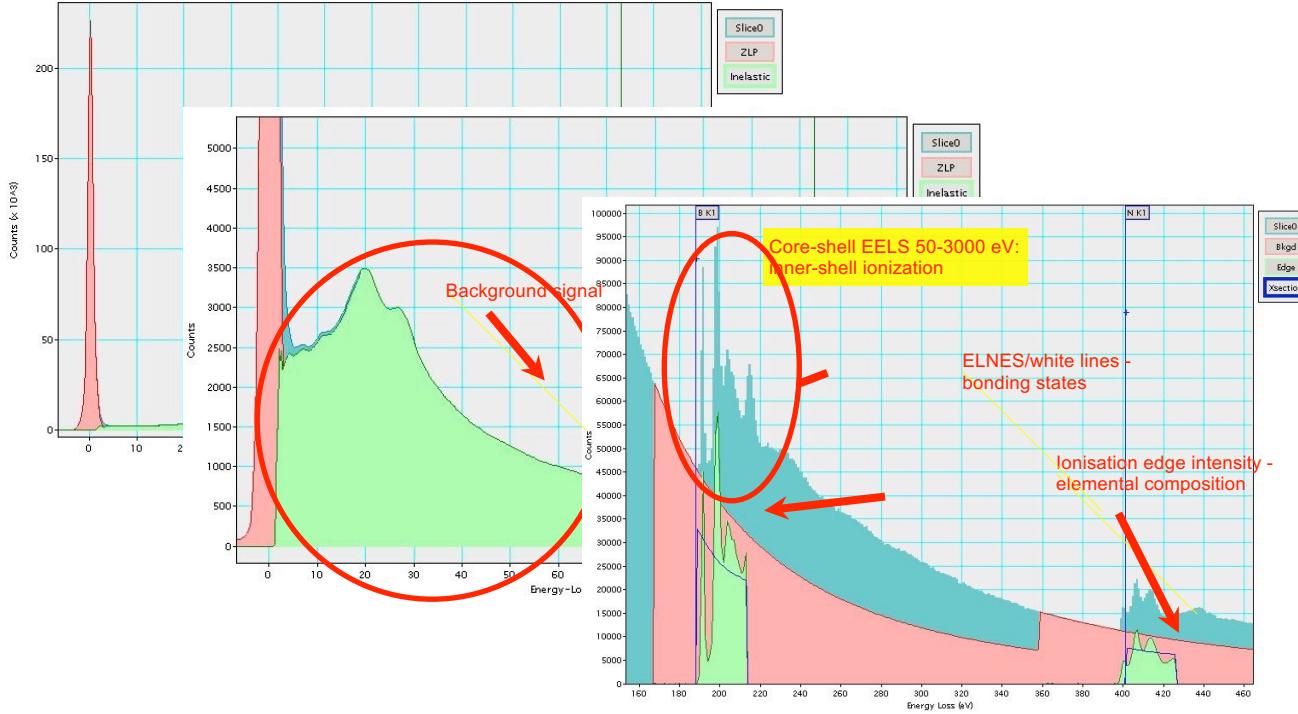
Magnetic prism separates electrons with different energy; produces spectrum of intensity  $J(E)$  vs energy loss  $\Delta E$ : **The energy-loss spectrum**



Zero-loss: Elastically-scattered  $e^-$   
Information about specimen thickness



Interaction with outer electrons (conduction/valence)  
Low-loss spectrum 0-50 eV: valence excitations -  
plasmons (bulk & surface),  
band gaps, optical properties



### Interaction with core electrons

Core-loss spectrum 50-3000 eV: inner shell ionization

ELNES/white lines: Bonding state

Ionisation edge intensity: elemental composition

Stimuli	Possible techniques	Information
<ul style="list-style-type: none"><li>• gas</li><li>• liquid</li><li>• temperature</li><li>• magnetic field</li><li>• current/voltage</li><li>• mechanical load</li><li>• light</li><li>• ...</li></ul>	<ul style="list-style-type: none"><li>• (S)TEM imaging</li><li>• diffraction</li><li>• electron energy-loss spectroscopy</li><li>• energy-filtered TEM</li><li>• energy-dispersive X-ray spectroscopy</li><li>• Lorentz microscopy</li><li>• holography</li><li>• ...</li></ul>	<ul style="list-style-type: none"><li>• microstructure</li><li>• crystallography</li><li>• chemistry</li><li>• diffusion/migration</li><li>• optical properties</li><li>• electric fields</li><li>• magnetic fields</li><li>• ...</li></ul>



Different dedicated holders

**Liquid phase processes**

Hydration and other liquid phenomena  
Crystal growth from the liquid phase  
Corrosion

**Phase transformations**

Solid state phase transformations  
Crystallization and melting  
Structural transformations  
Solid state reactions  
Solid state transformations in nanostructured materials  
Size-dependent transformations in embedded nanoparticles  
Shape and phase transformations in free-standing nanoparticles  
Sintering of nanoparticles

**Elastic and plastic deformation**

Microscopic phenomena during plastic deformation  
Deformation of polycrystalline materials  
The effect of gas environment on deformation  
Grain boundary motion  
Deformation phenomena in single crystals  
Deformation of multilayers  
Relaxation of epitaxially strained materials  
Mechanical properties of nanostructures, thin films and surfaces  
In situ indentation and straining of thin films  
Mechanical properties of nanostructures  
Surfaces: Tribology and Nanomanipulation

**Surface reactions and crystal growth**

Modification of surface structure  
Oxidation and other chemical reactions at surfaces  
Growth of nanostructures  
Carbon nanotubes  
Thin film growth and defect formation  
Epitaxial growth  
Polycrystalline growth  
Crystal growth on patterned substrates

**Domain wall motion and flux dynamics**

Magnetic domain switching  
Flux motion in superconductors  
Switching phenomena in ferroelectrics

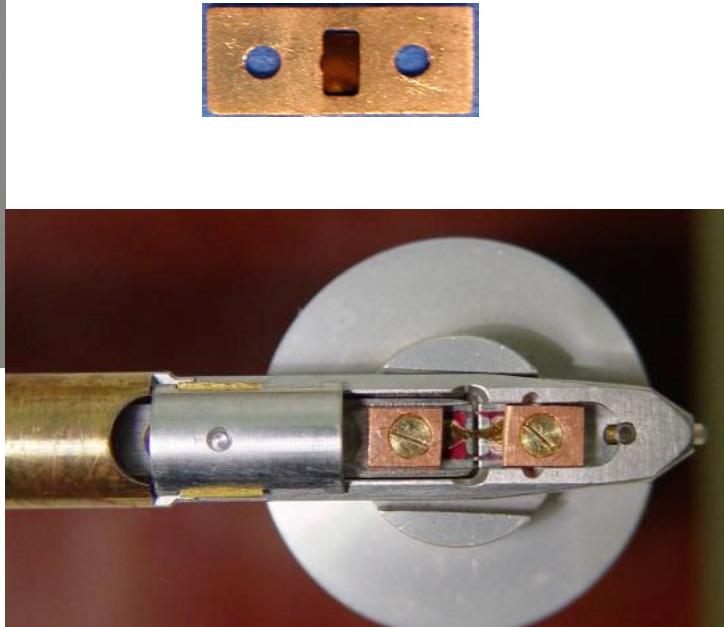
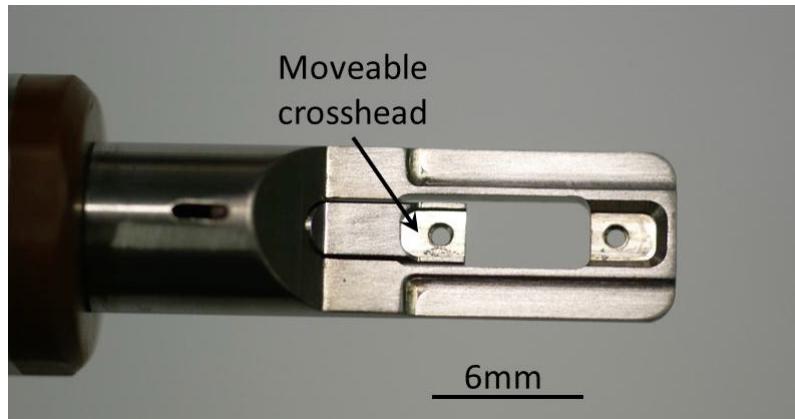
**Correlation of structural and electronic properties of materials**

Electrical measurements on TEM samples: samples as devices  
Electrical measurements on individual nanostructures

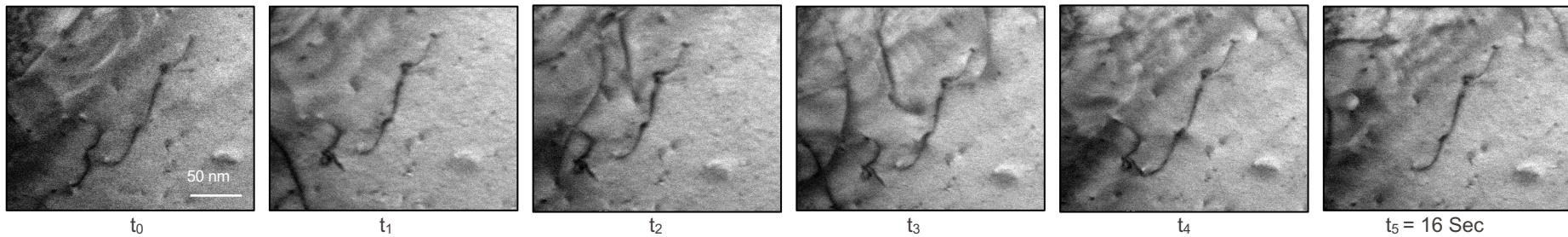
**Beam induced processes**

Electron beam induced phenomena  
Interaction with the vapour above the specimen  
Formation of point defects  
Beam induced transformations, surface reactions, growth  
Radiation enhanced dislocation motion  
Hole drilling  
Ion implantation  
High energy ion accelerators  
FIB in the TEM

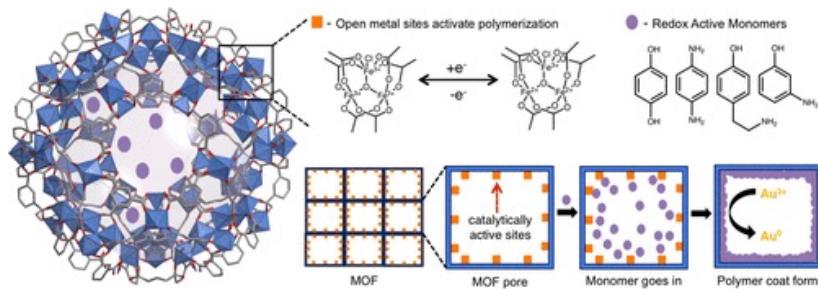
Conventional straining holder



Dislocations interaction  
Specimen: Cu foil | *in situ* deformation



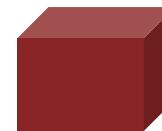
- A window into the behavior of materials under real processing conditions.
- A continuous view of a process, which may take the place of multiple post-mortem measurements.
  - It is easier to catch a transient phase or observe a nucleation event.
  - Specific and detailed kinetic information, such as the motion of individual dislocations under known stress, or growth rates of individual nanocrystals.
- But this unique information comes at the cost of increased experimental complexity.
- **Expensive: machine time, holders**



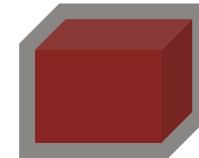
Without polymerisation



Polymer\* inside



Polymer on surface



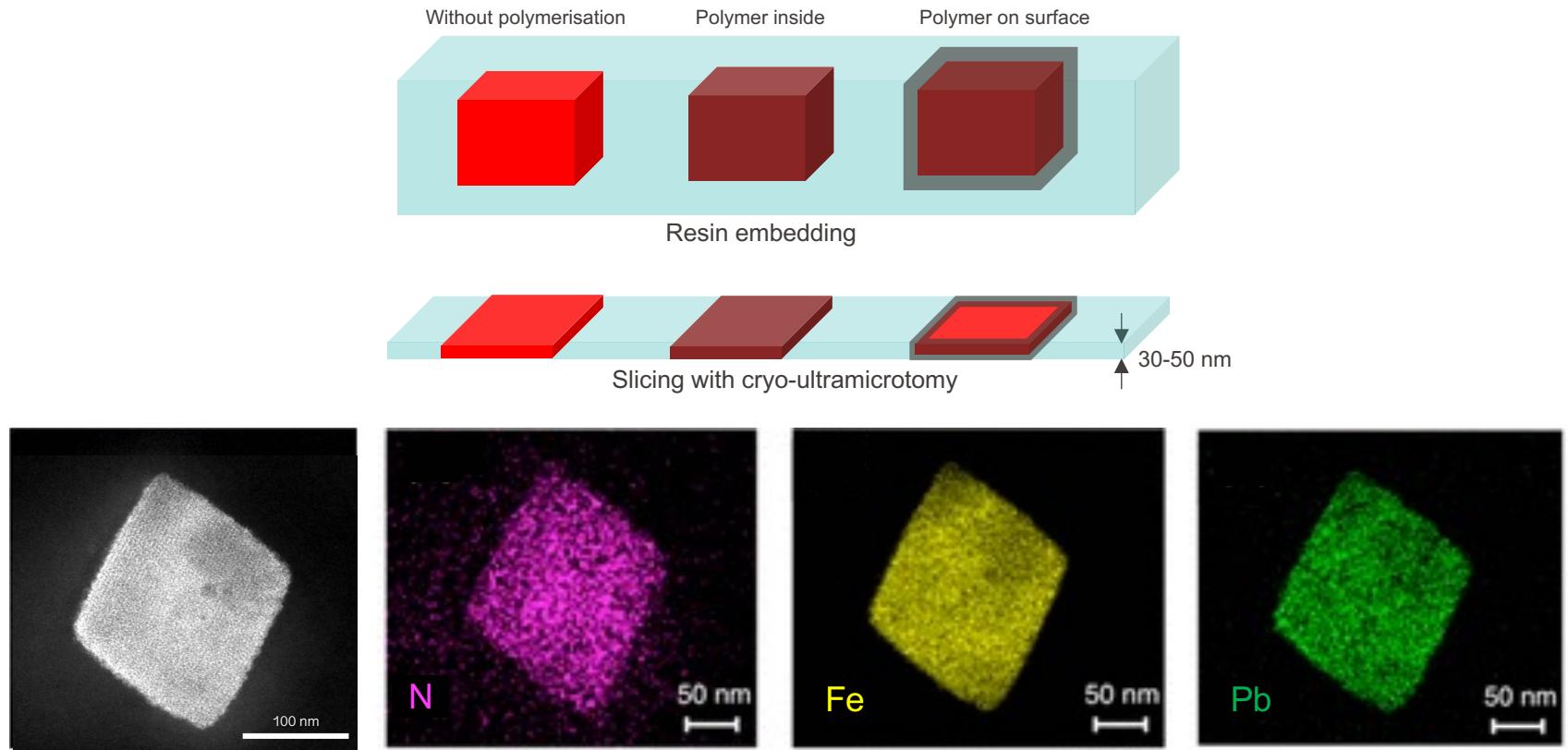
MOF: Fe-BTC

\*Para-phenylenediamine (pPDA)



2D projection of 3D objects

Due to projection problem,  
not possible to determine whether the polymer is inside the particle or on its surface



# EPFL • Projection problem

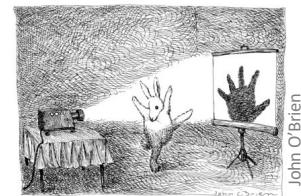
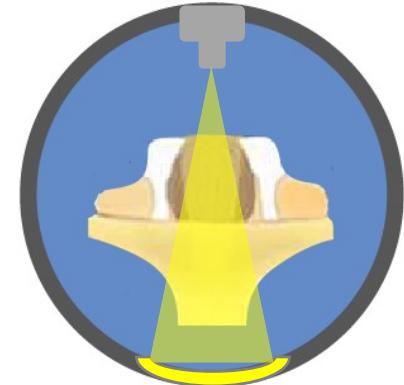
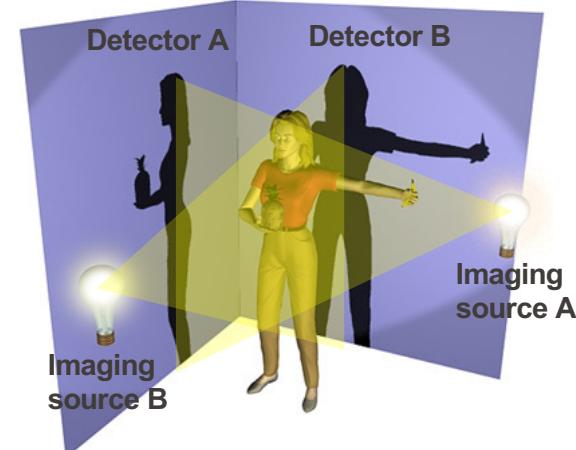
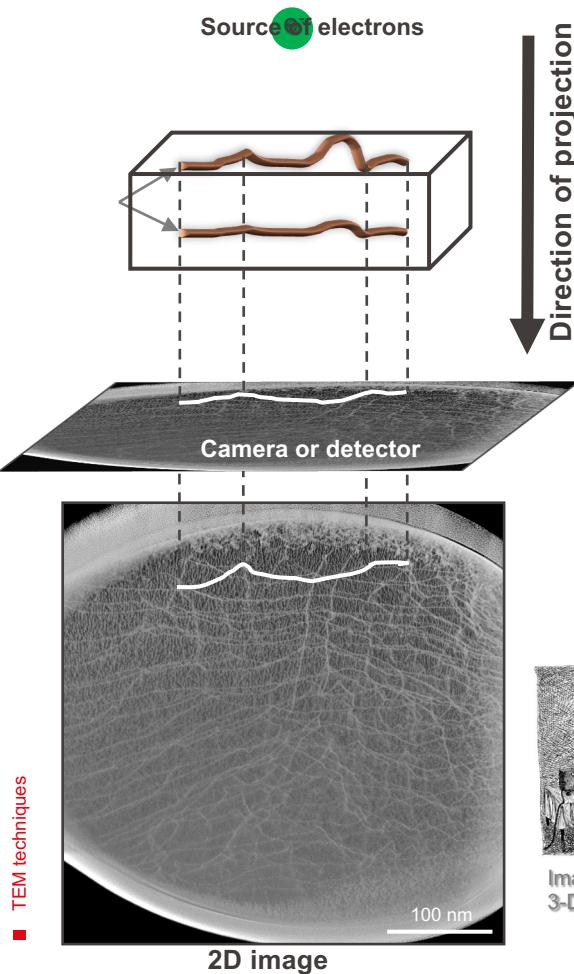
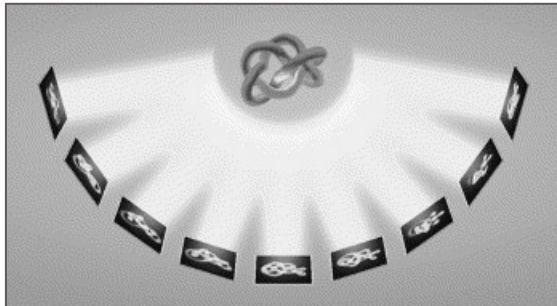
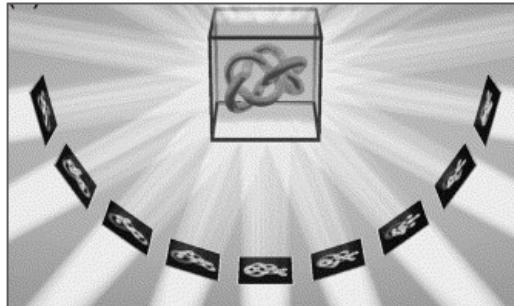


Image is the projection of a 3-D object onto a 2-D plane

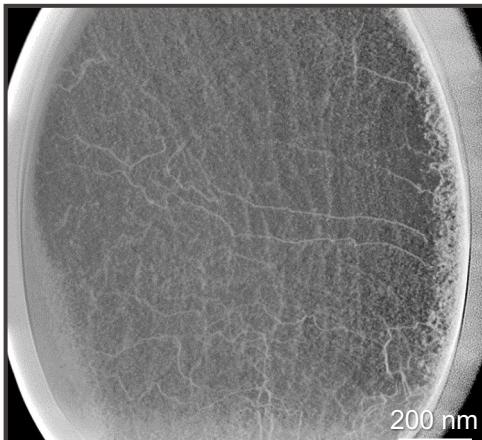




Series of projections



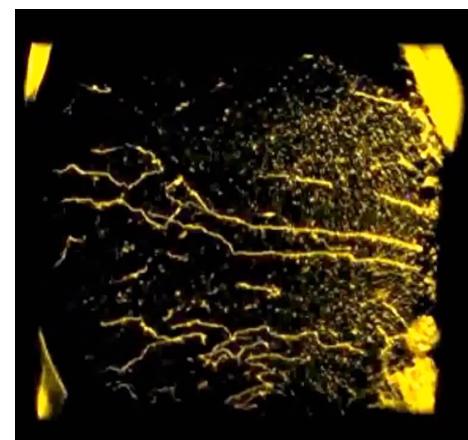
Back-projection



2D image



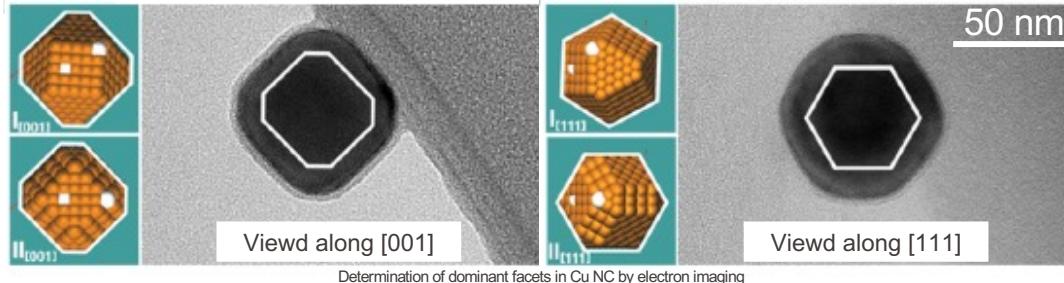
Series of tilted images  
-35°/+35° tilt arc | 1° interval



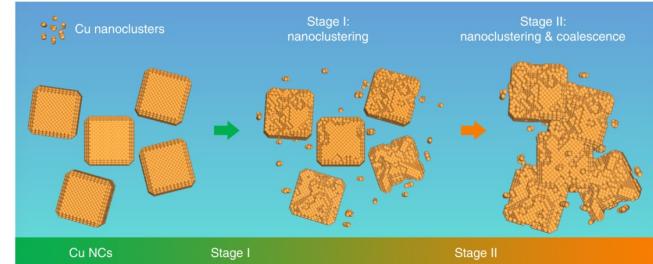
3D reconstruction  
SIRT 30 iterations

# EPFL • Electron microscopy to study degradation process of Cu NPs

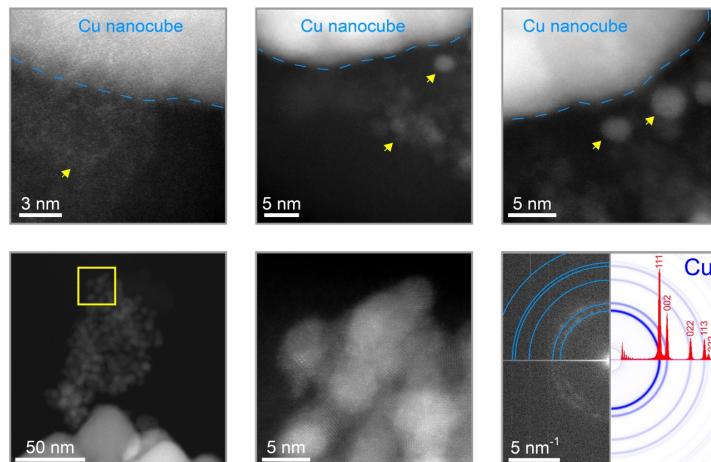
Advanced microscopy techniques and image simulation enabled researchers to uncover potential-driven nanoclustering degradation pathway Cu nanocubes during electrochemical  $\text{CO}_2$  reduction.



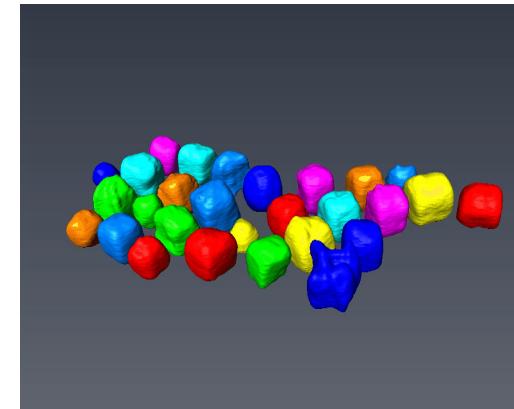
Determination of dominant facets in Cu NC by electron imaging



Followed morphological evolution during operation and correlate it with the electrocatalytic performance



■ TEM techniques

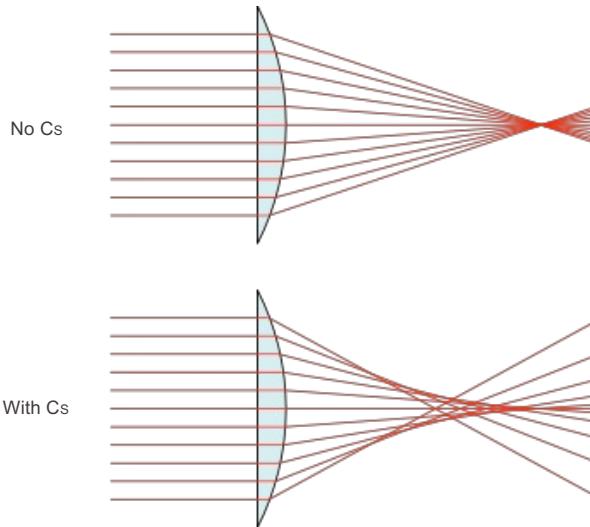


Determination of morphological changes using electron tomography

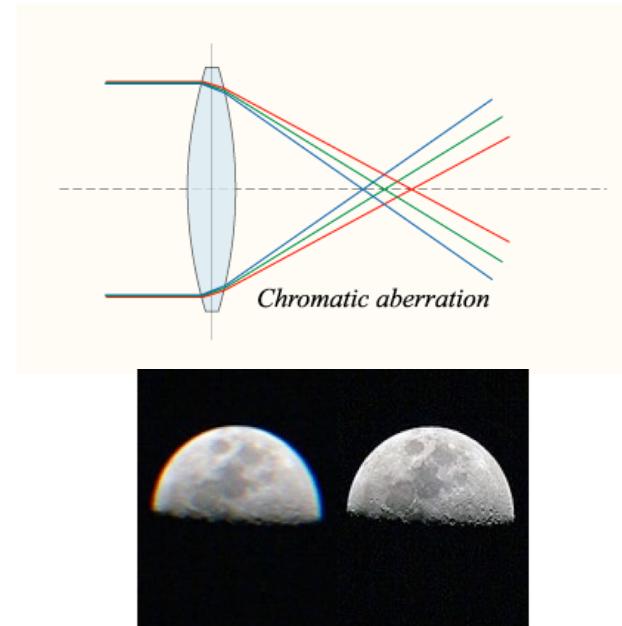
Electromagnetic lenses in TEM column are toroidal

Lenses inherently convergent

→ Spherical aberration ( $C_s$ ) and Chromatic aberration ( $C_c$ )



Core of the galaxy M100 ©NASA

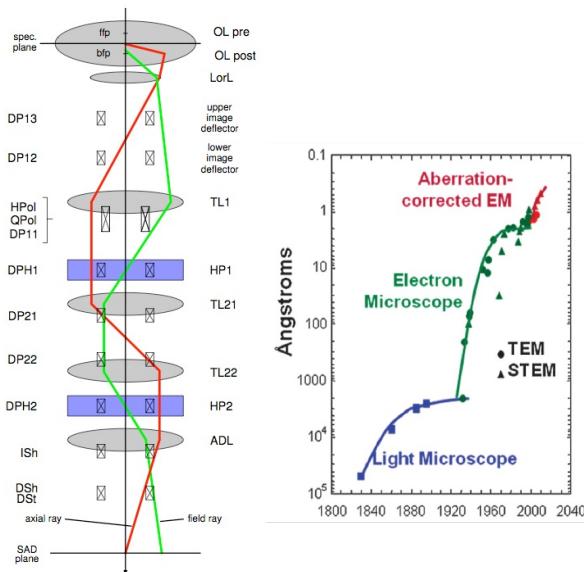


Parallel rays that pass through the central region of the lens focus farther away than the rays that pass through the edges of the lens. Results in multiple focal points and thus a blurred image.

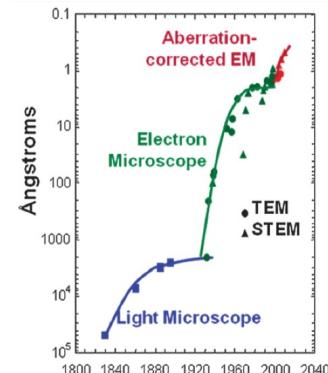
Lens cannot focus all energies (wavelengths) to the same convergence point.

Combination of standard radially-symmetric convergent lenses with multipole divergent lenses (e.g. tetrapoles, hexapoles) to tune Cs

Like “glasses” for TEM (or the Hubble)

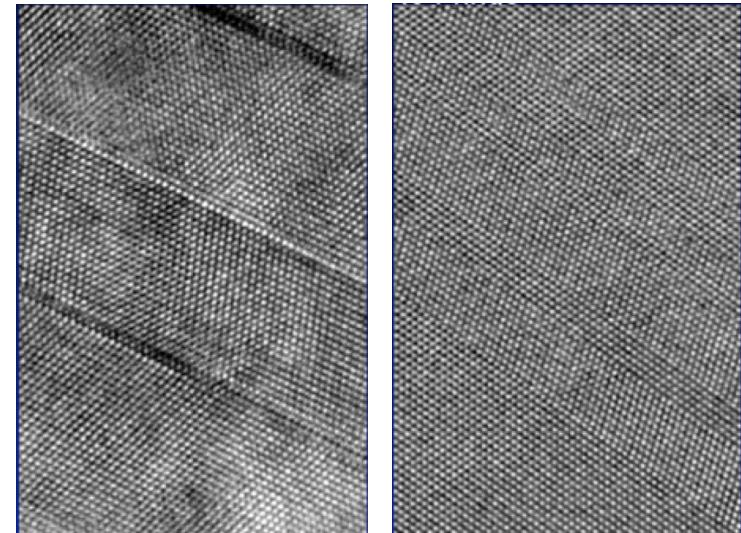


CEOS corrector



→ Resolution jumps to sub-Å!

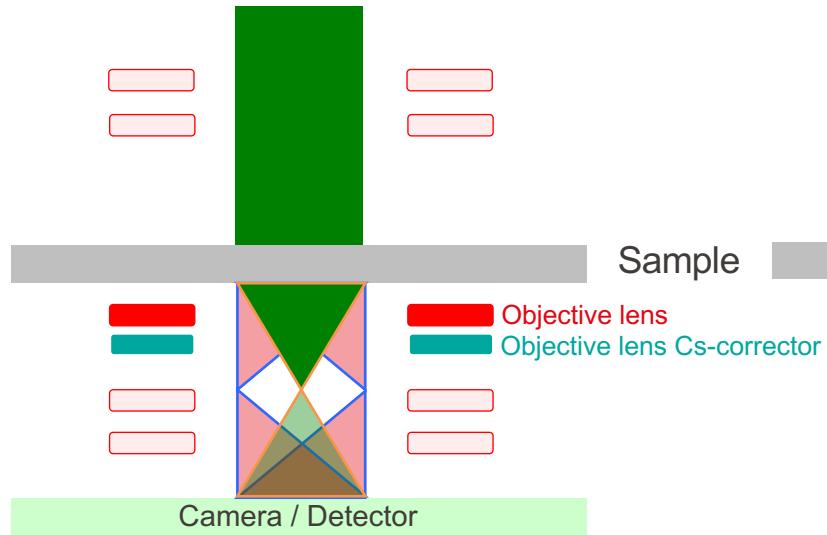
Example:  $\Sigma 3$  grain boundaries in Al  
Uncorrected Cs-corrected



Images: Oikawa, JEOL

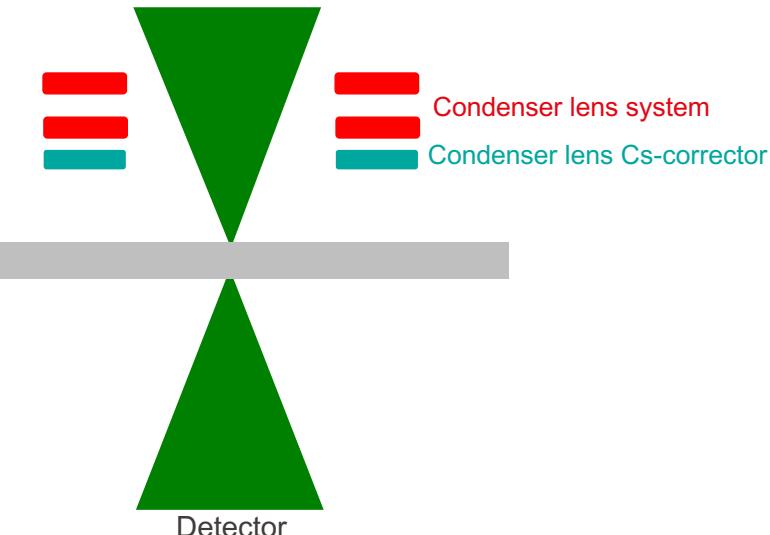
### TEM (Image-corrected)

Electron beam (60-300 keV)



### STEM (Probe-corrected)

Electron beam (30-300 keV)



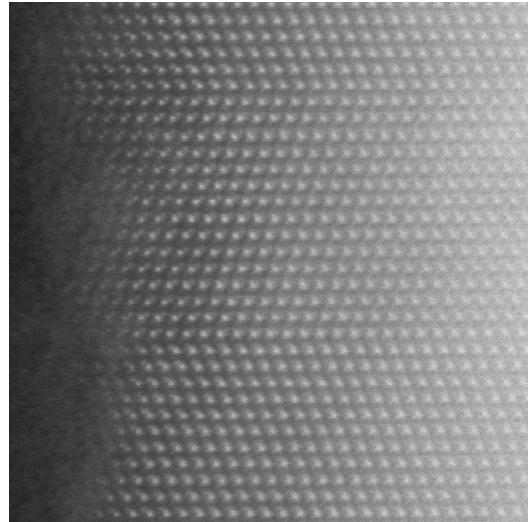
Example: (Al)GaAs nanowires imaged at 300 kV in STEM mode

### HR-STEM with sub-Å Cs-corrected probe:

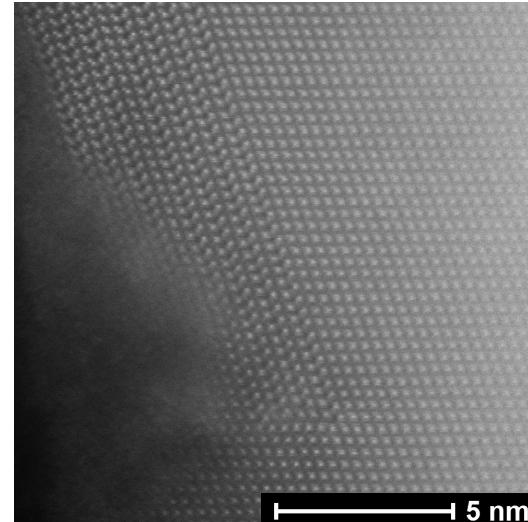
Simple (quantitative) atomic column contrast

Reduced sensitivity to small changes in sample orientation

Image either in focus or out of focus (no contrast reversals with defocus, thickness)

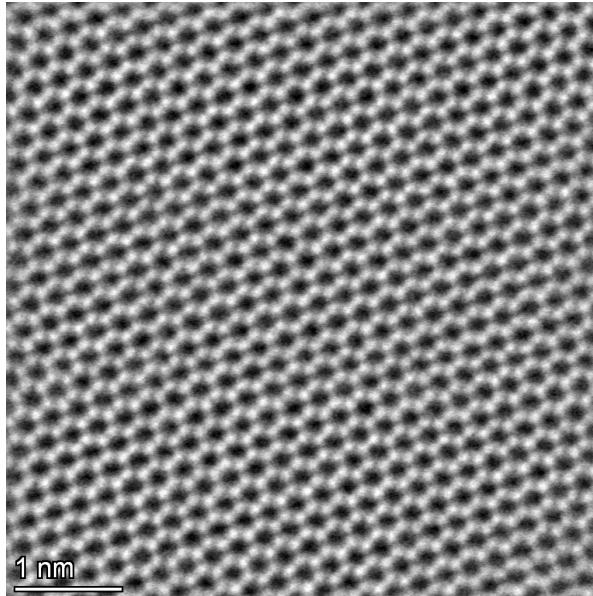


FCC twinning



HCP inclusion and dislocation

- Cs-correction and monochromatic illumination to reduce  $C_c$  allow sub-Å resolution even at lower kV where electron wavelength  $\lambda$  is greater
- Beam sensitive, low contrast materials can be imaged
- Here a monolayer graphene is imaged with 80 keV in STEM mode (IDPC)



TEM is now not one technique that can be easily summarised, but is split into many specialisms. In essence this is due to the many types of interaction of the electron beam with the atoms of a sample.

Modern TEM instruments are arguably the most versatile analytical tools. Many other possible uses (e.g. imaging of magnetic domains, optical plasmon mapping, in-situ studies) exist..

Aberration-correction and improved instrument stability give sub-Å resolution in TEM and STEM. With lower beam voltages lighter elements can be analysed without beam damage.

Faster, more sensitive spectrometers give unprecedented access to composition, chemistry and physics of materials.

Computer interfaces and software allow acquisition and processing of large datasets.

*With the latest instrumentation, the sample and specimen preparation are often the limiting factor!*

# Some useful literature

Transmission Electron Microscopy by D.B. Williams and C.B. Carter (Springer)

Large Angle Convergent Beam Electron Diffraction by J.-P. Morniroli

Aberration-corrected imaging in transmission electron microscopy: an introduction by R. Erni

Scanning Transmission Electron Microscopy by S.J. Pennycook and P.D. Nellist (eds) (Springer)

Diffraction Physics by J.M. Cowley (North Holland/Elsevier)

Science of Microscopy by C.W. Hawkes and J.C.H. Spence (eds) (Springer)