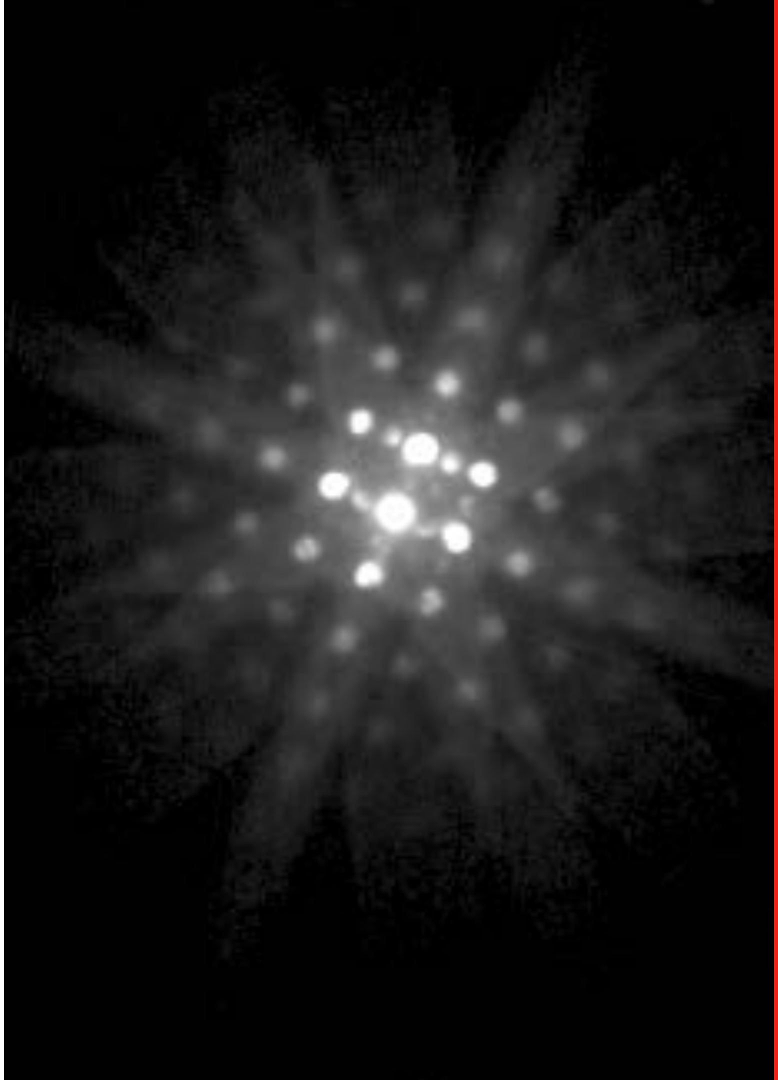
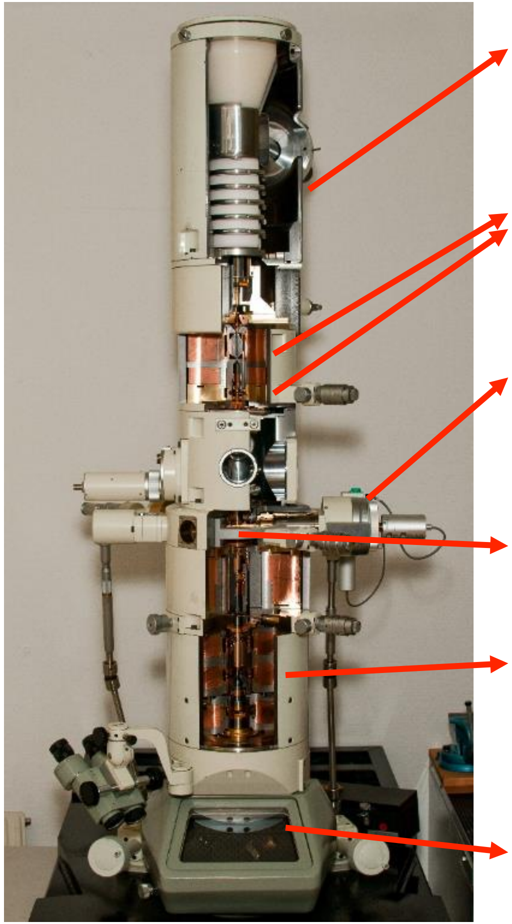


Imaging modes in TEM

November 2019

Emad Oveisi



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

What do you see in this image and how to interpret the contrast?

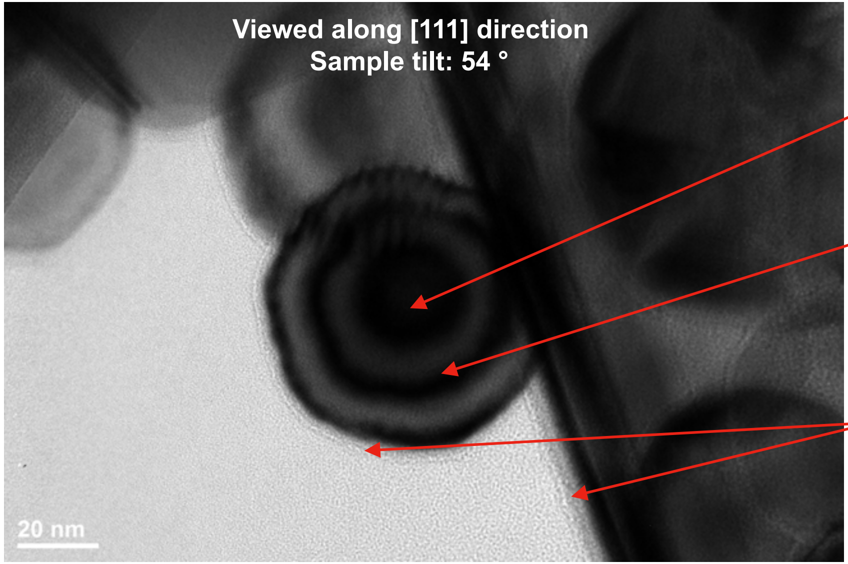
1- Why cubic particles appear as hexagons?

2- Why Cu particles are darker than the carbon background?

3- Why the middle particle is darker than the rest?

4- What is the origin of dark oscillatory contrast inside the Cu particles?

5- What are the bright (could be dark) fringes at the edge of particles?

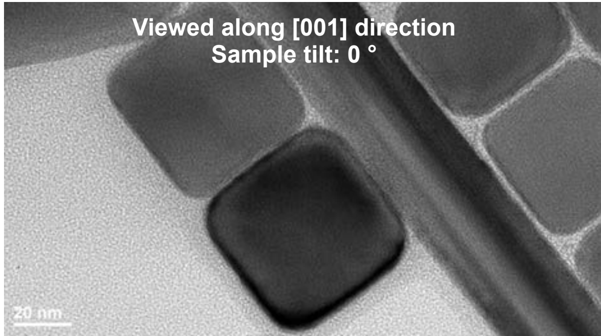


3- Diffraction contrast

4- Dynamical diffraction effect

5- Phase contrast (Fresnel fringes)

Bright-field TEM image of 54 nm cubic Cu particles



Outline

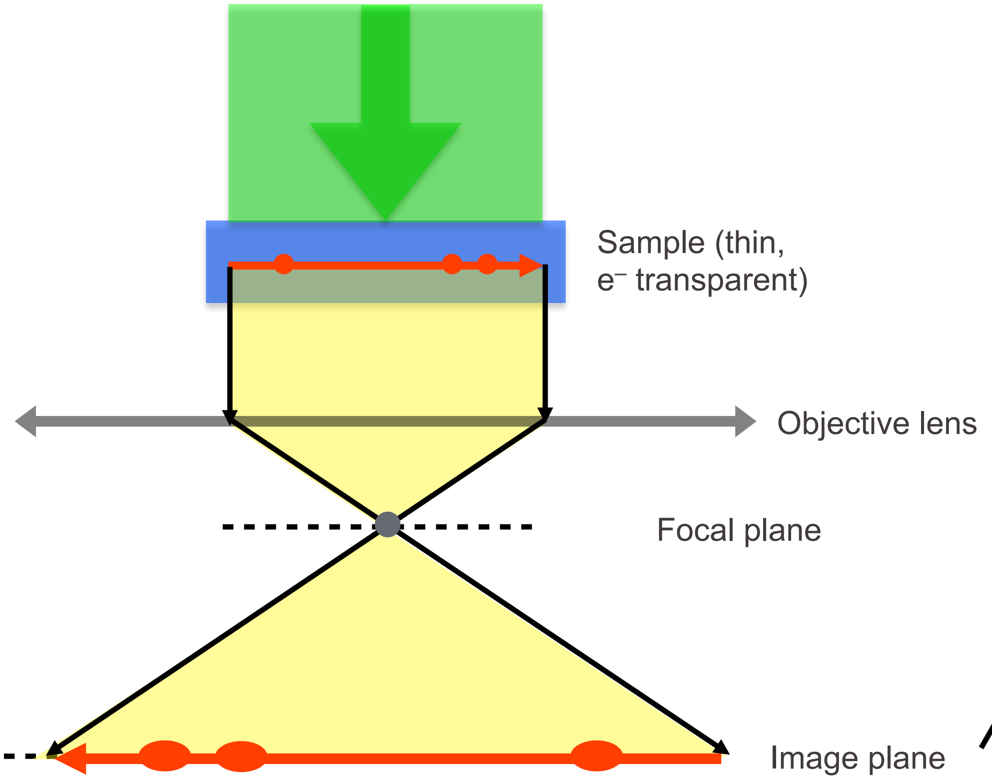
- **Image formation in TEM**
  - Image and diffraction modes
  - Bright- and dark-field modes
    - Strong- and weak-beam
  - High-resolution TEM

- **Image contrast in TEM**
  - Mass-thickness contrast
  - Diffraction contrast
  - Phase contrast

Image formation in TEM

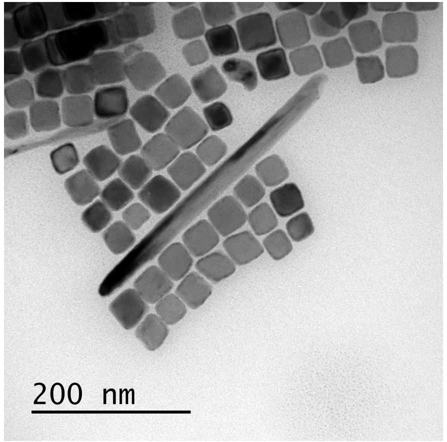
Emad Oveisi

Parallel incident e⁻ beam; $\lambda \approx 0.02–0.03$ Å



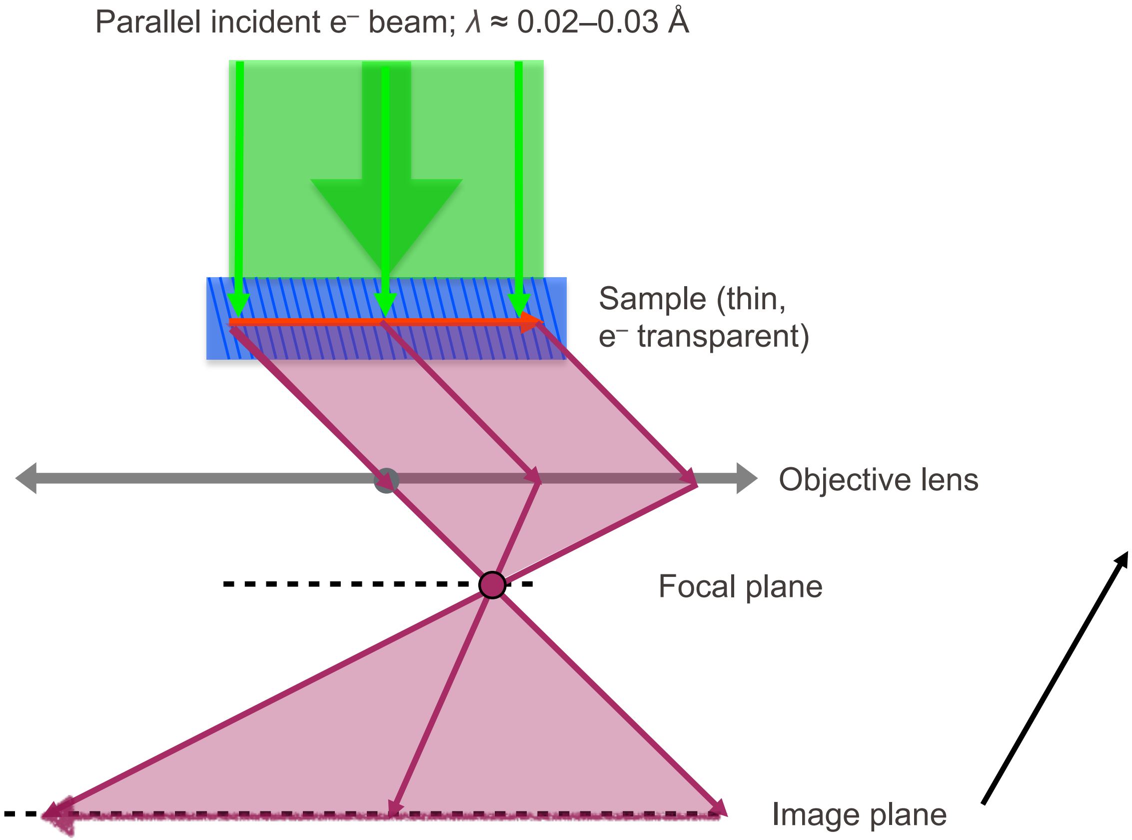
Why some cubic particles appear darker than others?

Cubic particles (≈ 40 nm) of Cu

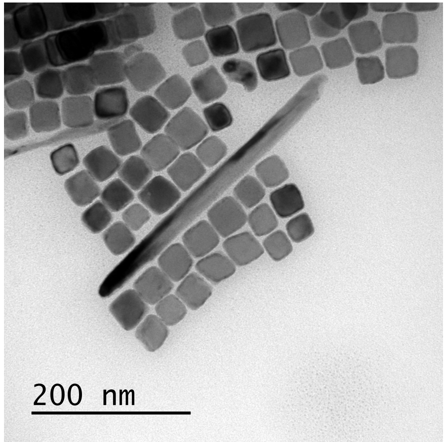


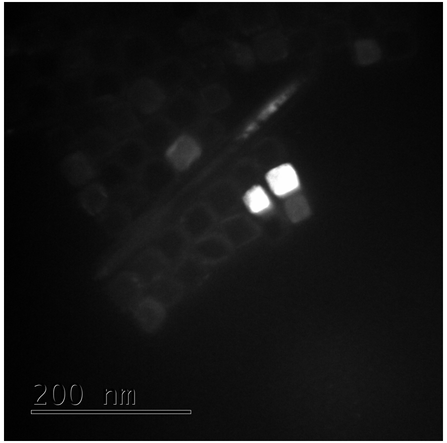
Bright-field image:
made by directly transmitted electrons

Image formation in TEM



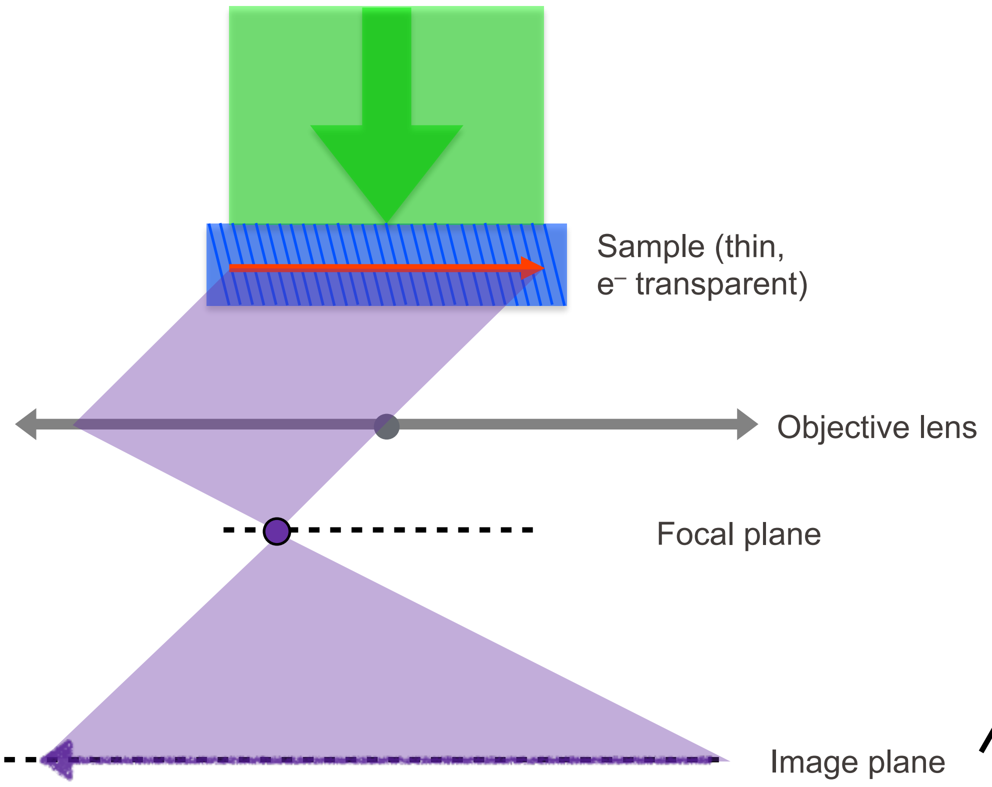
Bright-field image



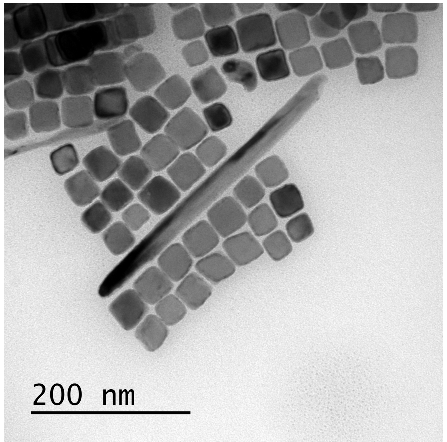


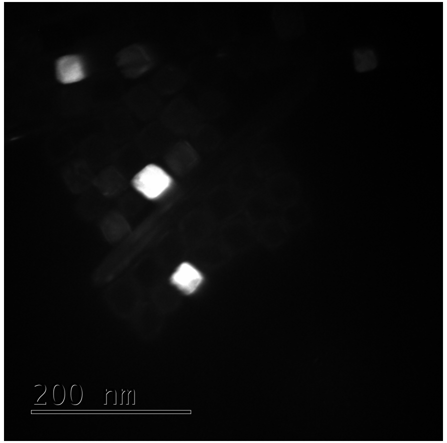
Dark-field image:
made by selected diffracted electrons

Image formation in TEM



Bright-field image





Dark-field image:
made by selected diffracted electrons

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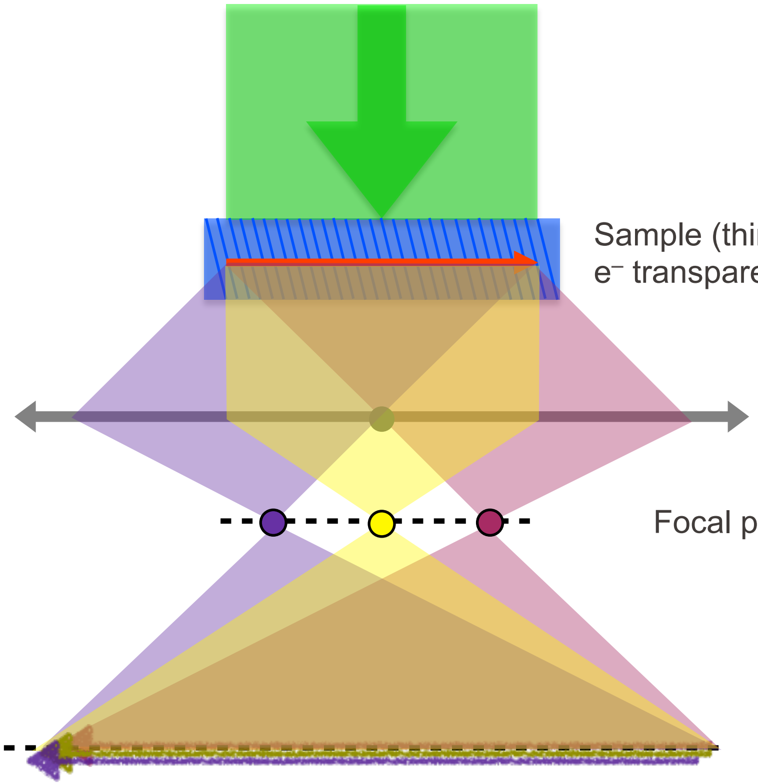
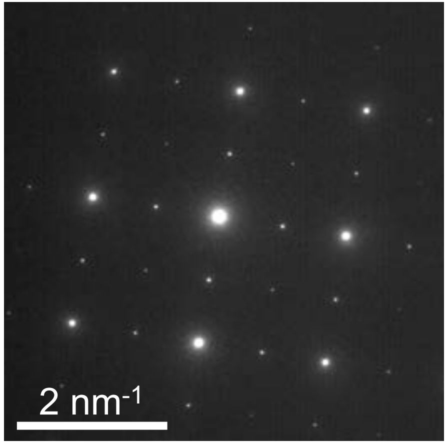
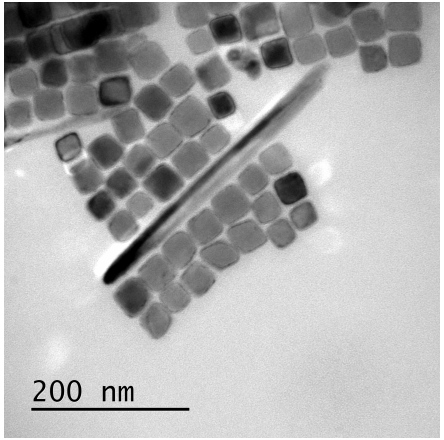
Image formation in TEM

Parallel incident e⁻ beam; $\lambda \approx 0.02–0.03$ Å



Electron diffraction pattern





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

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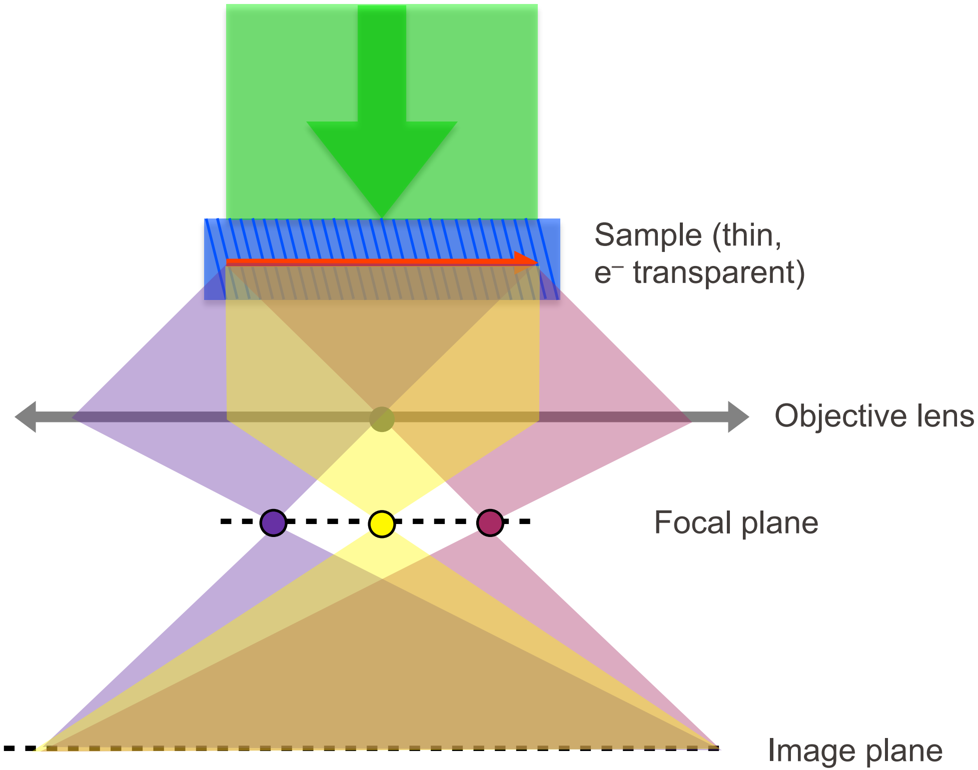
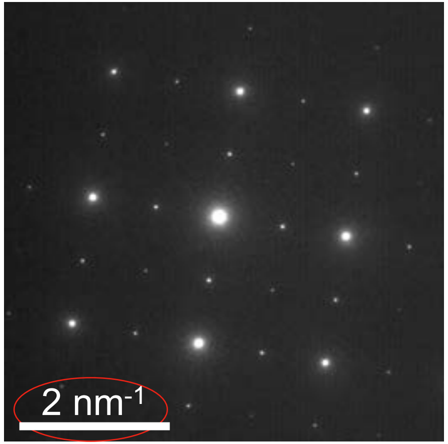
EPFL

Image formation in TEM

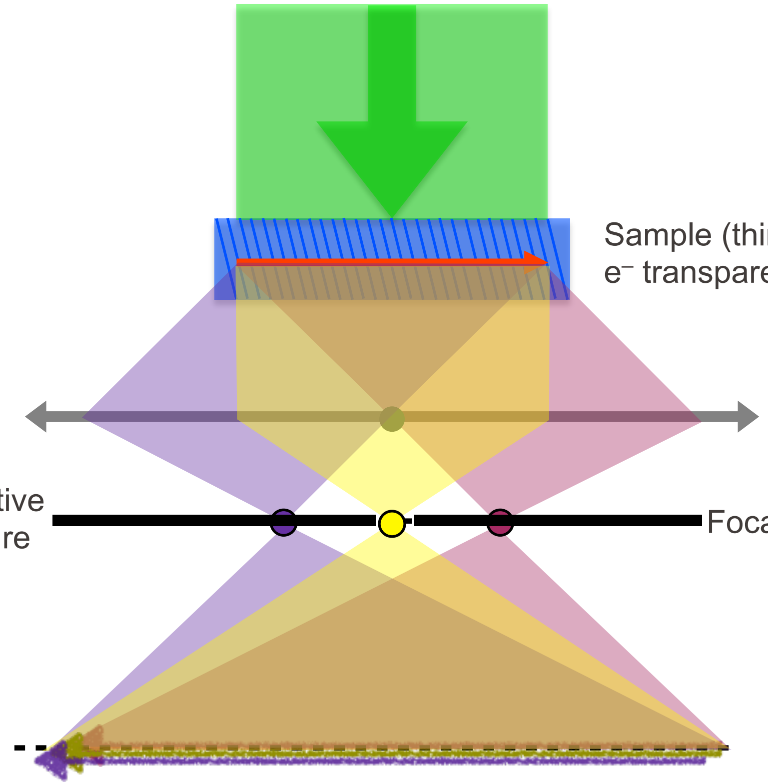


Electron diffraction pattern



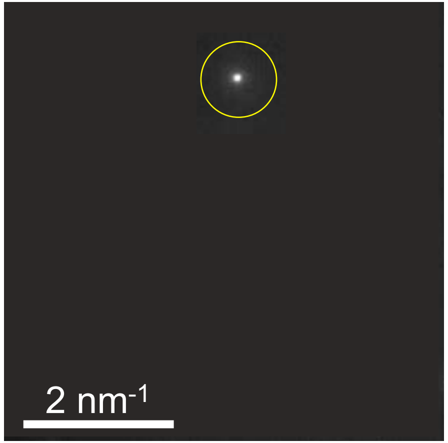
- *In back focal plane of objective lens parallel rays focused to point*
- *Diffraction – coherent scattering – creates sets of parallel rays from different crystal planes*
- *Focusing of these parallel rays in back focal plane creates spots of strong intensity:* ***the diffraction pattern***

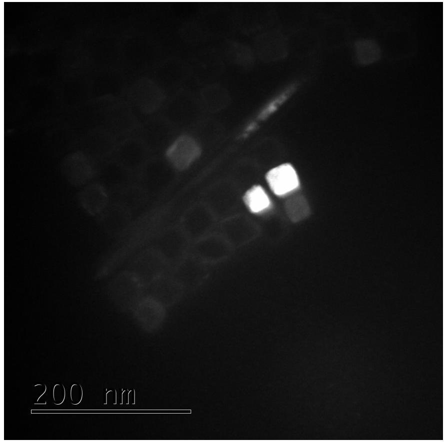
Image formation in TEM



Insertion of the "objective aperture" in diffraction plane allows imaging from just direct beam or from a selected diffracted beam.

Electron diffraction pattern





Dark-field image:
made by selected diffracted electrons

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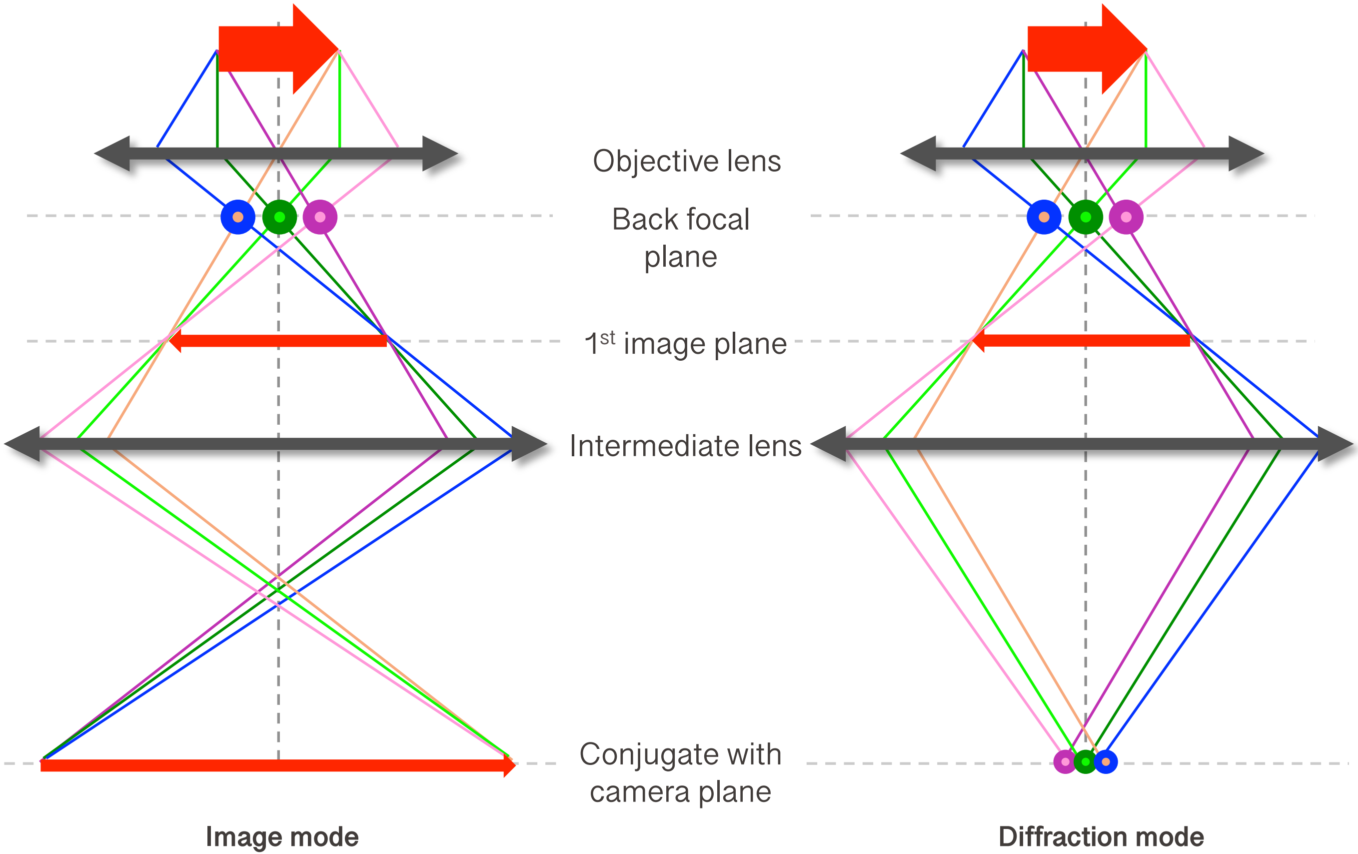
Imaging modes



Imaging modes: Selected area electron diffraction

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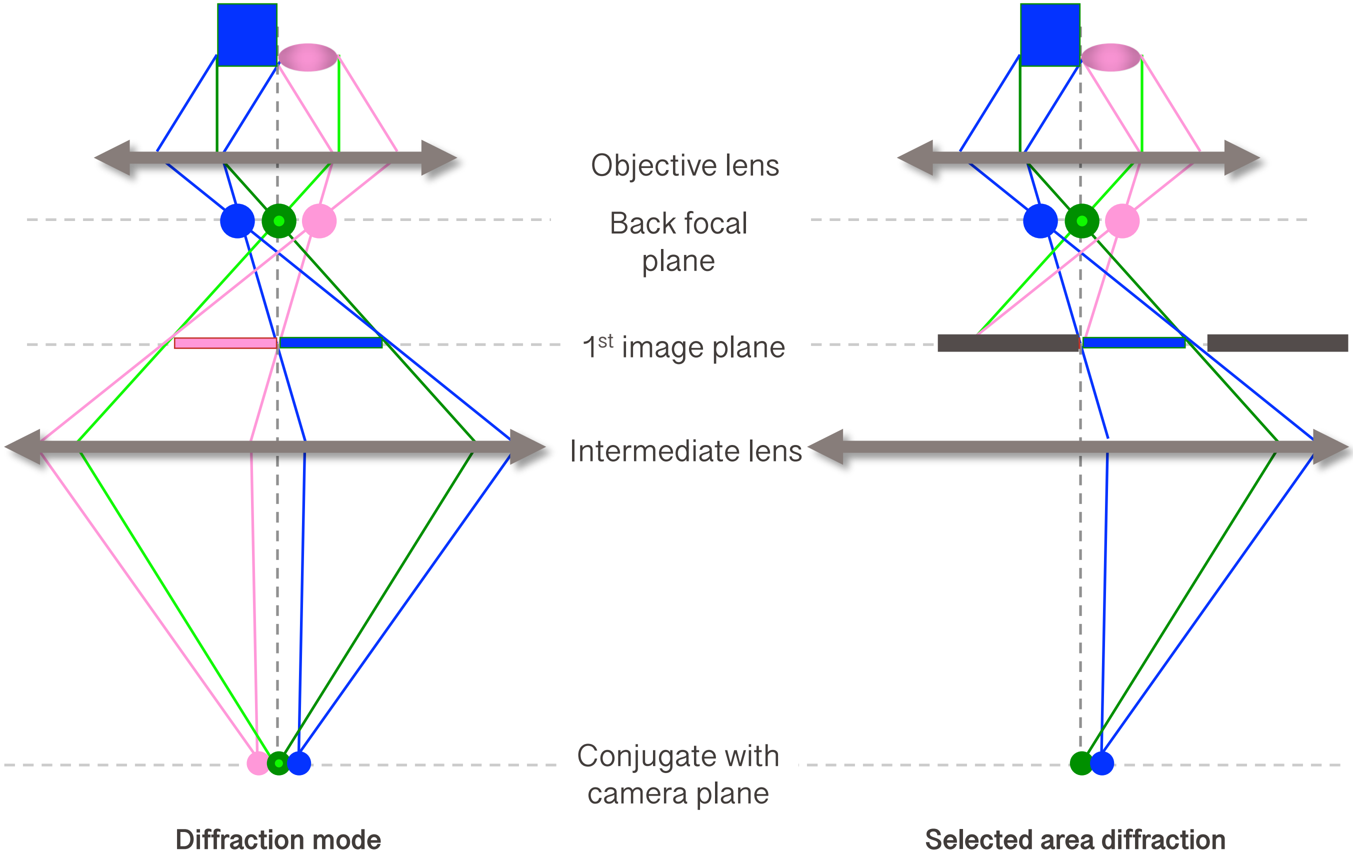
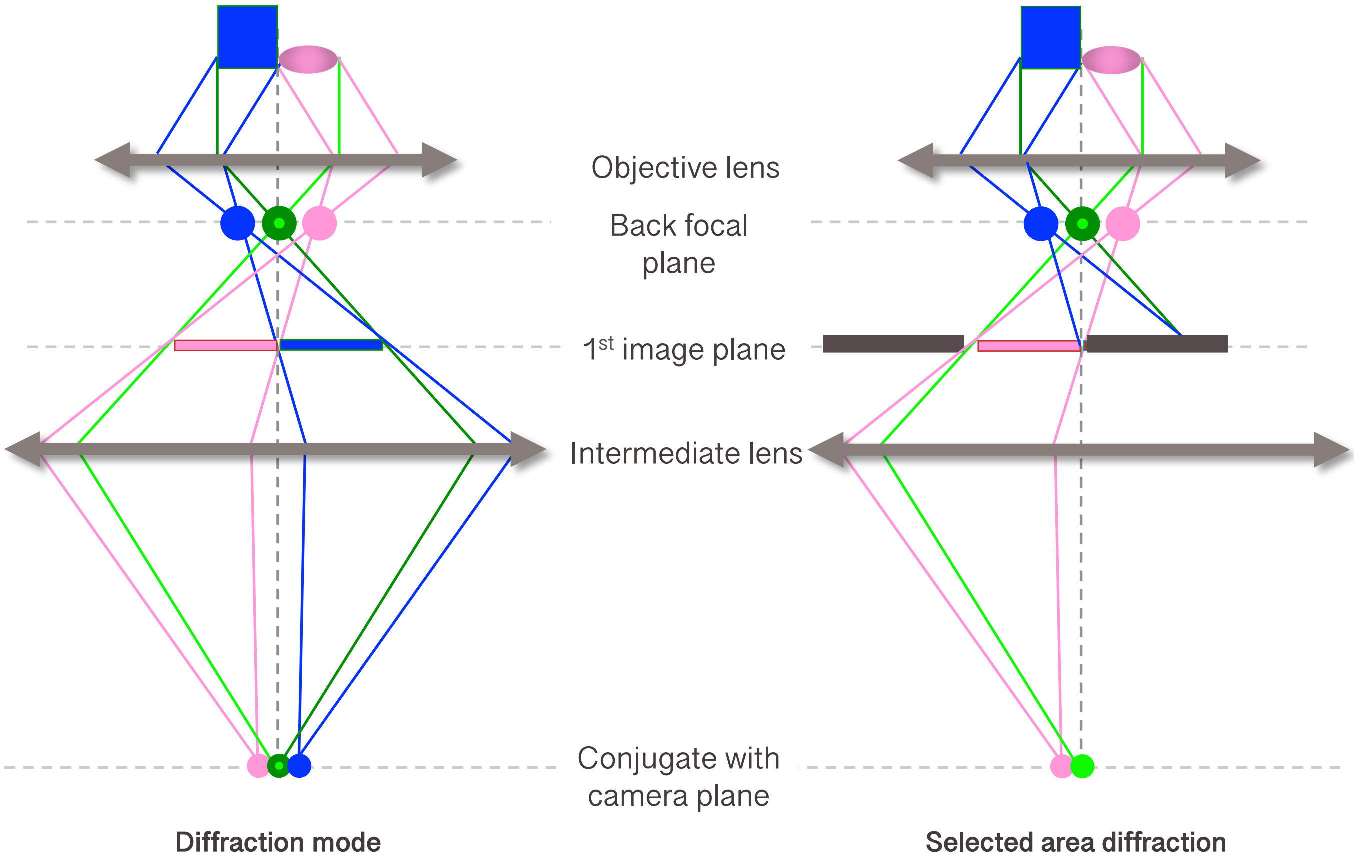
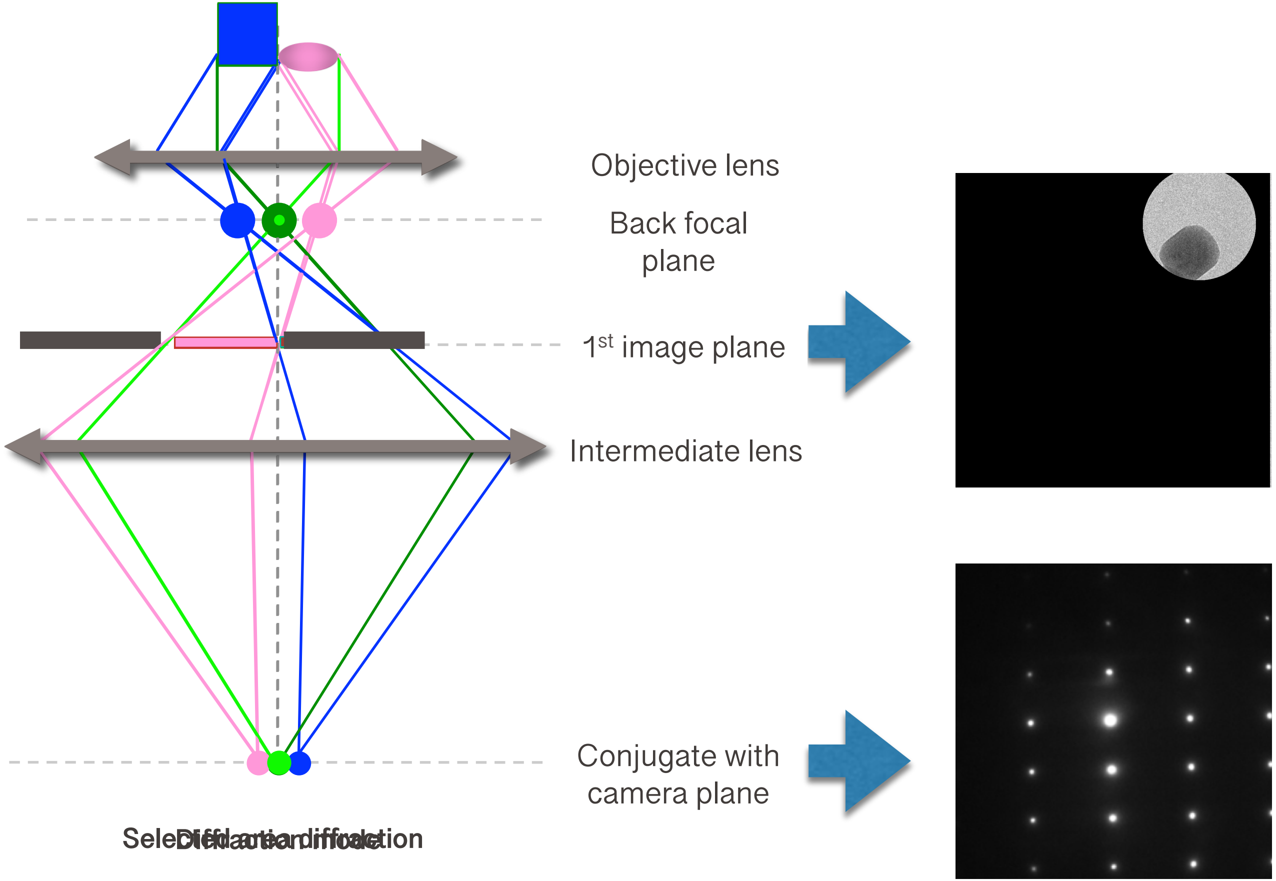


Imaging modes:
Selected area electron diffraction

Emad Oveisi



Imaging modes: Selected area electron diffraction



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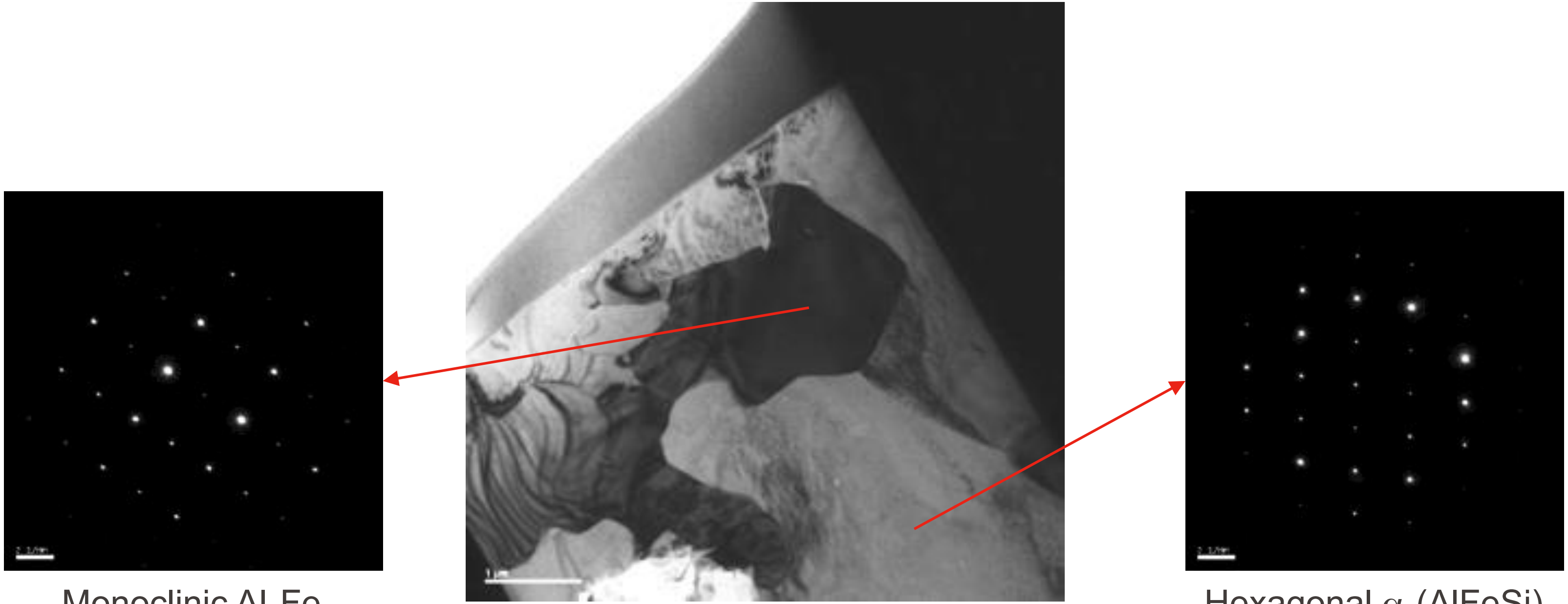
Imaging modes: Selected area electron diffraction



Monoclinic Al_3Fe

Hexagonal α-(AlFeSi)

Imaging modes: Selected area electron diffraction

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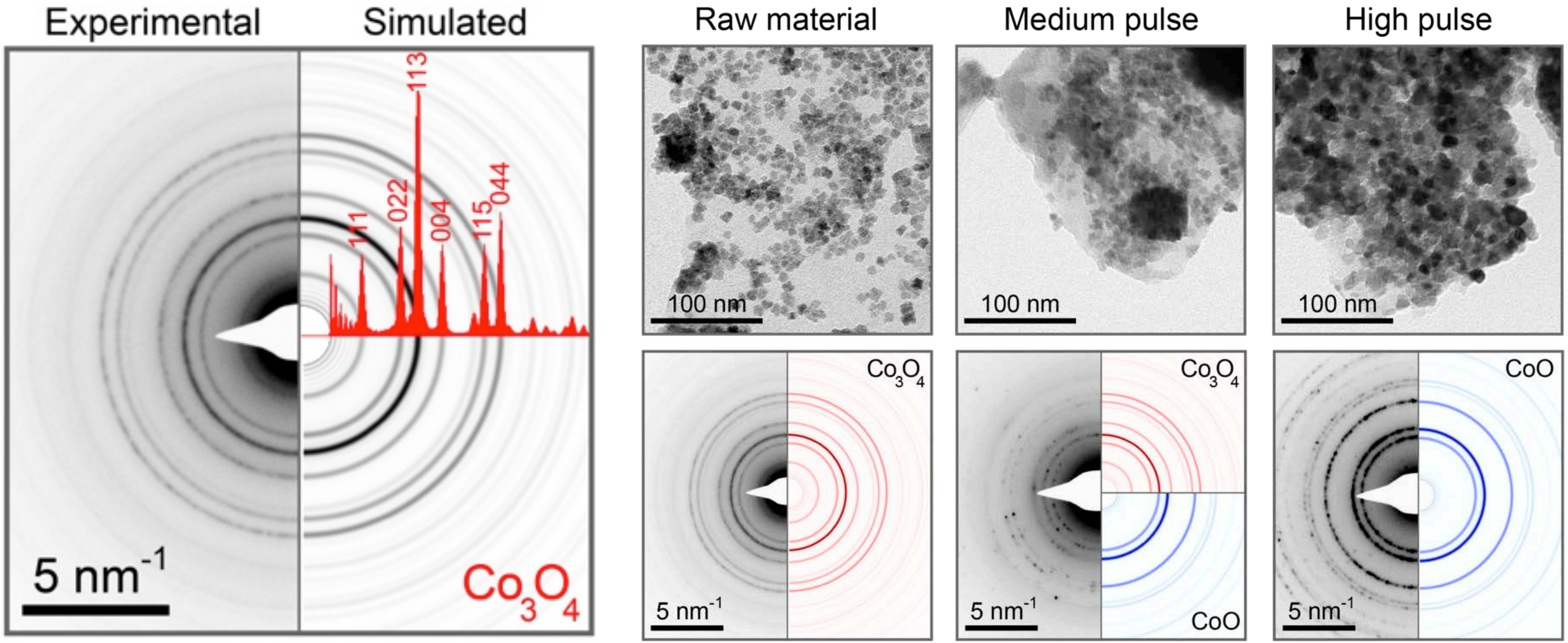
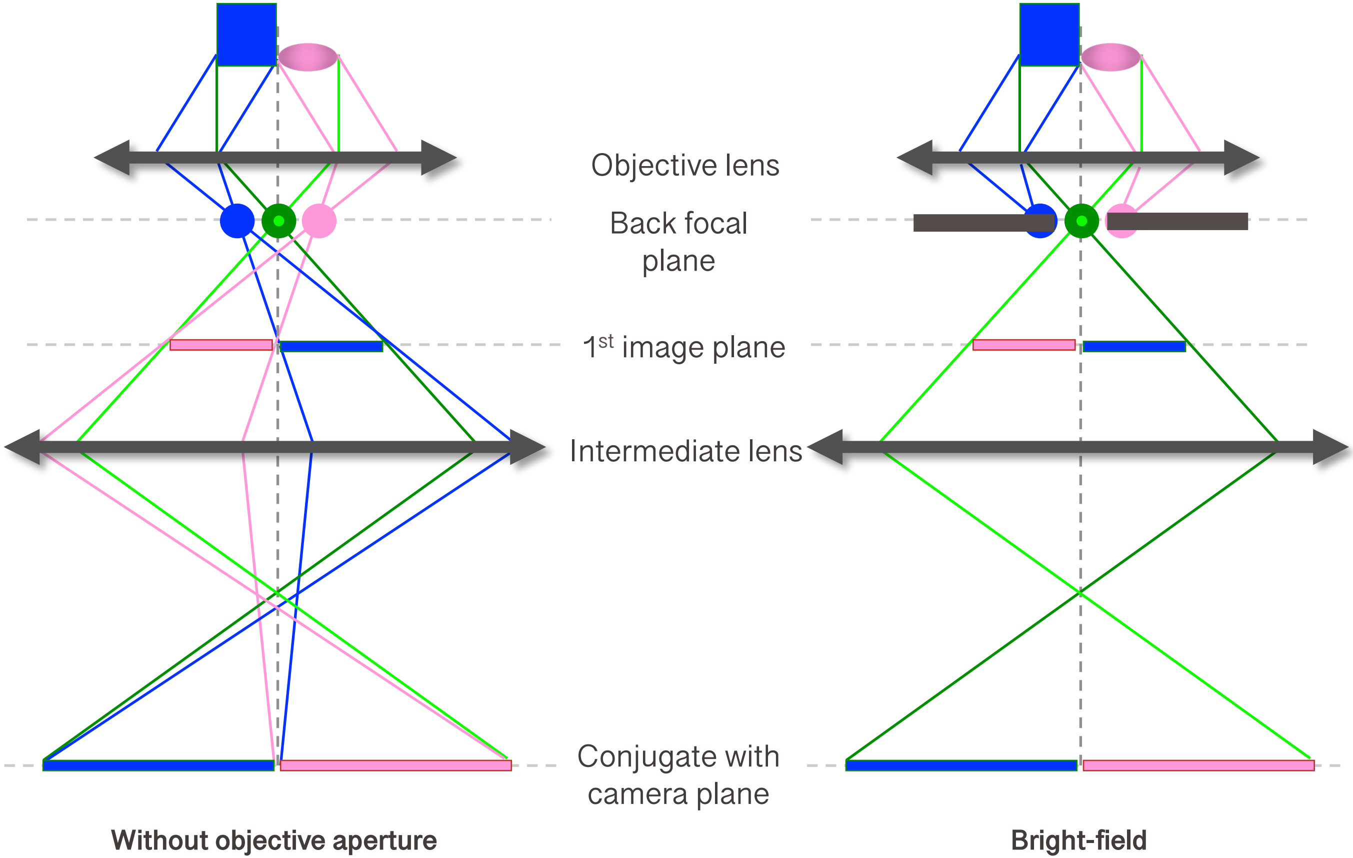
Example: Rapid inkjet printing of Co_3O_4/N-rGO layers for oxygen reduction reaction



In collaboration with LEPA-EPFL

Emad Oveisi

Imaging modes: Bright-field



Imaging modes: Dark-field

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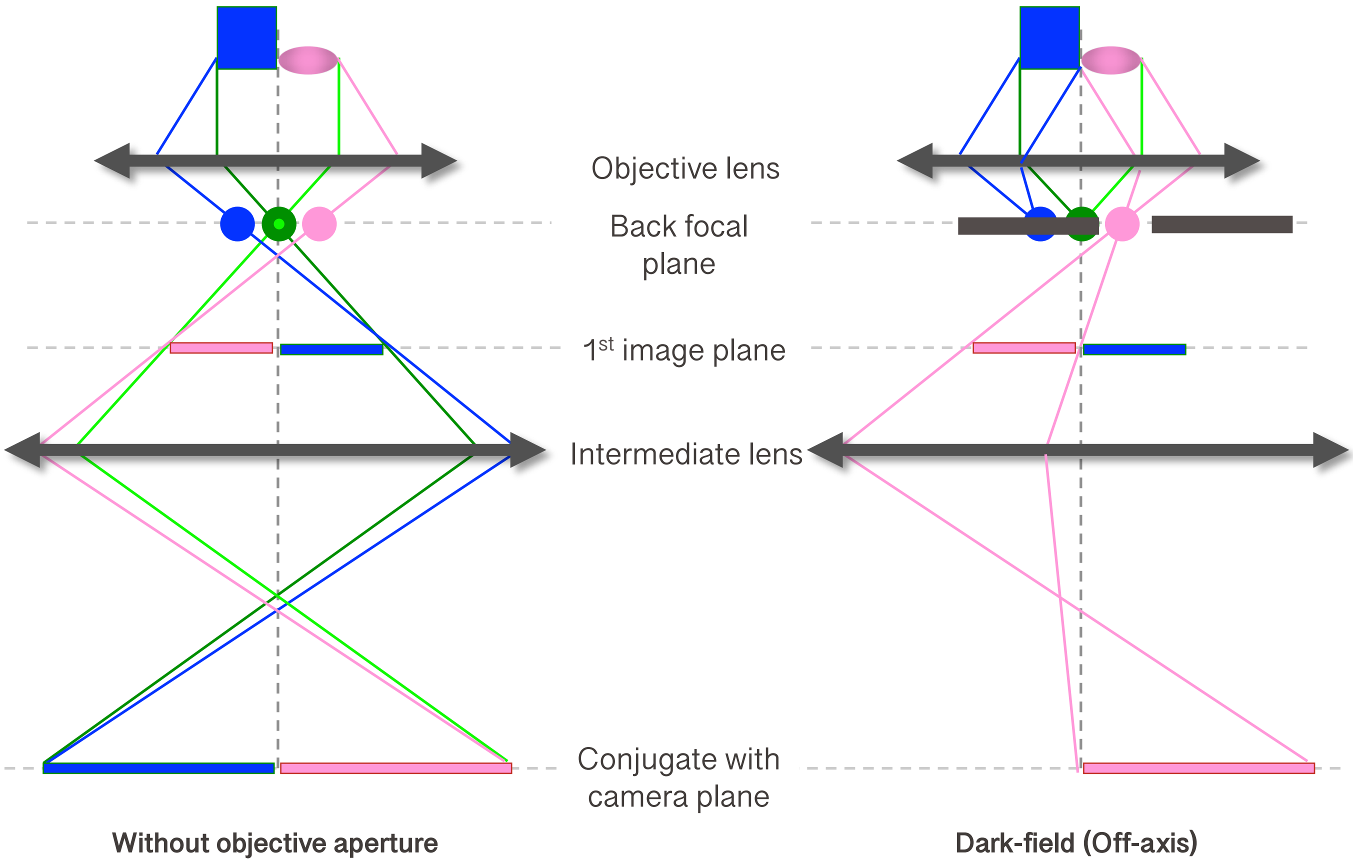
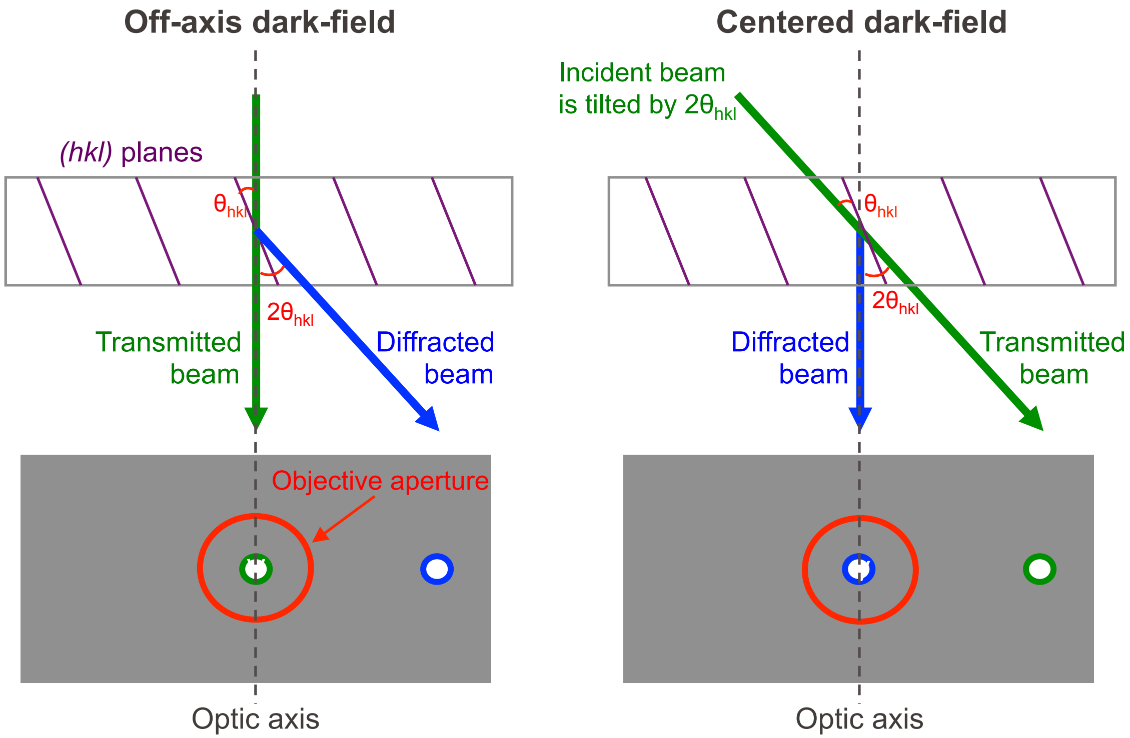


Dark-field imaging with tilted beam

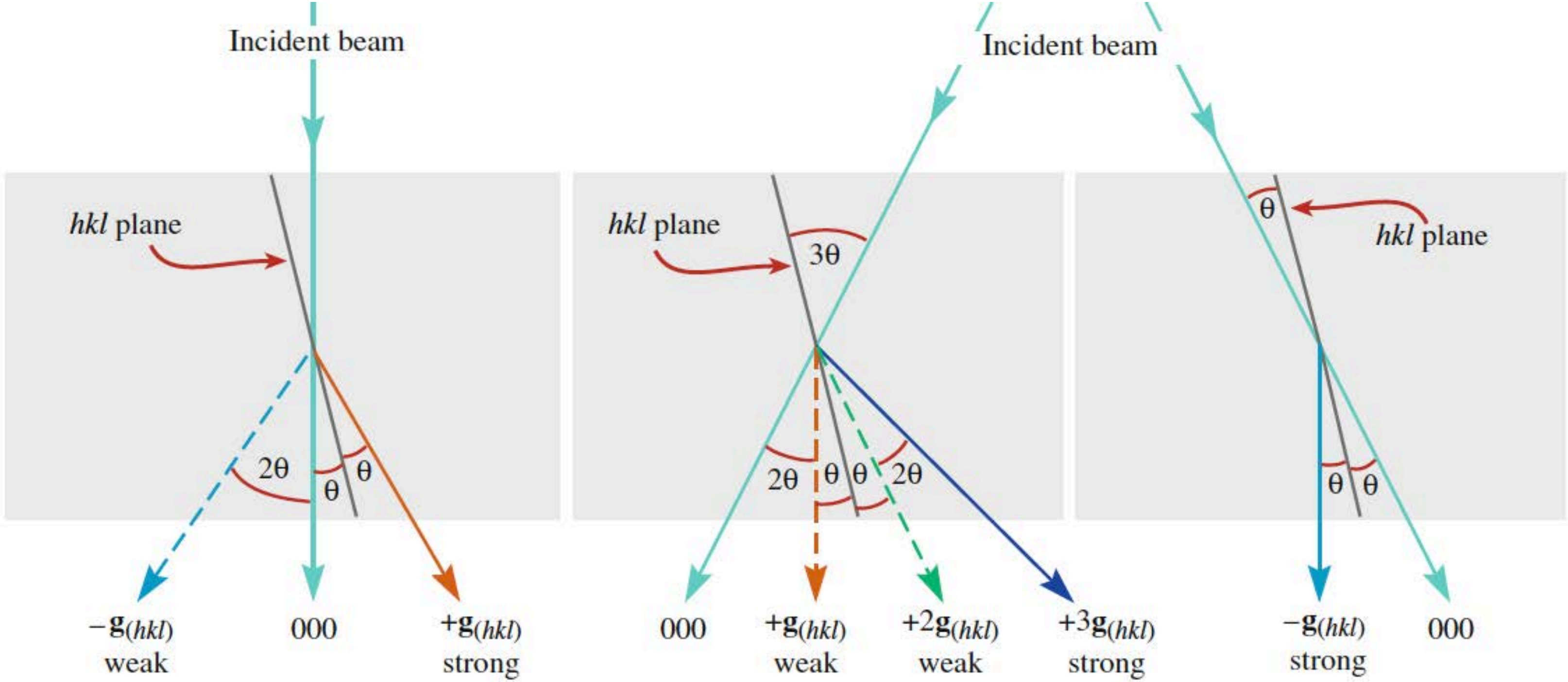
Two ways to setup a dark-field image:

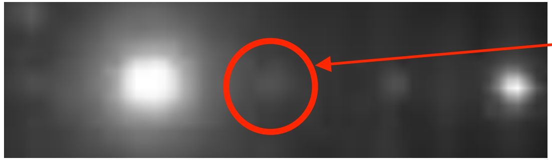
- Off-axis: Shift aperture to diffracted beam:
- Centered dark-field beam tilt: Use electromagnetic deflectors to tilt the incident beam on the sample
  ➔ diffracted beam on the optical axis



Which method produces fewer distortions, i.e. has better spatial resolution?

Imaging modes: Weak-beam dark-field





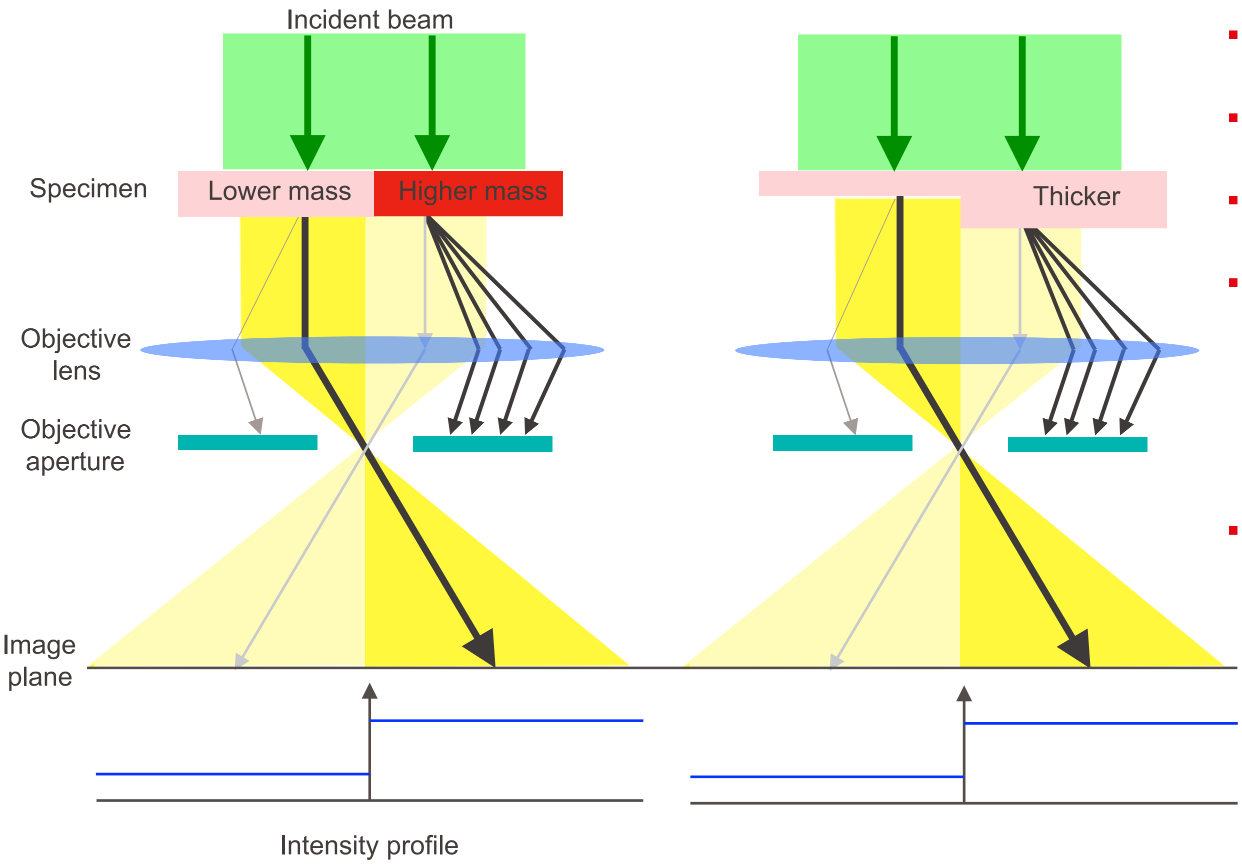
Objective aperture selects the weak reflection that is tilted to the the optic axis

From William and Carter's book

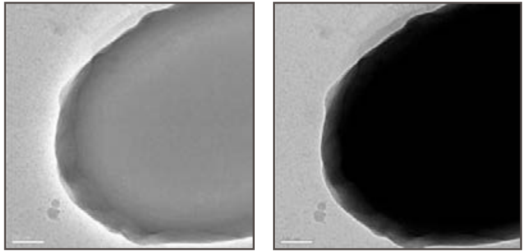
- **Image formation in TEM**
  - Image and diffraction modes
  - Bright- and dark-field modes
    - Strong- and weak-beam
  - High-resolution TEM

- **Image contrast in TEM**
  - Mass-thickness contrast
  - Diffraction contrast
  - Phase contrast

Mass-thickness contrast



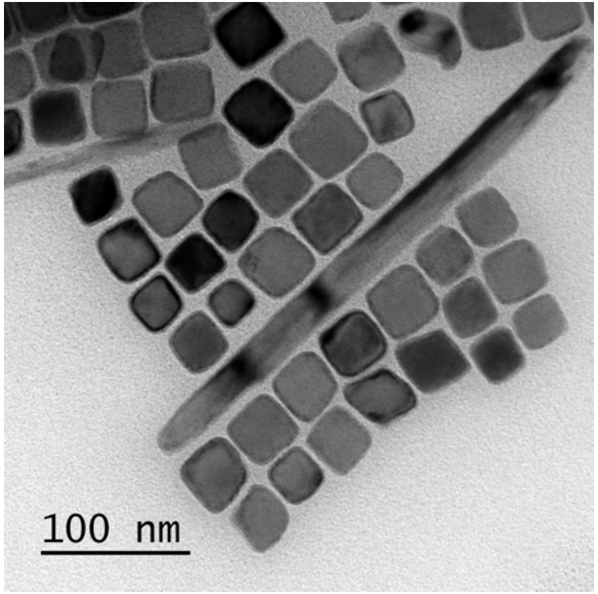
- Areas of higher mass/thickness scatter electrons more than others.
- Electrons are captured by the aperture and lost from the beam path.
- Areas of higher mass thickness will therefore appear dark in the image.
- This is known as:
  - **mass thickness contrast**,
  - **scattering contrast**,
  - **aperture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



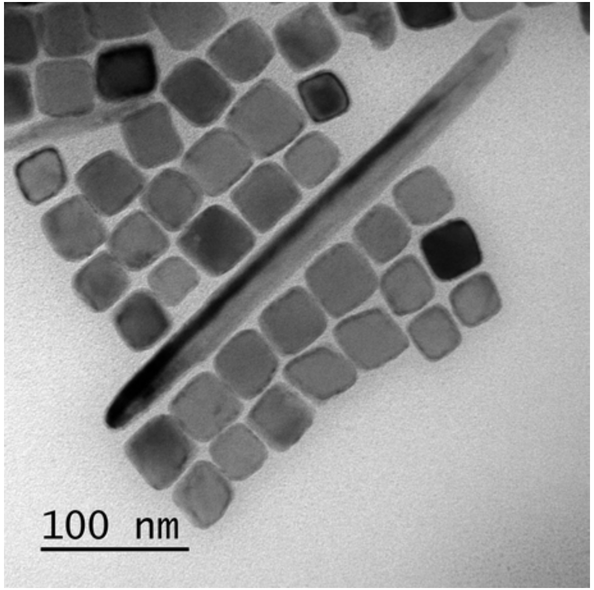
Without aperture　　With aperture

EPFL

Example: Cupper nano particles



Tilting the sample



Why some of cubic particles appear darker than others?

Note that contrast changes when tilting the specimen

Diffraction contrast

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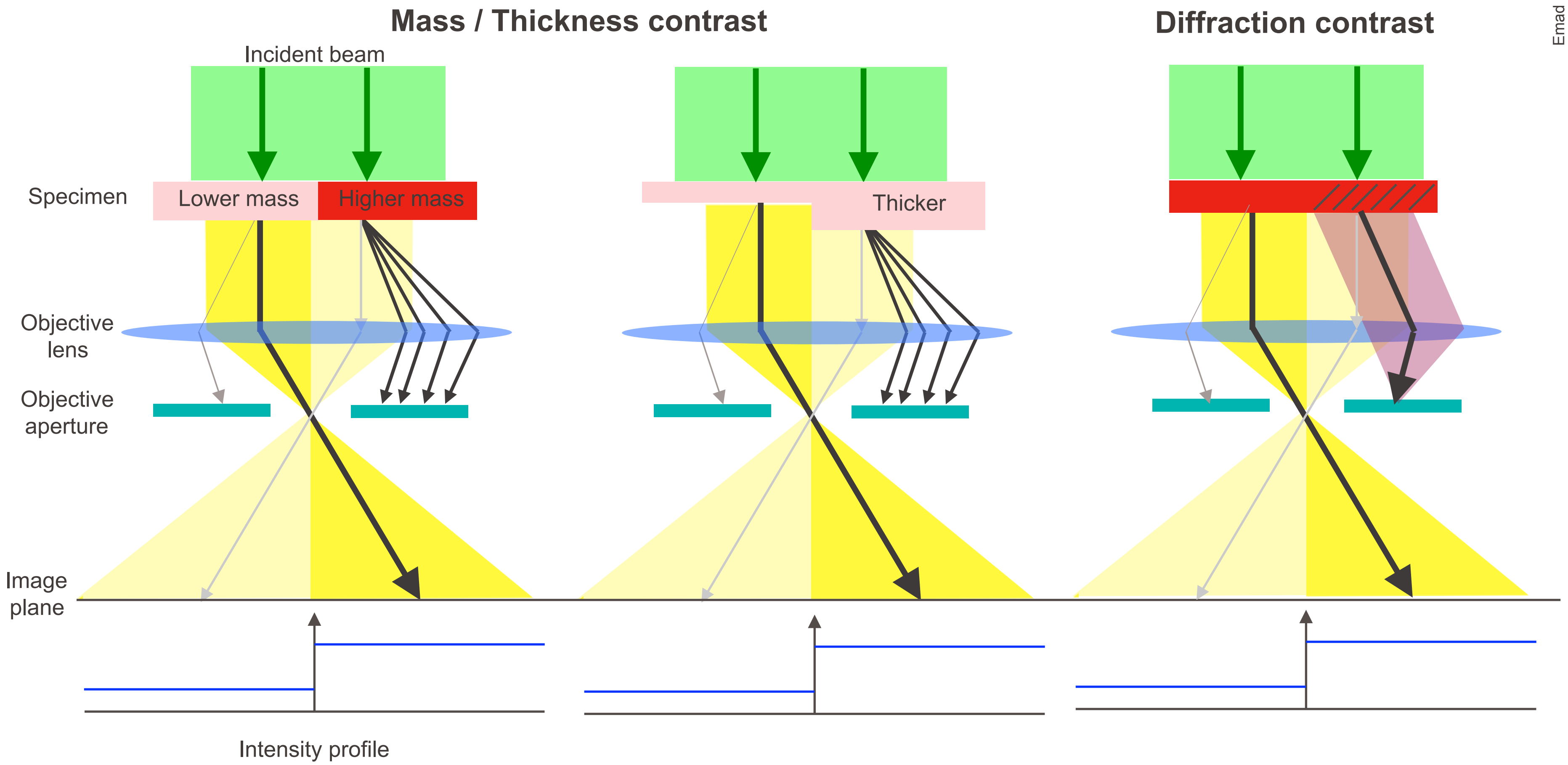
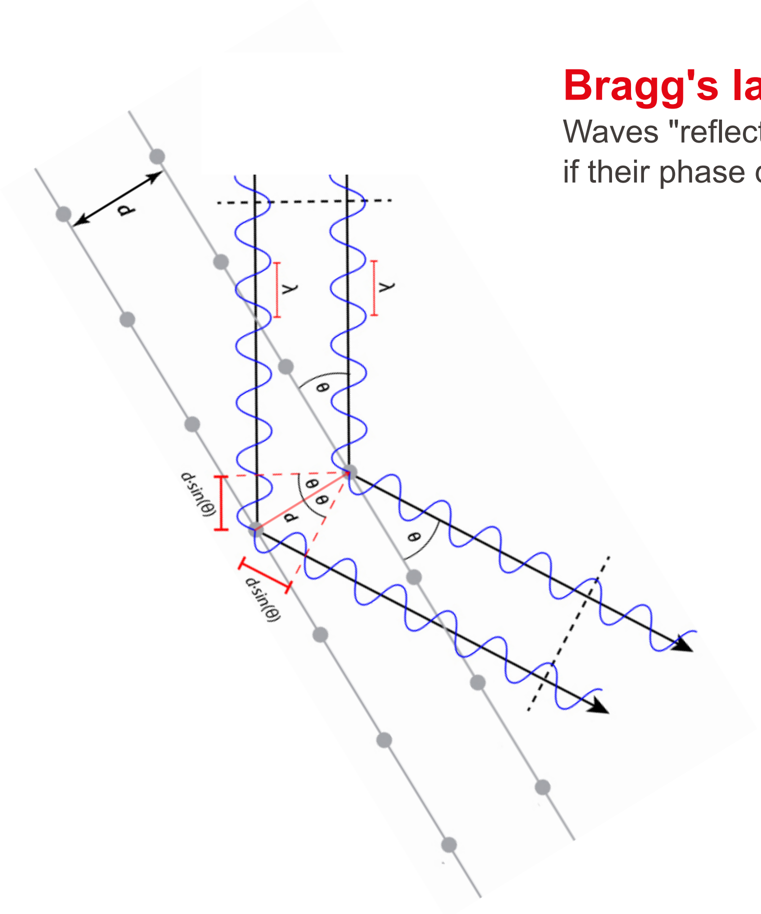
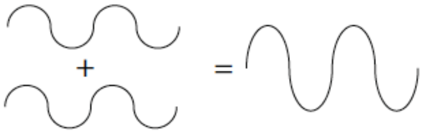


Electron diffraction



Bragg's law (William *Bragg, Nobel Prize Physics, 1915*)

Waves "reflected" of lattice planes with a spacing $d_{(hkl)}$ interfere constructively if their phase difference is a multiple of the wavelength λ.



Constructive intereference

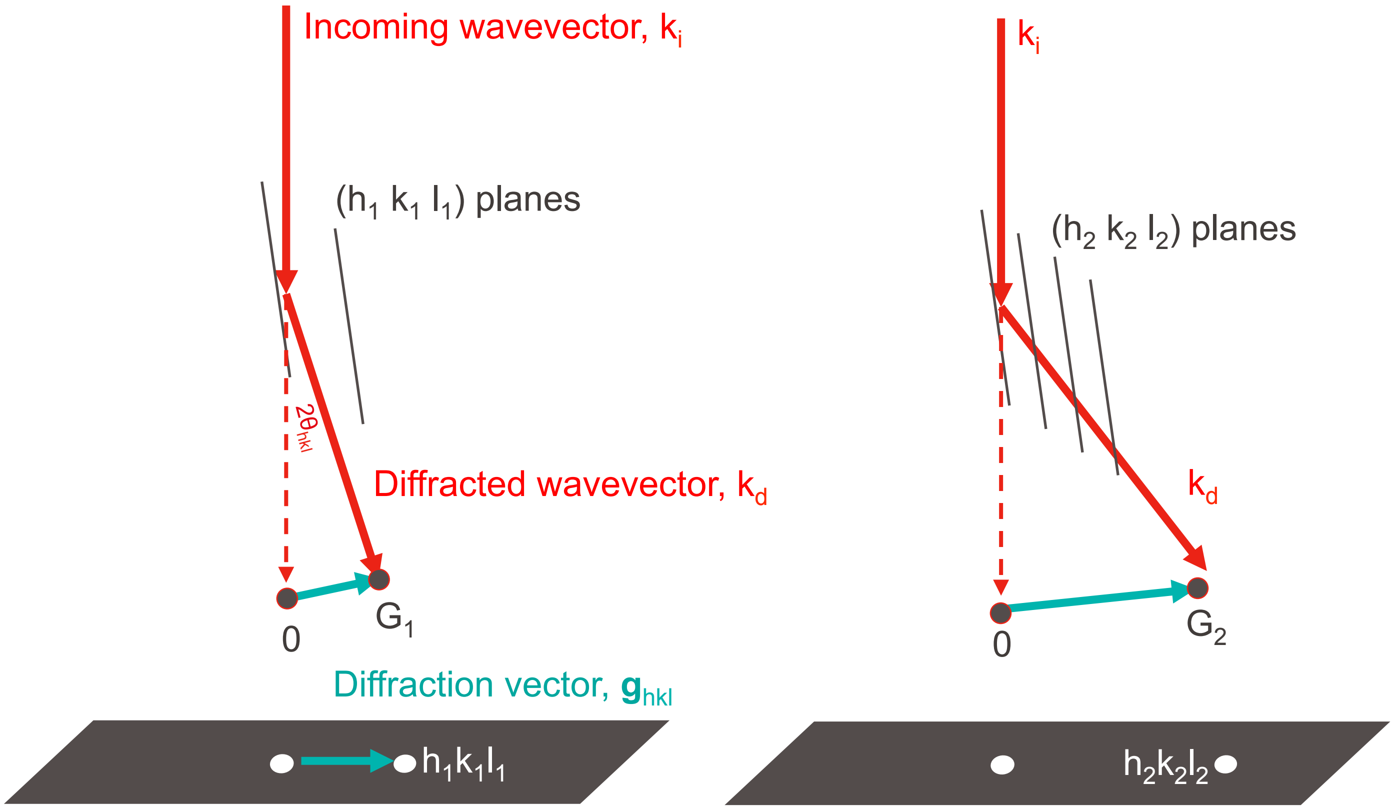
Bragg's law: $n\lambda = 2d_{hkl}\sin\theta$

Electron diffraction: λ ~ 2–3 pm
therefore: $\lambda \ll d_{hkl}$
⇒ small angle approximation: $n\lambda \approx 2d_{hkl}\theta$

Reciprocity: scattering angle $\theta \propto d_{hkl}^{-1}$

Atomic planes closer together => scattering angles greater

Electron diffraction

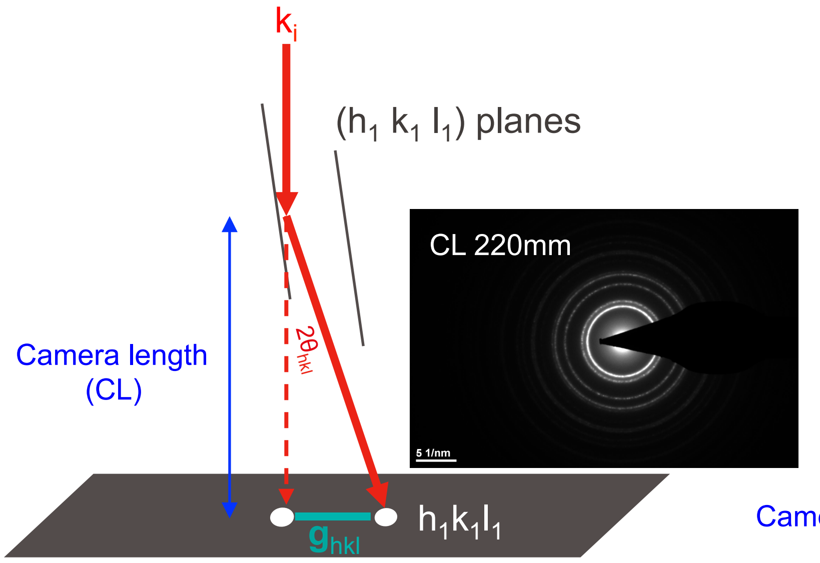


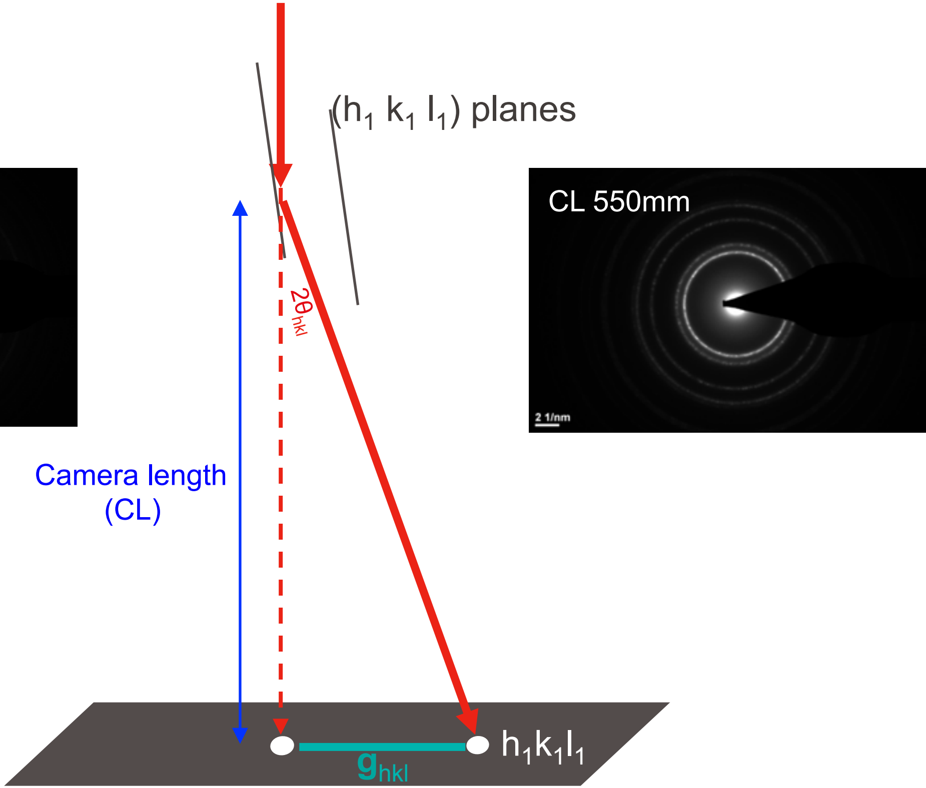
Atomic planes closer together = Greater scattering angle

Reciprocity: scattering angle $\theta_{hkl} \propto d_{hkl}^{-1}$

Electron diffraction

The magnification of the diffraction pattern (DP) is represented by the camera length CL.
The intermediate and projective lenses allow us to project the DP on the detector and change the CL.



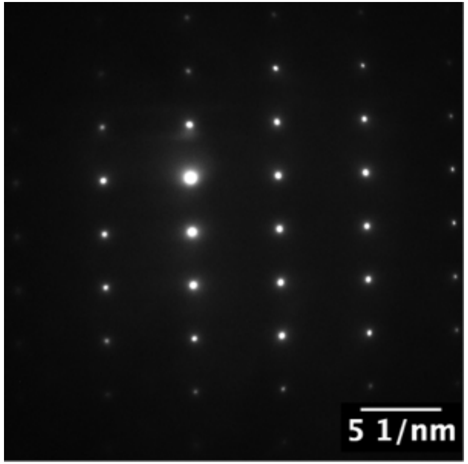


$\tan(2\theta_{hkl}) = \mathbf{g}_{hkl}/CL$
For small angles, $\theta \approx \sin\theta \approx \tan\theta \cong \mathbf{g}/CL$

Bragg's law: $2d_{hkl}\sin\theta_{hkl} = n\lambda$

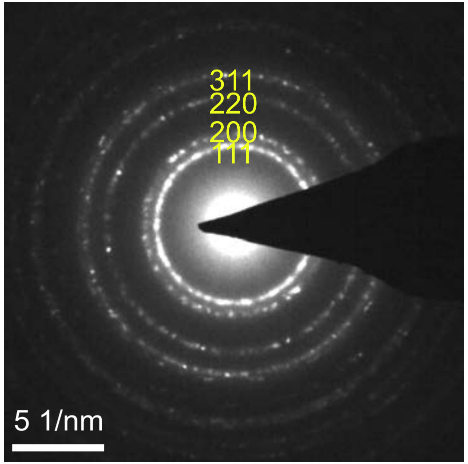
$d_{hkl}\,\mathbf{g}_{hkl} = \lambda CL$

Monocrystal



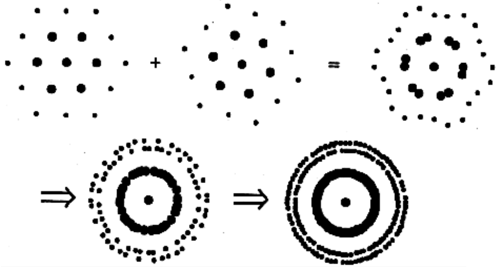
Oriented along a zone axis many (hkl) plans in diffraction conditions. Spot represent different (hkl) planes.

Polycrystalline

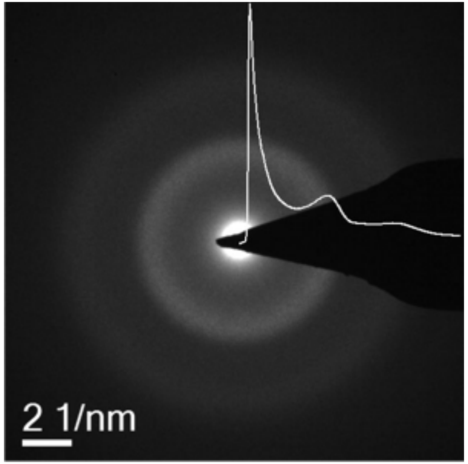


Ring pattern as many crystallites oriented differently in diffraction conditions. Each ring represents one set of (hkl) planes

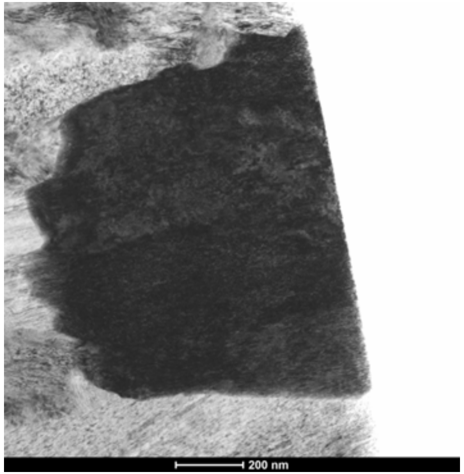
(hkl) ring's radius = d_{hkl}^{-1}



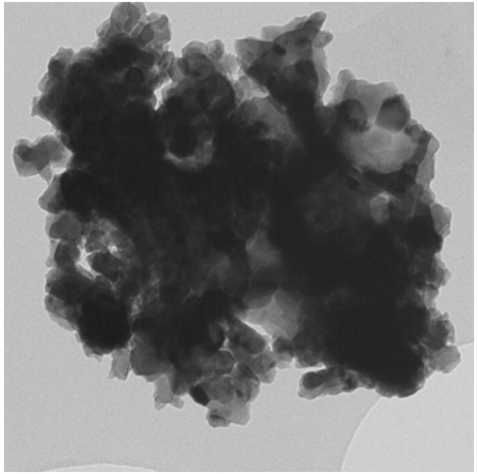
Amorphous

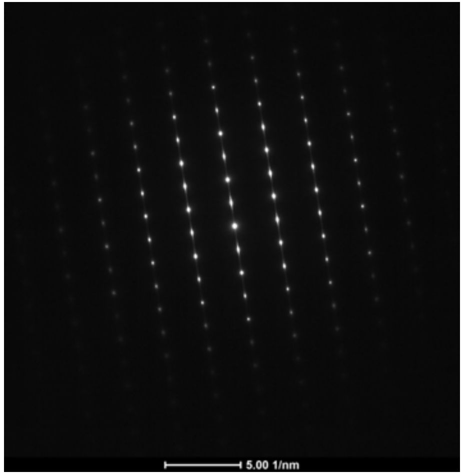


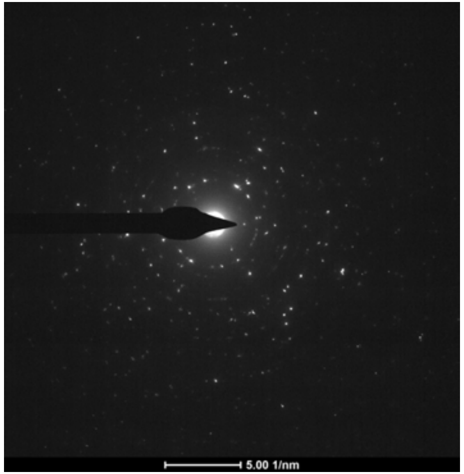
Diffuse ring due to short-range order as interatomic distance ~ constant
Radial distribution function

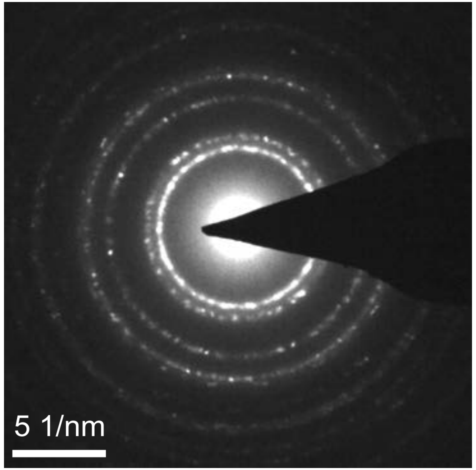

200 nm


200 nm



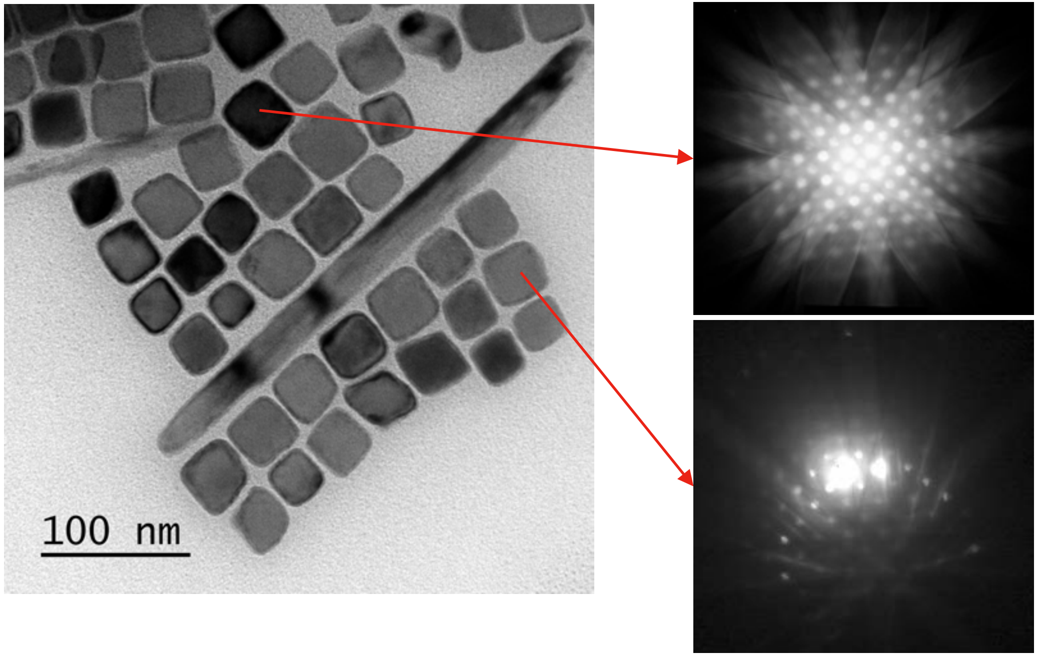

5.00 1/nm


5.00 1/nm


5 1/nm

Diffraction contrast

Example: Cupper nano particles



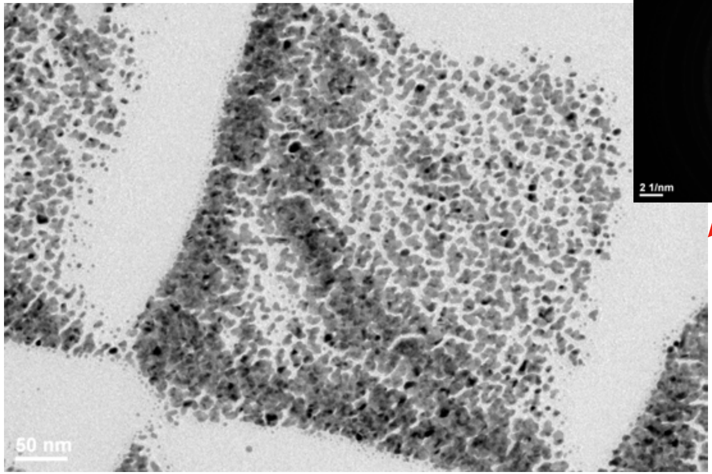
Crystal in the strongly diffracting condition:
Low intensity direct beam ➔
Dark crystal in the bright-field image

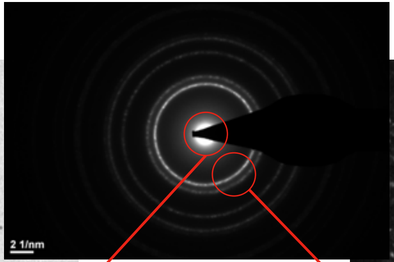
Crystal out of strongly diffracting condition:
Higher intensity direct beam

Diffraction condition changes when tilting the specimen

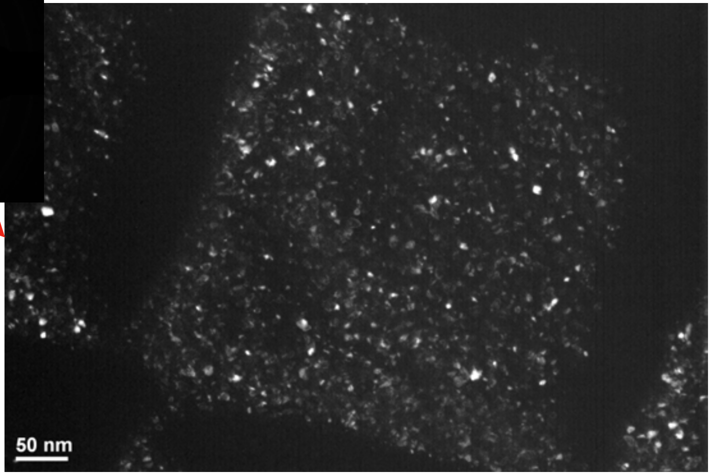
Diffraction contrast: Bright- and dark-field images

Bright-field image





Dark-field image

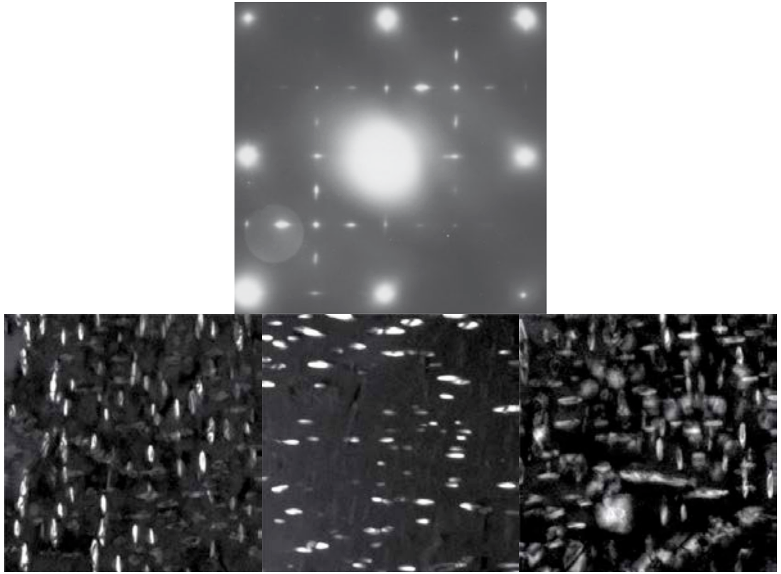


- Strongly diffracting crystals: dark
- Removes ghost image

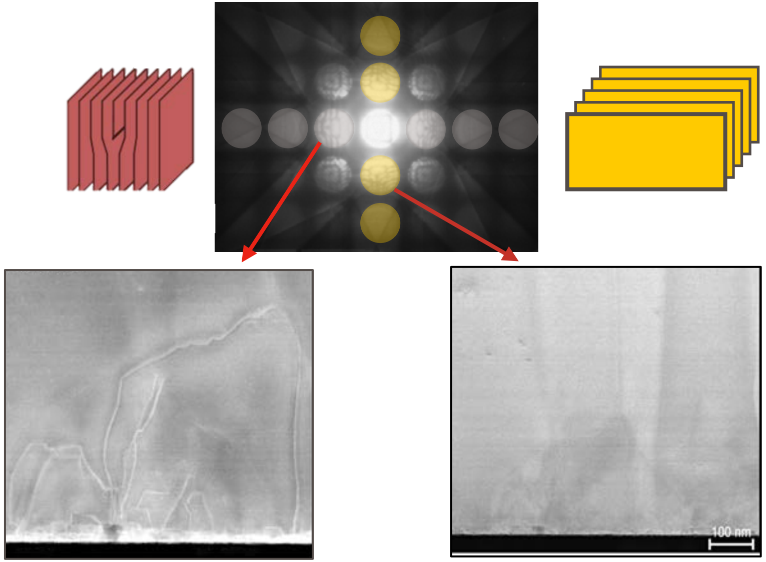
- Only crystals strongly diffracting into objective aperture: bright
- Possibility of crystal phase/orientation discrimination

Applications of diffraction contrast

Map intensity in the diffracted beam



Crystal phase/orientation discrimination

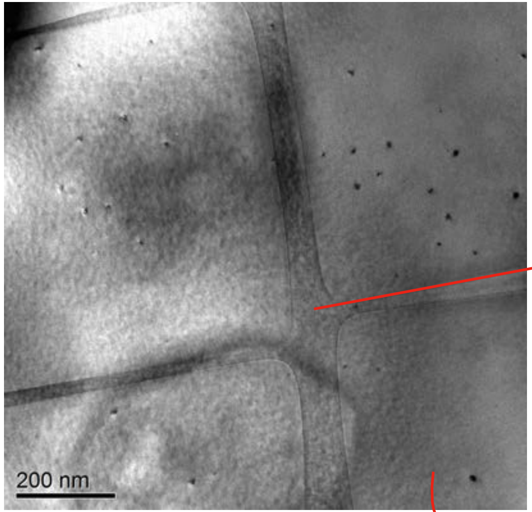


Crystal’s defect imaging/analysis

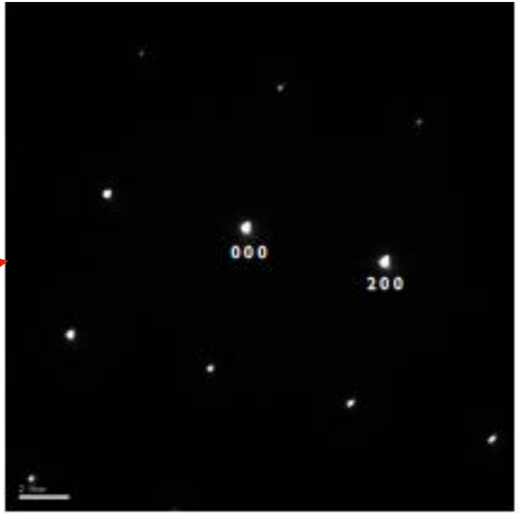
Applications of diffraction contrast

Crystal phase discrimination
Example: Ni_3Al-based superalloy

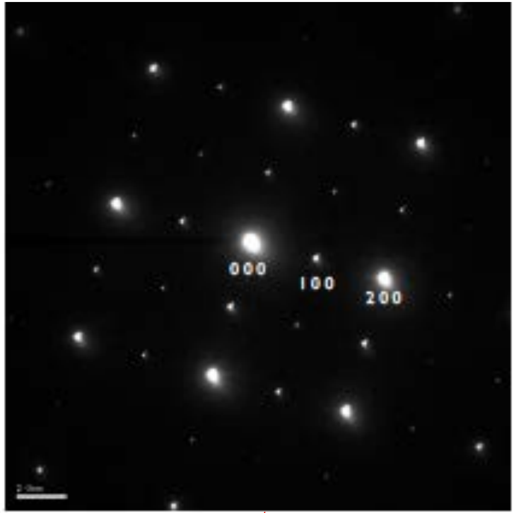
Bright-field



γ-phase matrix: FCC
(Ni, Al disordered on sites)



γ'-phase precipitate: primitive cubic
(Ni on face centres, Al on corners)



Note: EDX can't discriminate these two crystal phases!

Applications of diffraction contrast

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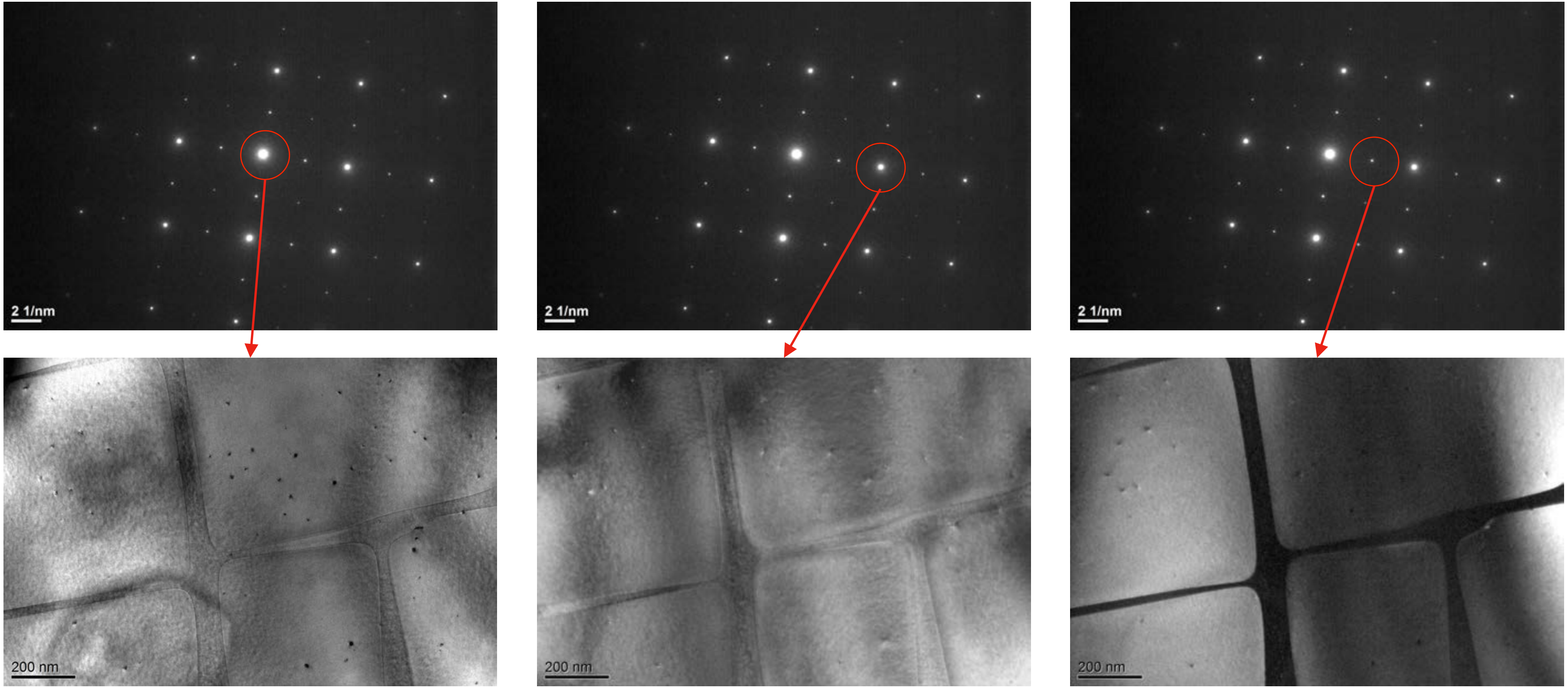
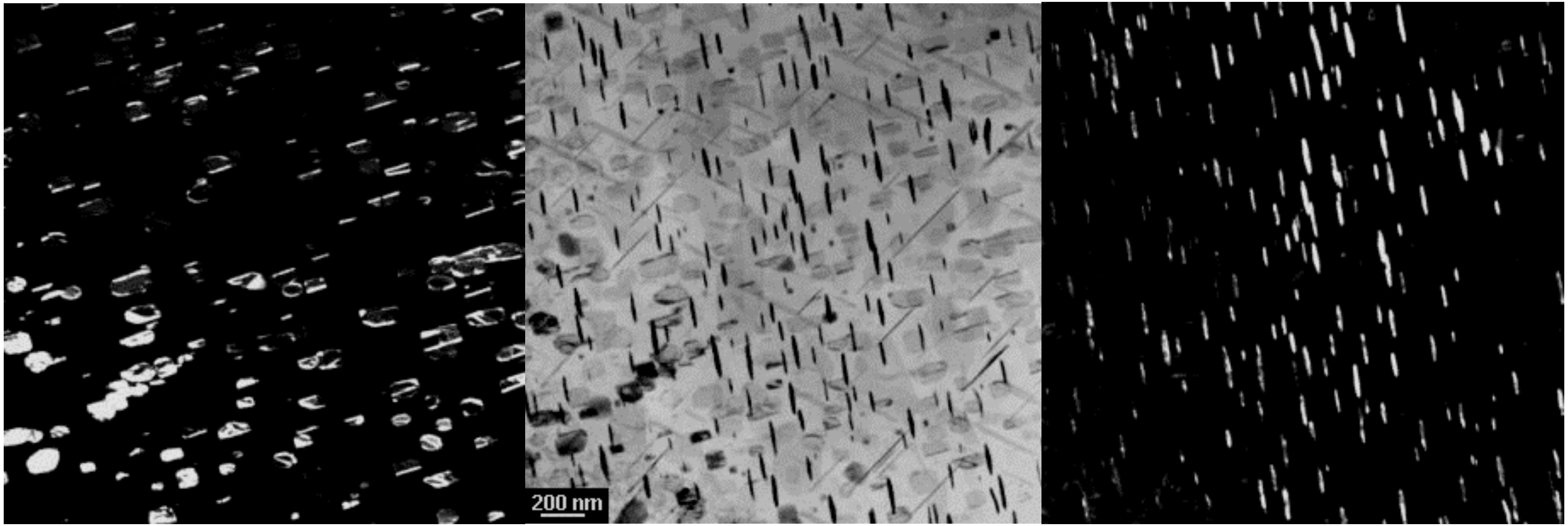
Crystal phase discrimination
Example: Ni_3Al-based superalloy



Bright-field image | Dark-field image **g** = (2 0 0) | Dark-field image **g** = (1 0 0)

Applications of diffraction contrast

Crystal phase and orientation discrimination
Example of aluminum alloy containing precipitates with preferential growth direction



Orientation relationship between the matrix and precipitates can be determined

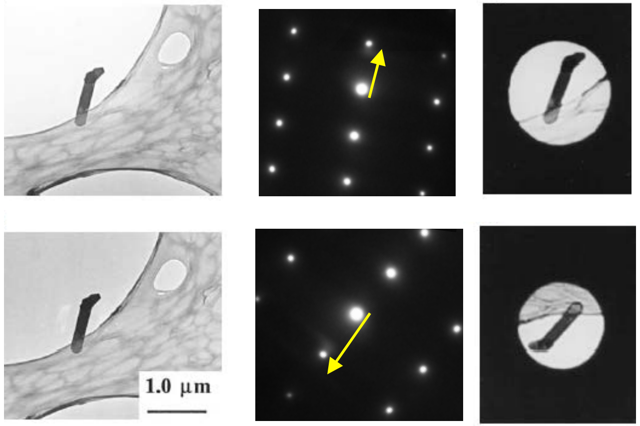
Calibration of rotation between the image and diffraction is necessary.

Rotation between image and diffraction

Image | DP | Defocused DP

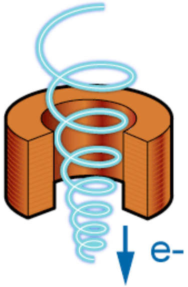
CL 220mm

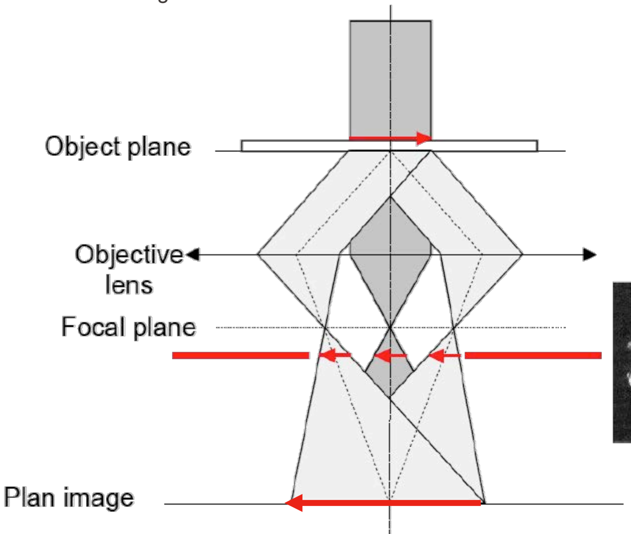
CL 550mm

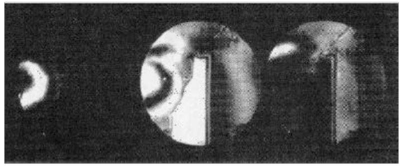


No rotation between image and DP

Rotation between image and DP







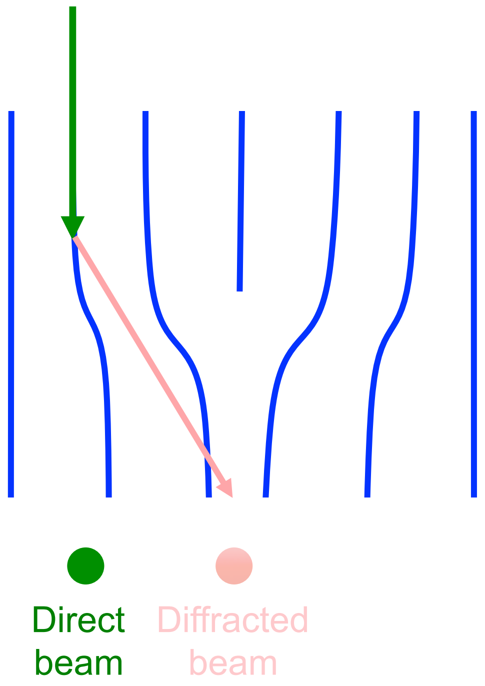
Defocused diffraction pattern

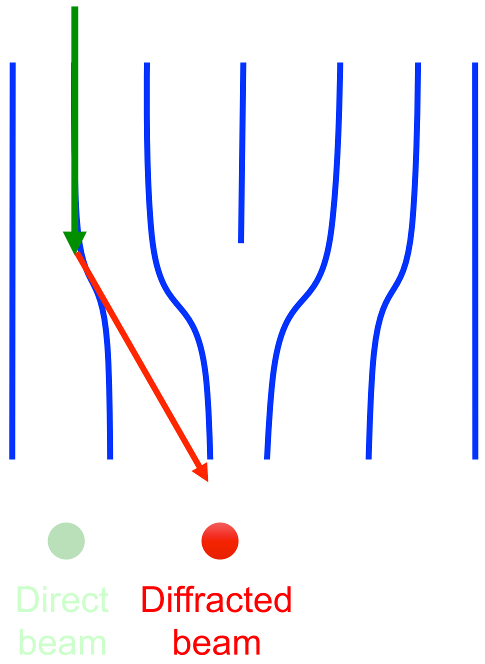
From William and Carter's book

Applications of diffraction contrast

Emad Oveisi

Crystal's defect imaging/analysis
Specimen is at near-Bragg condition

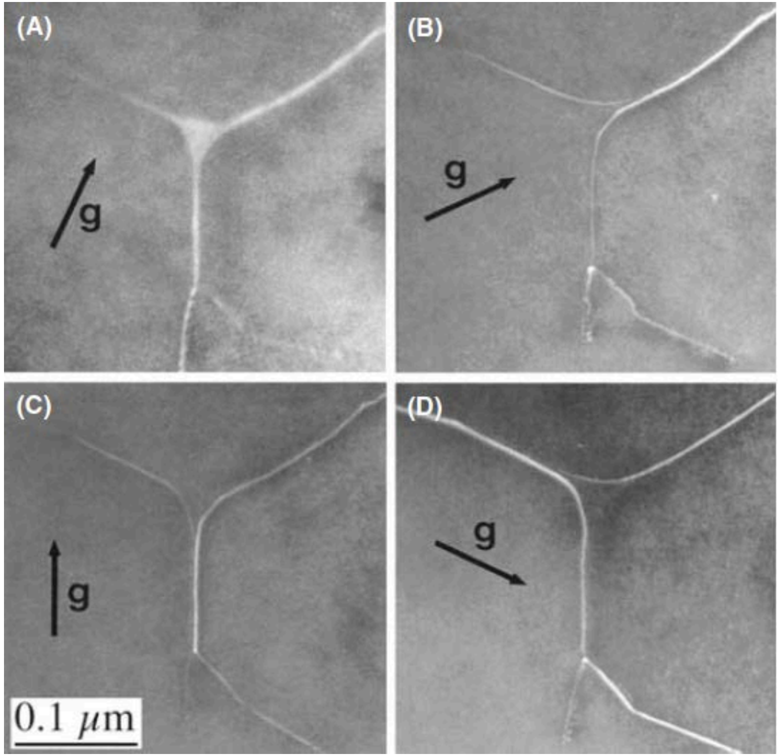




Near dislocation core, crystal is distorted and is at the exact Bragg condition: intensity in diffracted beam and hence image increases

Applications of diffraction contrast

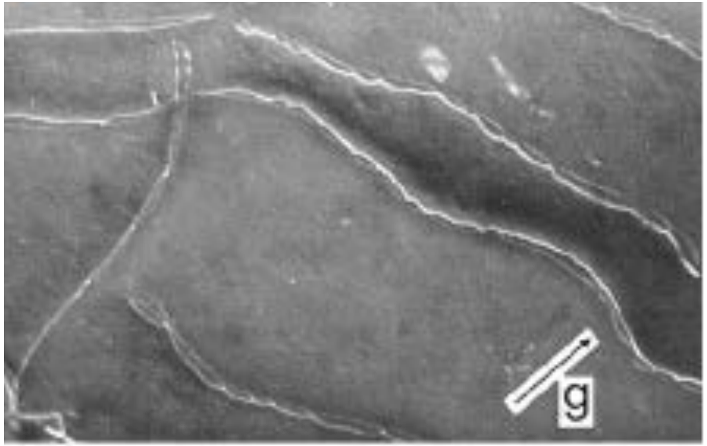
Crystal's defect imaging/analysis
Example: Pairs of dislocation nodes in a Cu alloy



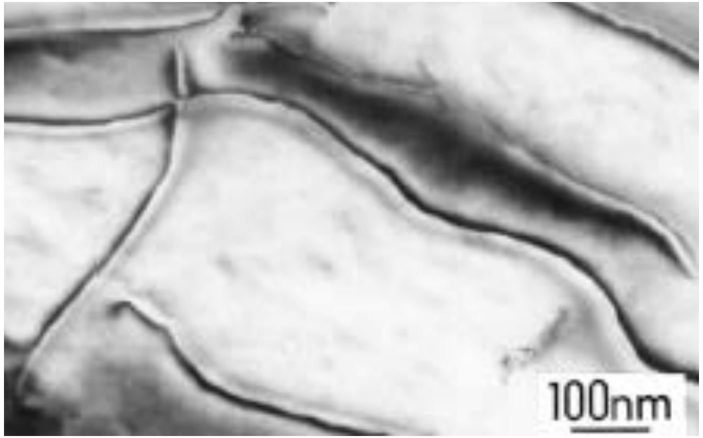
From William and Carter's book

Applications of diffraction contrast

Comparison of dislocation images in Cu alloy



Weak-beam dark-field



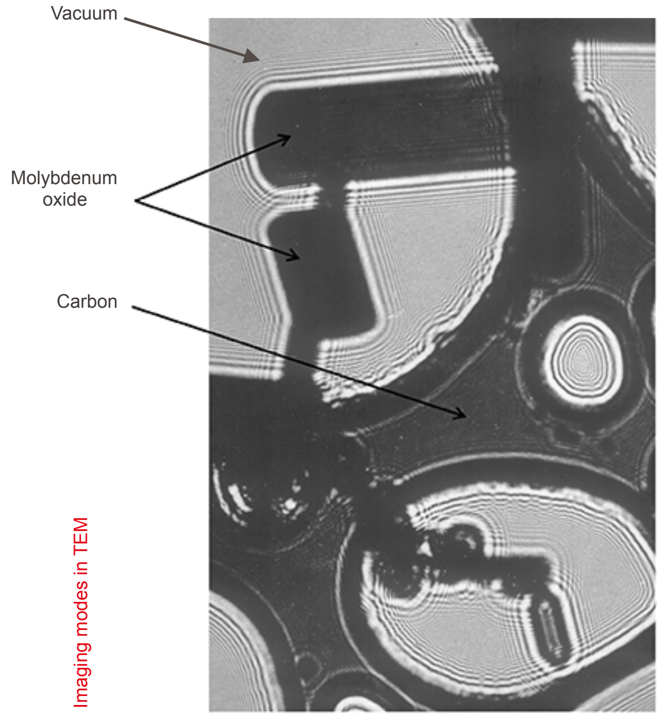
Strong-beam dark-field

Will be discussed on Wednesday by Dr. Duncan Alexander

From William and Carter's book

Phase contrast | Fresnel fringes

What happens when an electron wave passes through the sample?



Molybdenum oxide particles on a holey carbon grid at 80kV

Image from Jeol.co.jp

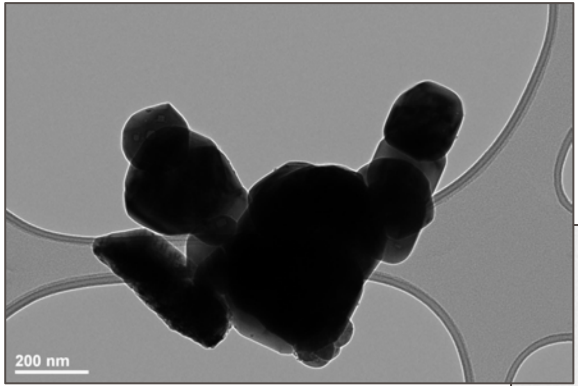
A spherical wave scattered from the edge of a specimen interferes with the incident wave. Then, interference fringes are produced whose period becomes narrow with the distance from the edge of the specimen.

Fringes arise due to the high coherence of the electron beam and interface of two electron waves that are scattered differently, e.g. one which is not scattered (vacuum) and one that scatters off the edges of sample features (the hole in this example), resulting in a path length difference.

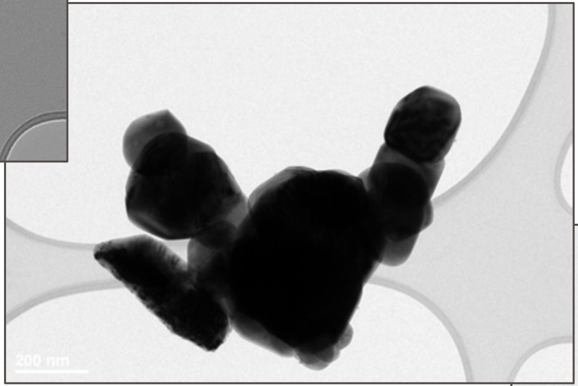
Phase contrast | Fresnel fringes

Emad Oveisi

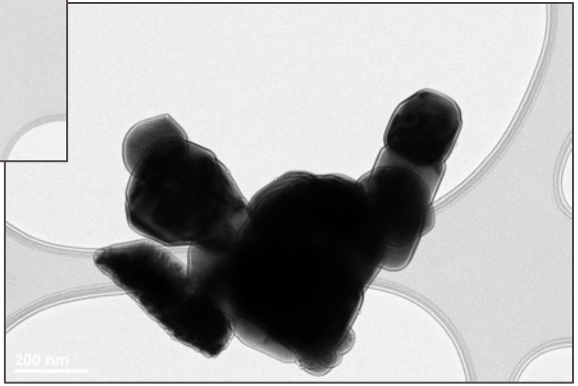
Bright or dark fringe thickness depends on focus!



Under-focused
Uniform white fringes



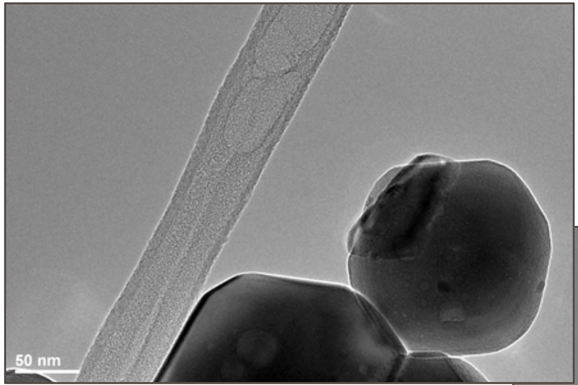
In focus
Min of contrast, no fringes



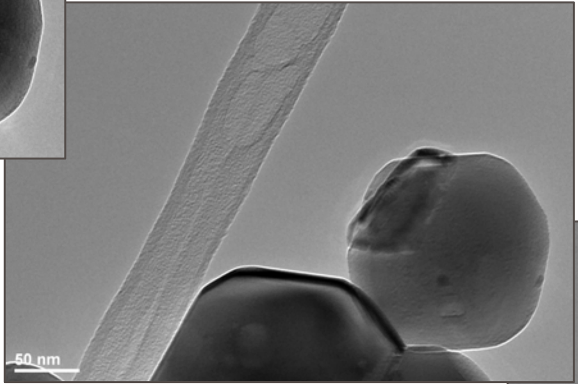
Over-focused
Uniform dark fringes

Phase contrast | Fresnel fringes

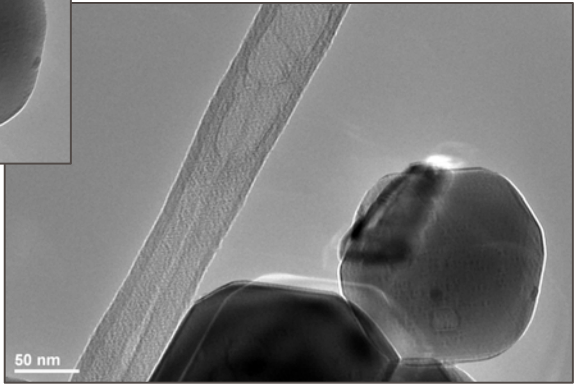
Fringes can also be used to observe and correct the astigmatism of the objective lens.



Without objective astigmatism
Uniform fringes



With objective astigmatism
Fringes varying thickness

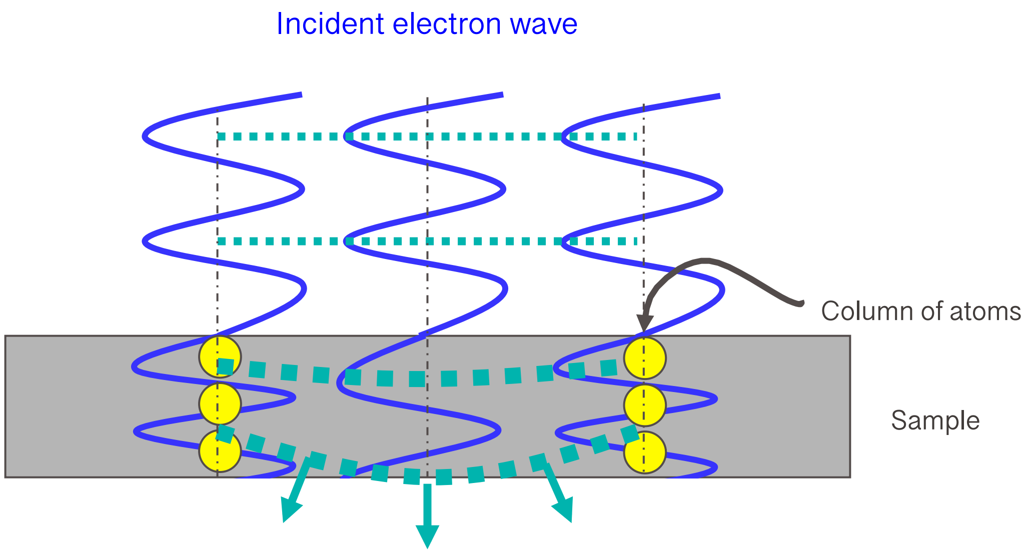


With large objective astigmatism
Fringes varying

Emad Oveisi

Phase contrast | High-resolution TEM

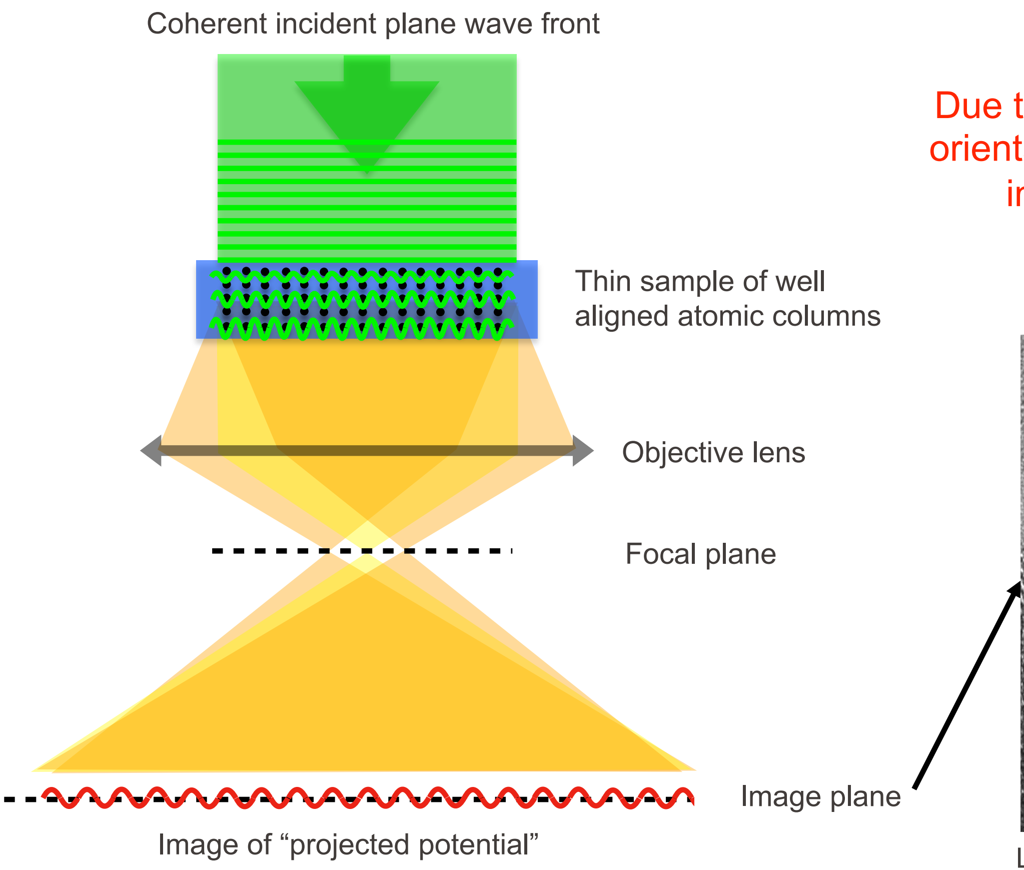
What happens when an electron wave passes through the sample?



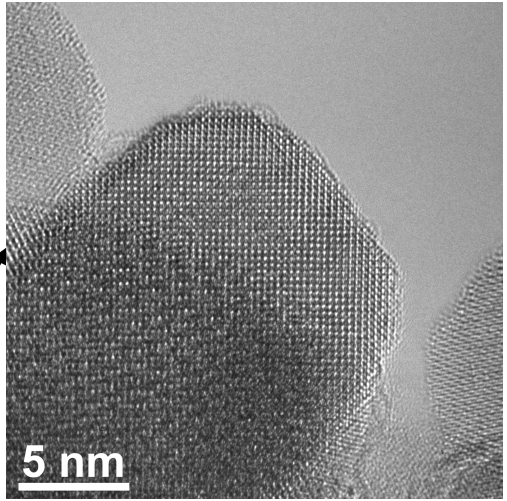
Variations in the projected potential produce local relative phase shifts of the electron wave. The wave front therefore bends as wave travels through medium.

The direction of propagation of the electron may change! ⇒ Diffraction!

Phase contrast | High-resolution TEM



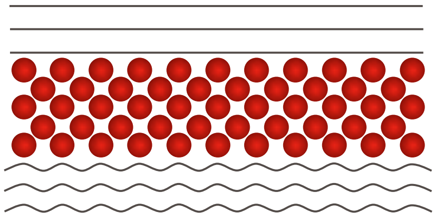
Due to changes in sample thickness and orientation, as well as lens imperfections, image interpretation is complex.

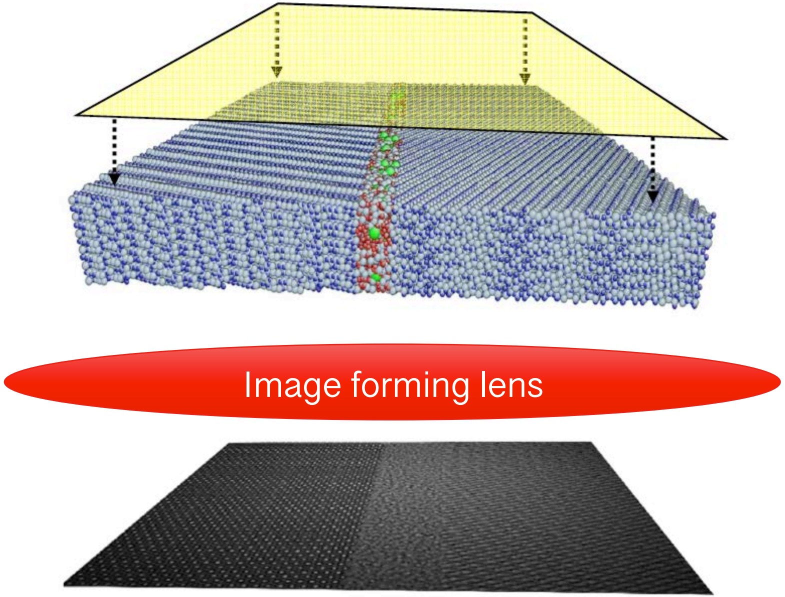


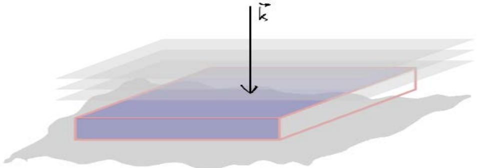
Lattice fringes in iron oxide nanoparticles

Phase contrast | High-resolution TEM

Emad Oveisi







But …
not a perfect lens!

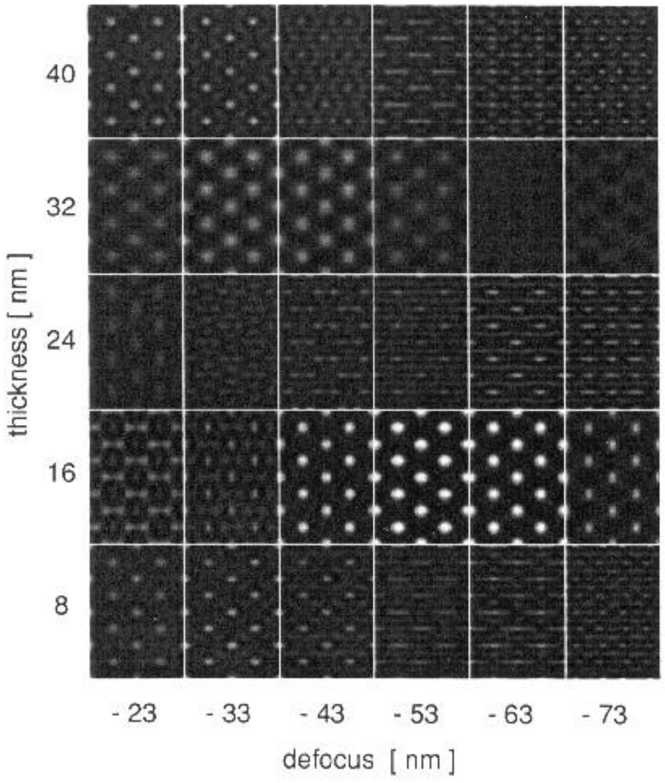
HRTEM can give you local structural information of your specimen.

However, do not expect straightforward interpretation (dots do not always correspond to atoms).

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

Schematic from C.T. Koch, QEM's lecture 2013.

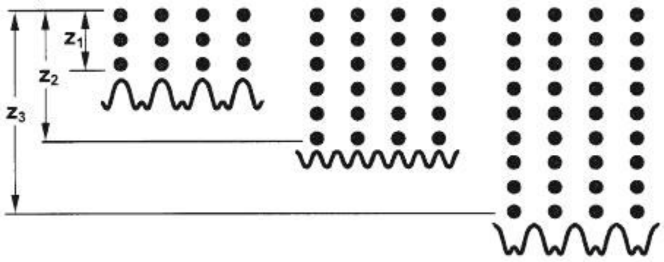
Phase contrast | High-resolution TEM



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.

Miroslav Karlík, Materials Structure, 8 (2001) 3

More details on HRTEM on Tuesday by Dr. Cantoni

Summary

Mass-Thickness contrast

- Applies for both crystalline and amorphous phase and scales with Z^2t

Diffraction contrast

- Bright-field: objective aperture centered on “unscattered” beam
- Dark-field: objective aperture centered on “diffracted beams”
- Weak Beam Dark-field: sample tilted to excited higher order “3g” reflections to higher intensity and objective aperture centered on low intensity “1g” spot

Fresnel contrast

- Developed by the interference of two waves, reference vacuum wave and one which scatters off the edges in the sample causing a path length difference and phase shift, resulting in fringe patterns that depend on the focus setting of the microscope
- Can be used to correct objective astigmatism

HR-TEM or 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