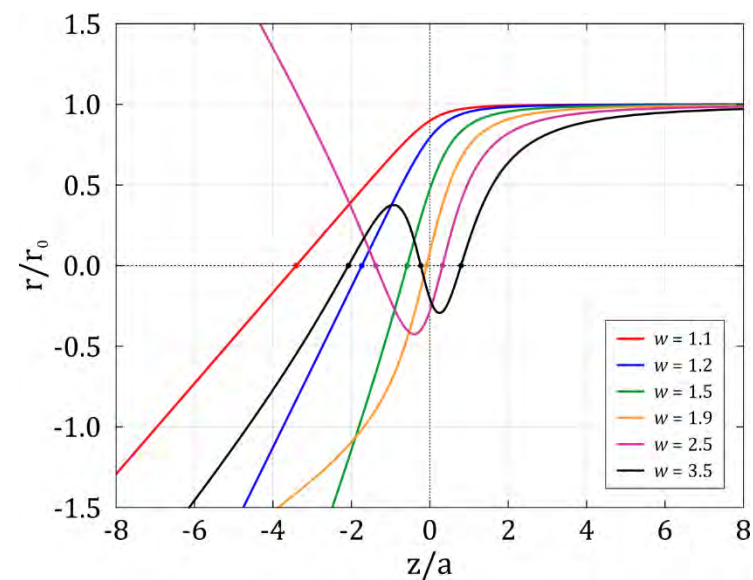
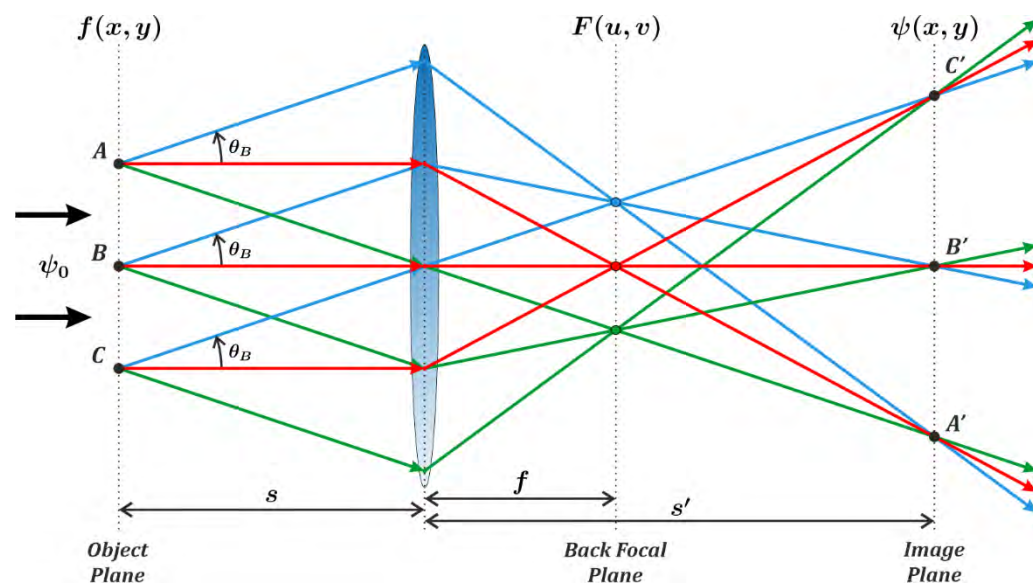


Quasi-Parallel and Precession Electron Diffraction: an Electron Optics Perspective

Sergi Plana Ruiz

*email contact: sergi.plana@urv.cat

NanED Workshop II, 7th of December 2022



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UNIVERSITÄT MAINZ



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BARCELONA



@SPlanaRui

❖ About me ...



as a baby microscopist ...

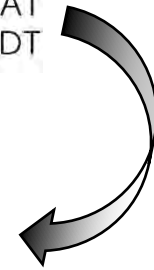


JOHANNES GUTENBERG
UNIVERSITÄT MAINZ



TECHNISCHE
UNIVERSITÄT
DARMSTADT

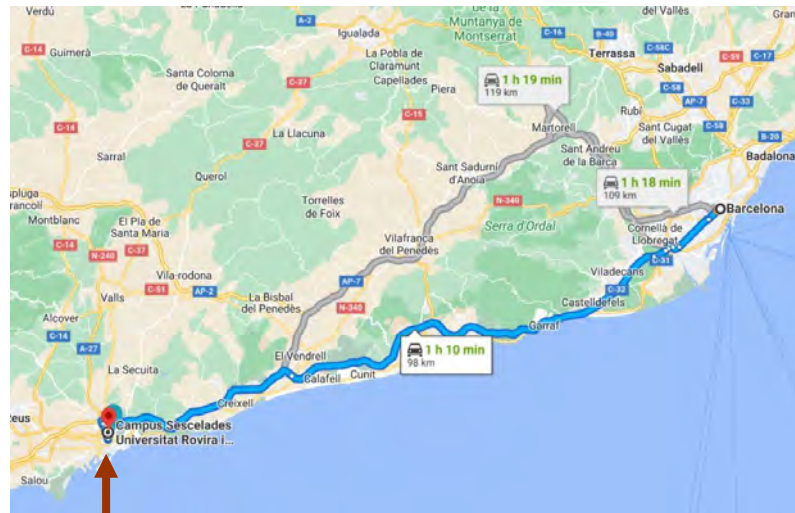
as a baby crystallographer ...



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NanoMEGAS
Advanced Tools for electron diffraction



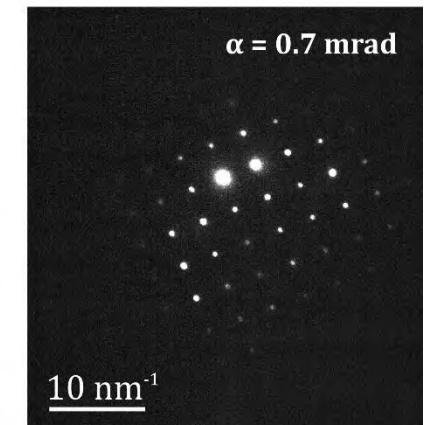
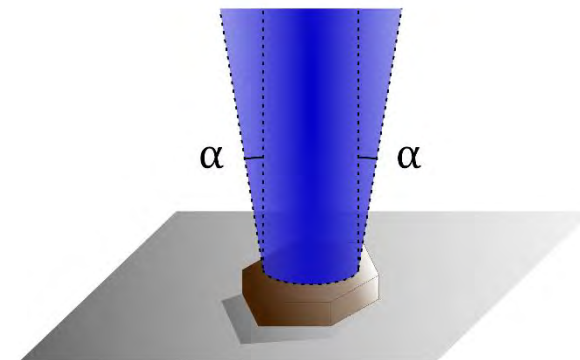
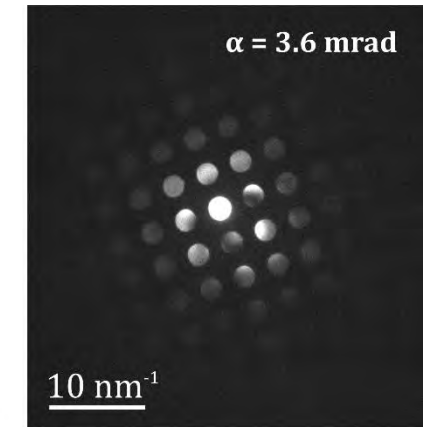
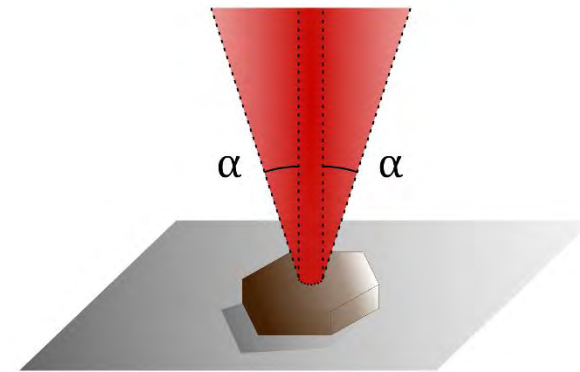
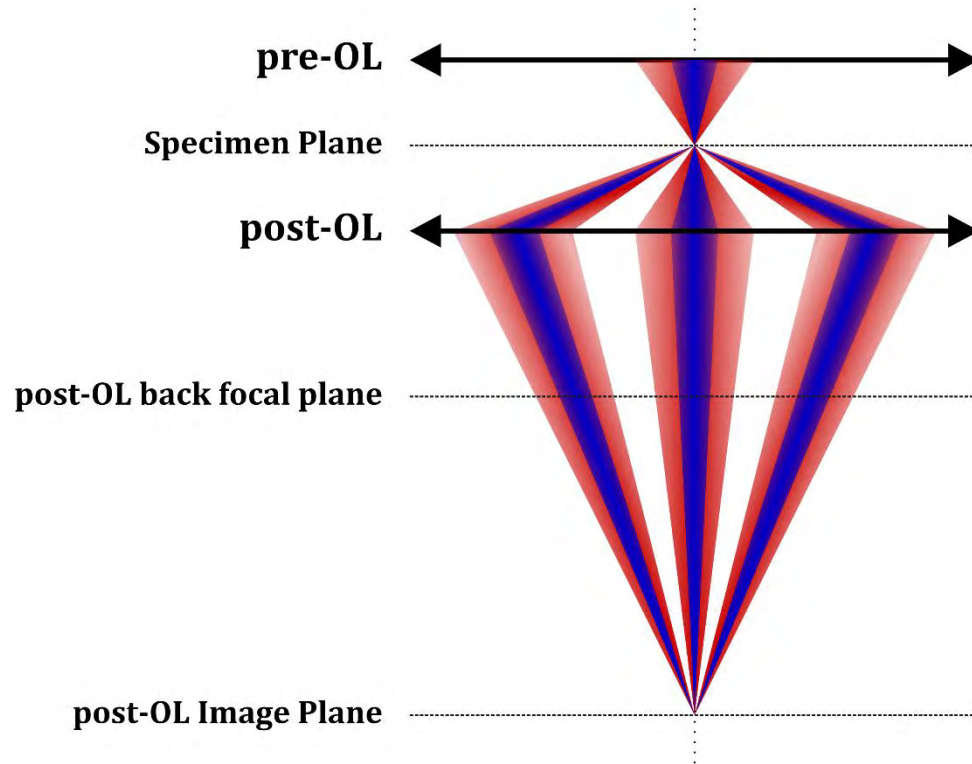
Tarragona



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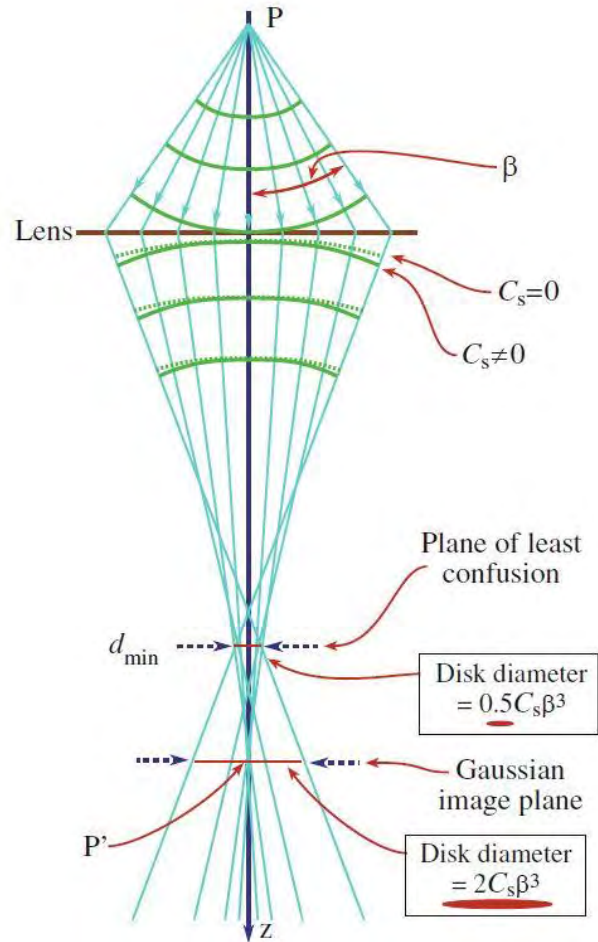
❖ Quasi-Parallel Electron Diffraction

- Why do we need a quasi-parallel electron beam?



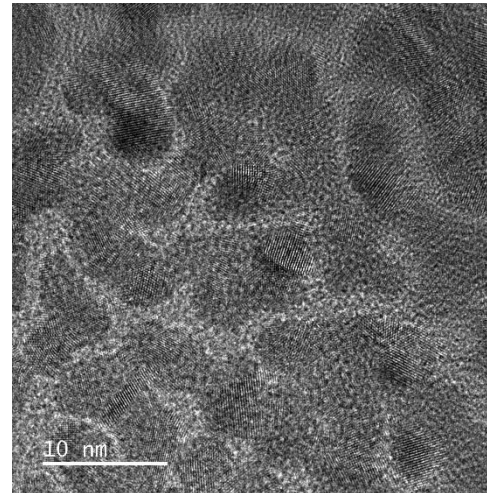
❖ Quasi-Parallel Electron Diffraction

- Why do we need a quasi-parallel electron beam?
 - In imaging mode ...

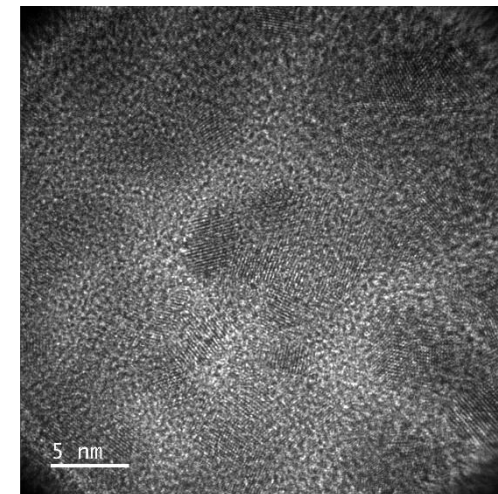
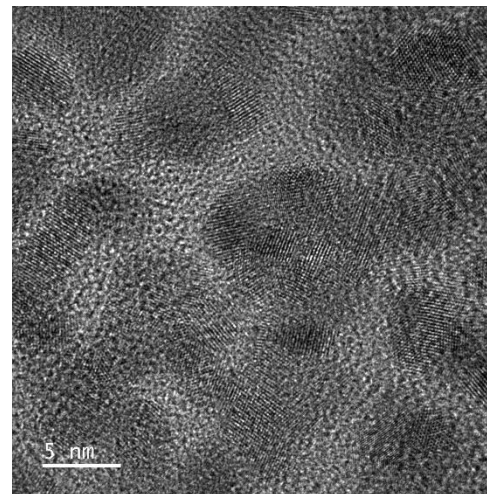
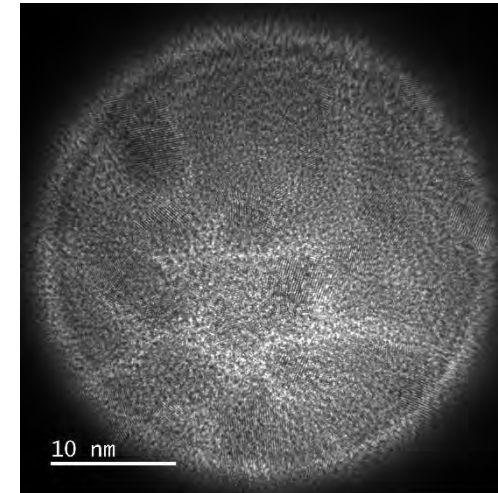


D. B. Williams & C. B. Carter, *Transmission Electron Microscopy*, 2008

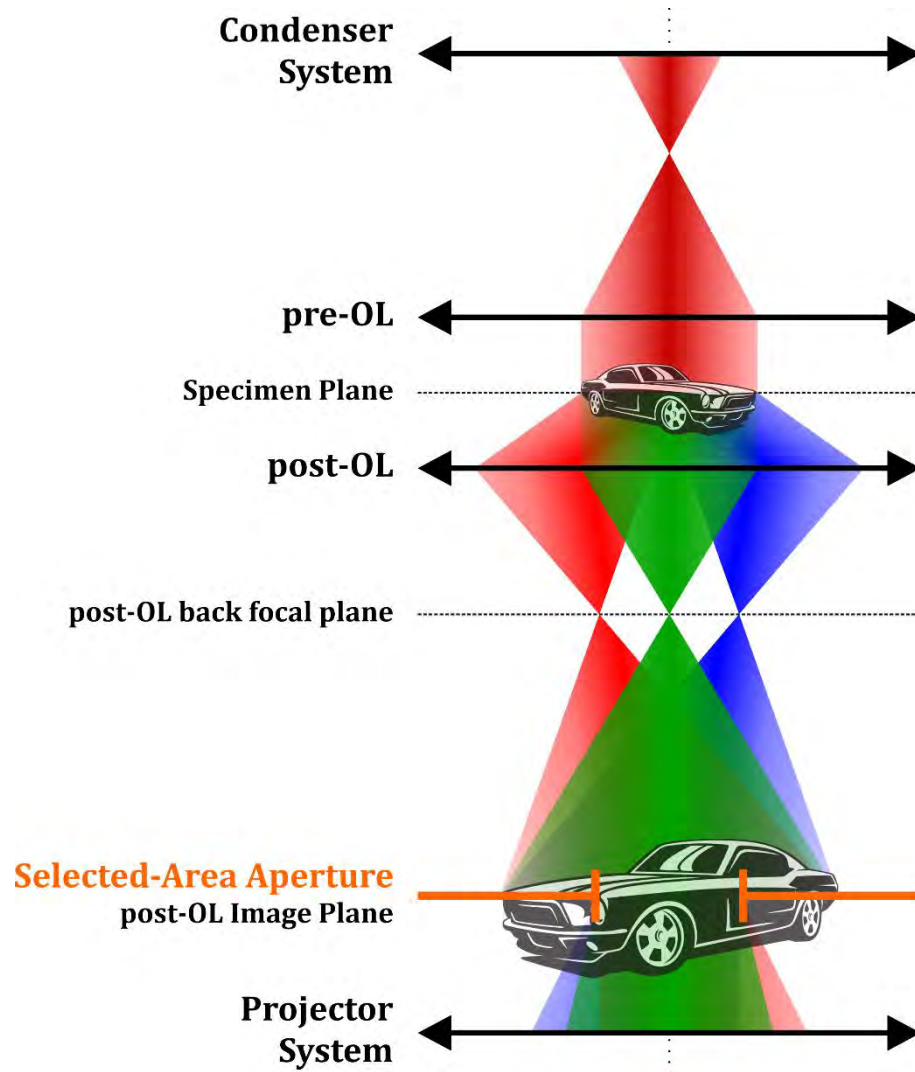
Spread illumination:



Closed illumination:

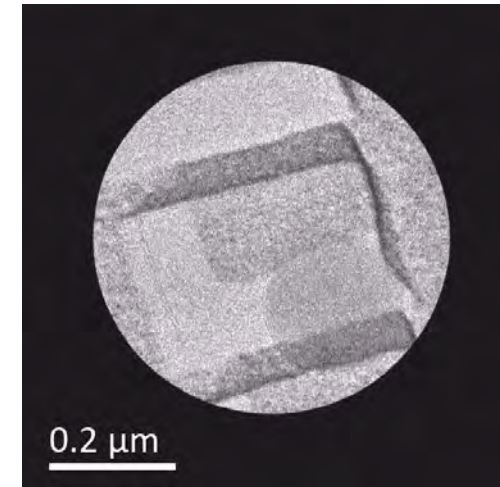


❖ Quasi-Parallel Electron Diffraction



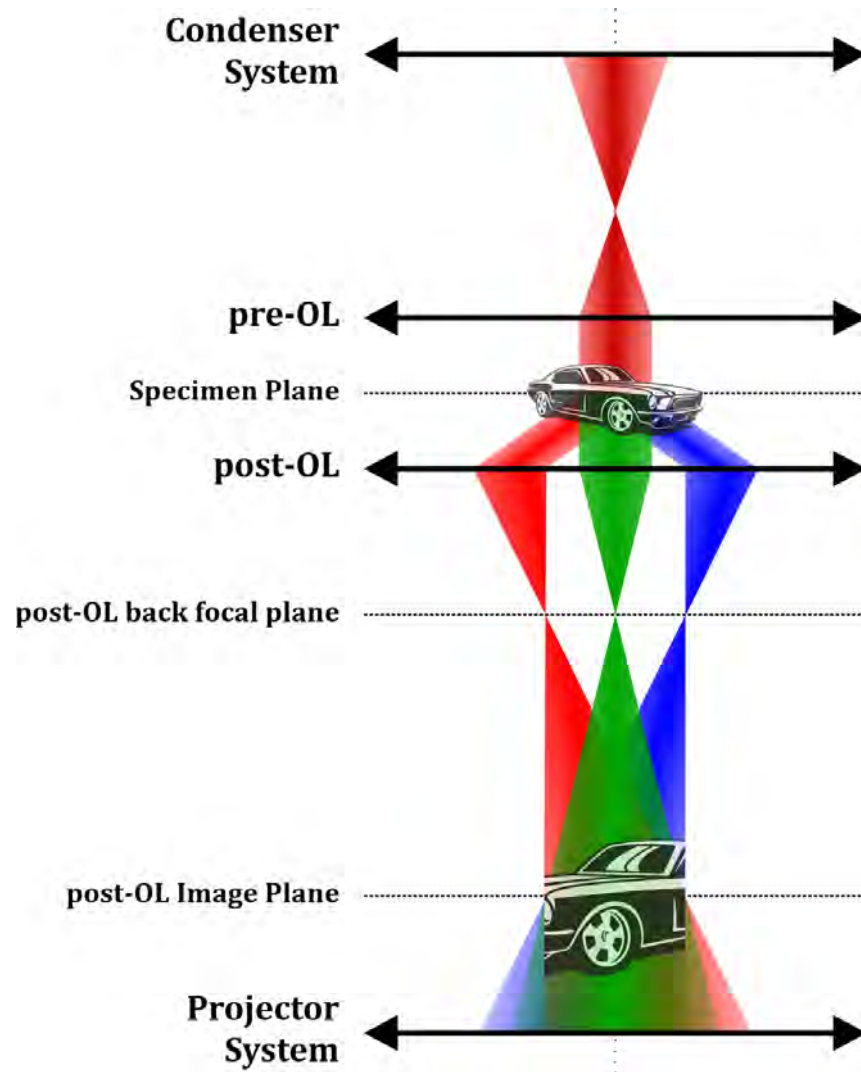
○ Selected-Area Electron Diffraction

- ✓ Easy to set and get ED patterns
- ✓ Available in almost any TEM
- × Probing of nanometre-scaled domains not possible
- × Illumination of areas not used for ED pattern formation
- × Heavily relies on well tomography-aligned stages



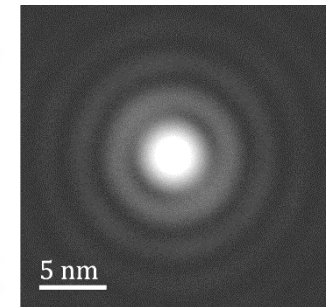
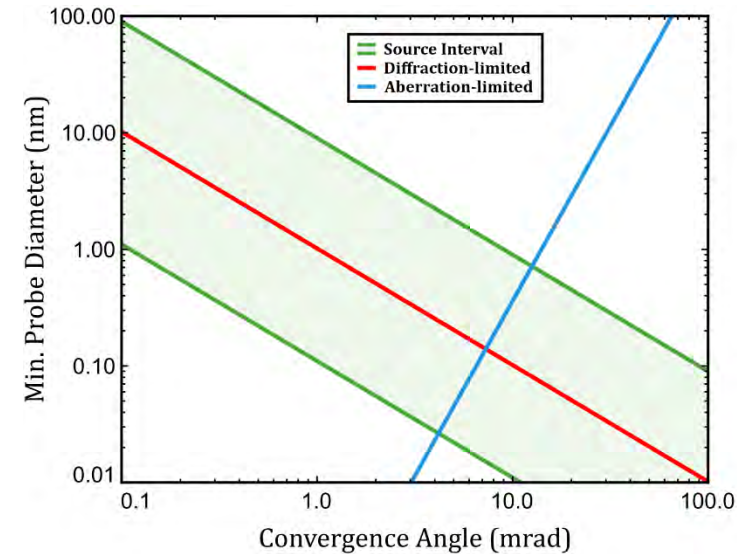
Continuous tilt between -50° to 50° at $1.68^\circ/\text{s}$
 $50\text{-}\mu\text{m}$ selected-area aperture

❖ Quasi-Parallel Electron Diffraction



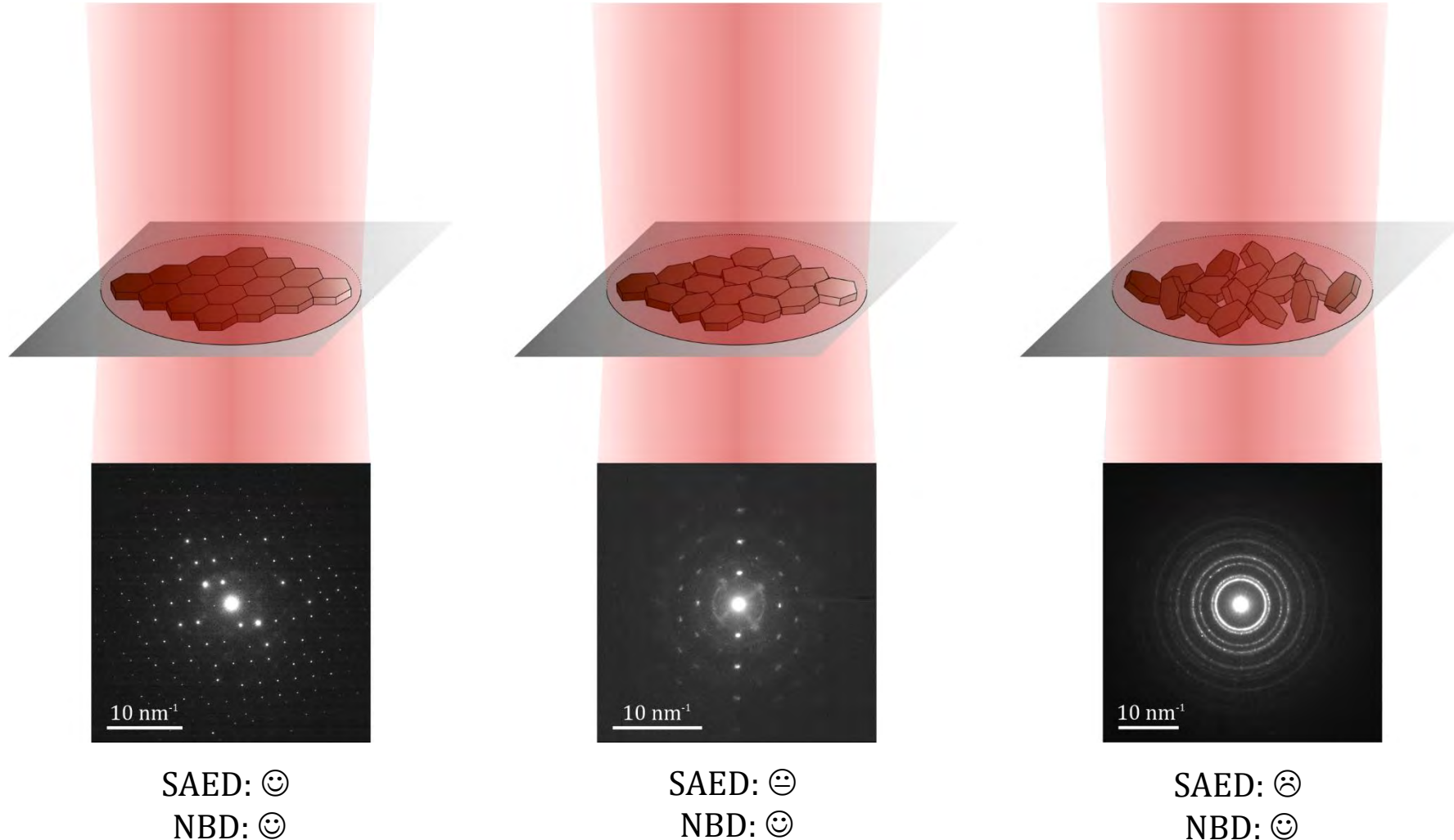
○ Nano-beam Diffraction

- ✓ Probe the smallest single-crystal domains
- ✓ Only illuminates where you want to collect ED info
- ✓ Very accurate in STEM mode
- × Not as easy to set for 3D ED acquisitions in an automated way



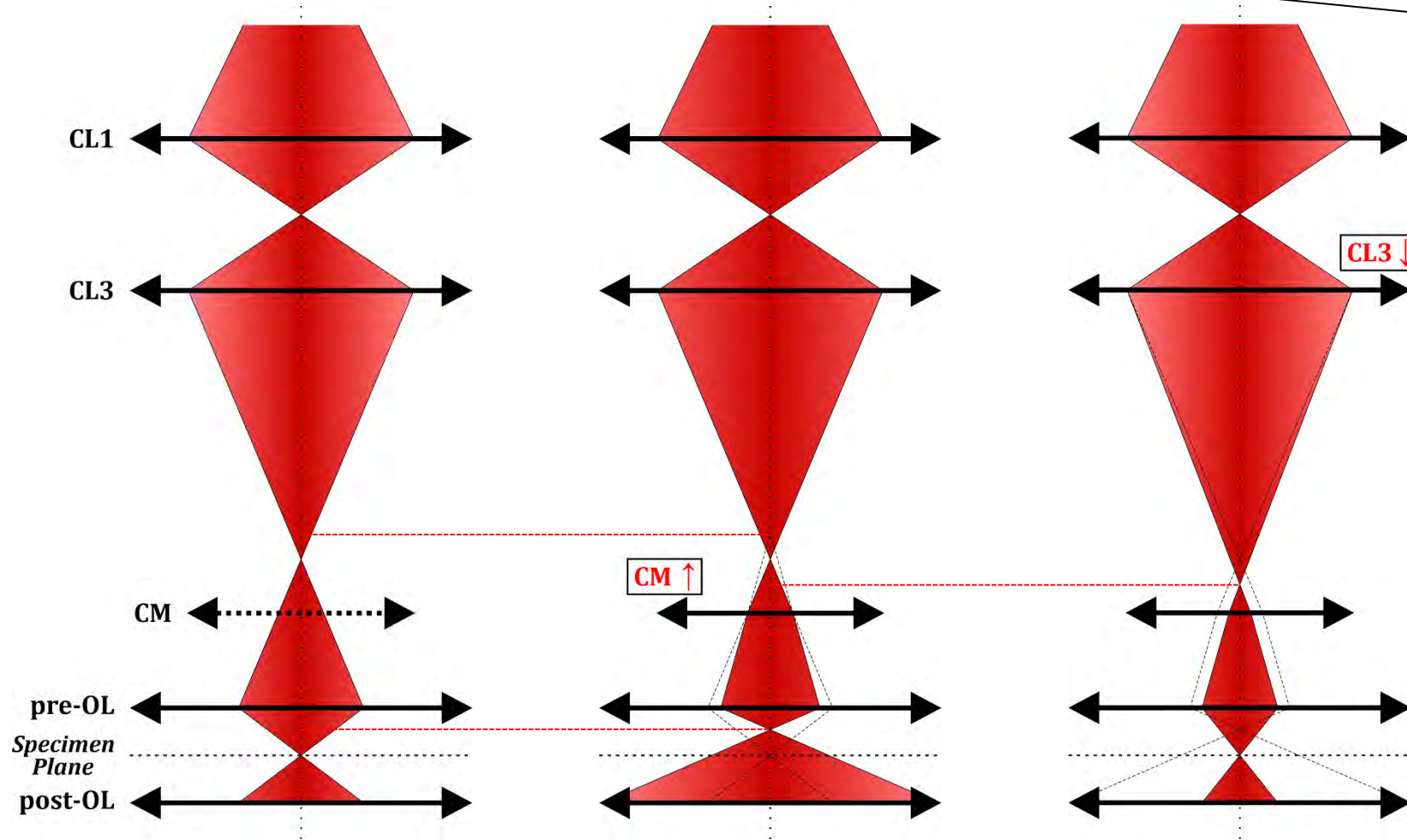
❖ Quasi-Parallel Electron Diffraction

- So ... SAED or NBD for single-crystal investigations?



❖ Quasi-Parallel Electron Diffraction

- How do we align a quasi-parallel beam?



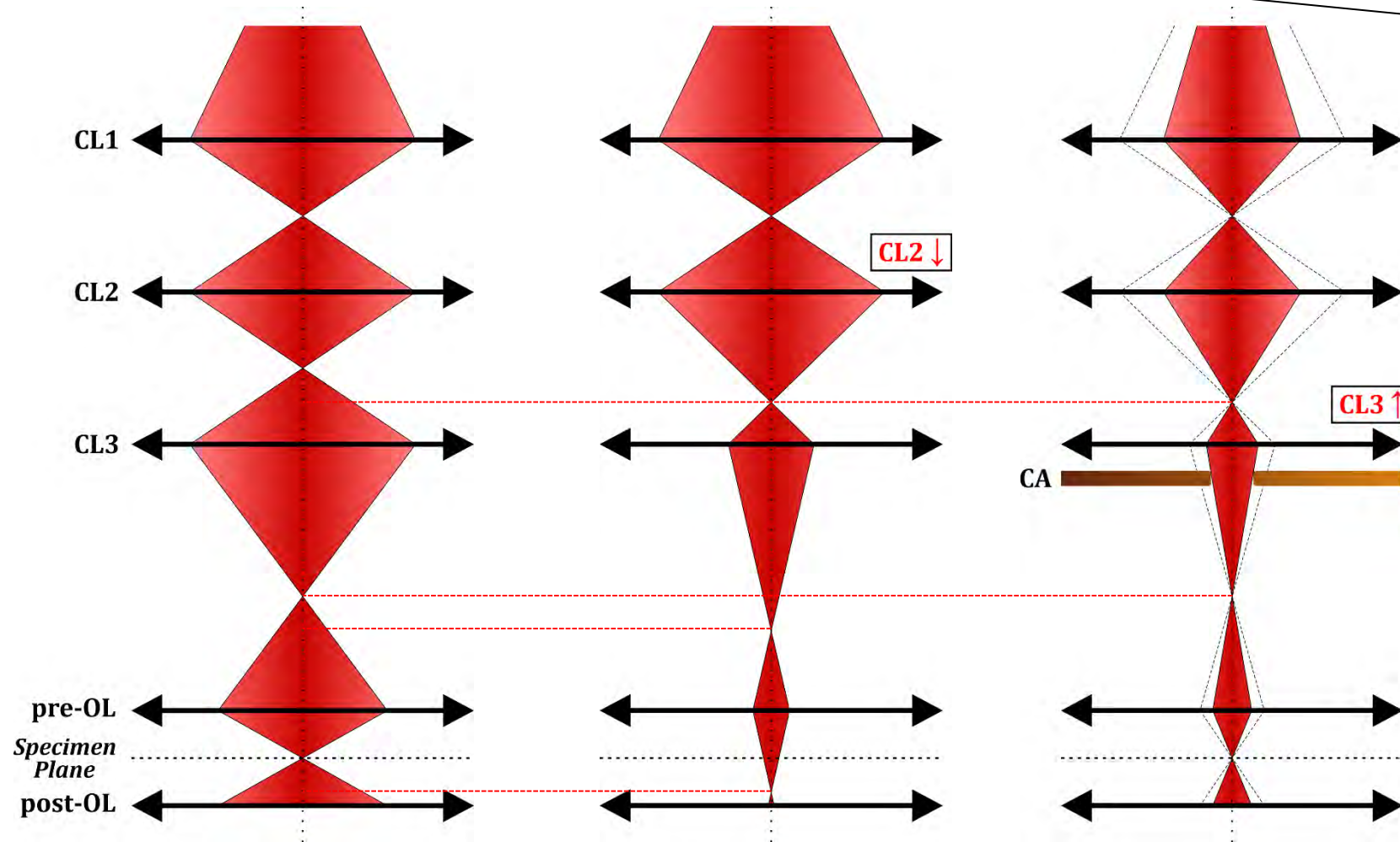
Plana-Ruiz et al., *Ultramicroscopy*, 193, 39-51, 2018

- Requirements:**
- Condenser System of 2CLs+1CM or 3CLs
 - 10 to 30 μm Condenser Aperture
 - Current Lens & Coils Control
 - Storage & Loading Capabilities

❖ Quasi-Parallel Electron Diffraction

- How do we align a quasi-parallel beam?

- Requirements:**
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Plana-Ruiz et al., *Ultramicroscopy*, 193, 39-51, 2018

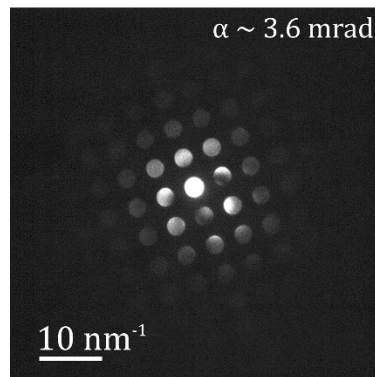
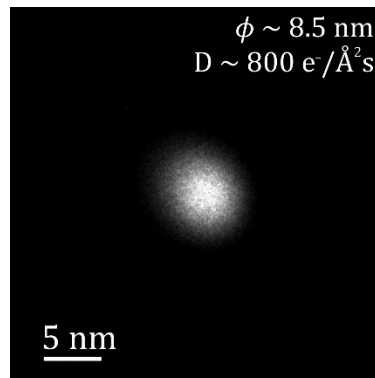
❖ Quasi-Parallel Electron Diffraction

- How do we align a quasi-parallel beam?

Requirements:

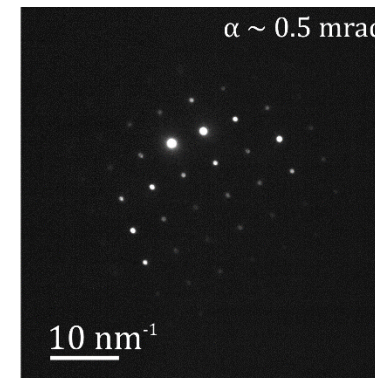
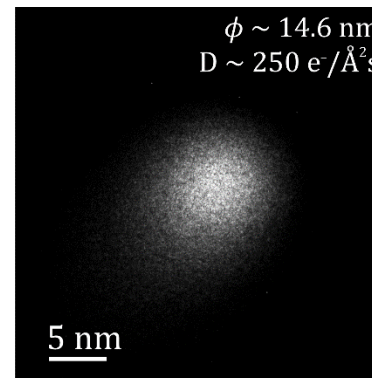
- Condenser System of 2CLs+1CM or 3CLs
- 10 to 30 μm Condenser Aperture
- Current Lens & Coils Control
- Storage & Loading Capabilities

Convergent STEM

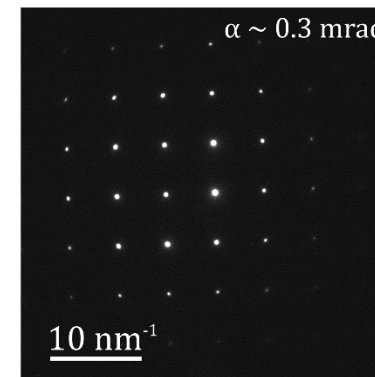
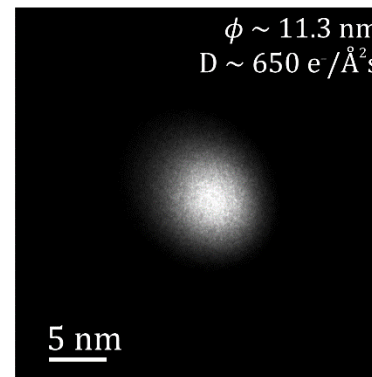


2CLs+1CM
→

Quasi-Parallel STEM



3CLs
→

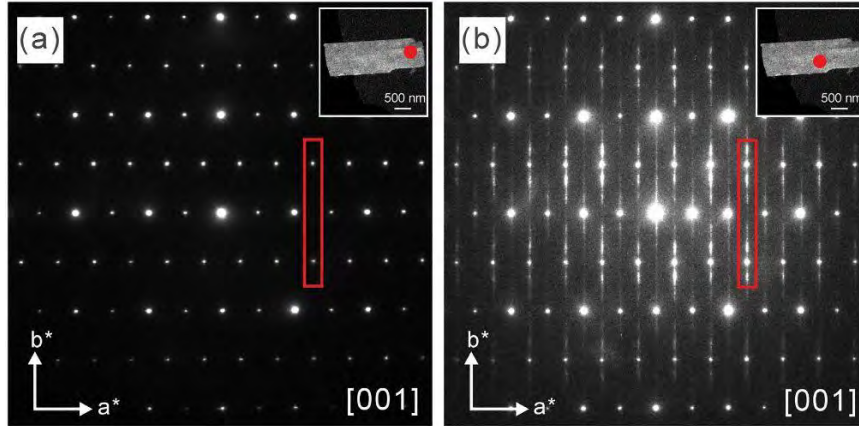


→ Measurements carried out in a JEOL 2100 LaB₆ at 200kV in Spot Size 3

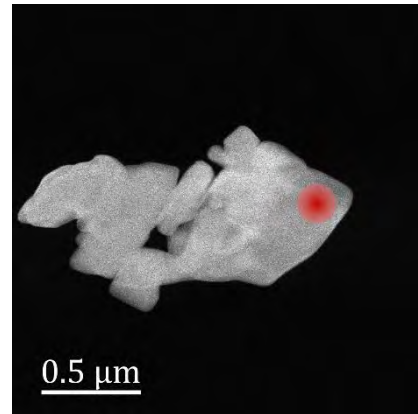
Plana-Ruiz et al., *Ultramicroscopy*, 193, 39-51, 2018

❖ Precession Electron Diffraction

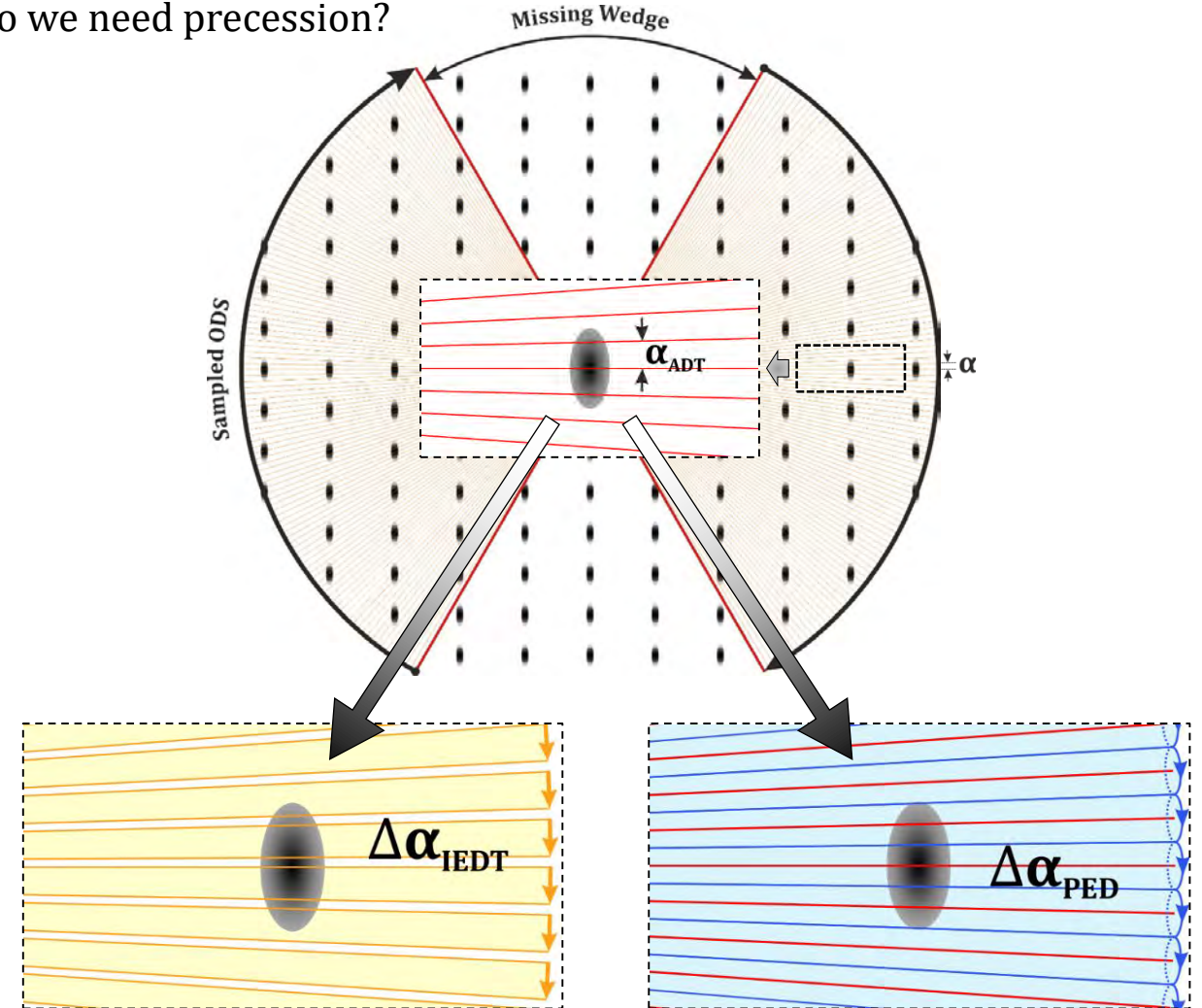
- If we can continuously tilt the TEM stage, why do we need precession?
 - Continuous tilt/rotation may not be an option



Zhao et al., *Journal of Solid State Chemistry* 249, 114–123, 2017

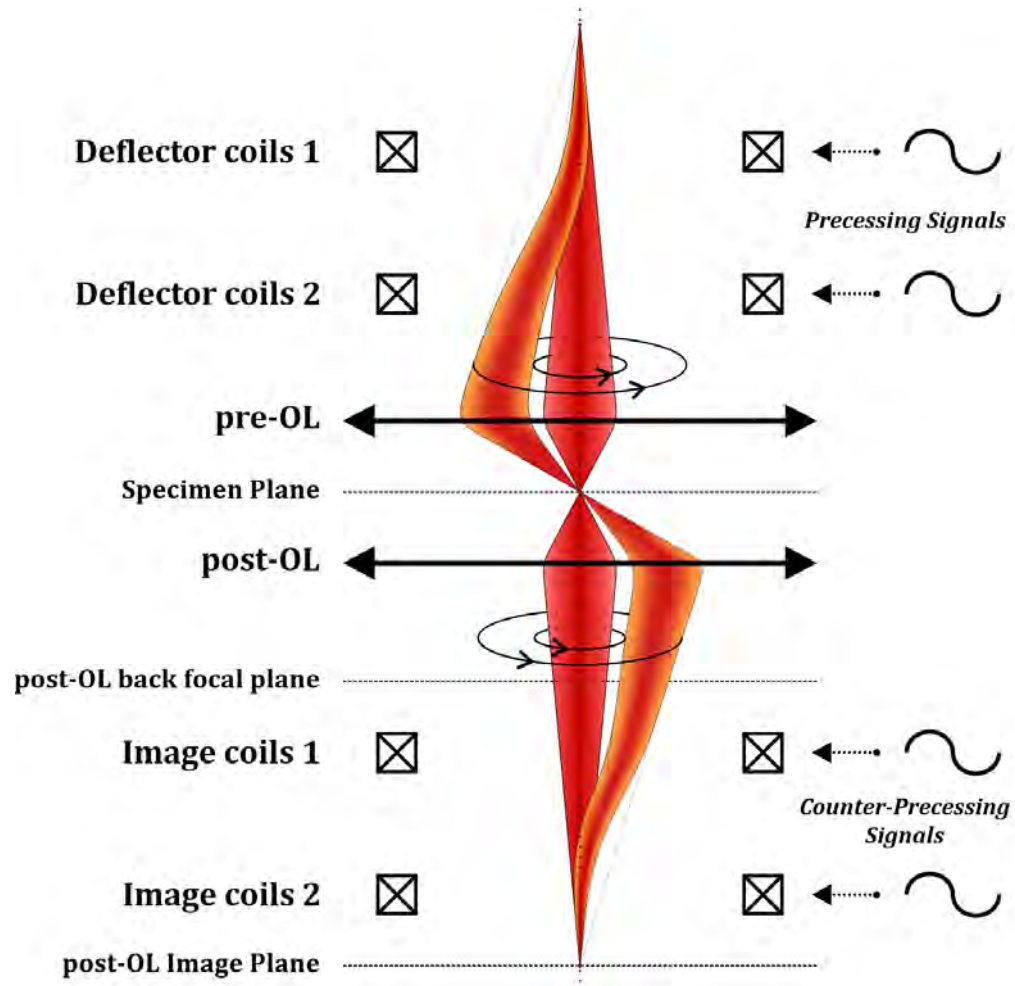


Sergi Plana-Ruiz., PhD thesis, 2021

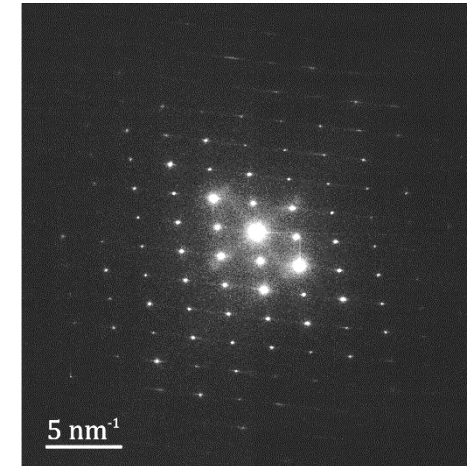


❖ Precession Electron Diffraction

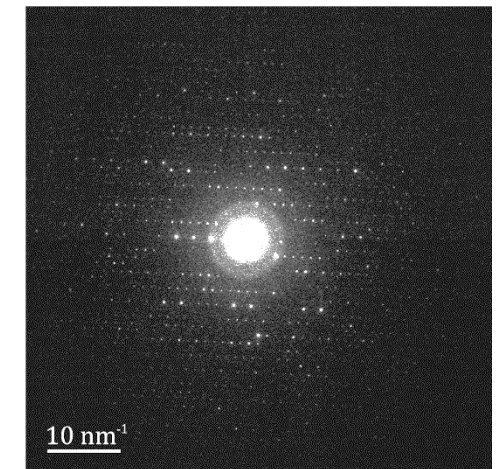
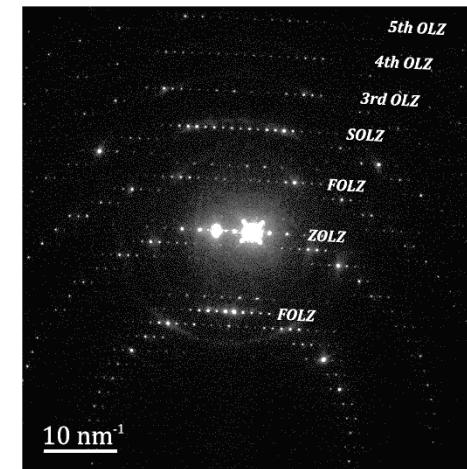
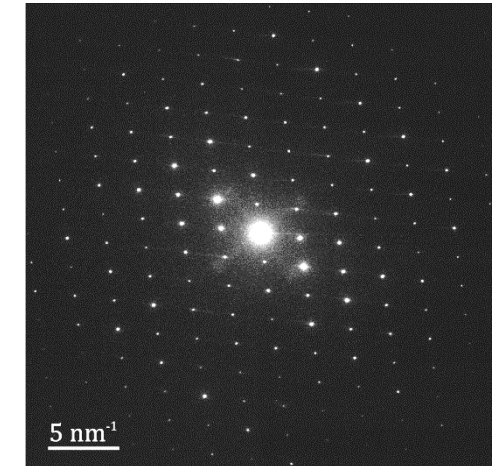
- What is the precession movement in a TEM?



Without precession:

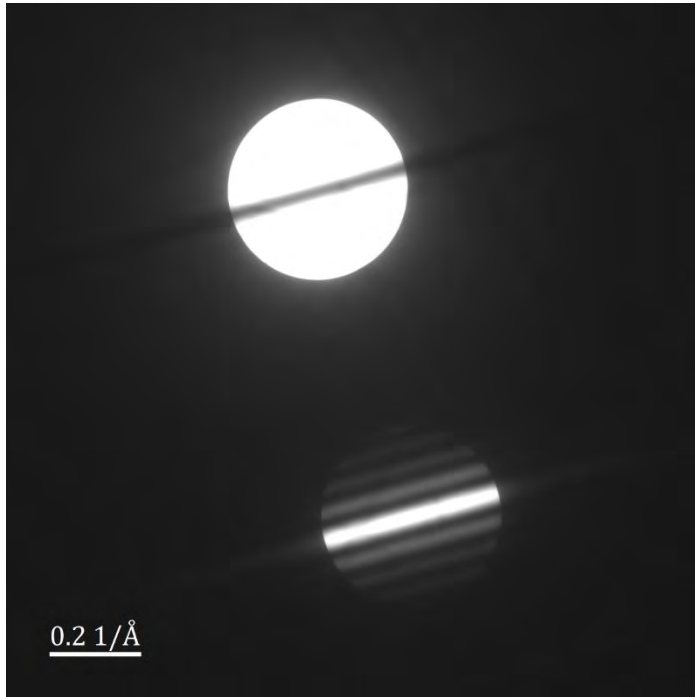


With 1° of precession:

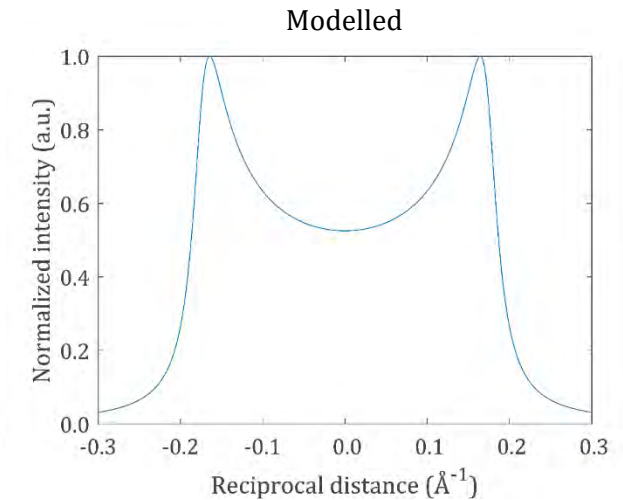
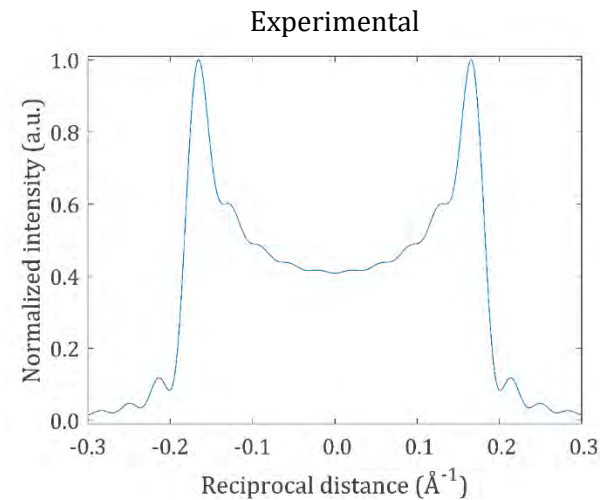
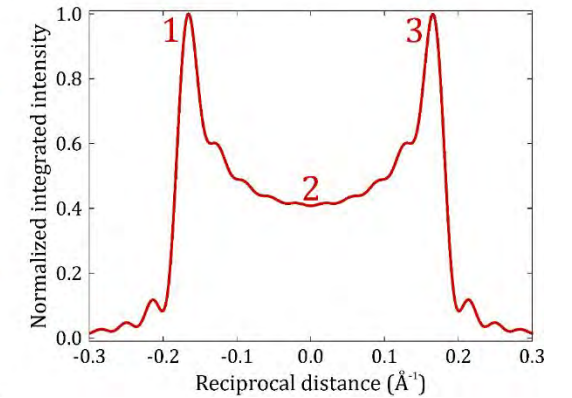
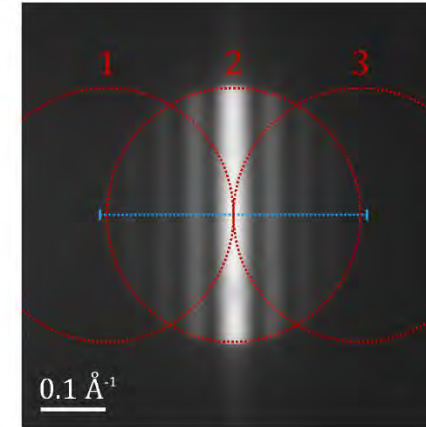
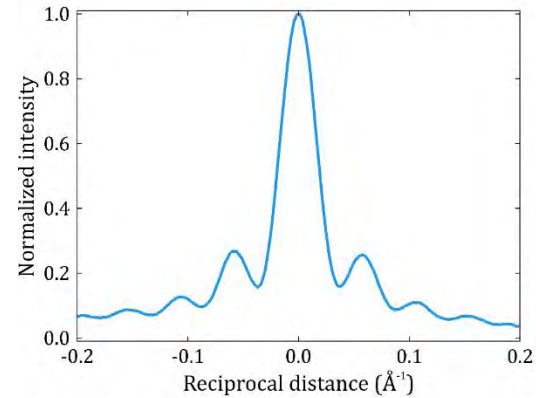


❖ Precession Electron Diffraction

- Rocking curves from precession-assisted 3DED data

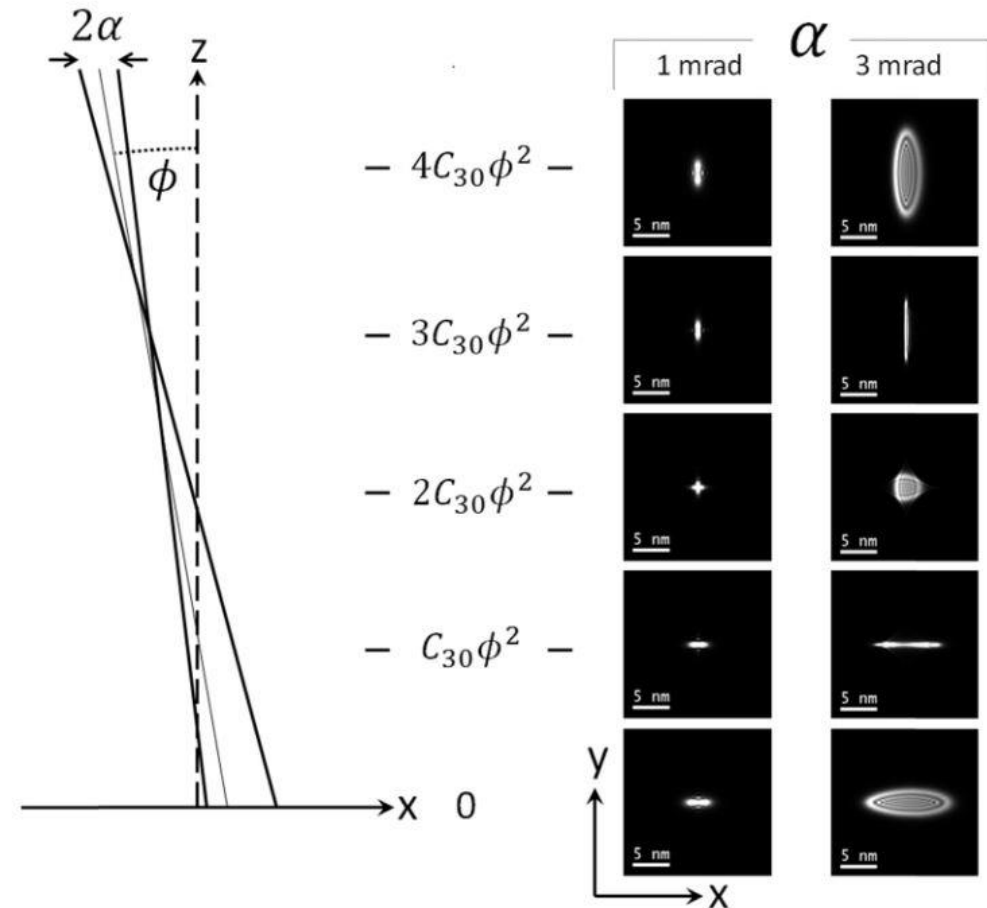
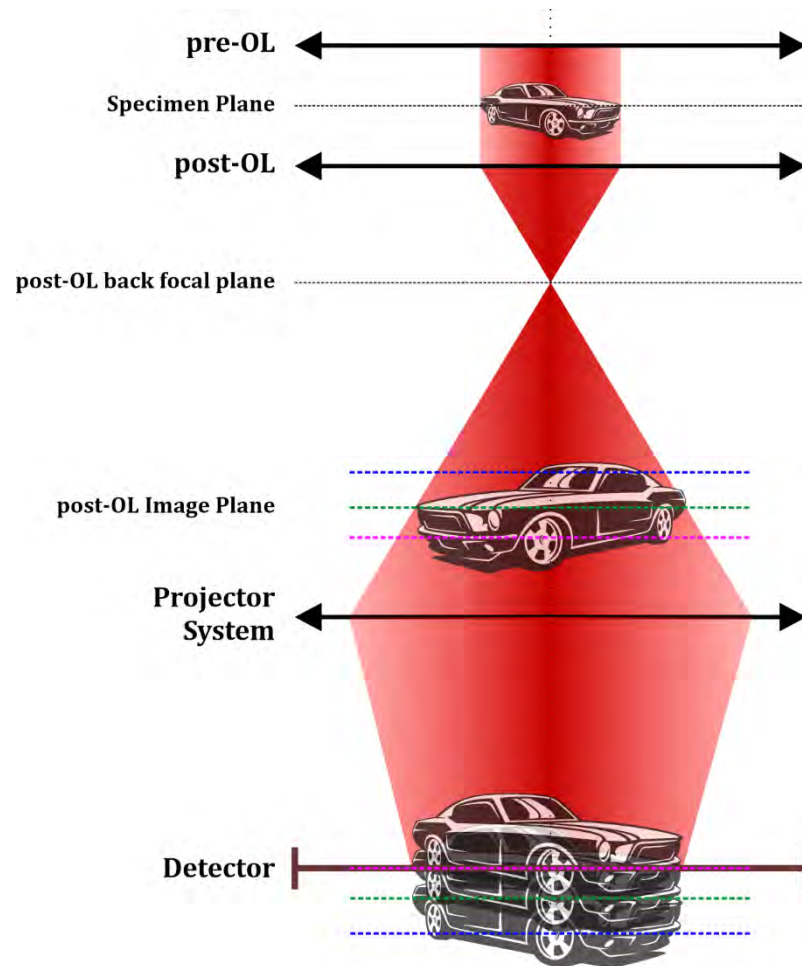


CBED pattern kindly provided by Dr. Lukáš Palatinus and Mariana Klementová from the Czech Academy of Sciences in Prague



❖ Precession Electron Diffraction

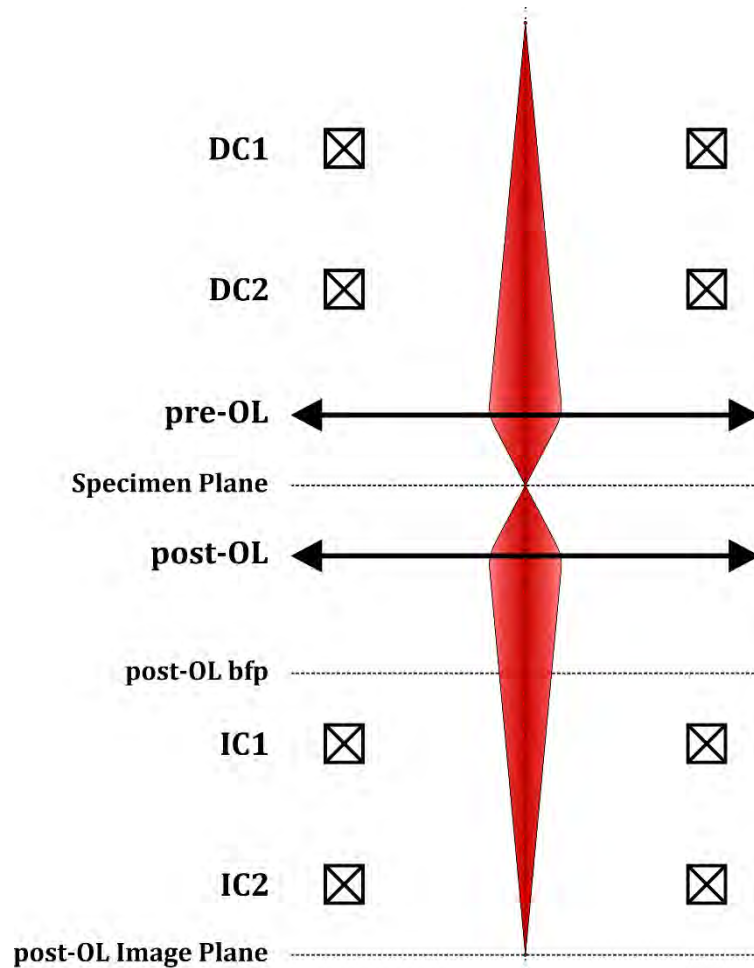
- How do we precisely align precession?
 - The image in the detector may not correspond exactly to the specimen plane – **STEM is preferred**



Barnard et al., *Ultramicroscopy*, 174, 79-88, 2017

❖ Precession Electron Diffraction

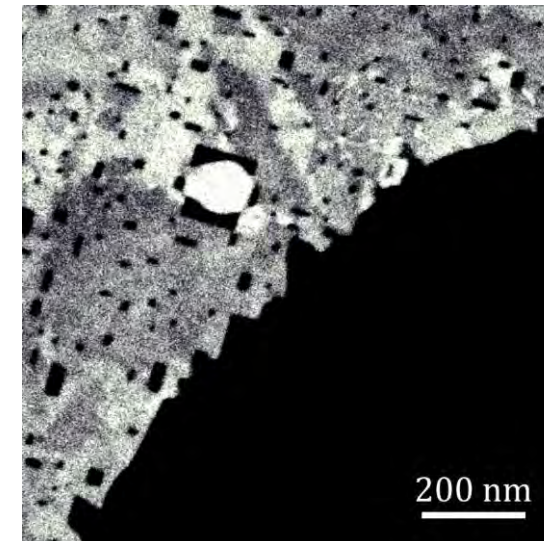
- Precession alignment in STEM mode



Plana-Ruiz et al., *Ultramicroscopy*, 193, 39-51, 2018

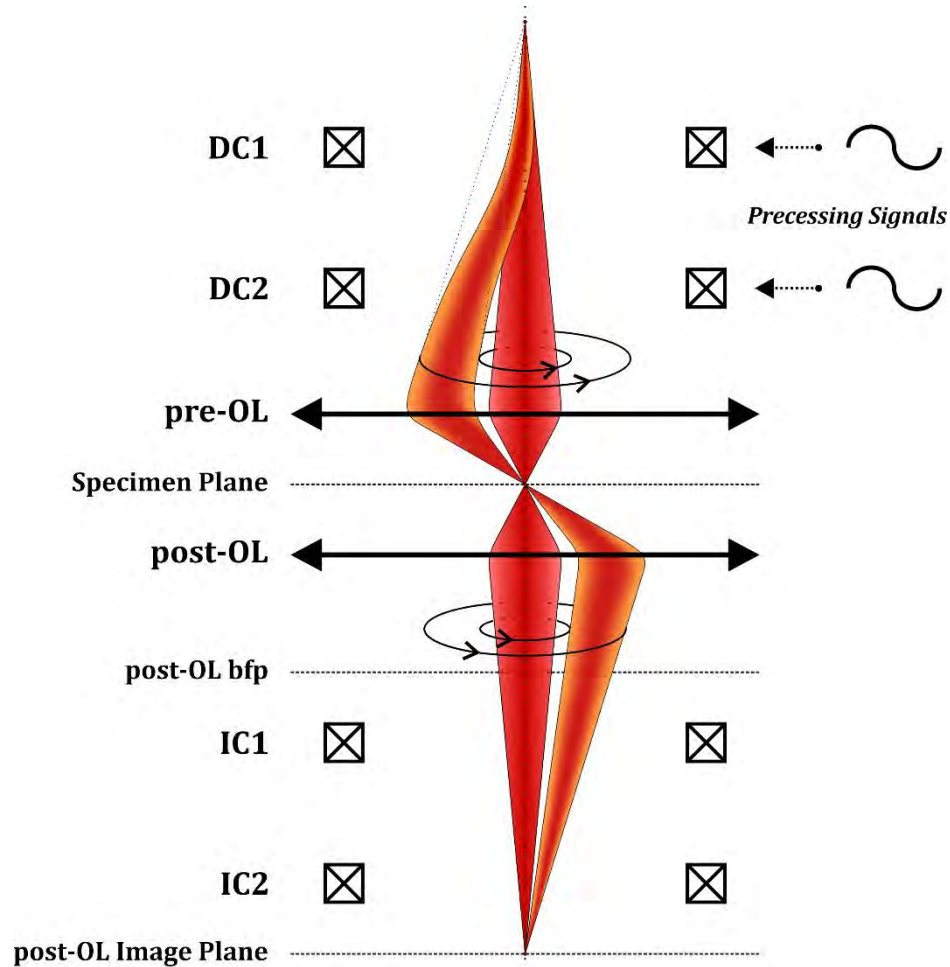
- Quasi-parallel beam is aligned in STEM mode following the usual steps (focus, HT centring, condenser stigmatism, beam tilt pivot points)
- Initial default strengths for precession and counter-precession signals are given but they are kept off.

Aligned Quasi-Parallel STEM

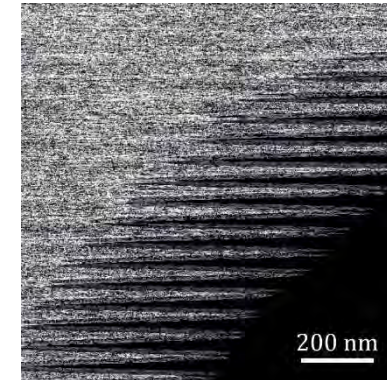


❖ Precession Electron Diffraction

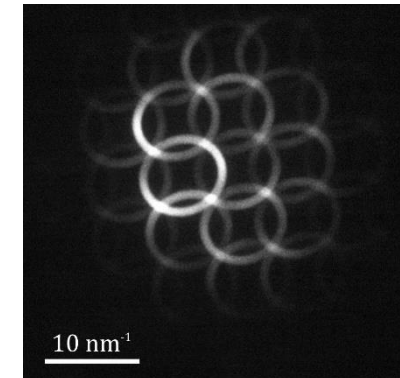
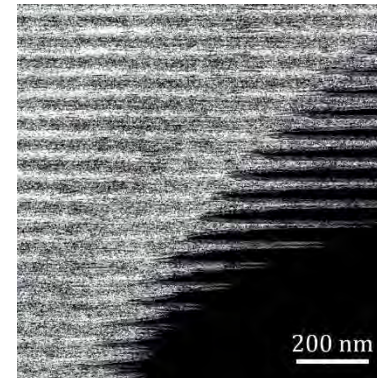
- Precession alignment in STEM mode



Misaligned Precession Signals



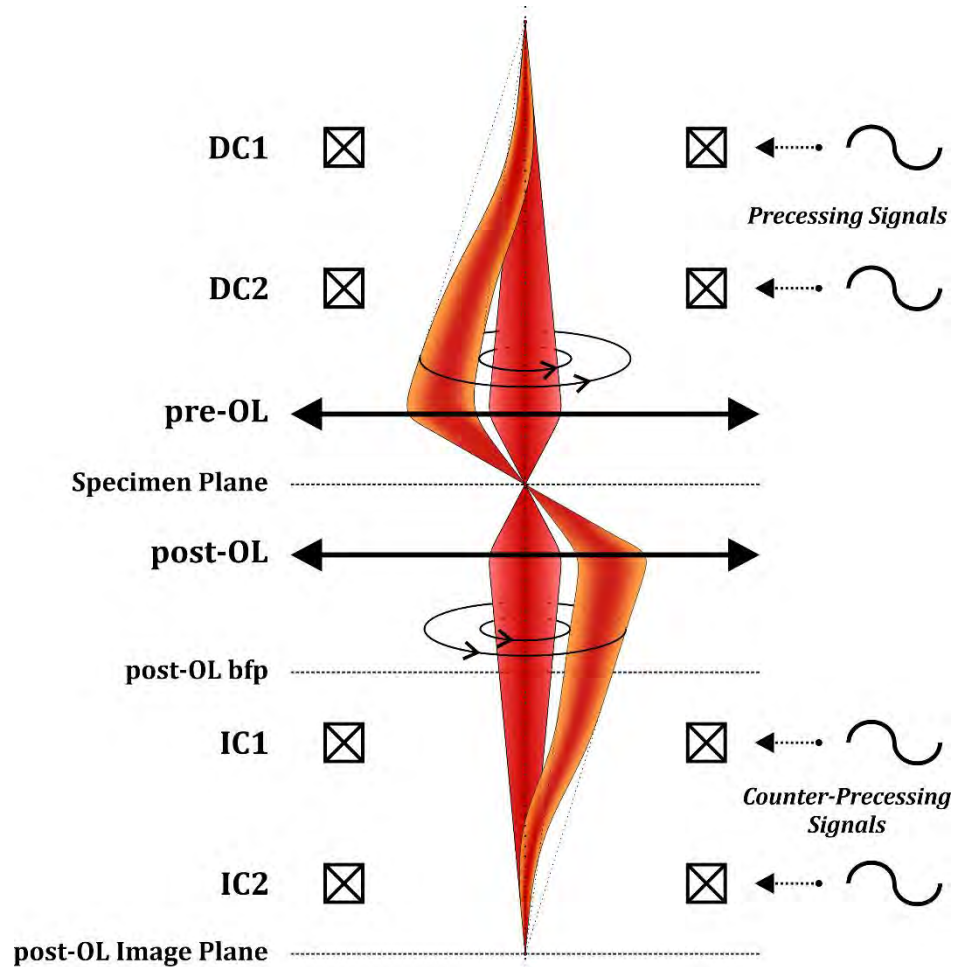
Aligning Precession Signals



Plana-Ruiz et al., *Ultramicroscopy*, 193, 39-51, 2018

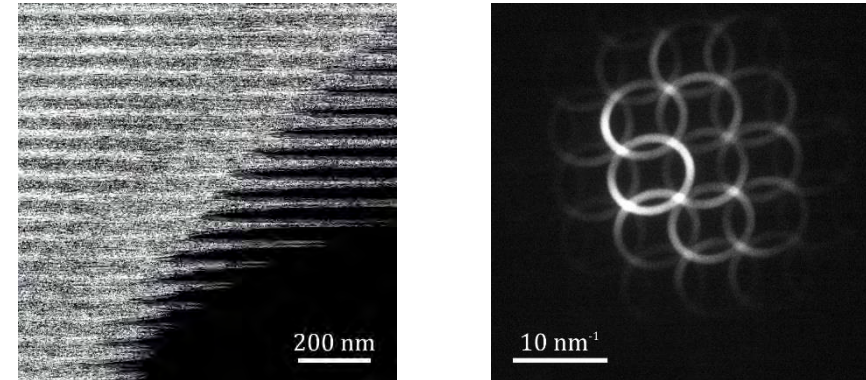
❖ Precession Electron Diffraction

- Precession alignment in STEM mode

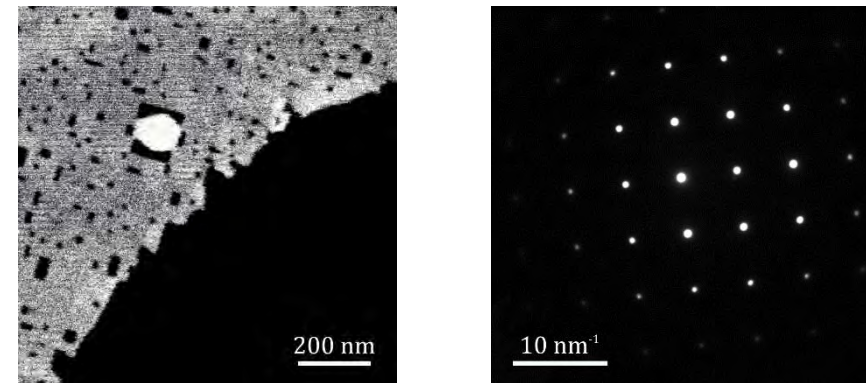


Plana-Ruiz et al., *Ultramicroscopy*, 193, 39-51, 2018

Aligning Precession Signals



Aligned Precession & Counter-Precession Signals

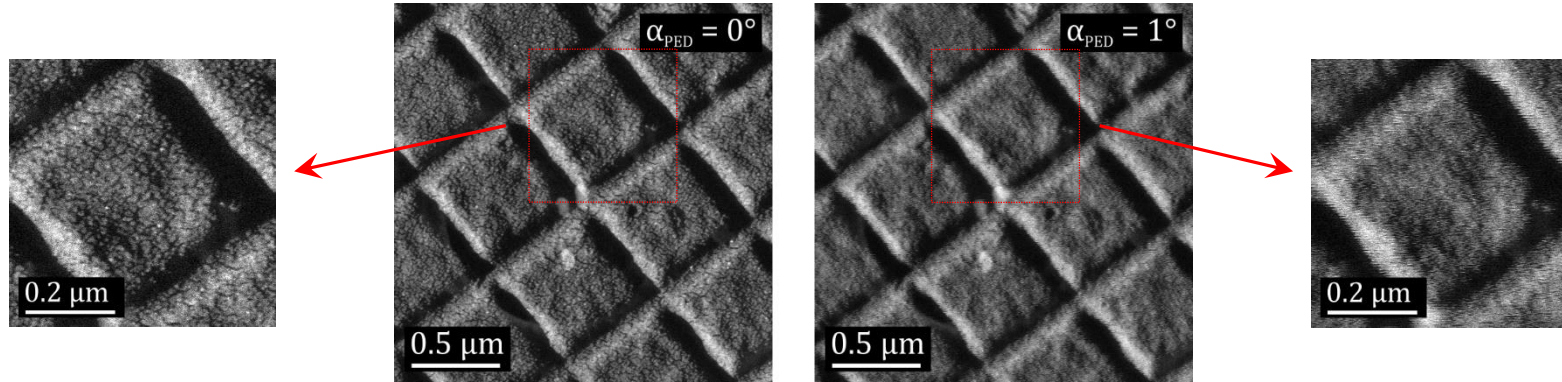


→ Results from a JEOL 2100 LaB₆ at 200kV

❖ Precession Electron Diffraction

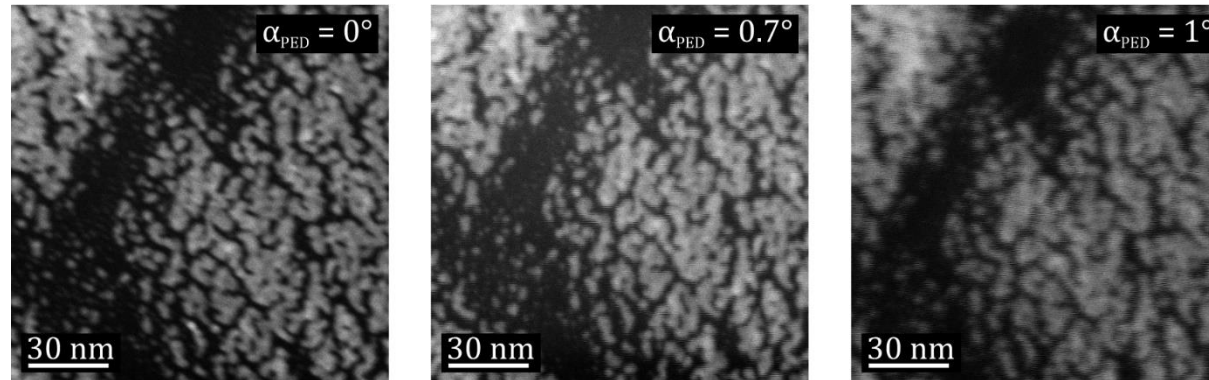
- Successful precession alignments

→ Results from FEI Tecnai F30 at 300 kV



*Au Grating Replica Sample

→ Results from JEOL ARM200F **Probe-Corrected** at 200 kV

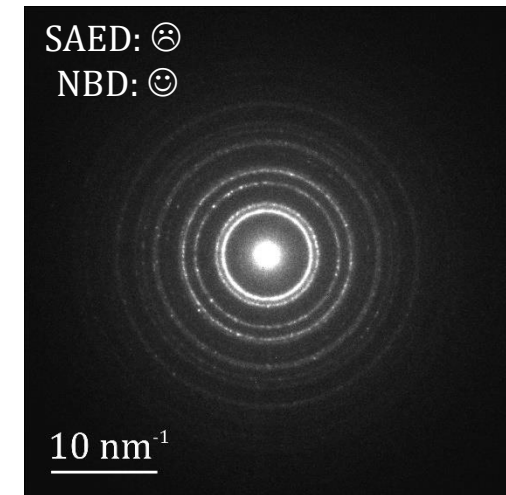
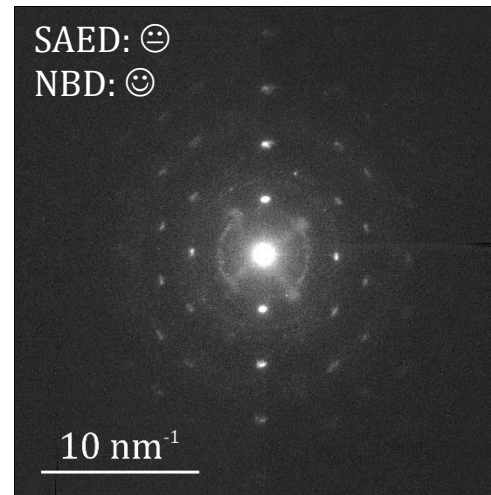
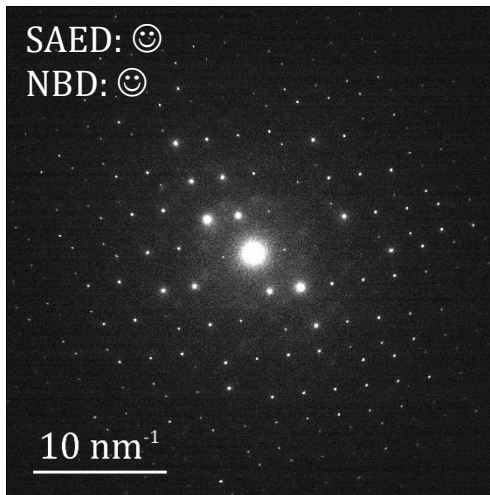


*Au Grating Replica Sample

Plana-Ruiz et al., *Ultramicroscopy*, 193, 39-51, 2018

❖ Conclusions

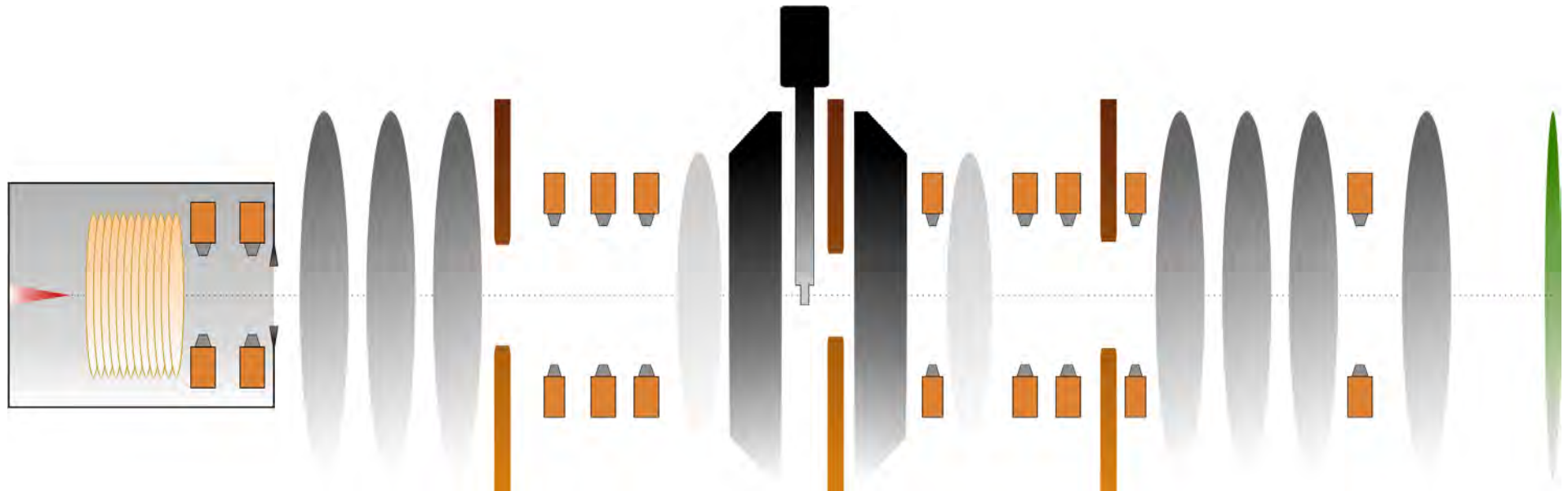
- **SAED vs NBD**: all depends on the crystalline domains that you are interested in
- Quasi-parallel electron beam - its alignment for probing the **smallest crystallites**
- Precession electron diffraction, an alternative to continuous tilt/rotation for the most challenging measurements
- **STEM preferred against TEM** to precisely image the sample plane
- The alignment of a precessed electron beam to nicely pivot on the sample plane



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NanED Workshop II, 7th of December 2022



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