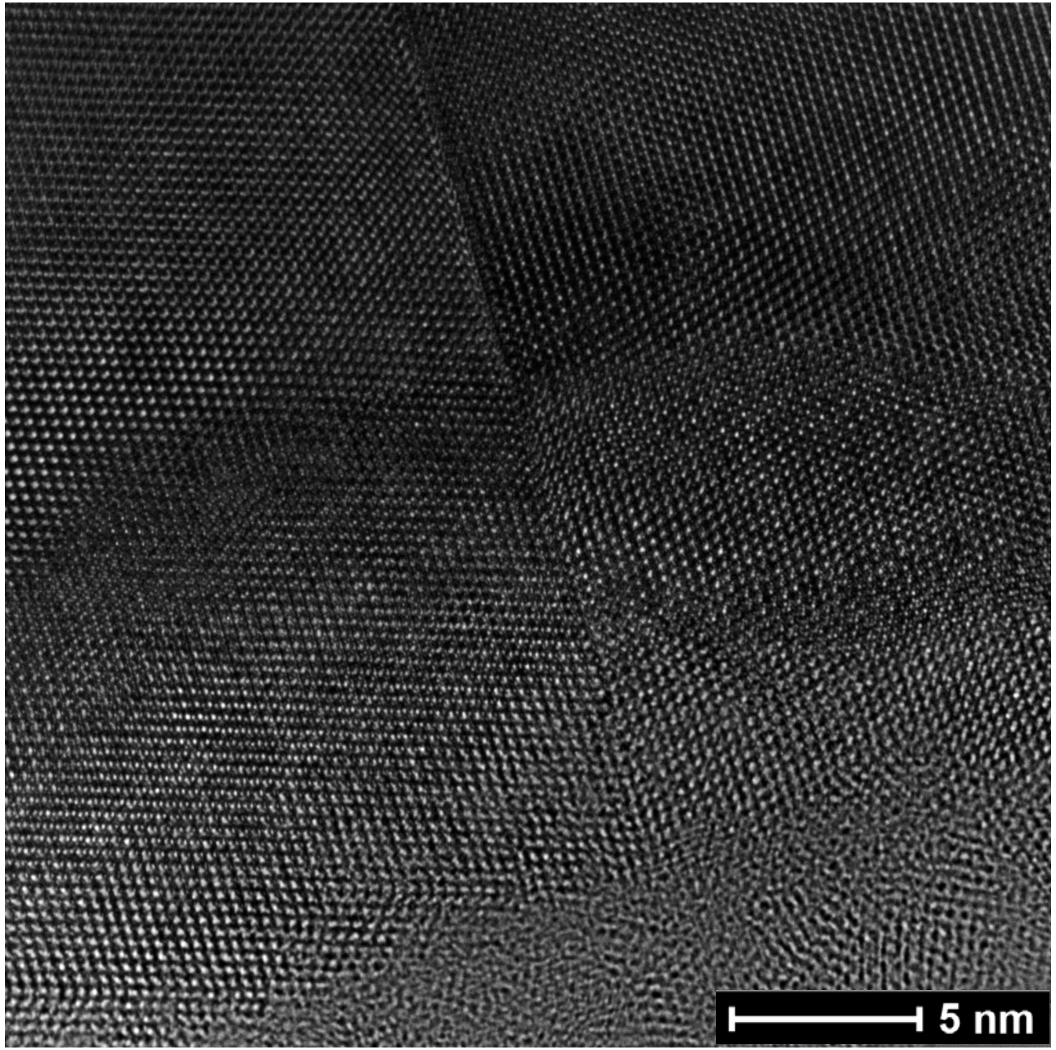
EPFL

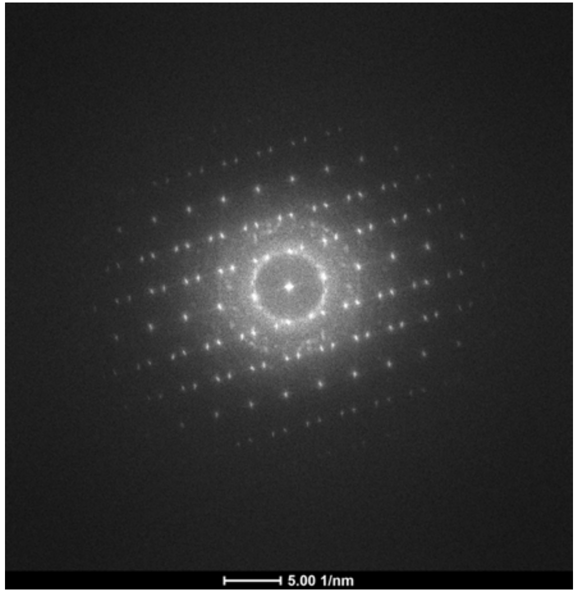
Scanning TEM (STEM) part I

Duncan Alexander

EPFL-IPHYS-LSME

Cs-TEM example: $(Al_xGa_{1-x})As$ nanowire





Sample courtesy of Yannick Fontana, Anna Fontcuberta-i-Morral, LMSC

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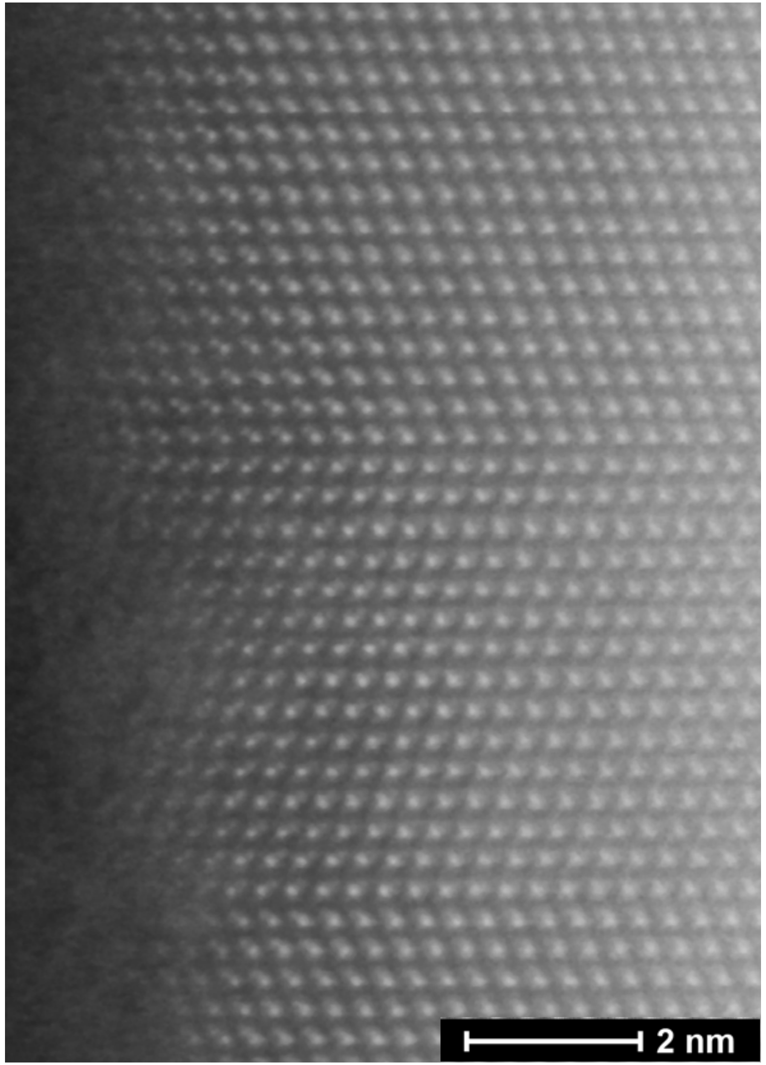


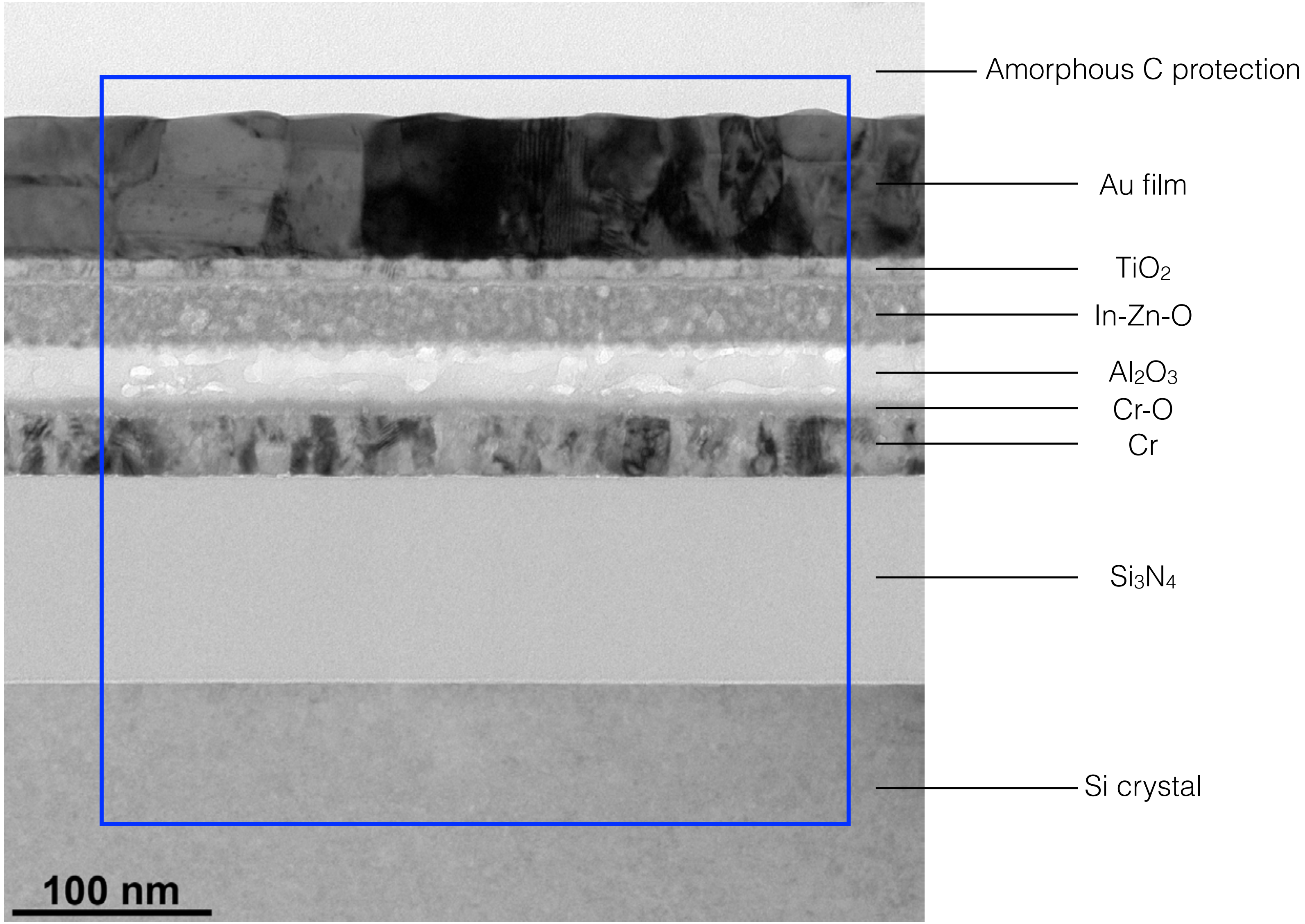
Sample courtesy of Yannick Fontana, Anna Fontcuberta-i-Morral, LMSC

STEM part I contents

- Example: STEM diffraction mapping of multilayer TEM specimen
- Principles of regular STEM imaging
- STEM detectors: BF, ADF
- Convergence and collection angles
- HAADF Z-contrast imaging
- Principle of reciprocity TEM vs STEM
- Optics and design of STEM
- The electron probe and electron source

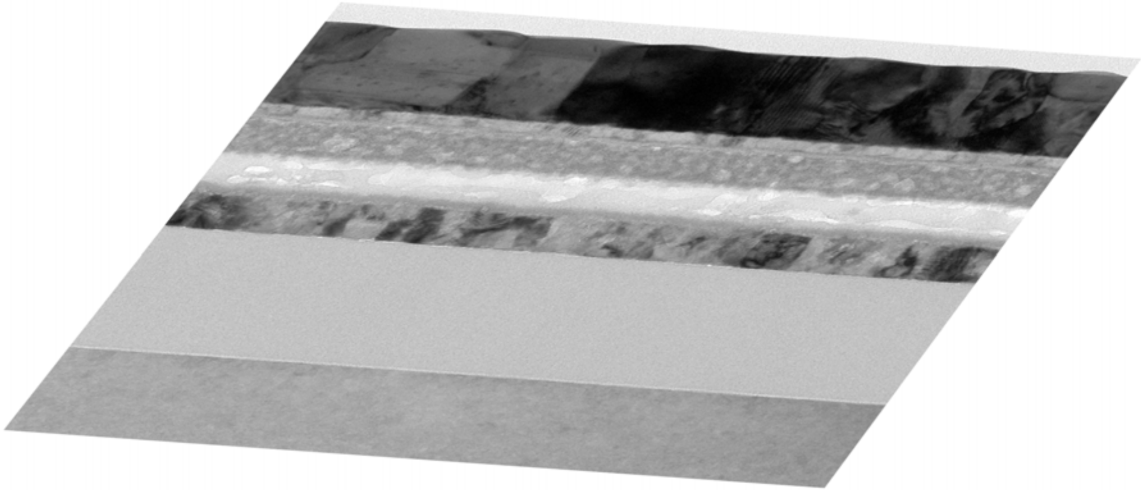
Example multilayer TEM specimen

- BF TEM image:



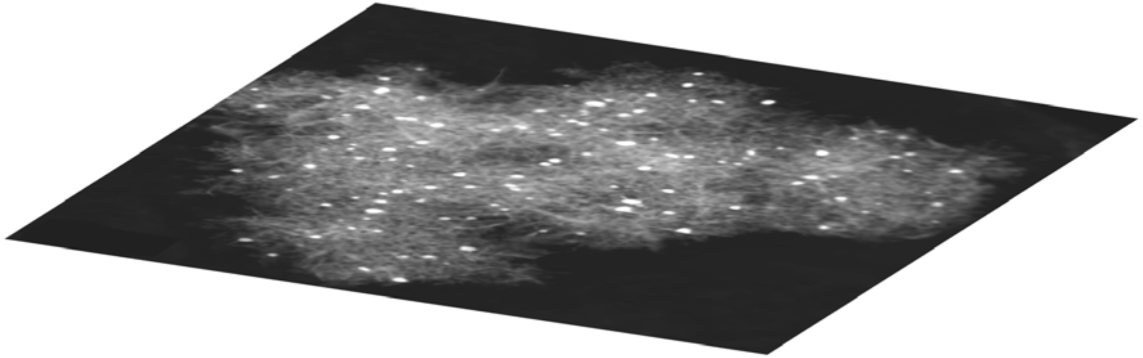
Scanning TEM diffraction mapping

- Focus converged e^--beam on sample
- Raster scan across an (x, y) grid
- For each pixel position, record a diffraction pattern
- We will look at the data using a Jupyter notebook!



Principle of regular STEM imaging

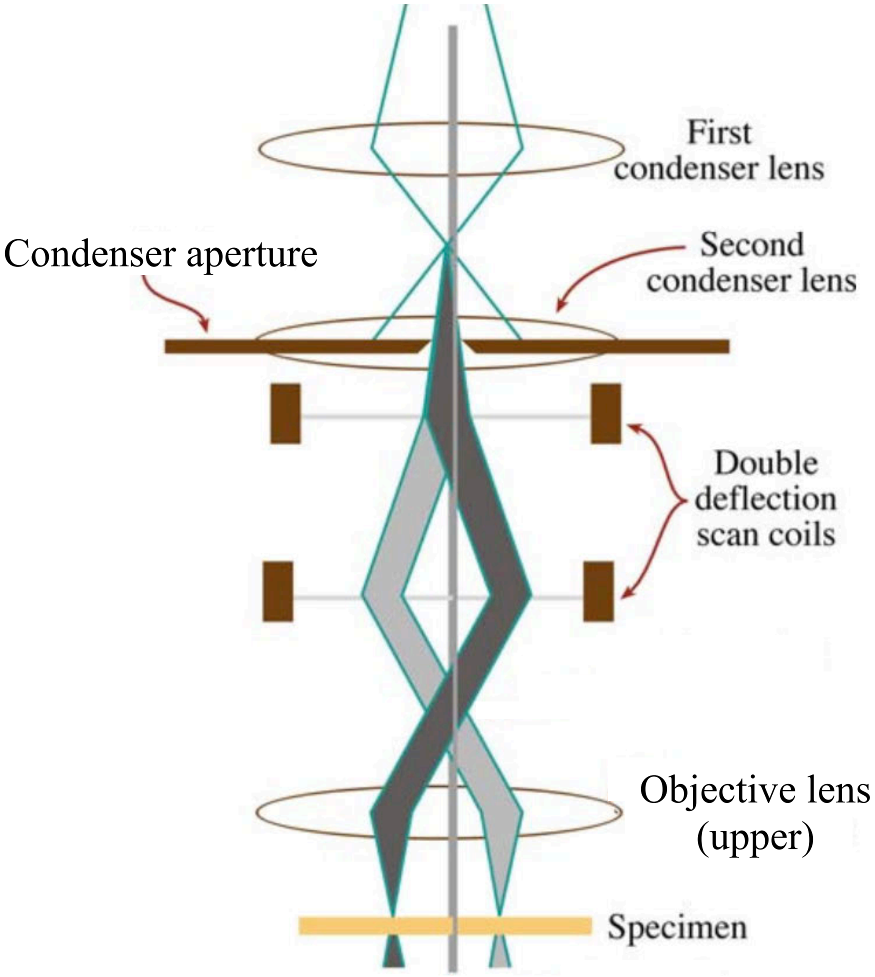
- Electromagnetic lens focuses electrons from FEG on to sample
- Beam deflectors scan beam across sample
- For each probe position (x, y) record signal intensity $I_{(x,y)}$ from radially-symmetric detector(s)
- Display/record image(s) of $I_{(x,y)}$
- *May also record an EELS or EDX spectrum at each probe position (CH lectures on analytical TEM)*



STEM electron detectors

- Detectors are located in the back focal plane (or a plane conjugated with the BFP)

STEM scan coils



STEM electron detectors

- “Short hand”: in STEM we often just consider scattering angles from specimen

Bright-field and annular dark-field STEM

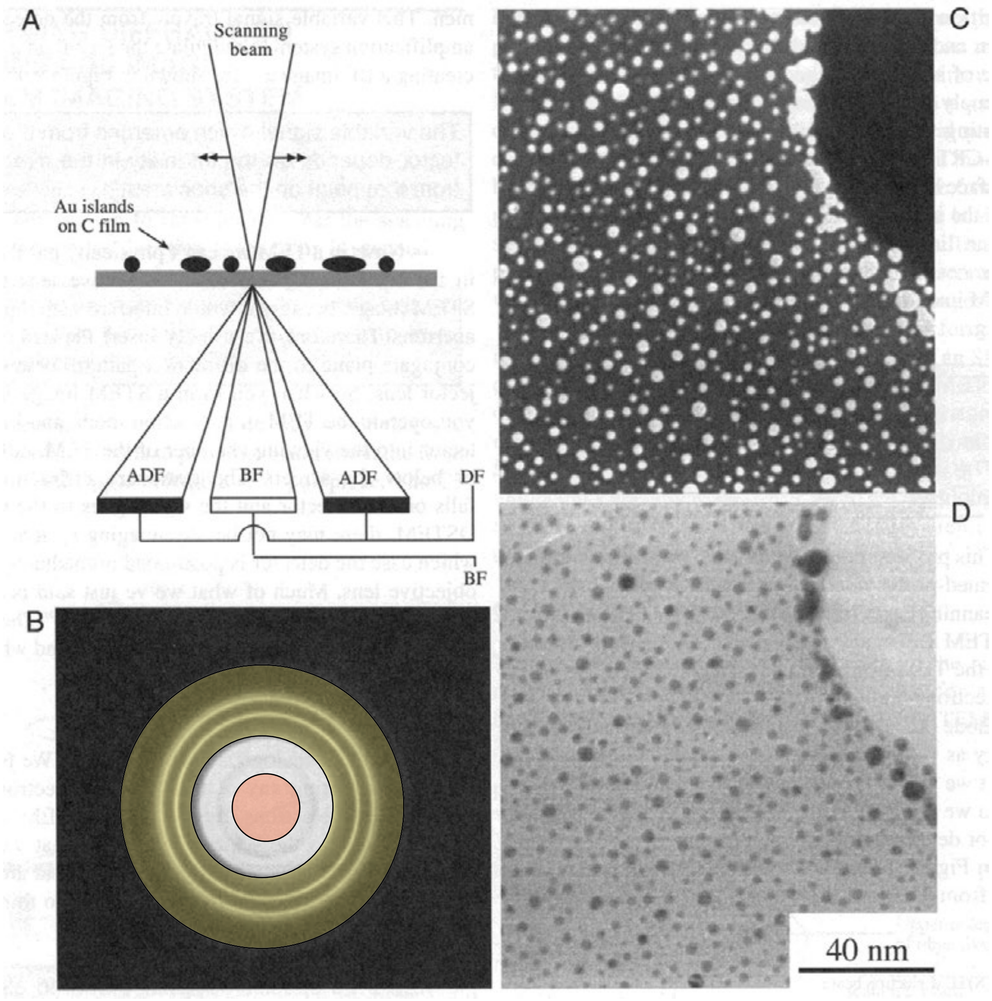
- Bright-field (**BF**) detector:
  solid disc (or square)

- Annular dark-field (**ADF**) detector:
  ring shaped – i.e. *annular*

- Both detectors characterised by their collection semi-angles ***β***

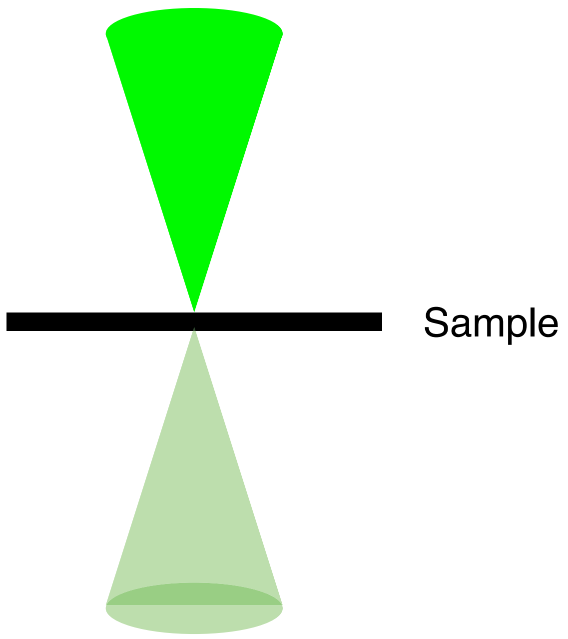
- If ADF detector is set up to collect diffracted beams we obtain a diffraction contrast dark-field image

- Diffraction intensity is an integration over all the selected beams – like an integration of multiple CTEM DF images



Convergence & collection angles

- The focused probe is a convergent e⁻ beam.
- The BF and ADF detectors are radially symmetric. They integrate signal over a range of scattering angles.
- Therefore all of them are characterised by angles
- The convergence semi-angle of the probe is called $\boldsymbol{\alpha}$
- Collection semi-angle for a detector is called $\boldsymbol{\beta}$
- Knowledge of these angles is important for STEM imaging



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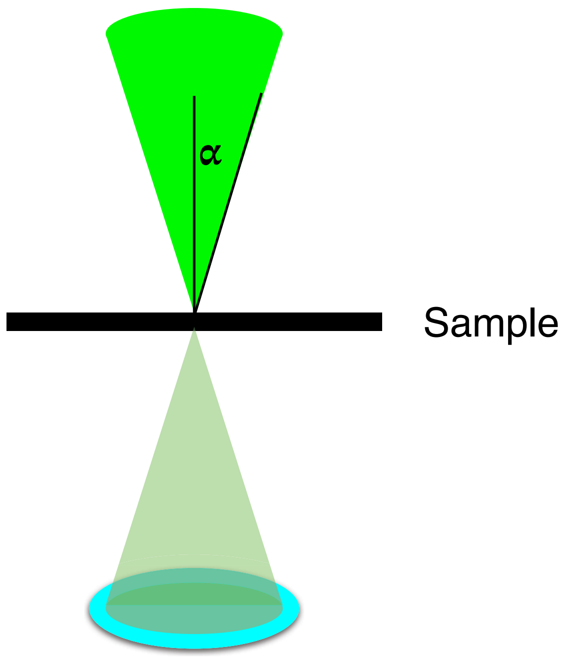

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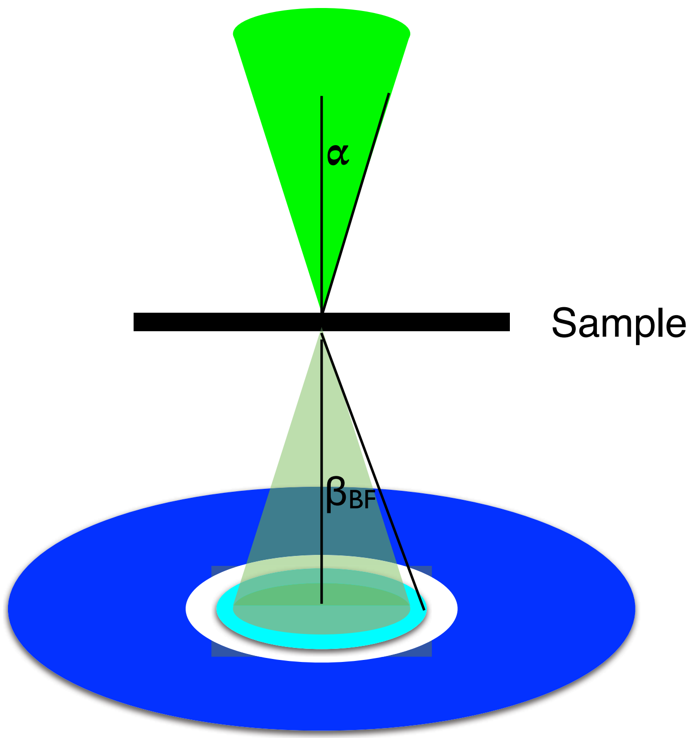
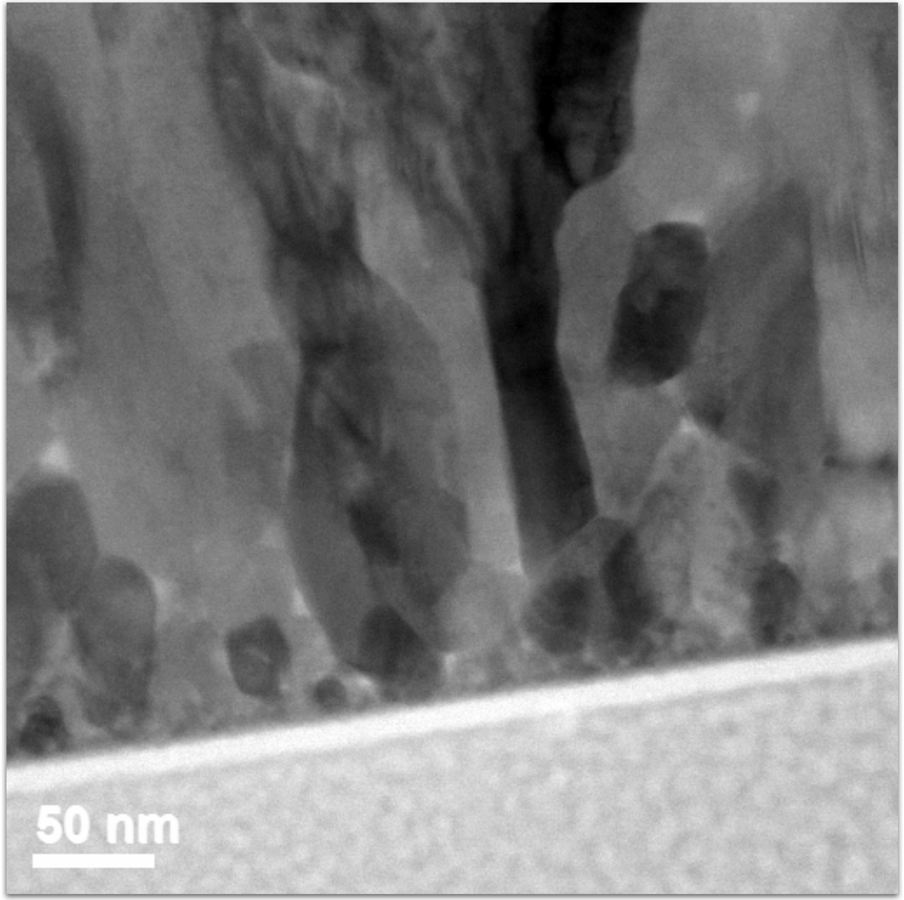


Bright-field STEM example

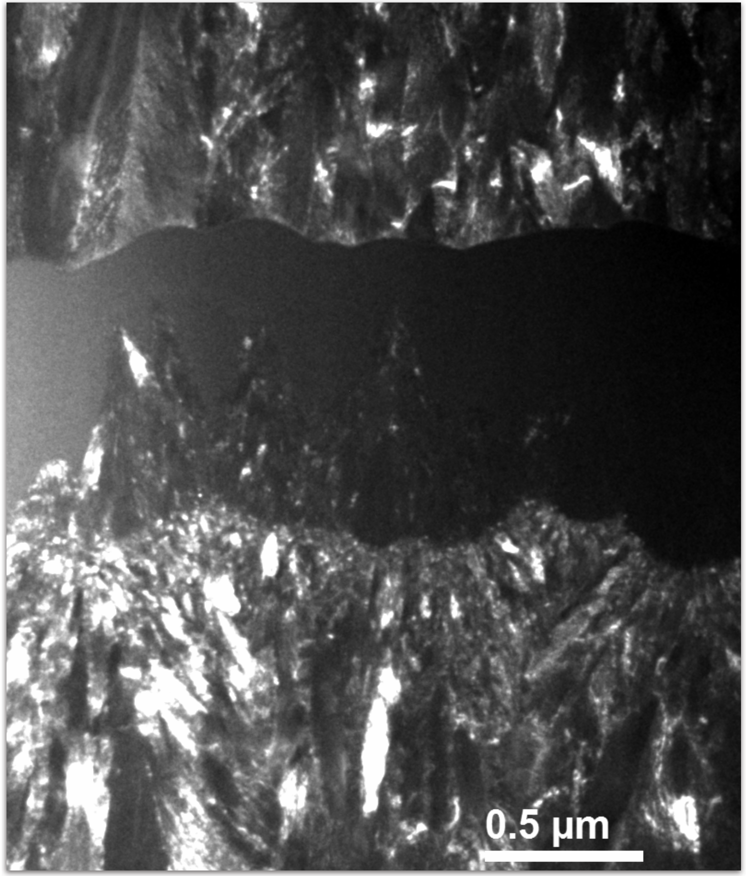
- Polycrystalline ZnO thin-film on glass substrate
- Image rather equivalent to that from BF TEM, but bit more “averaging” of grain contrast



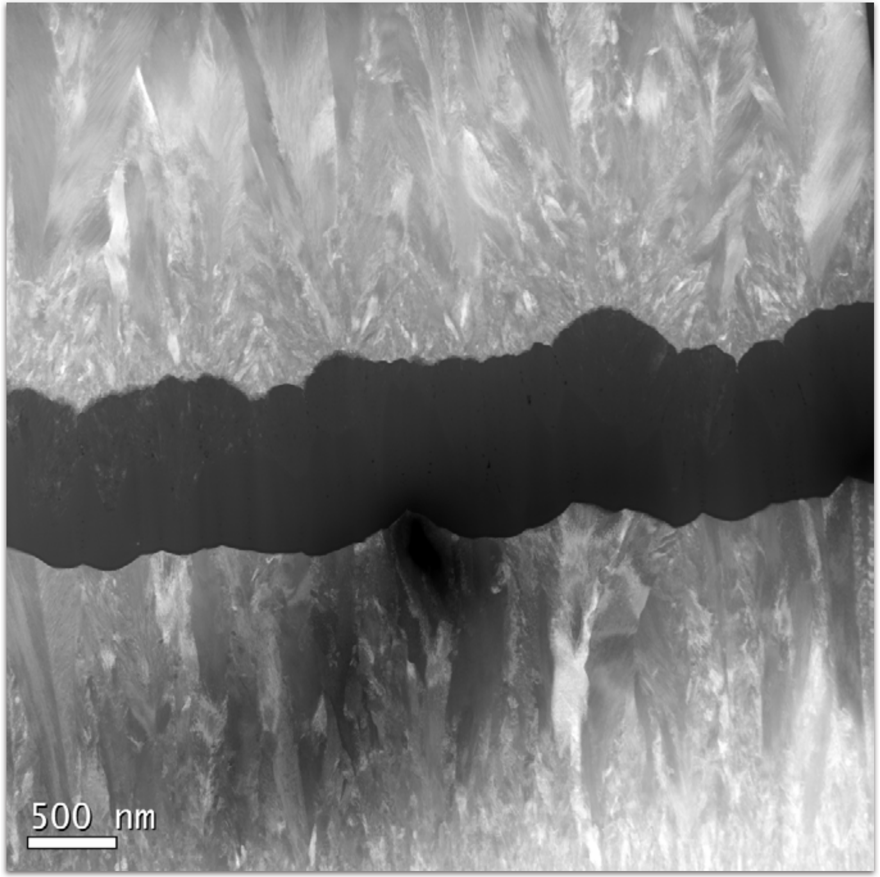
DF TEM vs ADF STEM example

- Photovoltaic stack (polycrystalline ZnO/proto-Si/polycrystalline-Si/ZnO)

DF TEM image: strong contrast, few grains have intensity

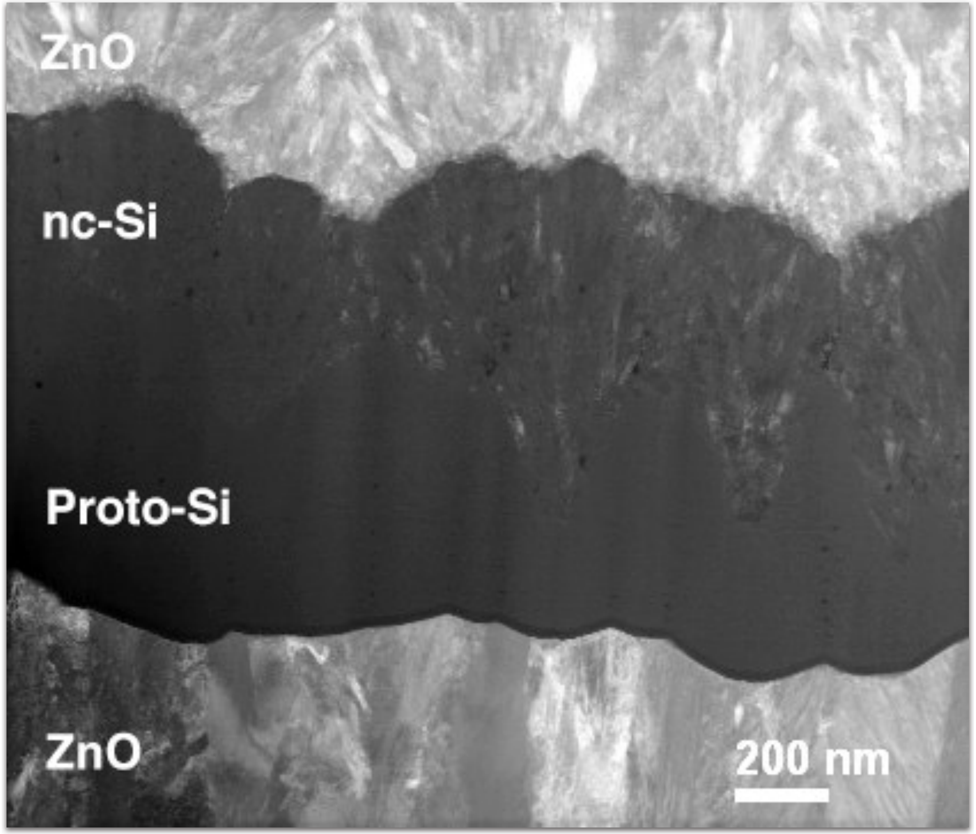


ADF STEM image: more grains have intensity, so more grain visibility & less contrast



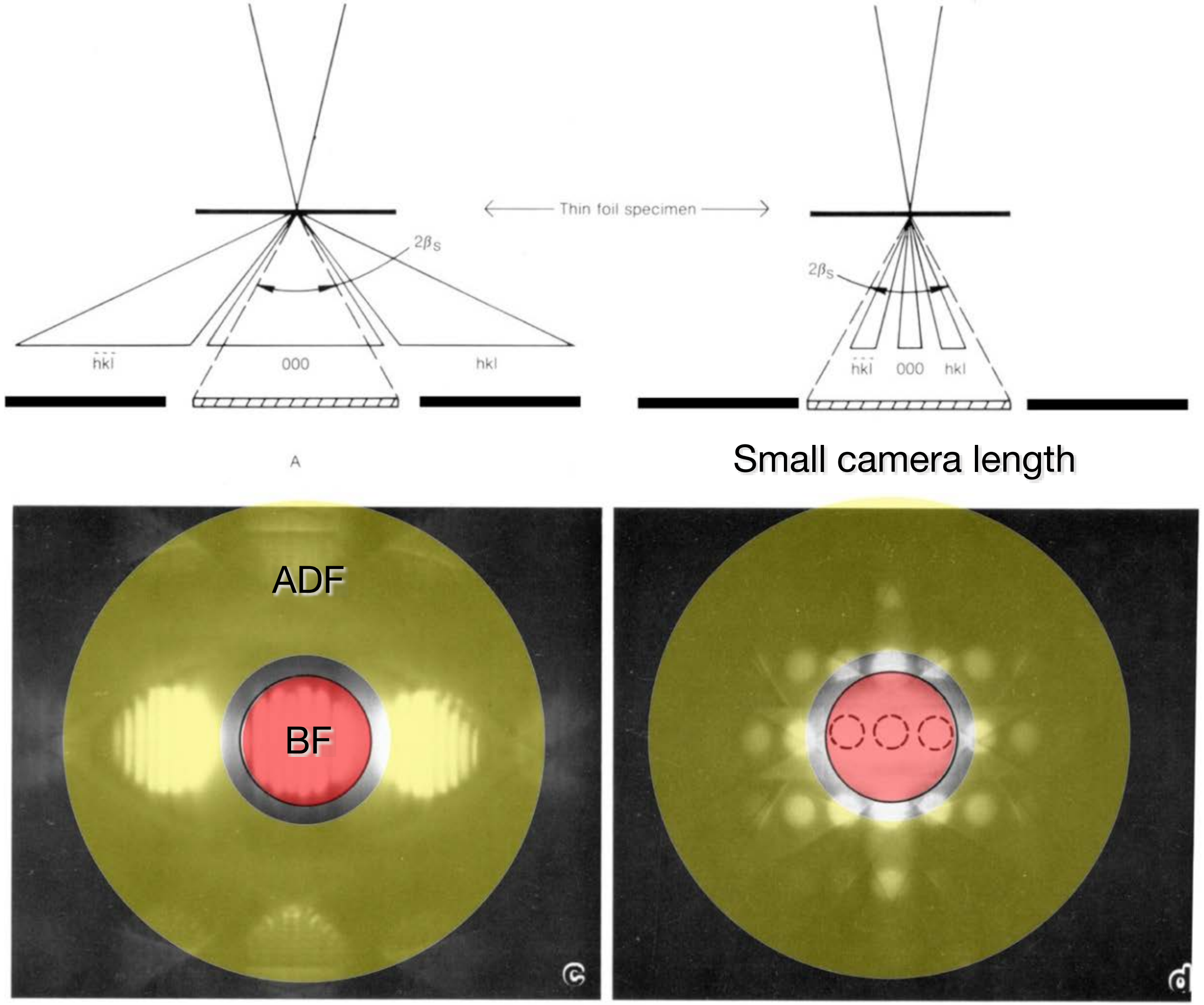
ADF STEM imaging example

- Photovoltaic stack (polycrystalline ZnO/proto-Si/polycrystalline-Si/ZnO)



Visible: protocrystalline/crystalline Si interface, B-doped layer in proto-Si, voids

Influence of camera length on BF/ADF



High angle scattering

- Low angle scattering intensity from crystalline samples dominated by coherent elastic scattering (“Bragg scattering”) – e.g. scattering angles up to ~20–30 mrad
- High angle scattering intensity instead dominated by “*thermal diffuse scattering*” (TDS)
- As first approximation, consider this as scattering by coulombic interaction with the nucleus (rather than e^- cloud)
- Relevant scattering angles: > 50–100 mrad (depending on HT)
- Commonly termed “*Rutherford scattering*”

Scattering amplitude/intensity from atom

- Mott formula for scattering amplitude of atom in terms of θ:

$$f(\theta) = \frac{1}{8\pi^2 a_0}\left(\frac{\lambda}{\sin\theta}\right)^2 \left[Z - f_x(\theta)\right]$$

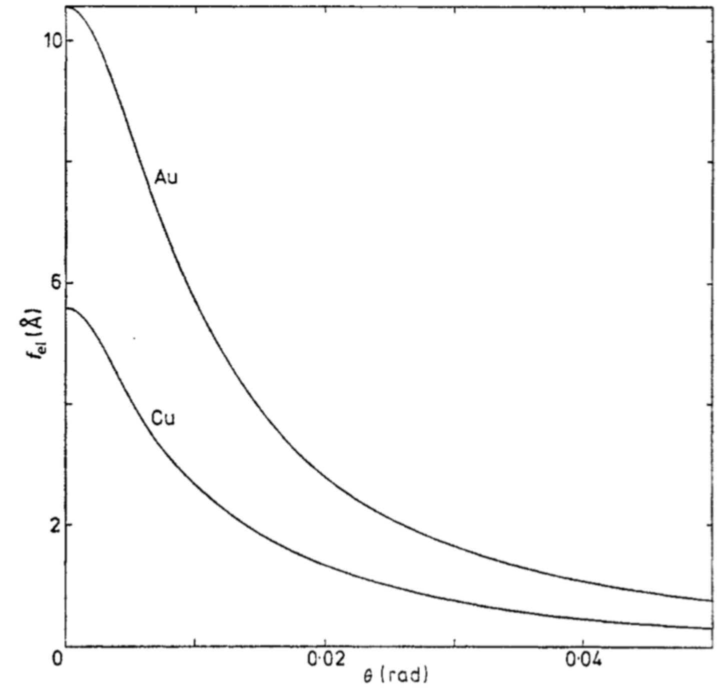


Figure 4. The first Born approximation scattering amplitude, $f^B(\boldsymbol{K}')$, as a function of θ for 100 keV electrons incident upon single atoms of gold and copper. The plots have been made using the relativistic Hartree–Fock scattering amplitudes of Doyle and Turner (1968).

Scattering amplitude/intensity from atom

- Scattering intensity at unit distance: $I(\theta) = |f(\theta)|^2$

- Also: $I(\theta) = \dfrac{d\sigma}{d\Omega}$

  σ: scattering cross-section – i.e. probability of scattering

- For large scattering angles: $f_x(\theta) \rightarrow 0$ hence:

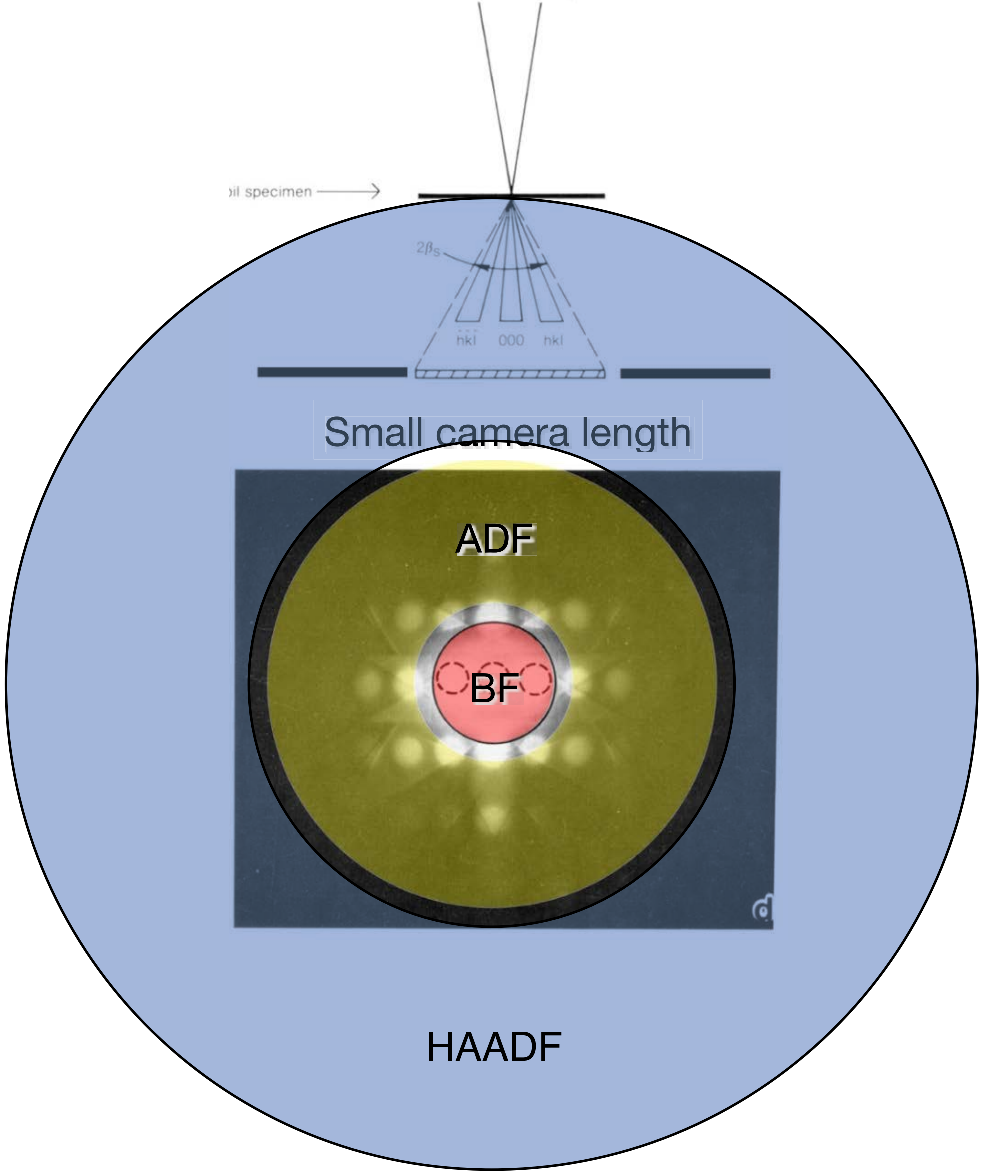
$$I(\theta)_{\theta \rightarrow \pi/2} = |f(\theta)|^2_{\theta \rightarrow \pi/2} = \frac{1}{(8a_0)^2}\left(\frac{\lambda}{\pi \sin\theta}\right)^4 Z^2 \propto Z^2$$

- Therefore Mott scattering ⟶ Rutherford scattering for high scattering angles

- Mechanism behind the high angle scattering is TDS, which is *incoherent* in nature

High-angle annular dark-field (HAADF)

- High-angle annular dark-field (HAADF) detector uses geometry to give large collection angles
- Aim: collect high angle scattered intensity dominated by incoherent “Rutherford-type” scattering
- Inner collection angle: $\beta_{\text{inner}} \approx$ 50–100 mrad (HT dependent)
- Outer collection angle typically: $\beta_{\text{outer}} \approx$ 200 mrad



HAADF and Z-contrast imaging

- By collecting "Rutherford-scattered" electrons, the HAADF detector produces an image which (ideally) gives an intensity:
  $I \propto t \cdot Z^2$
  for thickness t;
  average atomic number Z

- Diffraction contrast is eliminated (or at least much reduced…)

- Therefore often termed "Z-contrast imaging"

- In reality:
  $I \propto t \cdot Z^{1.6-2}$

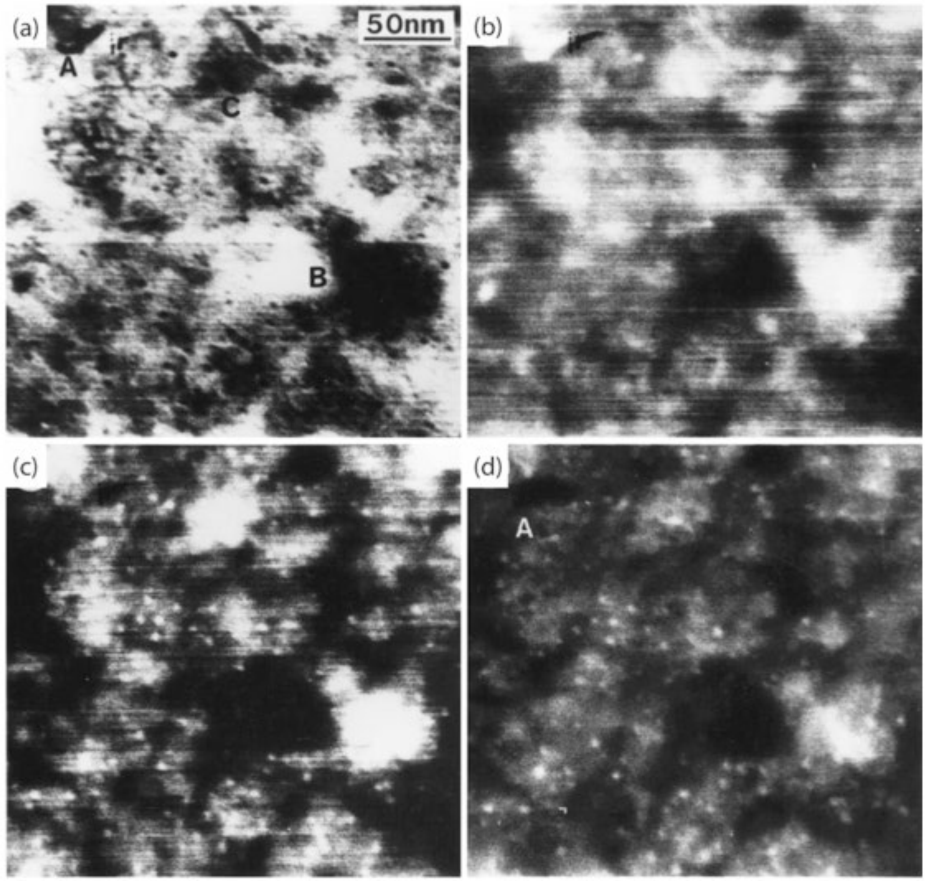
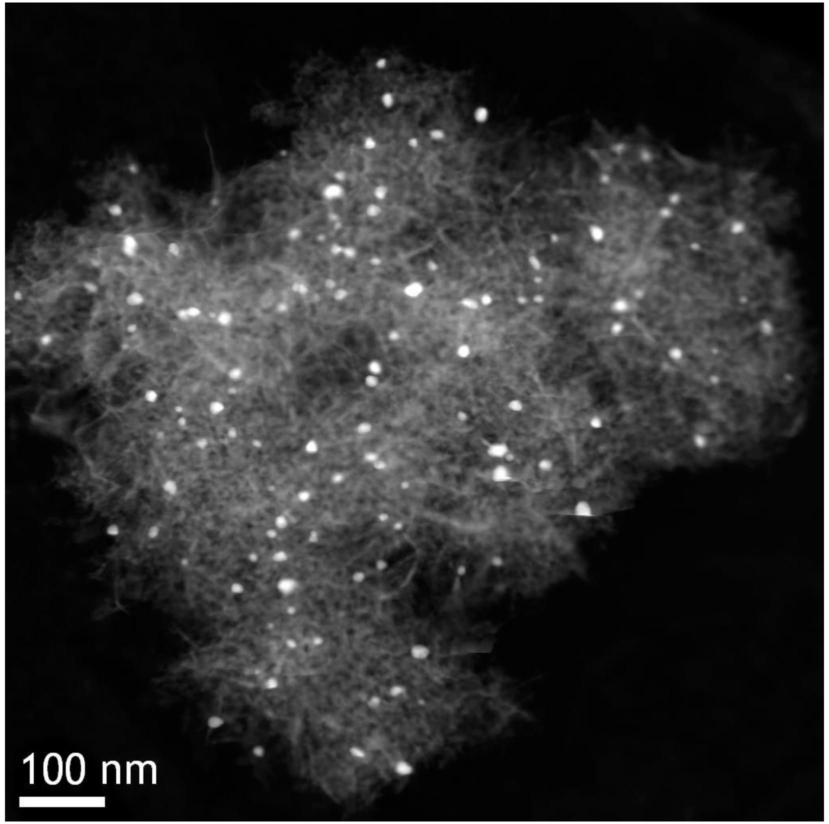


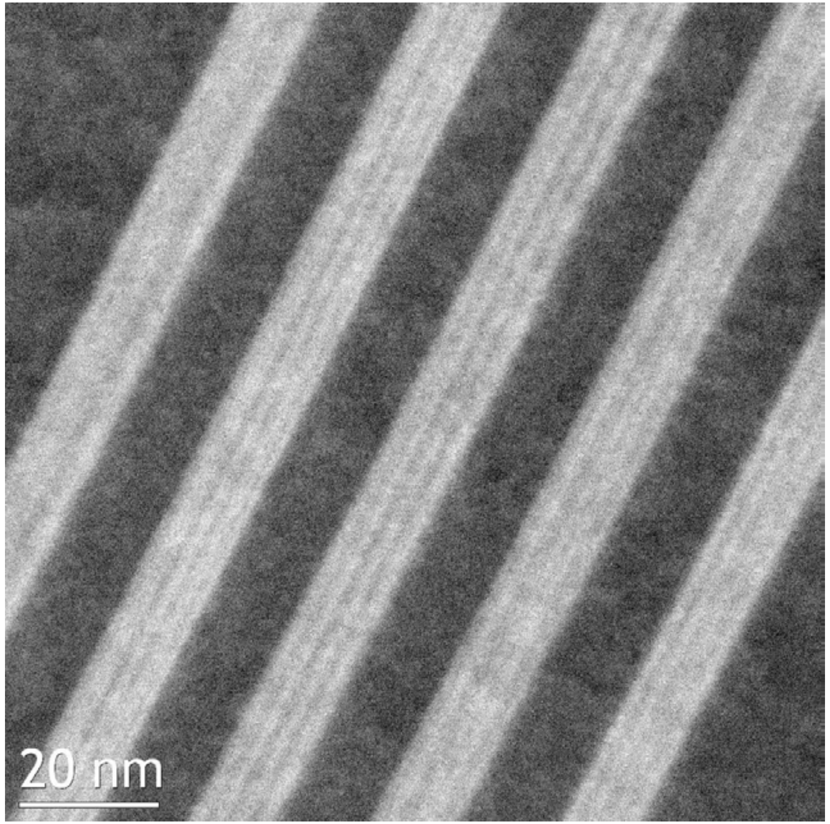
Figure 1–11. Images of a Pt particles on γ-alumina recorded in **(a)** bright field, **(b)** low-angle ADF, **(c)** HAADF, and **(d)** the ratio of high-angle to low-angle ADF signals. Particle contrast is highest in the HAADF image, reproduced from M. M. J. Treacy, PhD thesis, University of Cambridge, 1979, with permission.

HAADF and Z-contrast imaging

- Z-contrast imaging examples:



Pt catalyst on Al_2O_3



Si-Ge/Si multilayer

HAADF and Z-contrast imaging

- Z-contrast imaging examples:

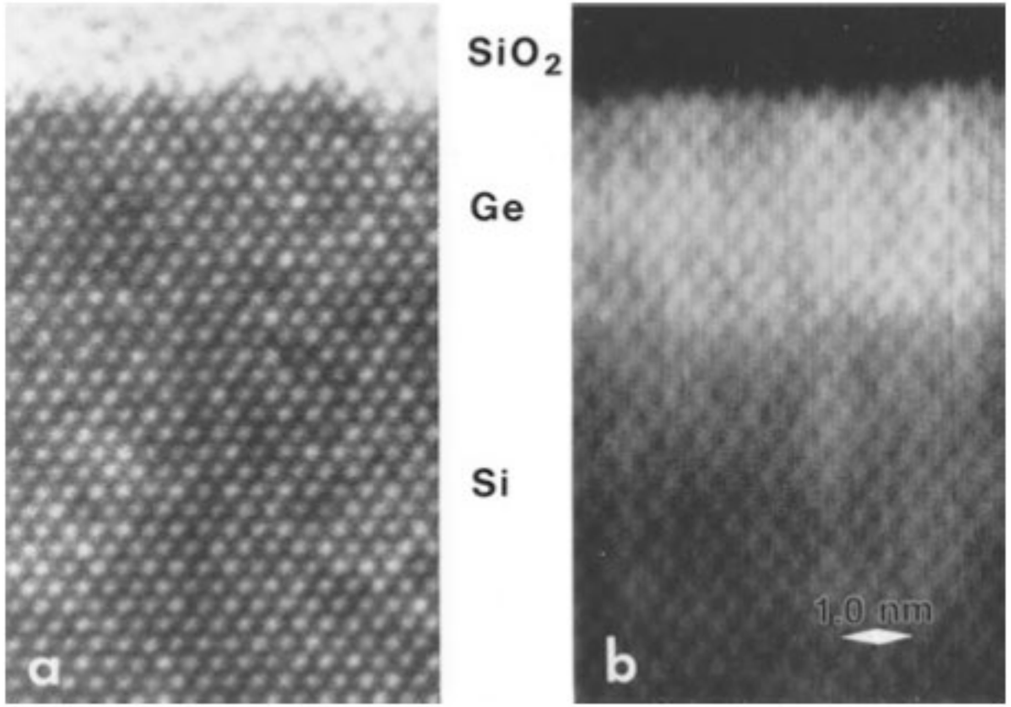


Figure 1–16. Images of a Ge film grown epitaxially on Si by an implantation and oxidation method. (**a**) Conventional TEM image from a JEOL 200CX, (**b**) Z-contrast image obtained with a VG Microscopes HB501UX clearly delineating the Ge layer, reproduced from Pennycook (1989a).

HAADF and Z-contrast imaging

- Z-contrast imaging examples:

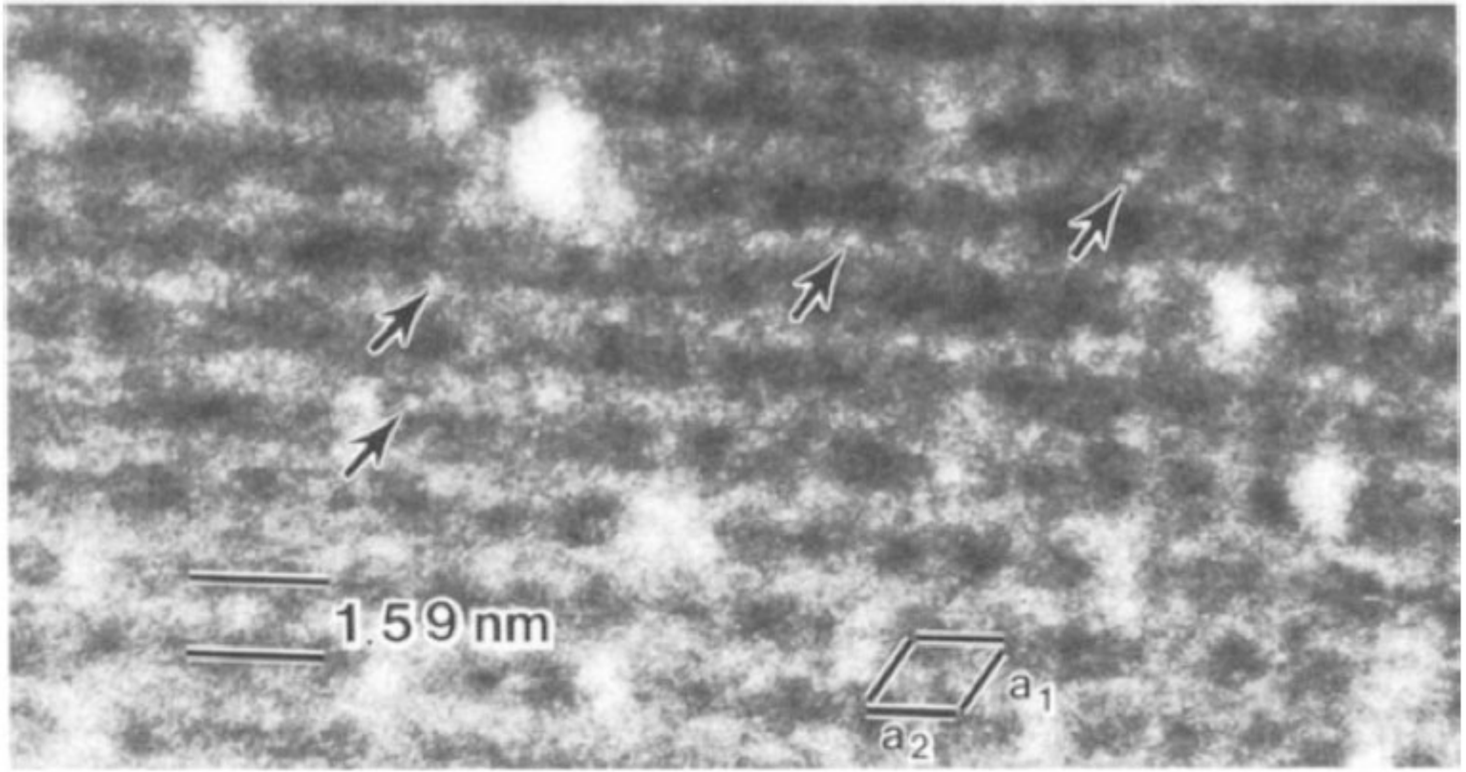
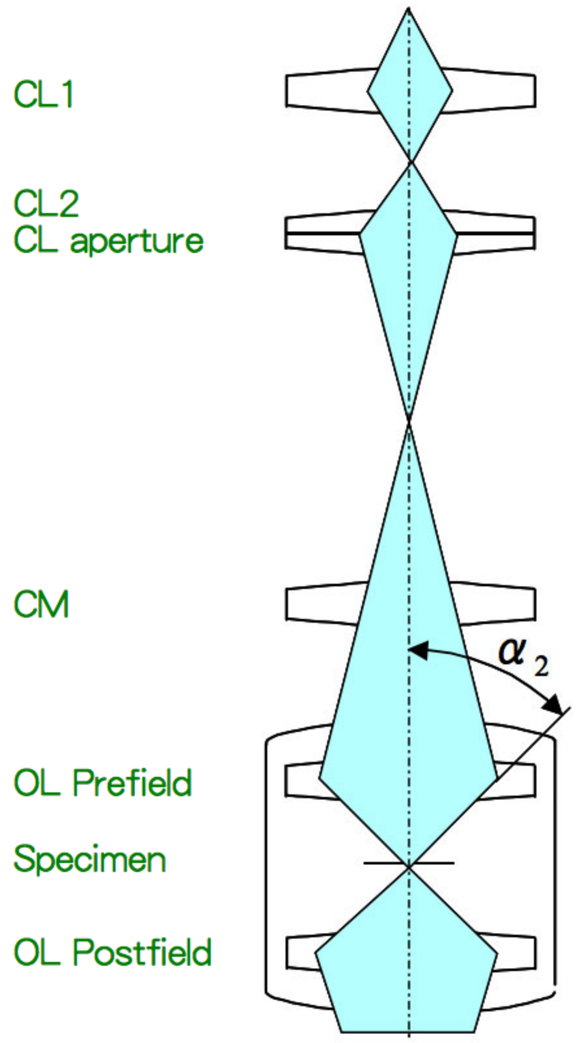


Figure 1–12. HAADF image showing individual Pt atoms in a zeolite framework, reproduced from Rice et al. (1990) with permission.

TEM/STEM design

- Many modern field emission gun TEM instruments can also be used in STEM mode
- STEM detectors are inserted in the back focal plane of the objective lens, or another plane conjugate to this in the intermediate/projector lens system
- Use nanoprobe (highly convergent) illumination – see next slide
- Scan beam with (S)TEM beam shift coils
- Modern STEM detectors send signal to computer acquisition software

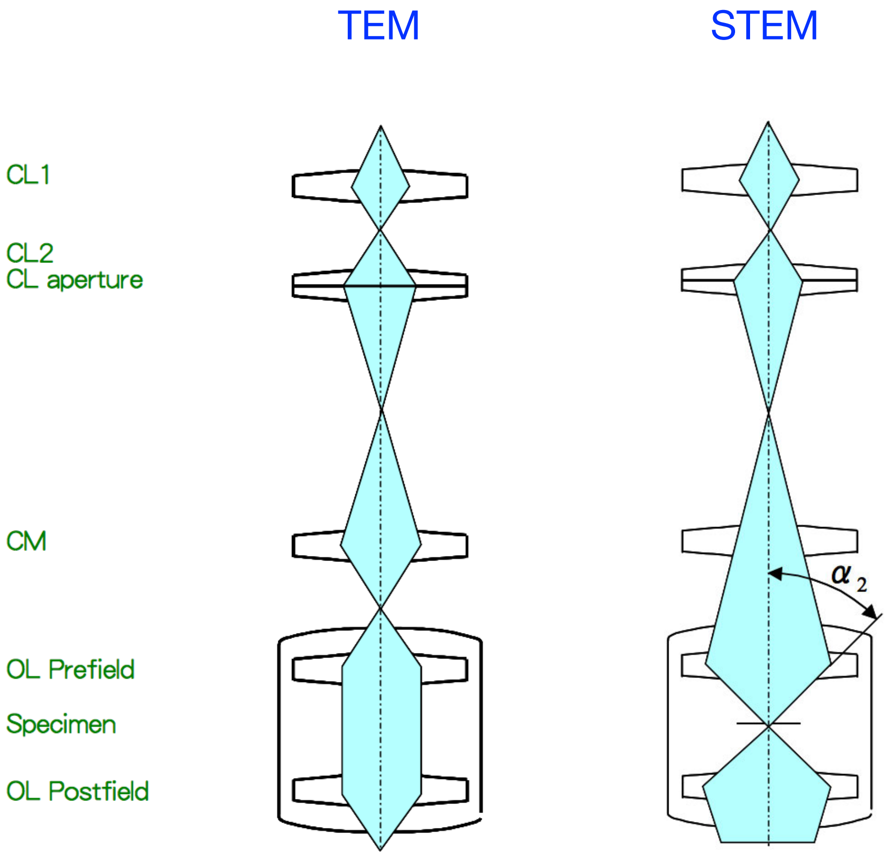


Condenser lens set up TEM vs STEM

- TEM mode: use 3 condenser lenses to achieve more parallel incident beam: C1 (“spot size”); C2 (“Intensity”/“Brightness”); Condenser minilens (CM)/ Minicondenser

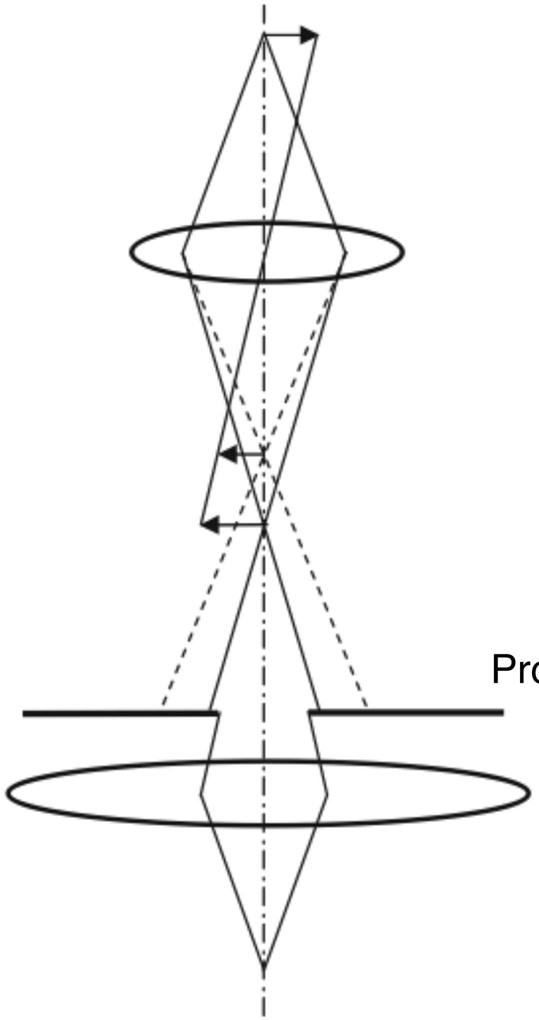
- Condenser minilens works in conjunction with upper pole piece of objective lens to make parallel rays transmitting across sample (i.e. parallel illumination)

- STEM mode: deactivate condenser minilens
$\Longrightarrow$ highly converged probe on specimen



The electron source and probe

- To benefit from these advantages of STEM we need a very well focused electron probe
- This requires: good optics and a small source
- *Why do we need a small source?*
- e^- probe is a **demagnified image of the source**
- 1st condenser lens (CL1) is typically responsible for demagnification ("spot size")
- Greater lens strength ⟹ move cross-over up
  ⟹ smaller e^- probe
- Cost: *reduced beam current and worse signal to noise ratio in image*
- ⟹ *Need field emission gun (FEG)!*



The electron source and probe

- First STEM microscopes by Albert Crewe (see right) and VG used cold field emission guns
- Cold field emission gun has smaller source, higher brightness than warm, Schottky field emission gun, but has worse beam current stability and more stringent vacuum requirements
- However, with new vacuum technologies, becoming popular again (Nion, JEOL ARM, Hitachi and now Thermo Fisher)
- FEI/Thermo Fisher has also X-FEG technology: high brightness Schottky FEG