Contrasts in TEM imaging

Duncan Alexander
EPFL-IPHYS-LSME

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  - Image delocalization
- Imaging of defects
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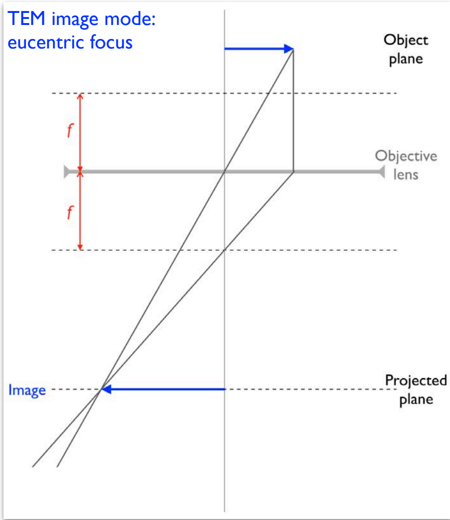
Objective lens focus

- In image mode, the image plane of the objective lens at *eucentric focus* forms the object plane for the next lens in the series (i.e. the first intermediate lens). The two lenses are *coupled* and the image plane is projected to the detector (camera or viewing screen) by the intermediate and projector lenses.



Objective lens focus

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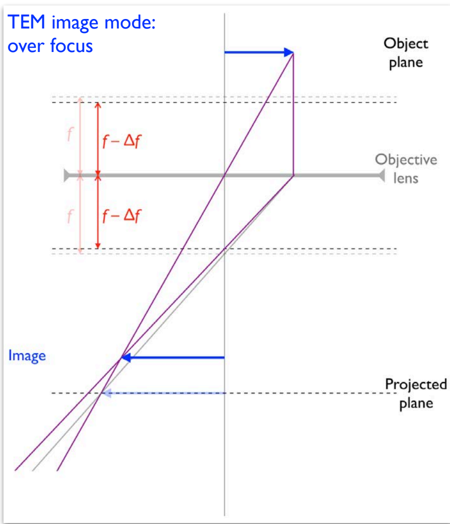
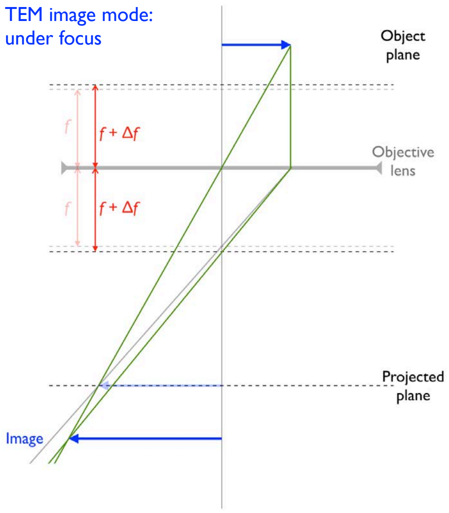
- If we increase objective lens strength, the image is formed above the projected plane. At the image plane there is an out of focus image which is then projected onto the detector. This image is called "*over focus*".



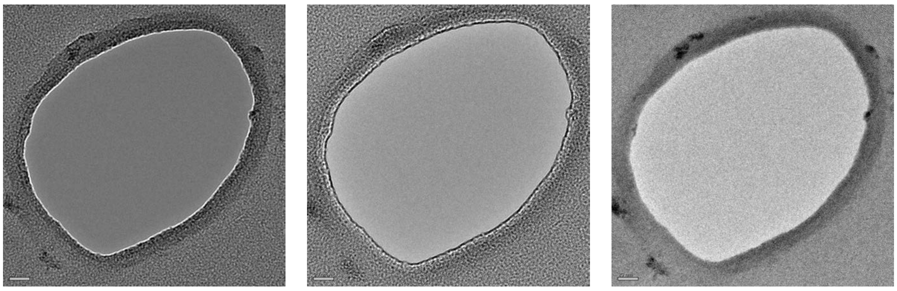
Objective lens focus

- In image mode, the image plane of the objective lens at *eucentric focus* forms the object plane for the next lens in the series (i.e. the first intermediate lens). The two lenses are *coupled* and the image plane is projected to the detector (camera or viewing screen) by the intermediate and projector lenses.
- If we increase objective lens strength, the image is formed above the projected plane. At the image plane there is an out of focus image which is then projected onto the detector. This image is called "*over focus*".
- If the objective lens strength is instead decreased the image is formed below the projected plane, and is "*under focus*".



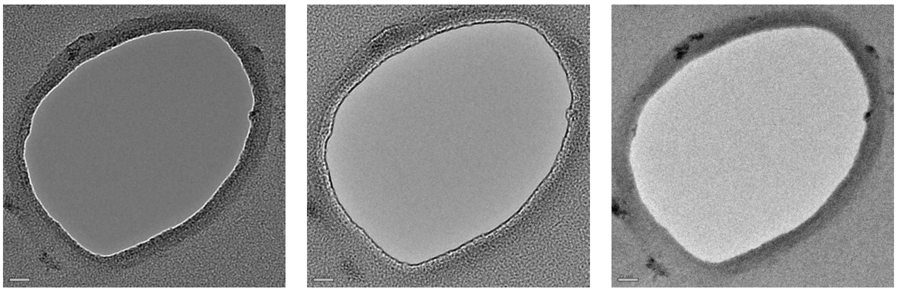
Objective lens focus

- Quiz: which of these images is in focus?



Objective lens focus

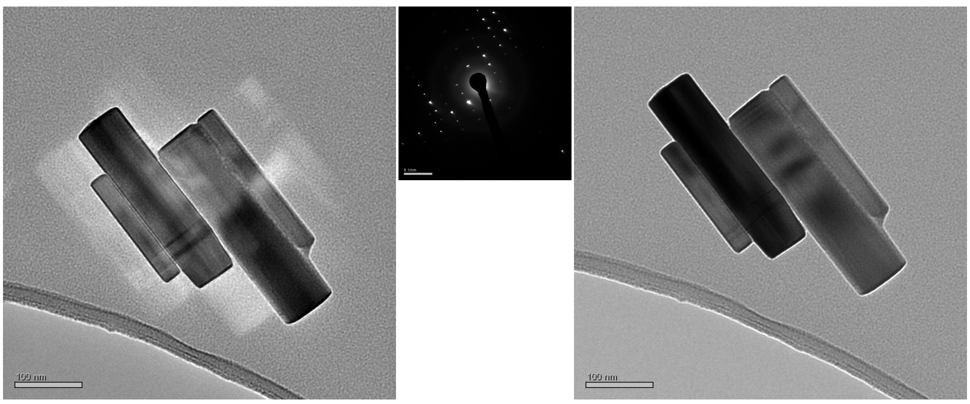
- *Very important: when the sample is in focus there is minimum contrast (see phase contrast lectures)*
- Quiz: which of these images is in focus?



1 2 3

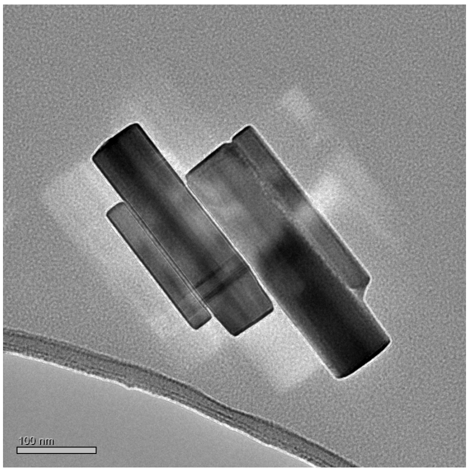
- *Image 3 is in focus: no Fresnel fringe at edge of hole, no specular ("speckled") contrast in the carbon film, therefore it has little contrast*

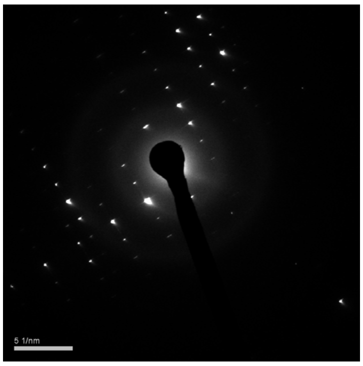
Which of these images of GaN nano-wires was taken with an objective aperture?



1 2

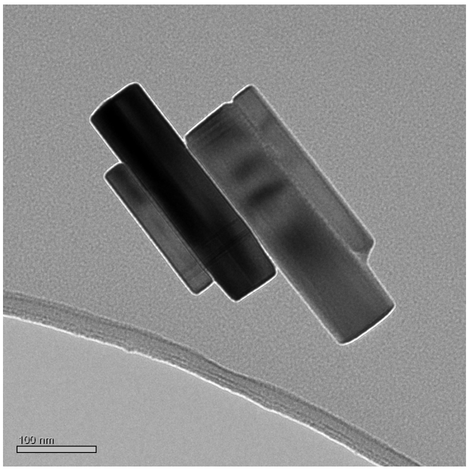
Image delocalization





TEM image with no objective aperture. Image formed from direct beam and diffracted beams. Dark-field images from diffracted beams delocalize from bright-field image of direct beam. Gives shadow images that move with objective focus (draw ray diagrams for out of focus image).

Image delocalization



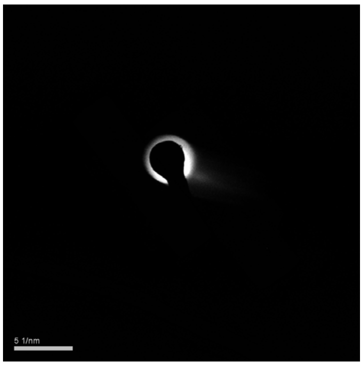
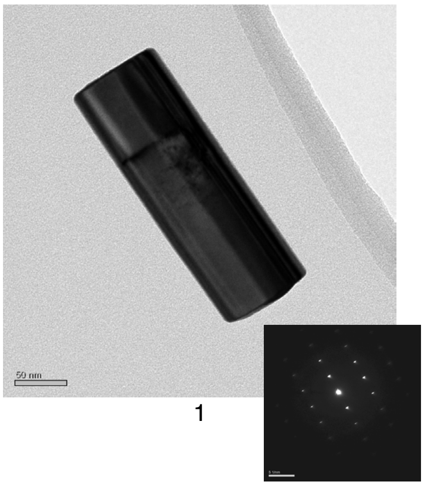


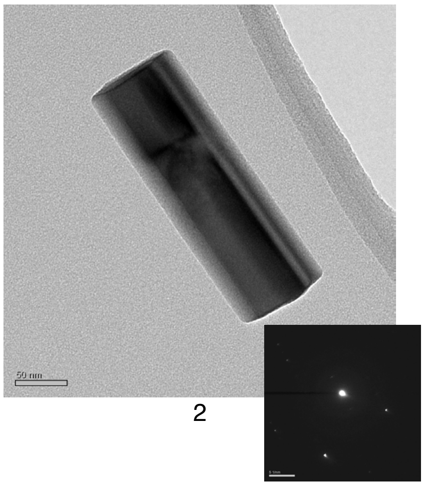
Image of same nanowires but with objective aperture to make bright-field image. No diffracted beams => no shadow images.
This is how you should take your TEM data!

Diffraction contrast on/off zone axis

In bright-field imaging, zone axis condition => more scattering to diffracted beams
Therefore intensity in direct beam goes down and bright-field image has strong contrast
Example: GaN nanowire



1



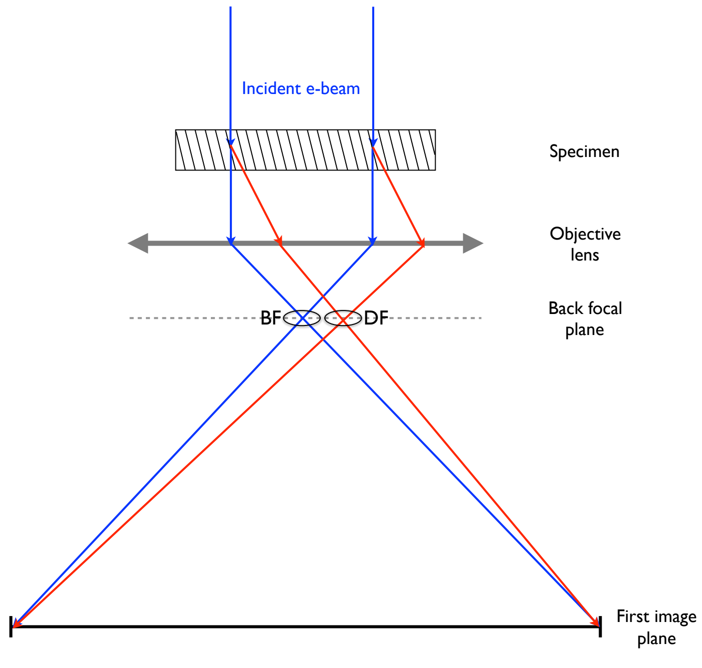
2

Diffraction contrast imaging of defects

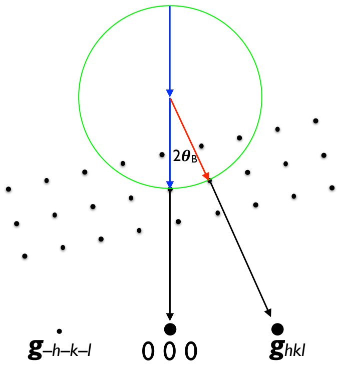
Principle of diffraction contrast imaging

- 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

2-beam displaced aperture dark-field imaging

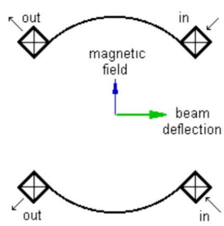


2-beam displaced aperture dark-field imaging



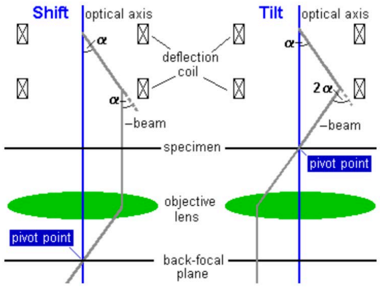
- Ewald sphere cuts reciprocal lattice node exactly
- Off-axis rays form DF image
  ⟹ aberrations and astigmatism
  ⟹ image moves when change objective lens focus

Beam deflection coils



- Deflection coils: set of coils either side of e⁻ beam
- Apply positive magnetic field to one, negative to the other
  ⟹ Deflection of e⁻ beam towards positive field
- Arcs used to generate homogeneous magnetic field
- Two perpendicular sets allow deflection in *X* and *Y* directions

Beam deflection coils

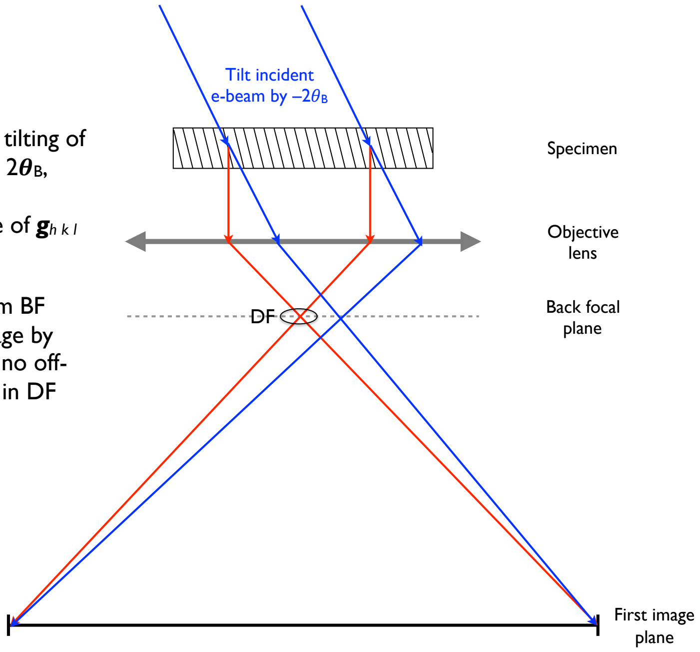


- Above objective lens have set of double deflection coils
- Can be used to:
  - ➡ Shift incident beam on sample
  - ➡ Tilt incident beam on sample

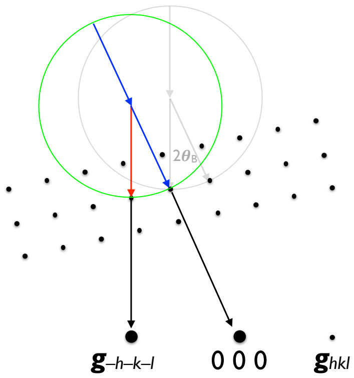
Centred aperture dark-field imaging

Corresponds to tilting of Ewald sphere by $2\theta_B$, excite $\mathbf{g}_{-h\,-k\,-l}$; 0 0 0 takes place of $\mathbf{g}_{h\,k\,l}$ in SADP

Can now go from BF image to DF image by pressing button, no off-axis aberrations in DF image



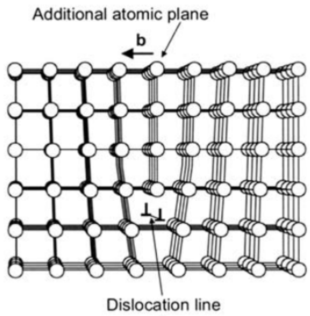
Centred aperture dark-field imaging



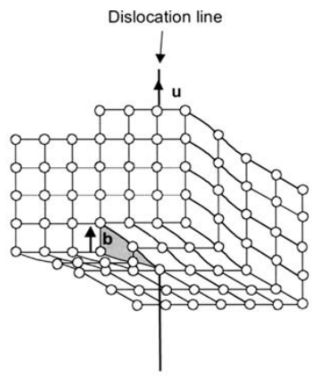
- Corresponds to tilting of Ewald sphere by $2\theta_B$, excite $\mathbf{g}_{-h\,-k\,-l}$; 0 0 0 takes place of $\mathbf{g}_{h\,k\,l}$ in SADP
- Can now go from BF image to DF image by pressing button, no off-axis aberrations in DF image

Imaging crystal defects: dislocations

Edge dislocation:
Burgers vector ***b*** perpendicular to dislocation line ***u***



Screw dislocation:
Burgers vector ***b*** parallel to dislocation line ***u***



- Note dislocations are often *mixed* in nature (both edge and screw components)

Diagrams from Morniroli, LACBED book

Imaging crystal defects: dislocations

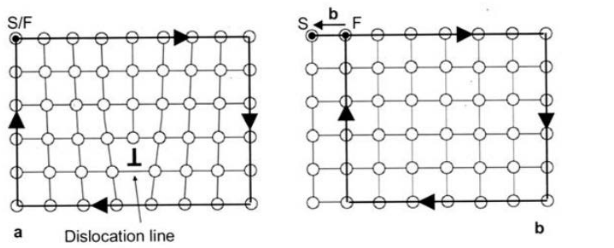


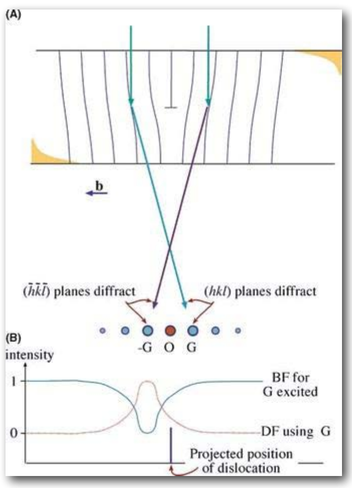
Figure XI.4 - Burgers circuit around an edge dislocation.
a - A closed right-handed circuit is drawn around the dislocation line.
b - The same circuit is drawn in a perfect crystal. The F →S gap characterizes the Burgers vector **b**.
On this diagram, the vector **u** points into the page.

- For dislocation analysis want to:
  - ➡ image dislocation
  - ➡ characterise both ***b*** and ***u***

Diagrams from Morniroli, LACBED book

Imaging crystal defects: dislocations

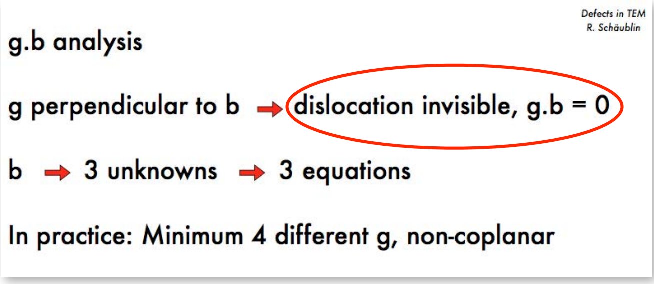
Local bending of crystal planes around the dislocation change their diffraction condition
This produces a contrast in the image => **g**.**b** analysis for Burgers vector

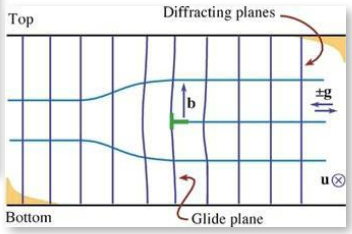


From Williams & Carter
Transmission Electron Microscopy

Crystal defects: dislocations - **g.b** analysis

- On a basic level, planes parallel to ***b*** are not distorted by the dislocation
  ⟹ these planes show no change in contrast
- This condition corresponds to ***g.b*** = 0 – the *invisibility criterion*

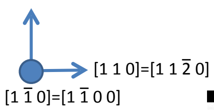




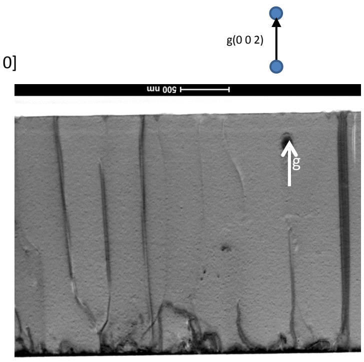
- Note: for edge dislocation the glide plane parallel to ***b*** can be buckled
  ⟹ still gives some contrast even for ***g.b*** = 0.
  Plane perpendicular to ***u*** and parallel to ***b*** gives no contrast: ***g***.(***b*** x ***u***) = 0

Crystal defects: dislocations - **g.b** analysis

Example: analysis of threading dislocations of hexagonal GaN grown on sapphire substrate



Condition1

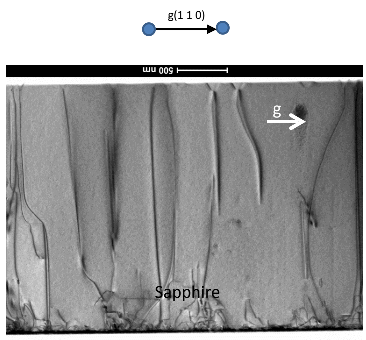


Sapphire

Visible:
Screw [0 0 0 1] and Screw[0 0 0 $\bar{1}$]
Mixed 1/3[2 $\bar{1}$ $\bar{1}$ 3],[$\bar{1}$ $\bar{1}$ 2 3],[$\bar{1}$ 2 $\bar{1}$ 3]

Invisible:
Edge 1/3[2 $\bar{1}$ $\bar{1}$ 0], [$\bar{1}$ $\bar{1}$ 2 0],[$\bar{1}$ 2 $\bar{1}$ 0]

Condition2



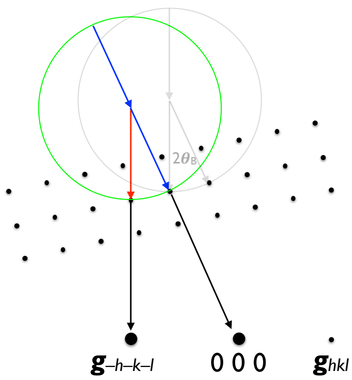
Visible:
Edge 1/3[2 $\bar{1}$ $\bar{1}$ 0], [$\bar{1}$ $\bar{1}$ 2 0],[$\bar{1}$ 2 $\bar{1}$ 0]
Mixed 1/3[2 $\bar{1}$ $\bar{1}$ 3],[$\bar{1}$ $\bar{1}$ 2 3],[$\bar{1}$ 2 $\bar{1}$ 3]

Invisible:
Screw [0 0 0 1] and Screw[0 0 0 $\bar{1}$]

Images by
Emad Oveisi, CIME

Weak beam imaging

Centred DF imaging (strong 2-beam $\boldsymbol{s}$ = 0)



- Corresponds to tilting of Ewald sphere by $2\theta_B$, excite $\mathbf{g}_{-h-k-l}$; 0 0 0 takes place of $\mathbf{g}_{hkl}$ in SADP
- Can now go from BF image to DF image by pressing button, no off-axis aberrations in DF image

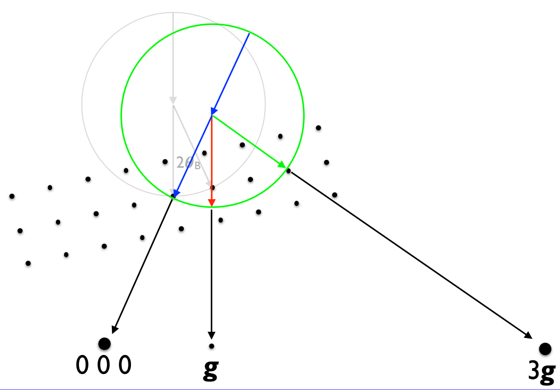
Weak beam imaging (*s* >> 0)

$$I_g = \left(\frac{\pi t}{\xi_g}\right)^2 \frac{\sin^2(\pi t s_{eff})}{(\pi t s_{eff})^2} \qquad s_{eff} = \sqrt{s^2 + \frac{1}{\xi_g{}^2}}$$

- Imaging with ***s*** = 0 gives contrast which is highly dynamical (s_{eff} varies strongly with ξ_g, I_g very sensitive to Bragg condition)
- Also images of dislocations are imprecise because you measure the whole strain field (see dislocation imaging, later)
- Can solve by imaging with large **s**, e.g. ***s*** ≈ 0.2 nm^{-1}
- This is called *weak beam imaging*

Weak beam imaging (*s* >> 0)

- Typical weak beam imaging conditions are **g**(3**g**) or 2**g**(5**g**)
- Example: **g**(3**g**). After tilting sample to excite g, beam/Ewald sphere is tilted $2\theta_B$ to excite 3**g** reflection. Then image with reflection **g** which now has large ***s***.

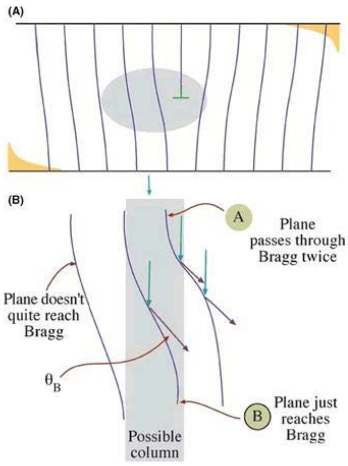


Principle of weak beam dislocation imaging

- The bright line corresponds to where planes are tilted towards satisfying the Bragg condition for diffraction vector **g**, which only happens close to the dislocation core
- The intensity peak is always displaced to one side of the dislocation core; if you go from **g** to –**g** it goes to the other side of the core
- Hirsch's kinematical approximation for screw dislocations finds half-width of dislocation given by:

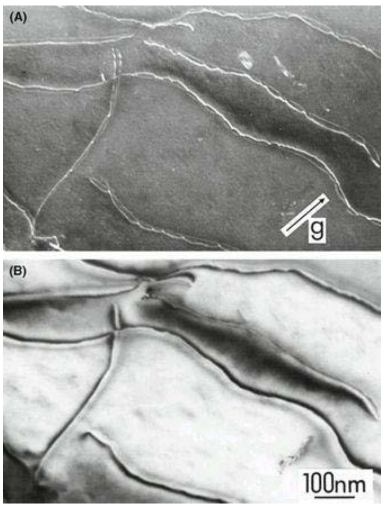
$$\Delta x = \frac{1}{\pi s_g} \cdot \frac{\xi_{\text{eff}}}{3}$$

- $\mathbf{s_g}$ = 0.2 nm⁻¹ => Δx = 1.7 nm (c.f. typically Δx > 10 nm for strong beam)



Weak beam imaging of strain fields

- When we use diffraction contrast to image dislocations we are in fact imaging their *strain fields* – i.e. the structural distortion of the crystal around their core
- In the weak beam dark field image, the dislocation shows as a sharp bright line on a dark background – *see exercises*

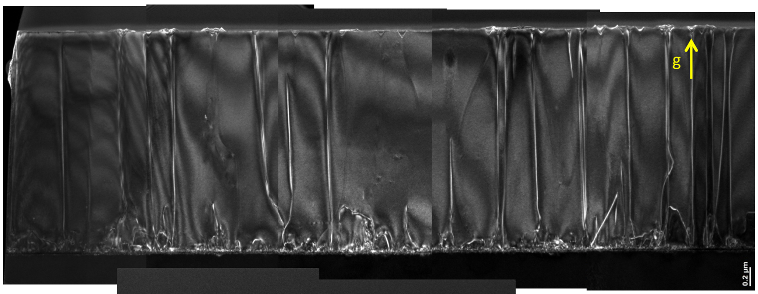


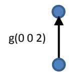
Weak beam image

Strong beam ($\mathbf{s_g}$ > 0) image

Comparison of dislocation images in Cu alloy

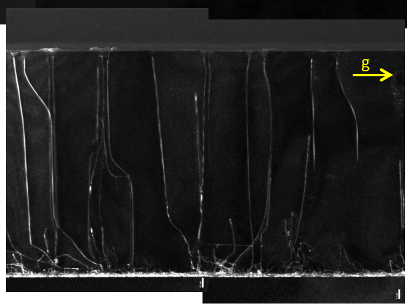
Weak beam example: **g.b** analysis on GaN

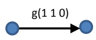




Visible:
Screw [0 0 0 1] and Screw[0 0 0 $\bar{1}$]
Mixed 1/3[2 $\bar{1}$ $\bar{1}$ 3],[$\bar{1}$ $\bar{1}$ 2 3],[$\bar{1}$ 2 $\bar{1}$ 3]

Invisible:
Edge 1/3[2 $\bar{1}$ $\bar{1}$ 0], [$\bar{1}$ $\bar{1}$ 2 0],[$\bar{1}$ 2 $\bar{1}$ 0]





Visible:
Edge 1/3[2 $\bar{1}$ $\bar{1}$ 0], [$\bar{1}$ $\bar{1}$ 2 0],[$\bar{1}$ 2 $\bar{1}$ 0]
Mixed 1/3[2 $\bar{1}$ $\bar{1}$ 3],[$\bar{1}$ $\bar{1}$ 2 3],[$\bar{1}$ 2 $\bar{1}$ 3]

Invisible:
Screw [0 0 0 1] and Screw[0 0 0 $\bar{1}$]

Summary on Contrasts in TEM imaging

- TEM is a contrast technique. We need contrast to see features in the sample. Often we have to take *defocused images* (i.e. under focus) in order to have the necessary contrast. The amount of defocus needed depends on the sample, and on the imaging conditions (e.g. magnification).
- Crystalline objects will give diffraction contrast in bright-field and dark-field TEM images; this contrast will depend on local variations in the intensity of the beam selected by the objective aperture.
- This diffraction contrast can be used for the precise analysis of crystal defects, such as 1-D defects called dislocations.
- The two main ways of imaging such dislocations are with:
  – strong beam centred dark-field imaging (excitation error s = 0)
  – weak beam dark-field imaging (excitation error s >> 0)
  The weak beam gives a sharper and better defined contrast.