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Dislocation imaging and characterisation in TEM

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What we will (try to) cover today

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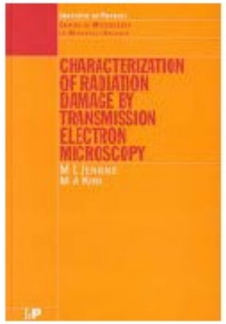
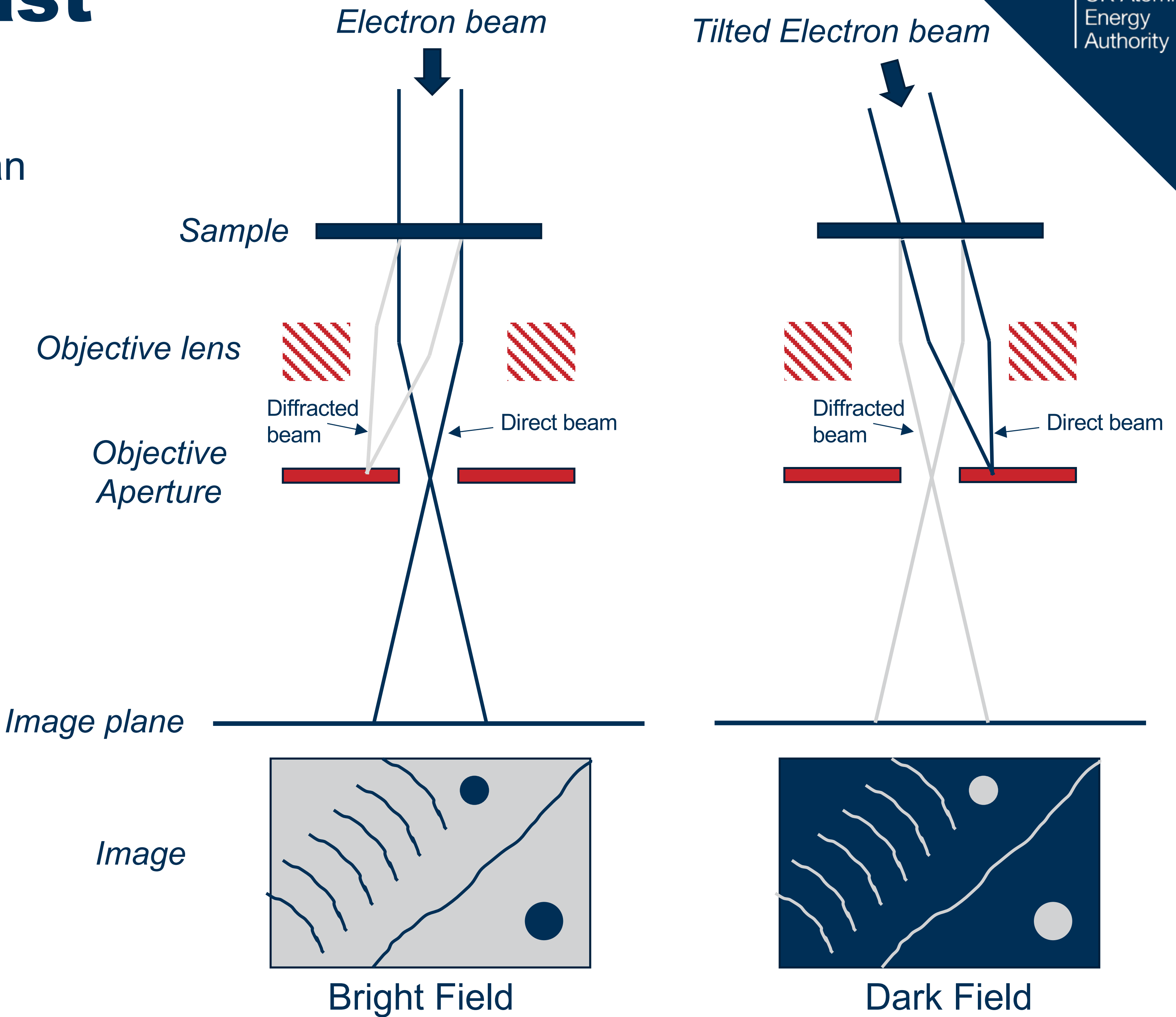
- Basic principles of diffraction contrast
- Weak-beam TEM
- Burgers vector analysis via invisibility criterion
- Artefacts in TEM
- STEM imaging of defects
- Burgers vector analysis via projection
- 4D-STEM of dislocations and loops
- Nature analysis of dislocation loops

Details of much of this talk are contained in the book : “Characterisation of radiation damage by transmission electron microscopy”, by M L Jenkins and M A Kirk



Diffraction Contrast

- By inserting a small aperture beneath our specimen, one diffracted beam can be selected and allowed to propagate down to our image plane.
- The resulting images are called **bright-field** or **dark-field**
- Contrast can be attributed to **diffraction from a specific plane**
- Can be used to identify **lattice defects**, like dislocations, or dislocation loops
- Also to identify other **phases**, like precipitates

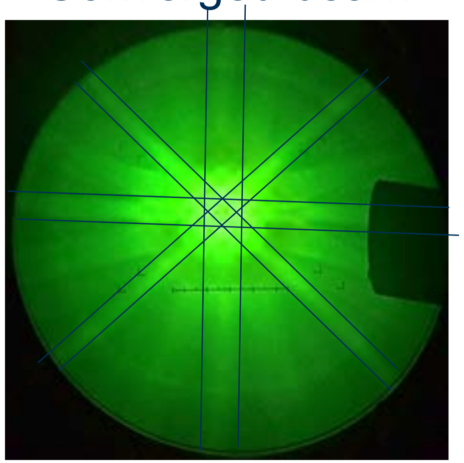


Two-beam diffraction conditions

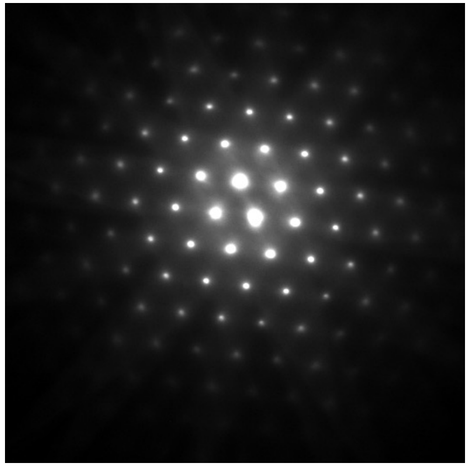
- Two-beam conditions are used in order to minimise the contributions of multiple beams to the contrast. Better to restrict the beam distribution to a single row of reflections

- Use **Kikuchi lines** to orient ourselves. Tilt along a Kikuchi band ~5-10 degrees

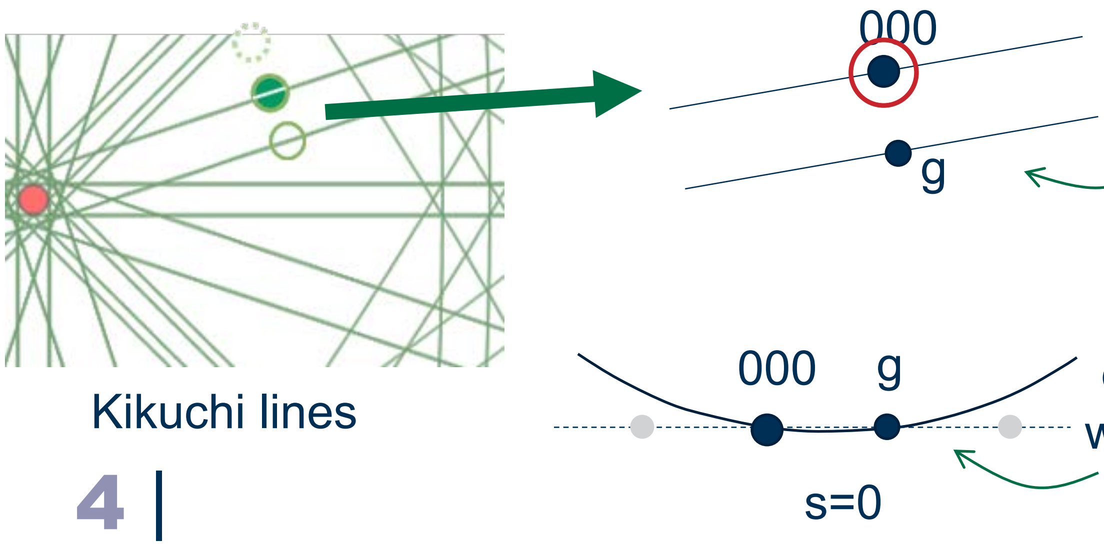
Converged beam



Parallel beam



Dynamical two-beam conditions

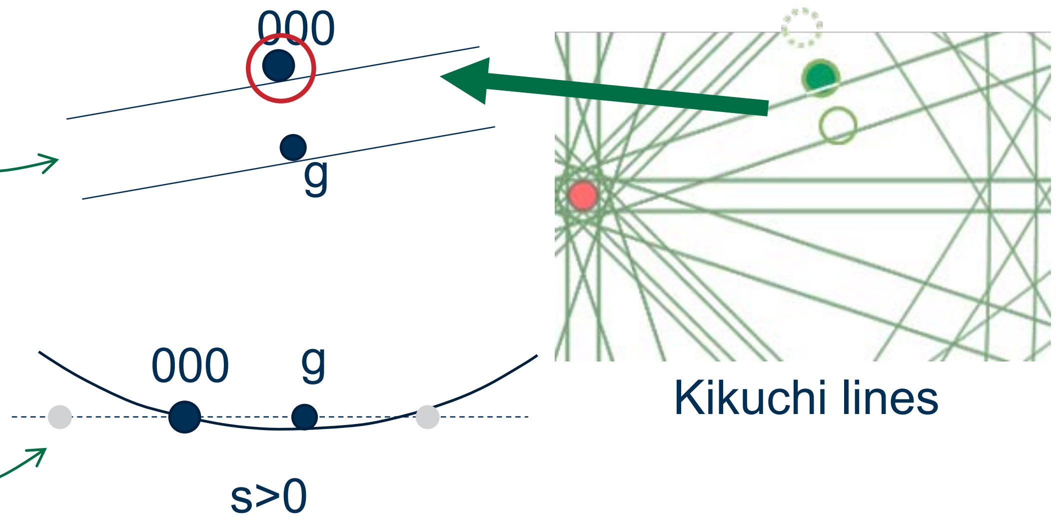


Kikuchi lines

Position of Kikuchi lines wrt diffraction spots

Position of diffraction spots wrt Ewald sphere

Kinematical two-beam conditions



Kikuchi lines

Effect on contrast on changing deviation parameter

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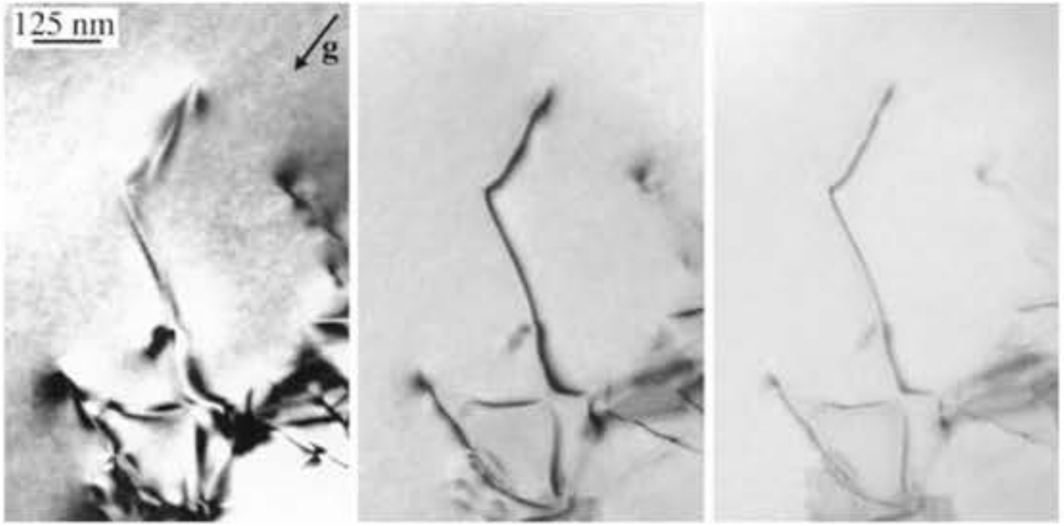
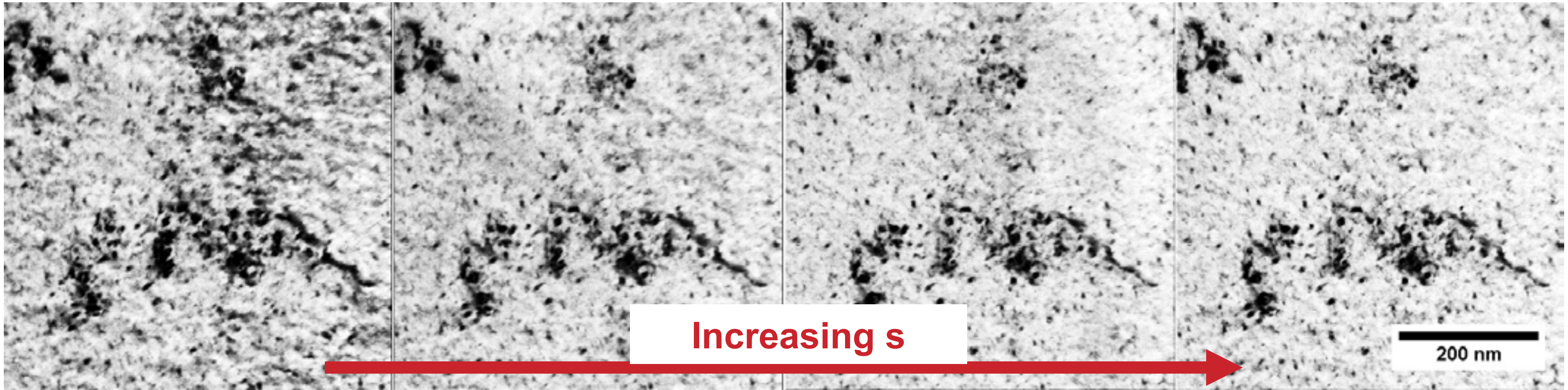
Dynamical bright field. s=0 | Kinematical bright field. s>0 and small | Kinematical bright field. s>0 and larger



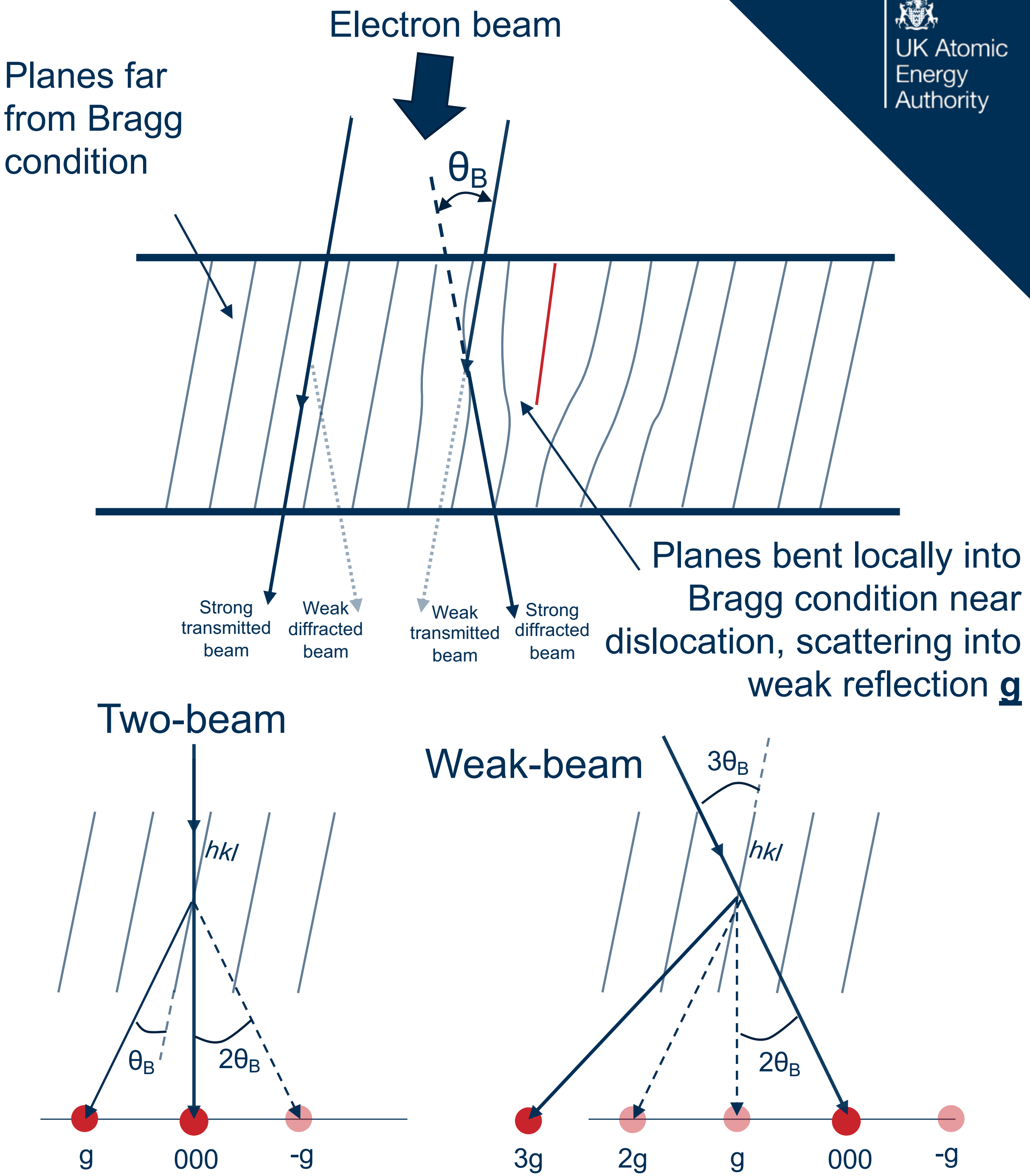
From Williams & Carter

Dislocation loops in irradiated FeCr

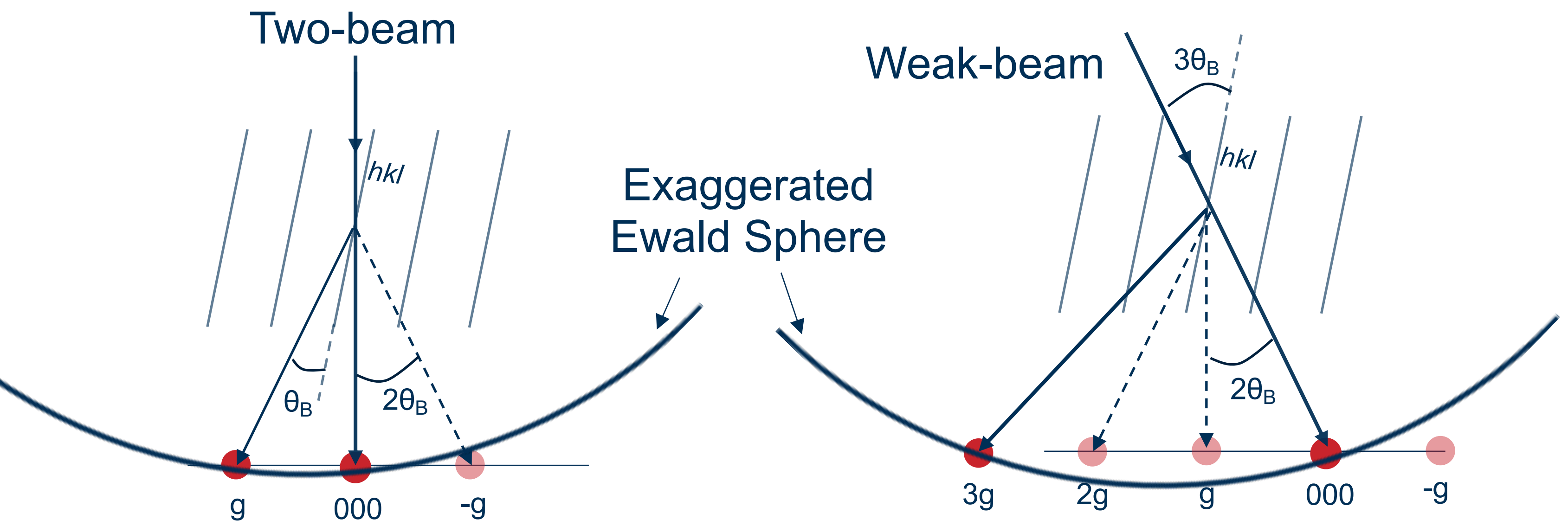


Weak-beam TEM

- Weak beam images are dark-field images, captured by selecting a weakly-excited diffraction spot
- The foil is tilted so that in regions absent of dislocations, the diffracting planes are far away from the Bragg condition. However, near a dislocation or loop, the local strain field is sufficiently high that it can rotate the planes back into the Bragg condition, scattering the electrons into the weak-beam.
- The result is an image with a narrow peak of high intensity relative to the background (but low absolute intensity)
- The larger the deviation parameter, the narrower the peak



Weak-beam TEM – what is s?



Ewald Sphere – a sphere in reciprocal space, of radius 1/λ. The reciprocal lattice points it passes through are those that meet the Bragg condition. Where it cuts a relrod, we see intensity from that reflection, despite the Bragg condition not being met

The deviation parameter s is the reciprocal-distance from the reflection of interest, **g**, to the Ewald sphere

1/s is the *Effective* Extinction length, ξ_{Eff}

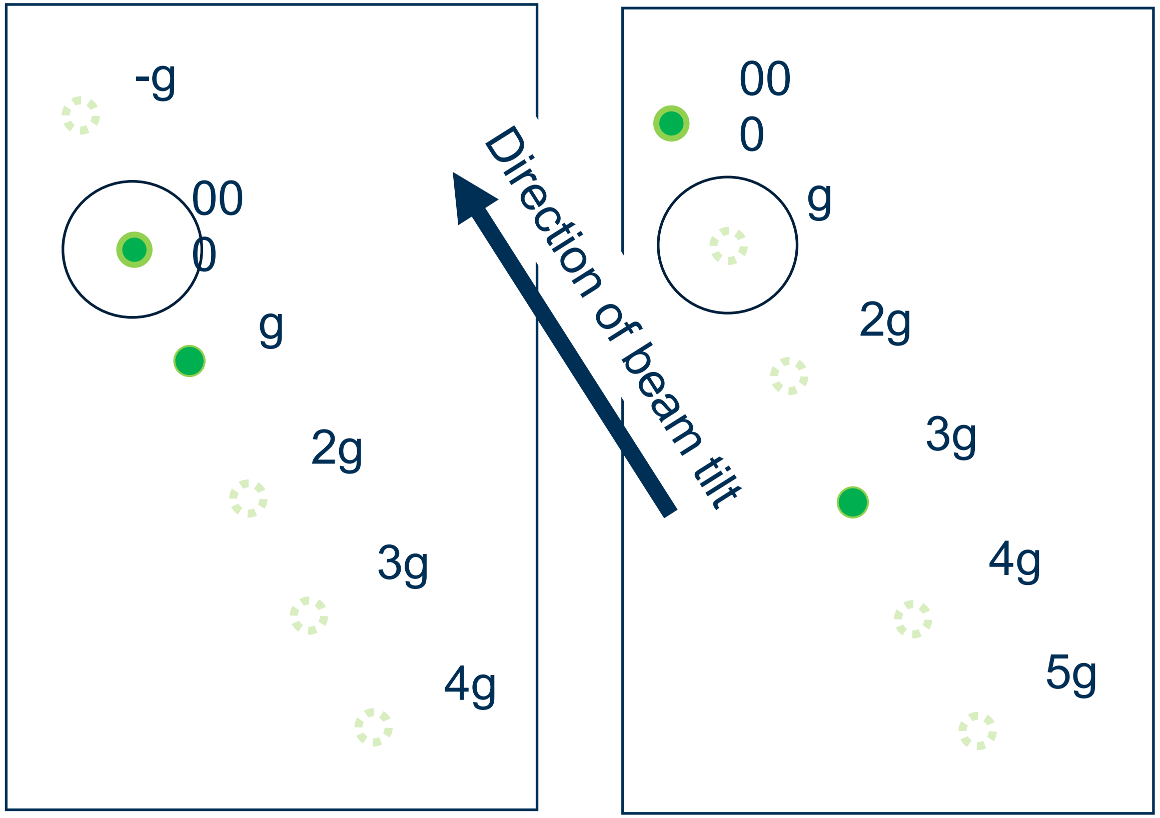
A weak-beam condition is usually notated as (g, ng)

e.g.
011 (g, 3g)
or 011 (g, 3-4g)
or 110 (g, 7.1g)
or 110 (2g, 5-6g)

Generally, avoid integer n, as there is a slight drop in contrast when n is integer

Weak beam dark field

Starting from a two beam diffraction pattern



Switch to dark field mode. **Beam is tilted** so that **+g** passes through the aperture

In this condition, **+g** will go from strong to weak as you beam tilt +g onto the optic axis

We have gone from a +g condition to a higher order ng. Here is shown a 3g condition where the third reflection is excited.

Weak beam dark field

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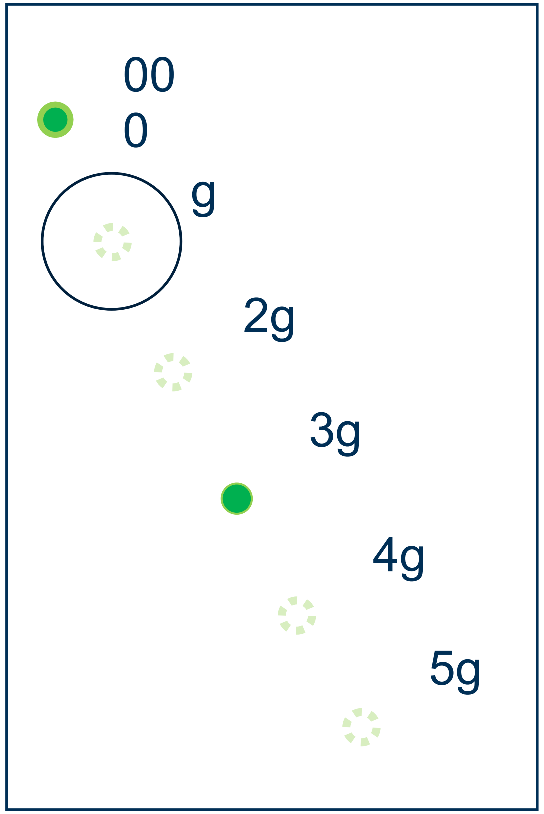

3g condition

Weak beam dark field

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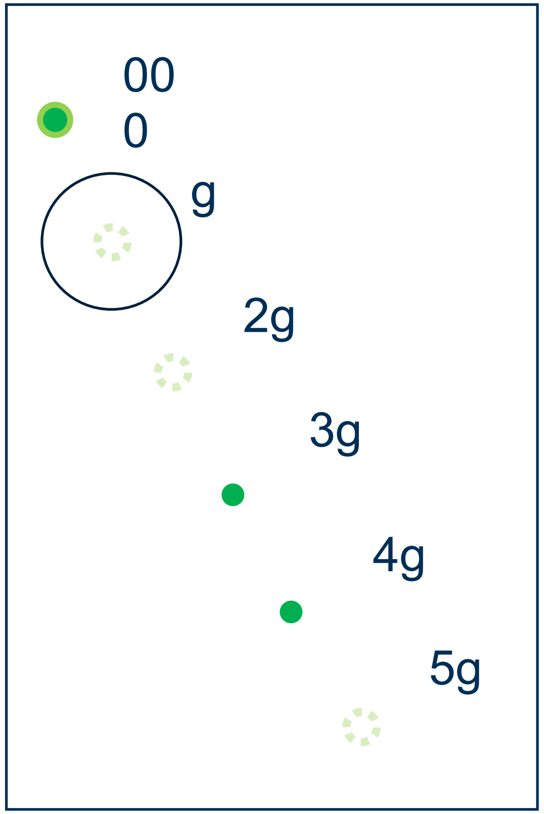
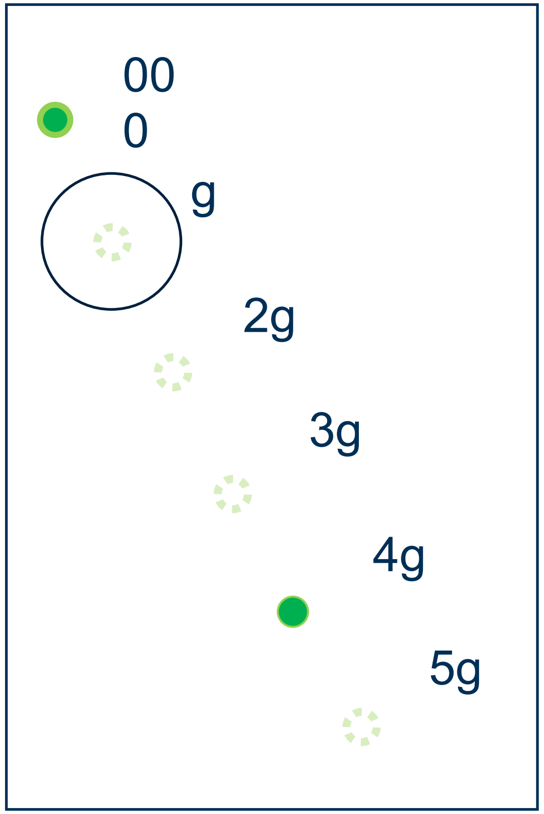


3-4g condition

Weak beam dark field

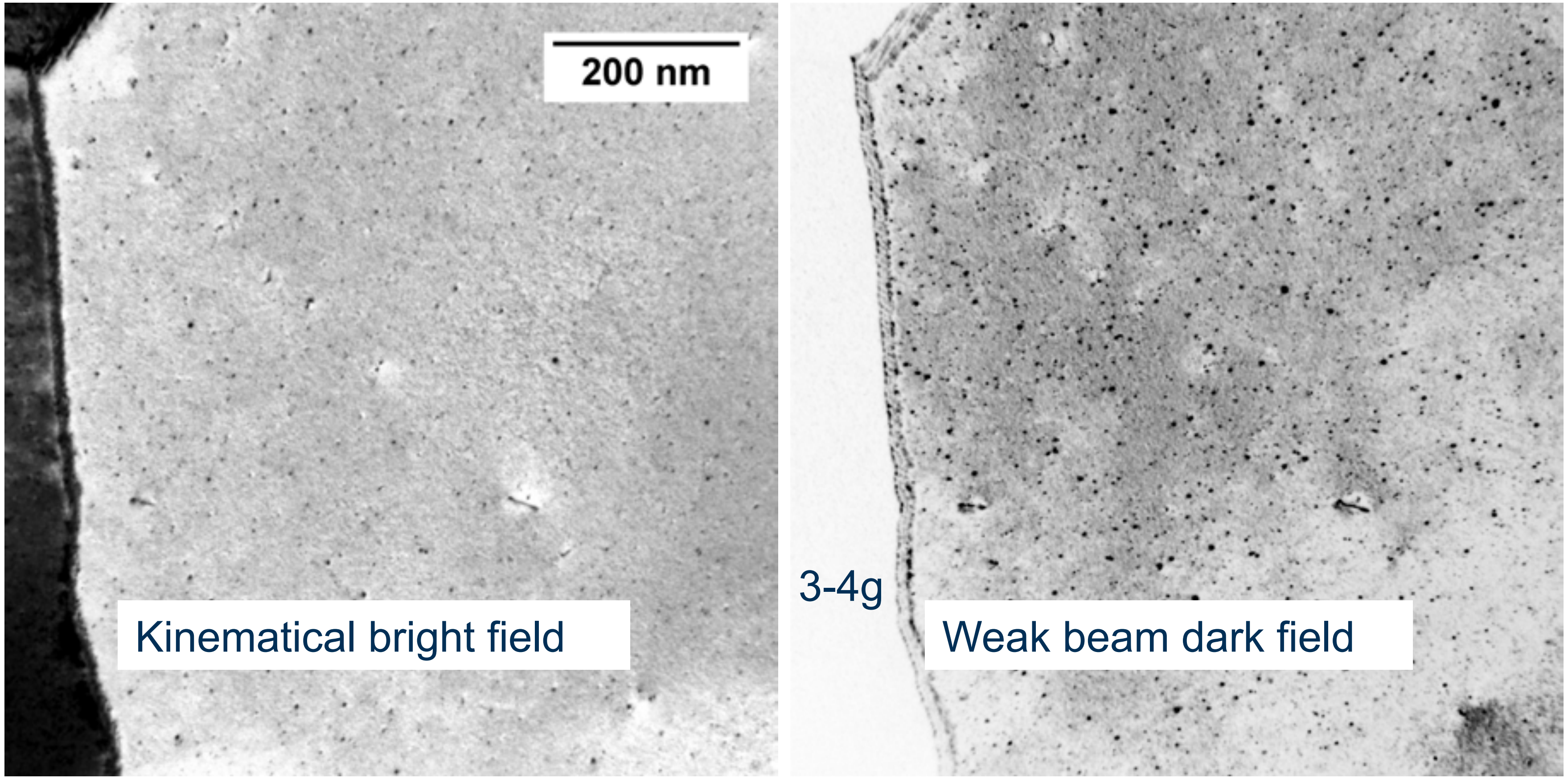


The position of the excited reflection isn't always clear, particularly for large n. You can keep track by noting that the Kikuchi lines pass half way between 000 and the excited reflection(s)

4g condition

Weak beam TEM

Example:



Note: Strictly speaking, a dark-field condition is only “weak beam” when s>0.2/nm

$$s = \frac{1}{2}(n-1)\frac{\lambda}{d^2}$$

@200kV, $\lambda = 2.5 \times 10^{-12}$m

Weak-beam dark-field is good for resolving finer detail in loops, and for counting loops when the population is small and relatively sparse – interpretation becomes very challenging if the density is high

Burgers vector analysis via invisibility criterion

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Dislocations have a displacement field $\underline{\mathbf{R}}$

$$\underline{\boldsymbol{R}} = \frac{1}{2\pi}\left(\underline{\boldsymbol{b}}\varphi + \frac{1}{4(1-\nu)}\left\{\underline{\boldsymbol{b}}_e + \underline{\boldsymbol{b}}\times\underline{\boldsymbol{u}}\,(2(1-2\nu)\ln r + \cos 2\varphi)\right\}\right)$$

Unit vector line direction

For a pure screw dislocation:

$$\underline{\boldsymbol{R}} = \underline{\boldsymbol{b}}\frac{\varphi}{2\pi} = \frac{\underline{\boldsymbol{b}}}{2\pi}\tan^{-1}\left(\frac{z - z_d}{x}\right)$$

And pure edge dislocation:

$$\underline{\boldsymbol{R}} = F(\underline{\boldsymbol{b}}) + G(\underline{\boldsymbol{b}}\times\underline{\boldsymbol{u}})$$

In the two-beam condition, with reflection $\underline{\boldsymbol{g}}$, the contrast is caused by $\underline{\boldsymbol{g}}.\underline{\boldsymbol{R}}$

Screw: contrast ~ $\underline{\boldsymbol{g}}.\underline{\boldsymbol{b}}$

Edge: contrast ~ $\underline{\boldsymbol{g}}.\underline{\boldsymbol{b}} + \underline{\boldsymbol{g}}.\underline{\boldsymbol{b}}\times\underline{\boldsymbol{u}}$

When $\underline{\boldsymbol{g}}.\underline{\boldsymbol{b}}$ = 0, there will be **no contrast** because the diffracting planes are parallel to $\underline{\boldsymbol{R}}$

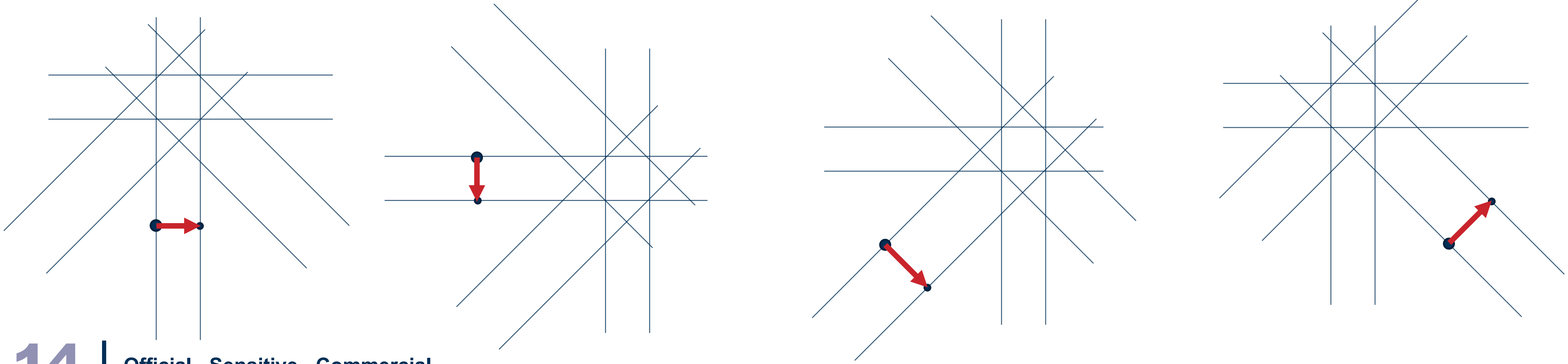
Burgers vector analysis via invisibility criterion

Example:

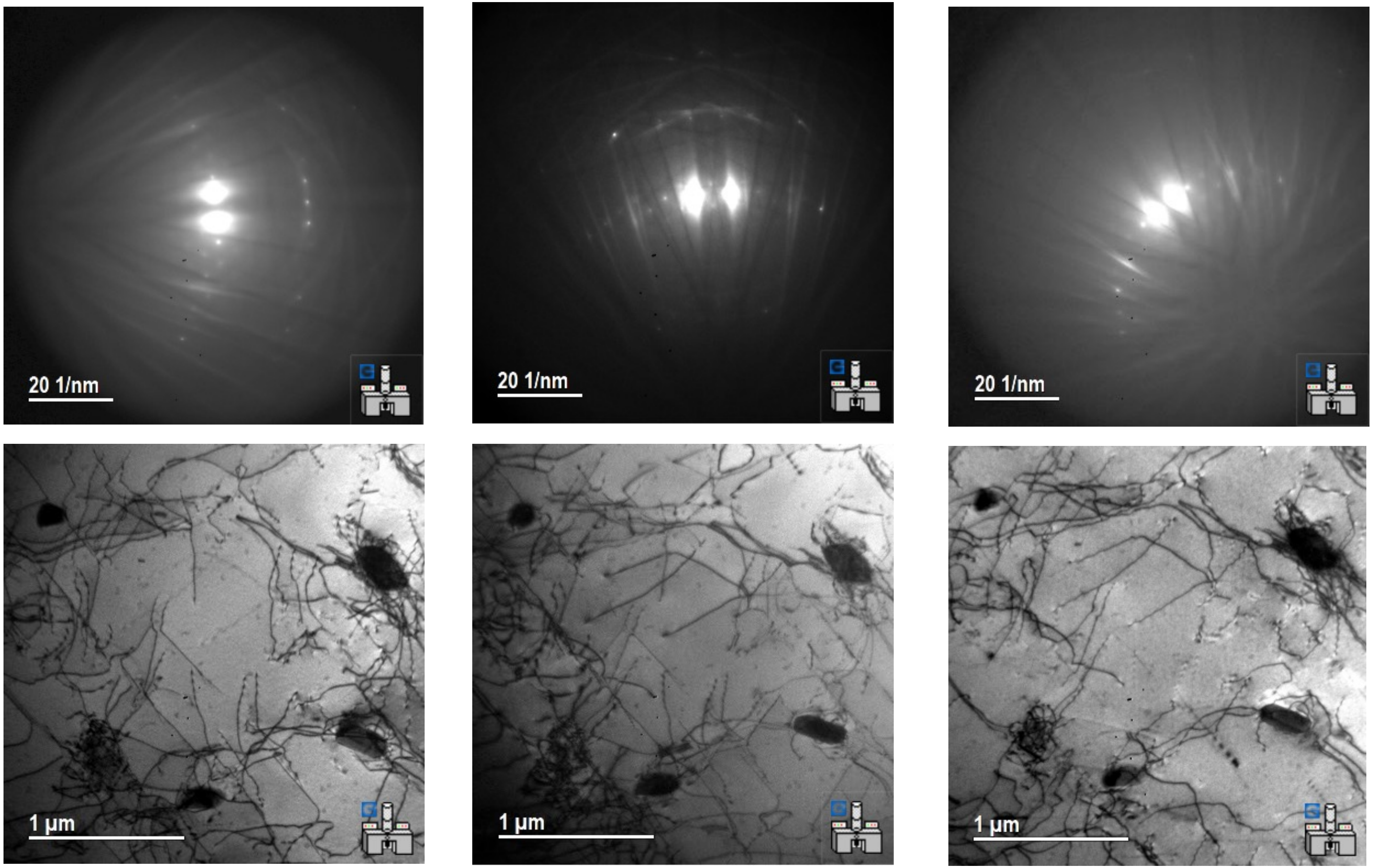
In BCC, there are two families of dislocation possible, $\underline{\boldsymbol{b}} = \frac{1}{2}\langle\mathbf{111}\rangle$ and $\underline{\boldsymbol{b}} = \langle\mathbf{100}\rangle$

Choose your reference crystallographic axes – e.g. what major zone axis is your foil oriented close to? Let's consider the $\langle\mathbf{001}\rangle$ zone axis. Let our upward-drawn direction be defined as $\underline{\boldsymbol{z}} = [\mathbf{001}]$

$\underline{\boldsymbol{g}} = \mathbf{200}$ $\underline{\boldsymbol{g}} = \mathbf{0\bar{2}0}$ $\underline{\boldsymbol{g}} = \mathbf{1\bar{1}0}$ $\underline{\boldsymbol{g}} = \mathbf{110}$



Two-beam diffraction conditions



Burgers vector analysis via invisibility criterion

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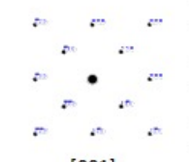
Example:

Construct a $\underline{g}.\underline{b}$ table:

Pole	Vector g			Burgers Vector b												
	h	k	l	1/2 [1 1 1]	1/2 [-1 1 1]	1/2 [1 -1 1]	[1 1 -1]	[1 1 0]	[1 -1 0]	[1 0 1]	[-1 0 1]	[0 1 1]	[0 -1 1]	[0 0 1]	[0 1 0]	[1 0 0]
	0	2	0	1.0	1.0	-1.0	1.0	1.0	-1.0	0.0	0.0	1.0	-1.0	0.0	2.0	0.0
	2	0	0	1.0	-1.0	1.0	1.0	1.0	1.0	1.0	-1.0	0.0	0.0	0.0	0.0	2.0
	1	1	0	1.0	0.0	0.0	1.0	1.0	0.0	0.5	-0.5	0.5	-0.5	0.0	1.0	1.0
	1	-1	0	0.0	-1.0	1.0	0.0	0.0	1.0	0.5	-0.5	-0.5	0.5	0.0	-1.0	1.0
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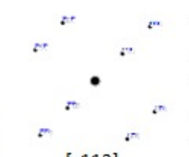	1	1	0	1.0	0.0	0.0	1.0	1.0	0.0	0.5	-0.5	0.5	-0.5	0.0	1.0	1.0
	2	-1	1	1.0	-1.0	2.0	0.0	0.5	1.5	1.5	-0.5	0.0	1.0	1.0	-1.0	2.0
	1	-2	1	0.0	-1.0	2.0	-1.0	-0.5	1.5	1.0	0.0	-0.5	1.5	1.0	-2.0	1.0
				-	-	-	-	-	-	-	-	-	-	-	-	-
				-	-	-	-	-	-	-	-	-	-	-	-	-
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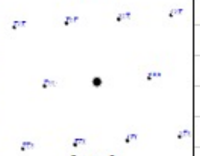	2	0	0	1.0	-1.0	1.0	1.0	1.0	1.0	1.0	-1.0	0.0	0.0	0.0	0.0	2.0
	1	2	-1	1.0	0.0	-1.0	2.0	1.5	-0.5	0.0	-1.0	0.5	-1.5	-1.0	2.0	1.0
	1	-2	1	0.0	-1.0	2.0	-1.0	-0.5	1.5	1.0	0.0	-0.5	1.5	1.0	-2.0	1.0
				-	-	-	-	-	-	-	-	-	-	-	-	-
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[012]				-	-	-	-	-	-	-	-	-	-	-	-	-

To know the Burgers vector of a particular dislocation within plus or minus $\underline{b}$, we need to capture a range of conditions to deduce the $\underline{b}$ without ambiguity.

Burgers vector analysis via invisibility criterion

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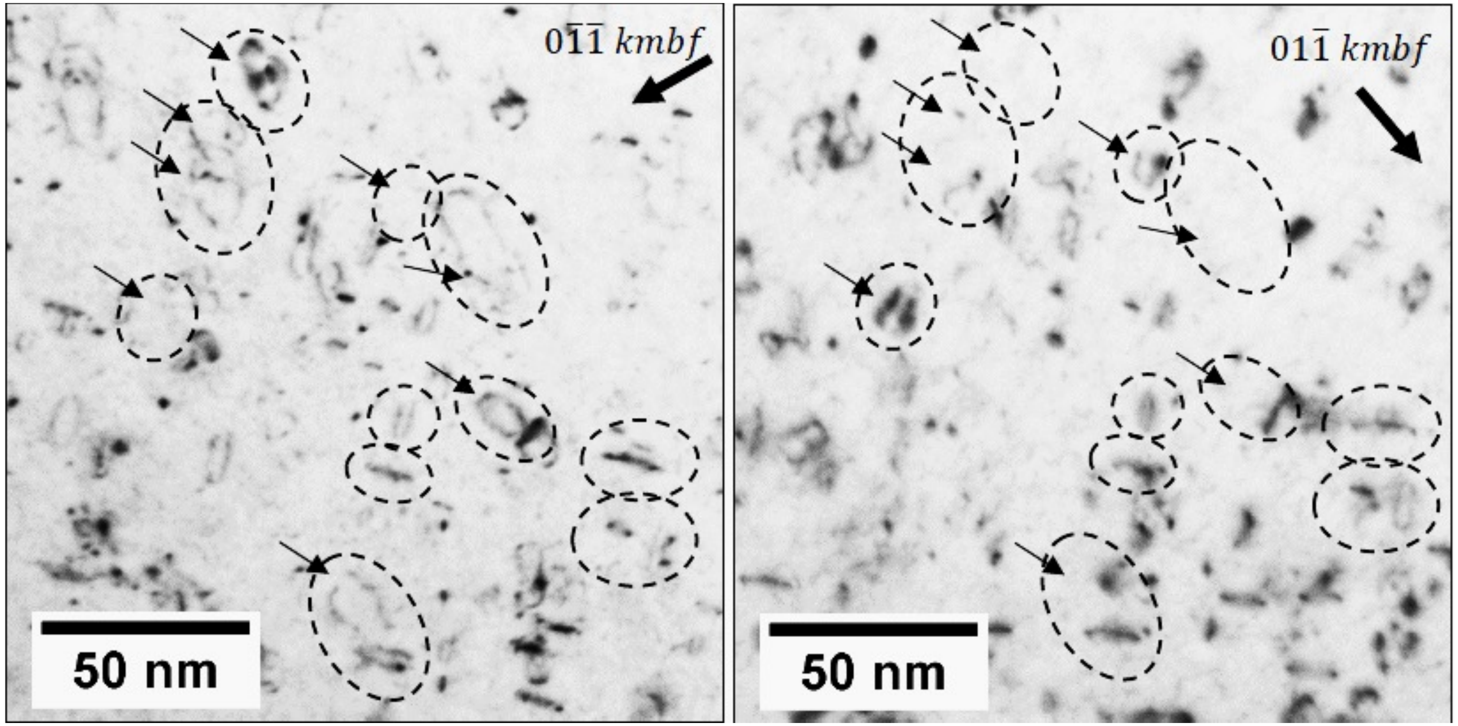
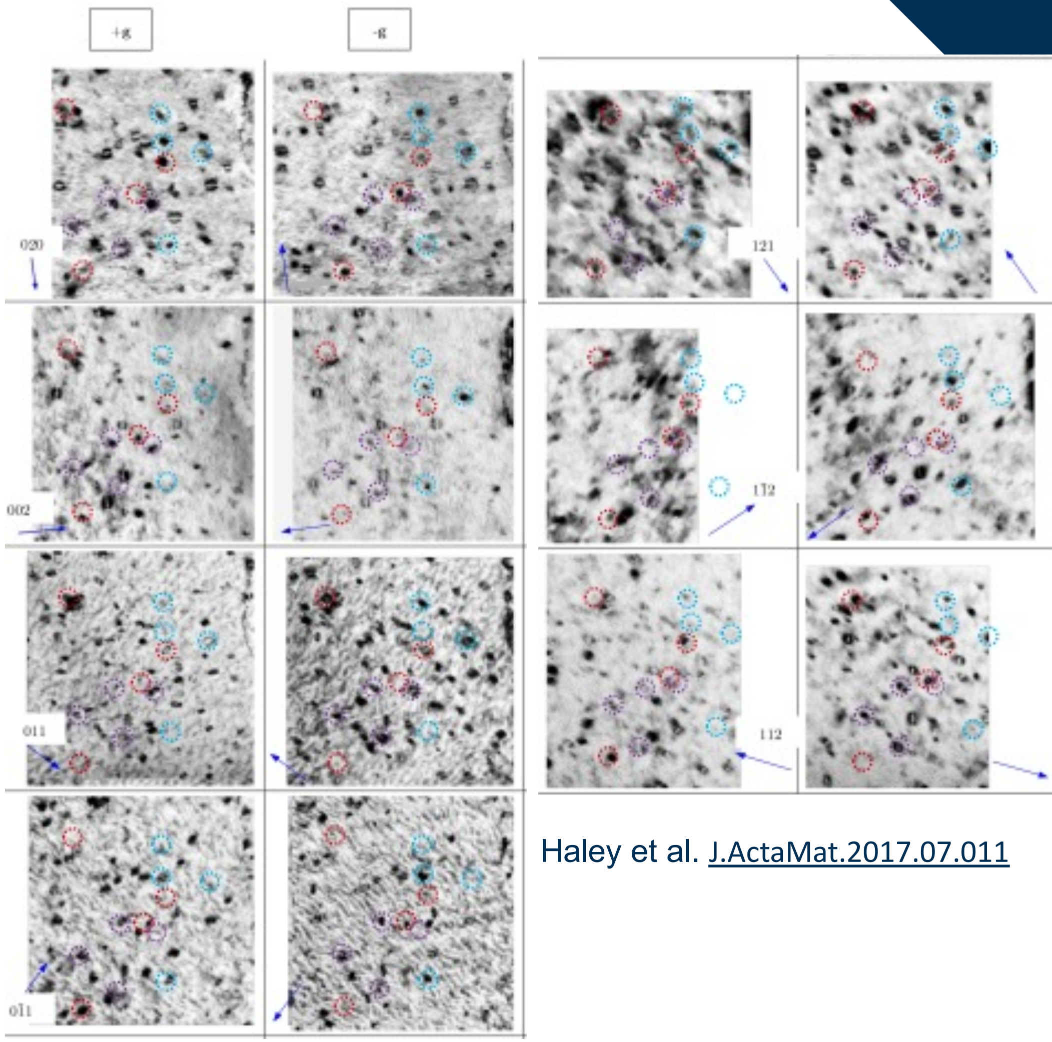
Example:

Simple analysis



Haley et al. J.NucMat.2020.152130

Full analysis



Haley et al. J.ActaMat.2017.07.011

Those present in both images are of type $\underline{b} = \langle 100 \rangle$, whereas those visible in only one are of type $\underline{b} = \langle 111 \rangle$

Caution: It is recommended to check both $+\underline{g}$ and $-\underline{g}$ conditions, as the contrast can be weak in one and strong in the other. A dislocation loop thus might be misinterpreted as $\underline{g}.\underline{b} = 0$

Burgers vector analysis via invisibility criterion – Tips and tricks

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- Always check both $+\underline{\boldsymbol{g}}$ and $-\underline{\boldsymbol{g}}$ conditions, as the contrast can be weak in one and strong in the other. A dislocation loop thus might be misinterpreted as $\underline{\boldsymbol{g}}.\underline{\boldsymbol{b}} = 0$
- - artefacts, like surface oxide, or FIB damage, might also be weaker in one condition over another
- Capture everything! Try varying the deviation parameter and see how that affects the contrast – combining many images with varying deviation parameter can suppress background artefacts and improve visibility of loops
- For EVERY condition, capture the diffraction pattern once you have finished imaging in that condition.
- Capture multiple magnifications of the region you are looking. A low-mag image is particularly important to help you identify the same region from a different imaging condition – microstructures can look very different using a different g!
- Don't try and do too much analysis on the microscope – know what you want to capture, and prioritise getting the images. These experiments take a long time, you can worry about analysing them later.
- Use crystallographic software that can generate diffraction patterns and stereograms to help you navigate your sample, and ensure you indexing is consistent. A Kikuchi map is incredibly useful too for deducing how your sample is oriented (PTCLab is open source and produces nice maps)
- When doing the analysis on dense structures, a big challenge is knowing if you are looking at the same defect in two different conditions, particularly if a lot of tilting is involved. It can be useful in this case to capture a tomographic series, by tilting along a Kikuchi band and capturing images of the microstructure at regular intervals with the same g two-beam condition. This allows you to see where the projection of a loop moves to. Sometimes, though, it's just not possible to say with confidence, yes this is the same defect.

Quantifying defects

ImageJ tips: Use “elliptical” selection, and press “t” to add to ROI-Manager.
Tools like thresholding, or Weka Segmentation are useful, but are not reliable

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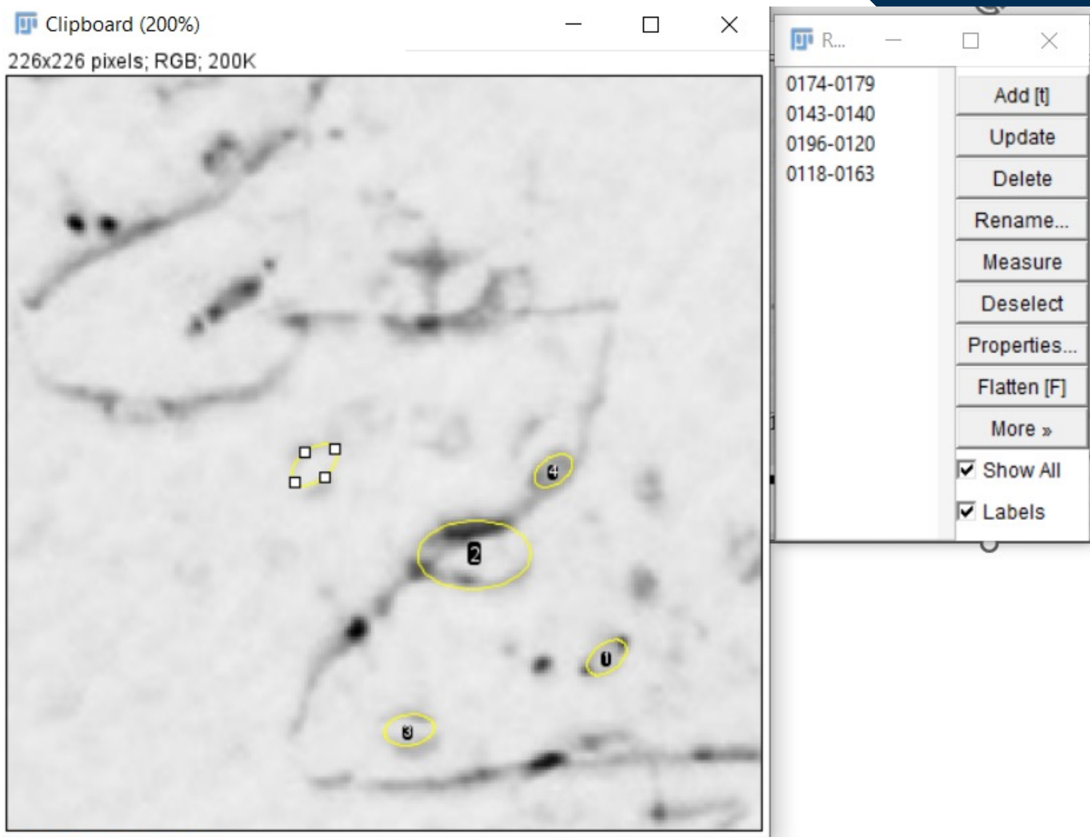
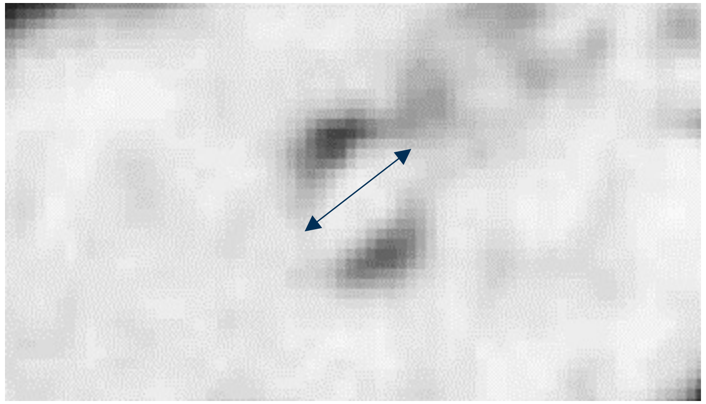
Due to $\underline{\boldsymbol{g}}.\underline{\boldsymbol{b}} = 0$, if you count dislocation loops in a given micrograph, you will be missing a portion of the total dislocation loop population.



To obtain a true dislocation density:

- Count and classify every dislocation loop, by tracking its visibility/invisibility in multiple conditions: e.g. by comparing 01-1 and 011
- Choose a condition where only one family of dislocation loops are visible: e.g. in hexagonal materials, only <a>-type loops are visible when g=01-10
- Image multiple conditions containing different proportions of the different loop types, and solve via least squares



The major axis of a loop is between the coffee-beam lobes, and is an ok measure of the size. Compare ±g for precise sizing

Quantifying defects – statistical analysis

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For example: In BCC, we know we might have <111> and/or <100> loops

G=011, we count 56 dislocation loops:

- g.b tells us we expect 2/3 of the <100> loops and 2/4 of the <111> loops to be visible

G=002 we count 76 dislocation loops:

- g.b tells us we expect 1/3 of the <100> loops and all of the <111> loops visible

G=112 contain 78 loops

- g.b tells us we expect all of the <100> loops and 3/4 of the <111> loops visible

We can re-write this using a series of equations:

$$N_g = f_1 N_{b1} + f_2 N_{b2}$$

$$N_{011} = \frac{2}{3} N_{\langle 100\rangle} + \frac{2}{4} N_{\langle 111\rangle} = 56$$

$$N_{002} = \frac{1}{3} N_{\langle 100\rangle} + \frac{4}{4} N_{\langle 111\rangle} = 76$$

$$N_{112} = \frac{3}{3} N_{\langle 100\rangle} + \frac{3}{4} N_{\langle 111\rangle} = 78$$

$$\boldsymbol{N_g} = \boldsymbol{M} \cdot \boldsymbol{N_b} \Rightarrow \boldsymbol{M^{-1}} \cdot \boldsymbol{N_g} = \boldsymbol{N_b}$$

$$\begin{pmatrix} N_{011} \\ N_{002} \\ N_{112} \end{pmatrix} = \begin{pmatrix} 2/3 & 2/4 \\ 1/3 & 4/4 \\ 3/3 & 3/4 \end{pmatrix} \cdot \begin{pmatrix} N_{\langle 100\rangle} \\ N_{\langle 111\rangle} \end{pmatrix}$$

In Matlab: syntax $\boldsymbol{M} \backslash \boldsymbol{N_g} = \boldsymbol{N_b}$

In Python: syntax $scipy.linalg.lstsq(M, N_g)$

Practical counting tips

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There is no standardised method to count dislocation loops

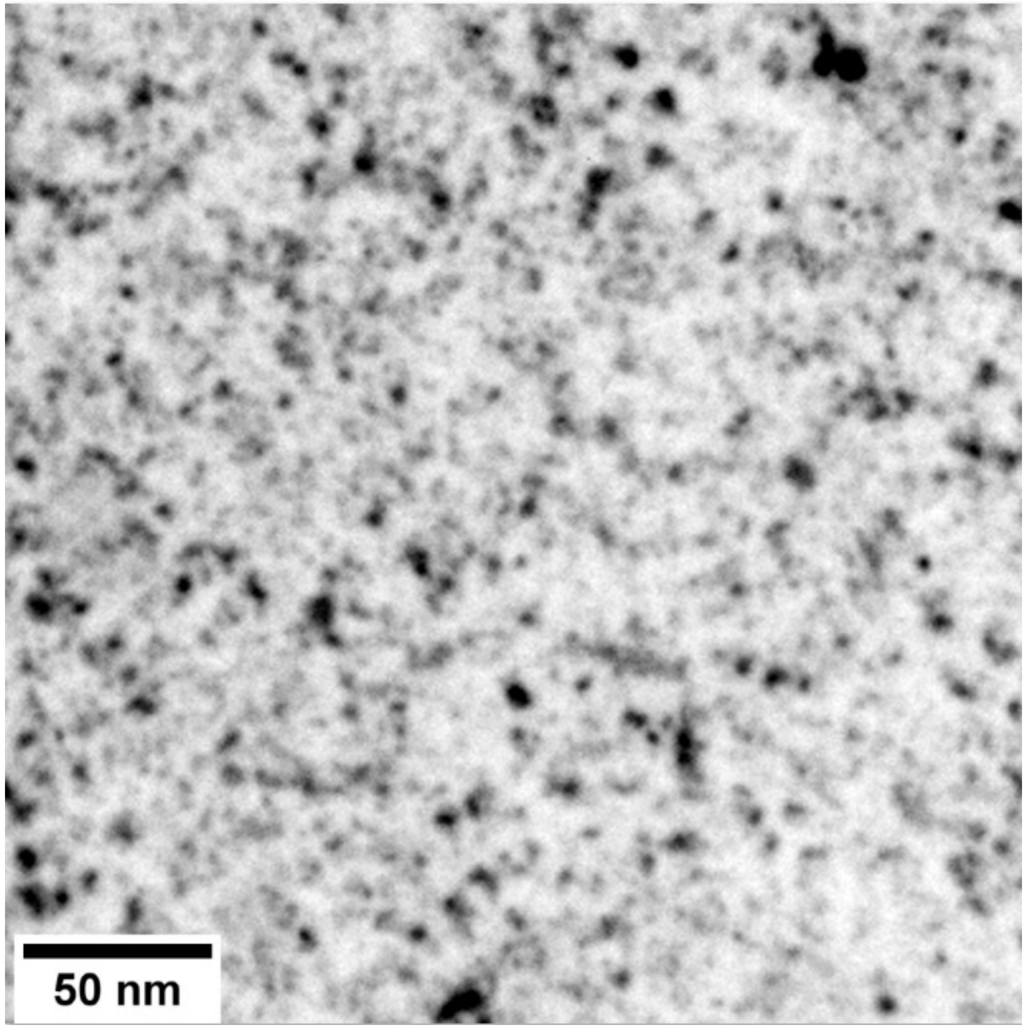
As we've noted, loop visibility is highly variable. Particularly for dense microstructures, the displacement field can produce rogue contrast, or a large defect can appear broken and easily mistaken for several. For this reason, for high dislocation densities, the uncertainty can be very large

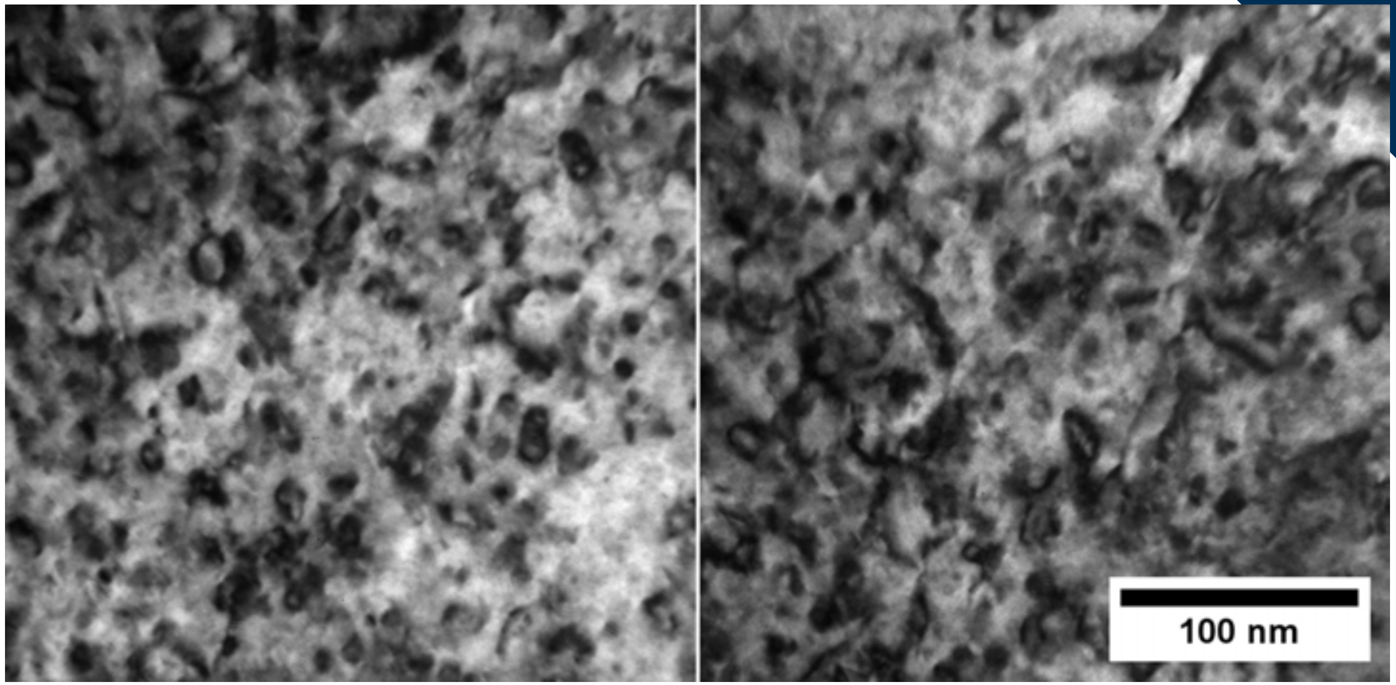
Counting small defects is highly uncertain, as the defects approach the resolution limit, or have a similar appearance to the background fluctuations (e.g. FIB damage, surface artefacts etc.). Good practice is to define a lower-bound size, above which you have confidence in the count.

Be honest – what is your uncertainty?

- Thickness could be 10-20%
- Count multiple regions – do you get the same density?
- Ask a colleague to count the same region – do you get the same?
- What makes a defect difficult? Contrast? Vicinity to others? Past examples from literature of classifying "definitely" loops and "maybe" loops, with a density of 100% "definitely" loops + 50% "maybe" loops

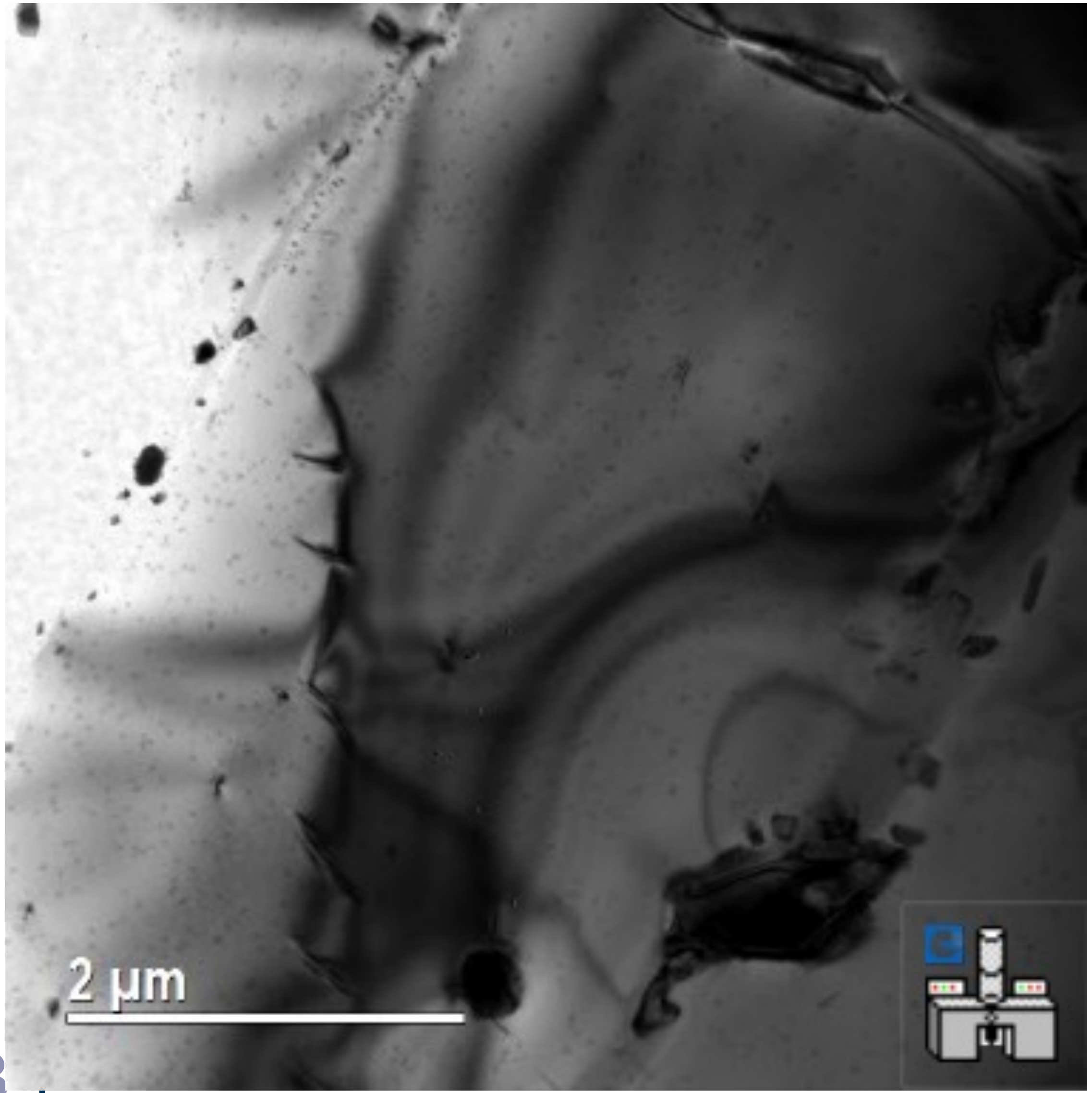
Examples of difficult micrographs



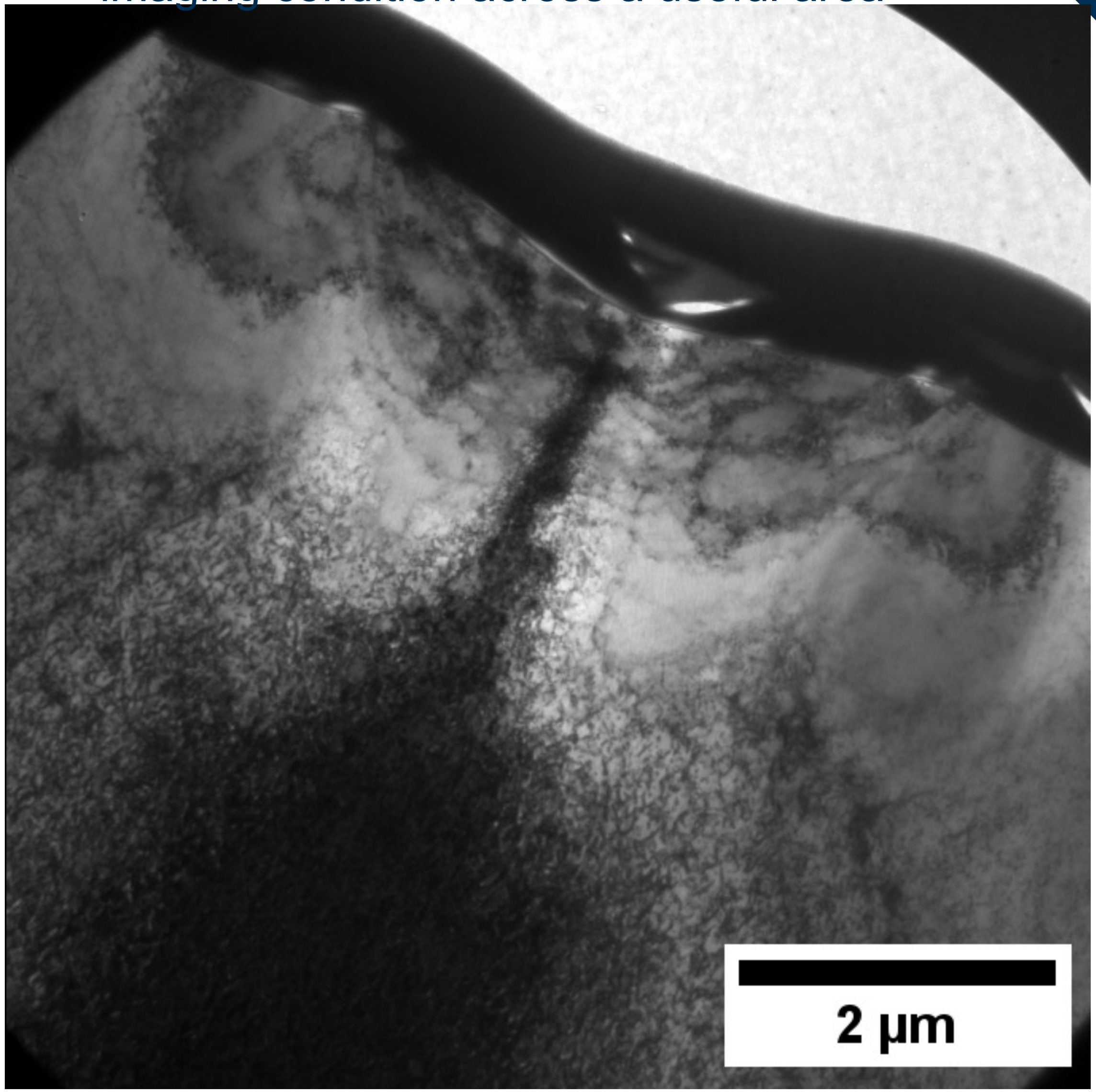


Artefacts in the TEM

Bend contours at a thin section of an electropolished foil



Bending beneath an indent in copper makes it impossible to achieve a consistent imaging condition across a useful area



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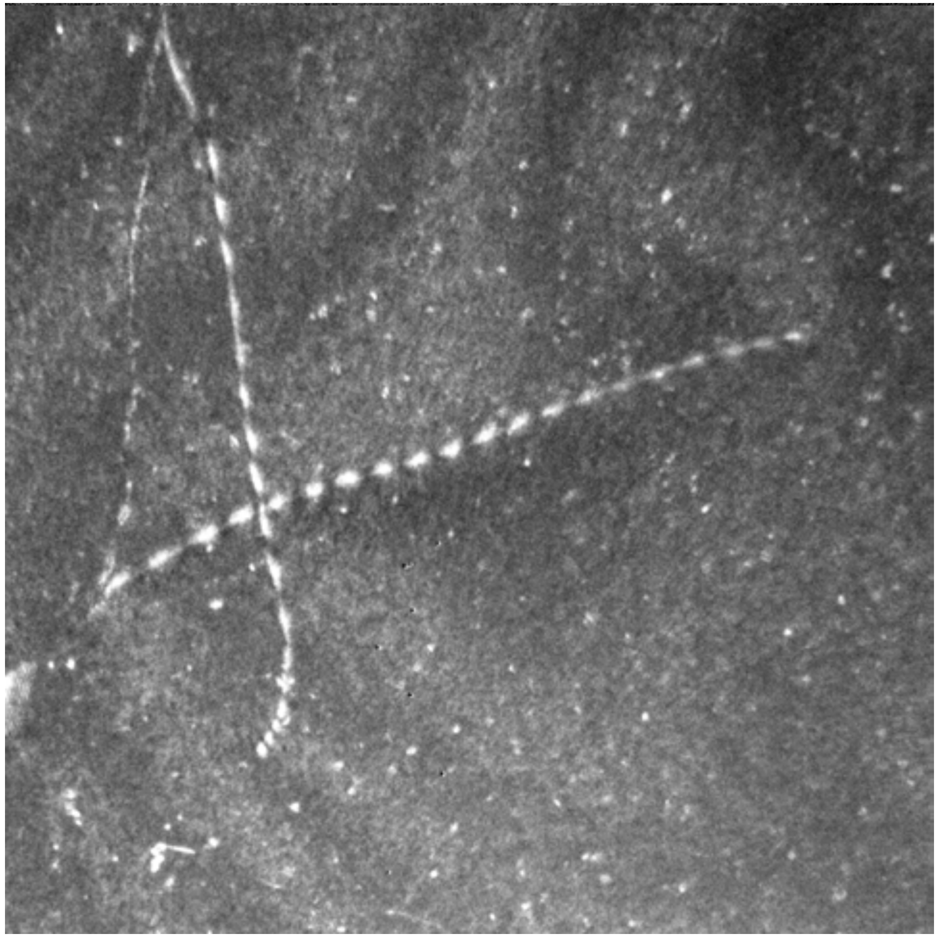
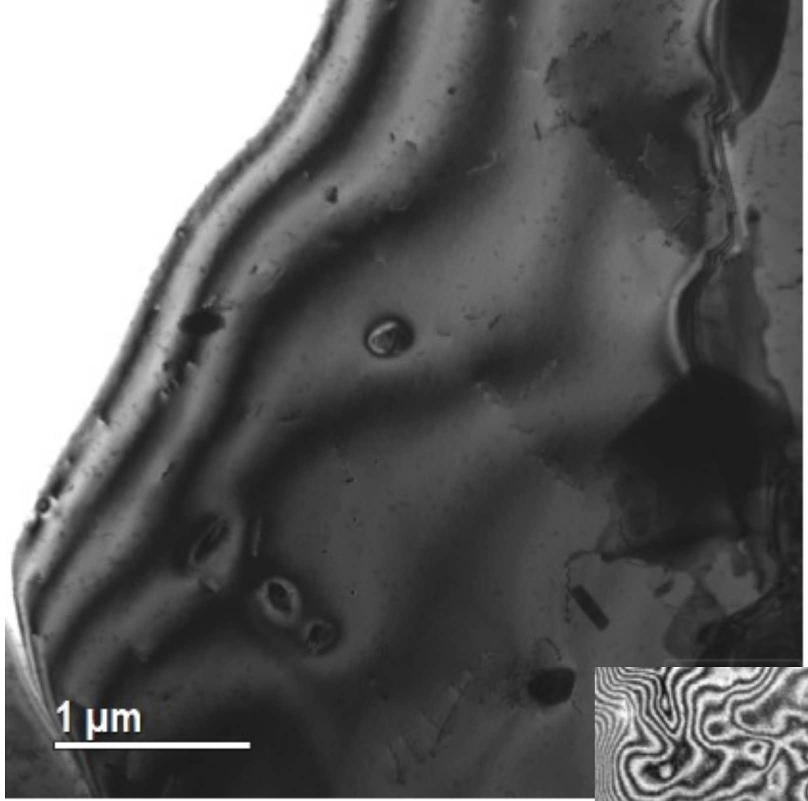
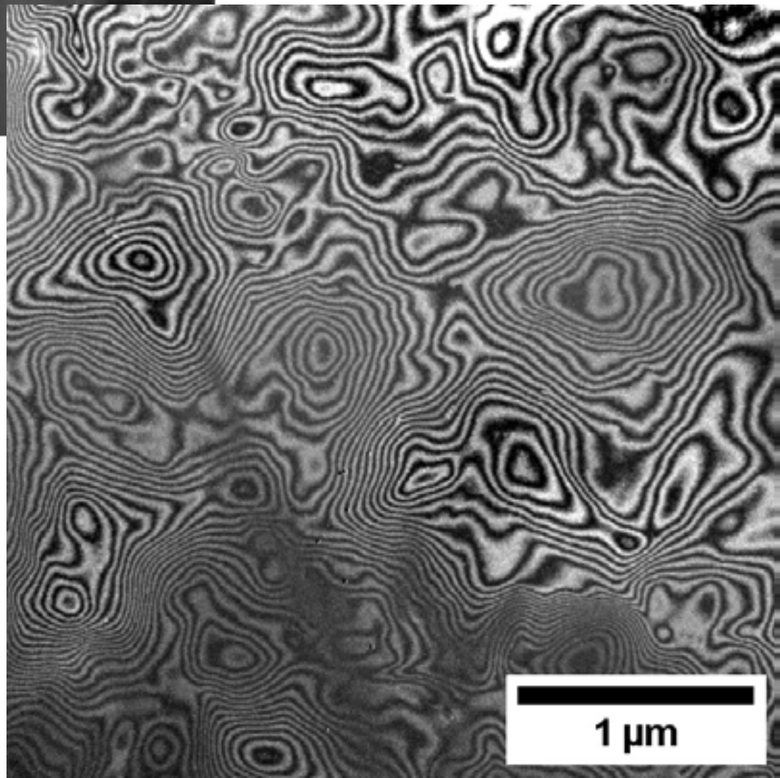
Artefacts in the TEM

Extinction effects!



(side note, no radiation damage in
this electropolished foil!)





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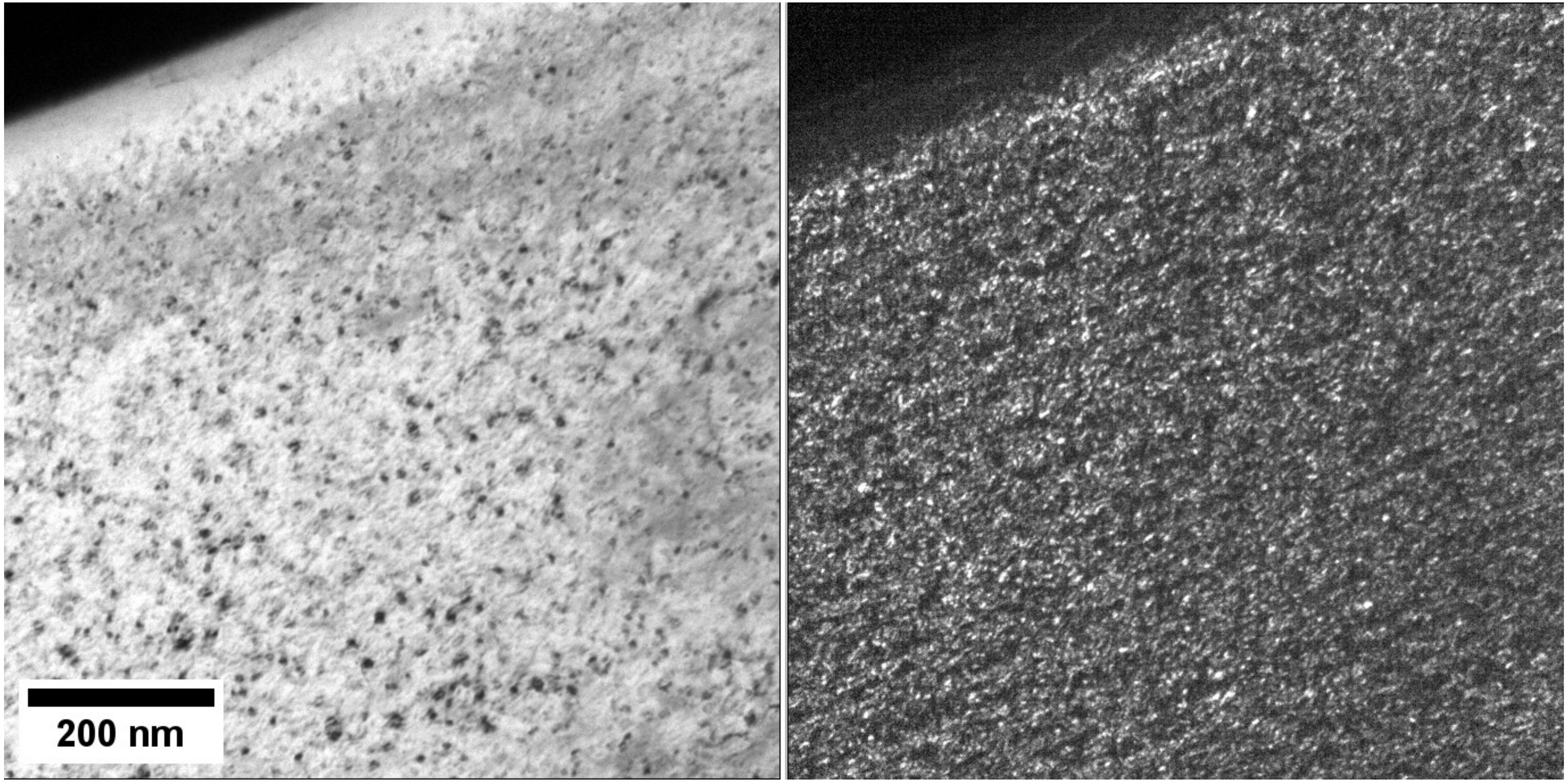
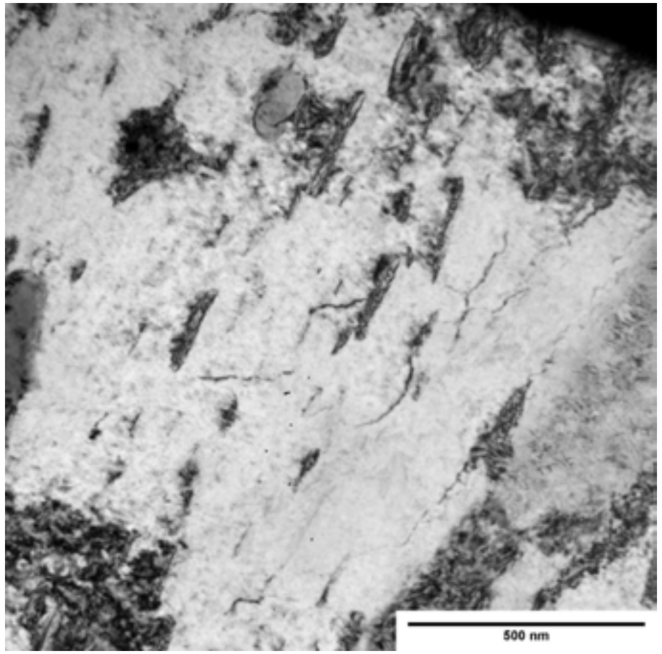
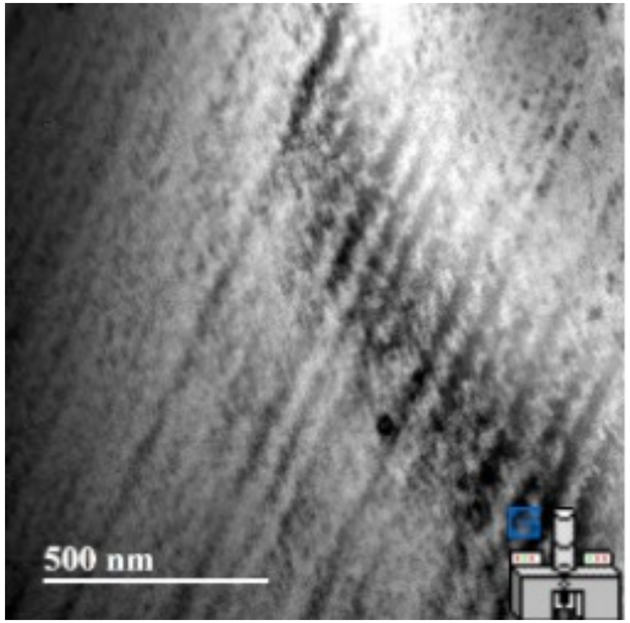
Artefacts in the TEM



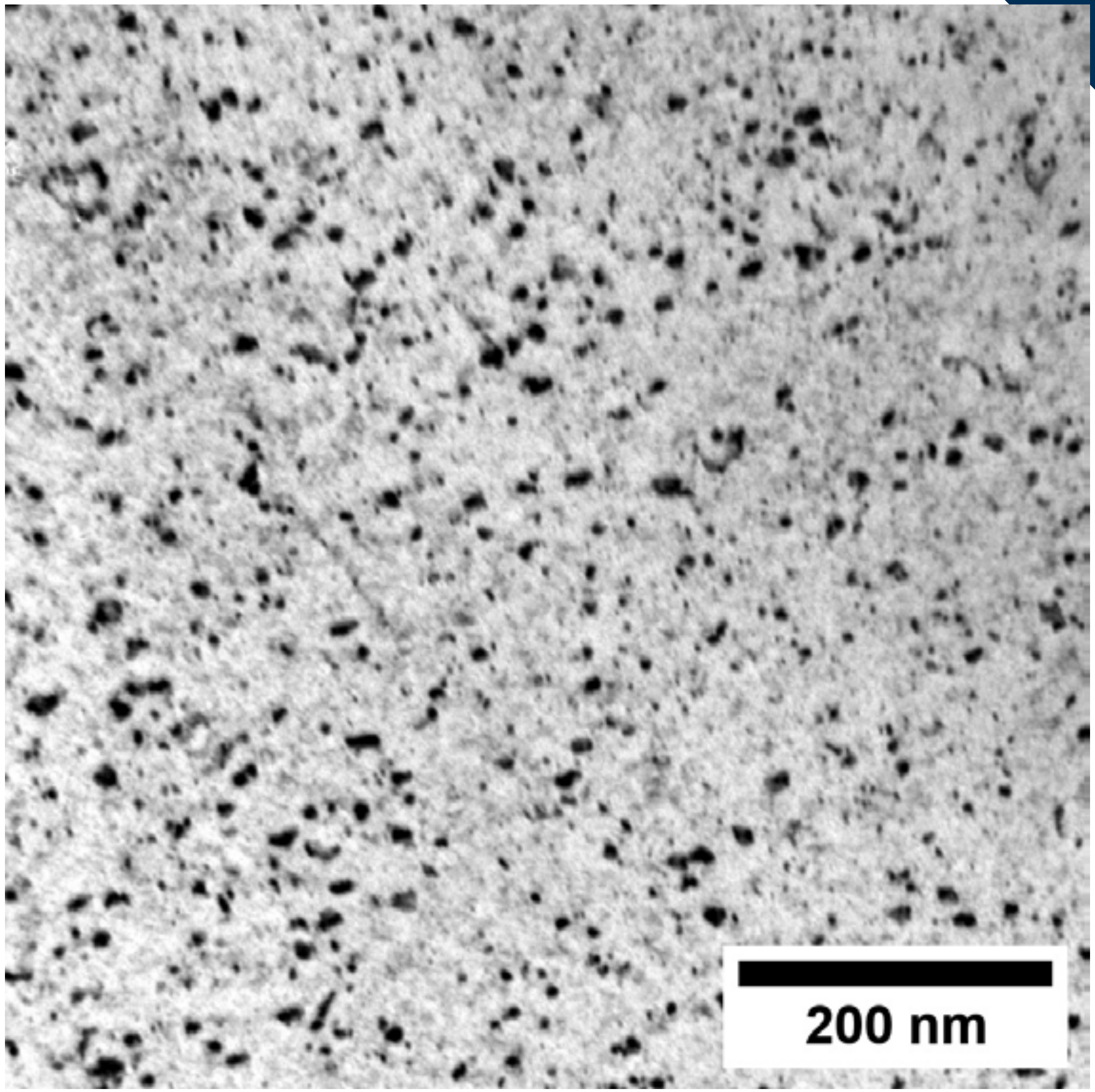
If the dark-field doesn't look remotely like the bright-field, then this is a strong indication another phase is dominating the contrast

Artefacts in the TEM





Milling for too long at 30kV can produce very nice dislocation loops!



If you observe any kind of streaking, or defect that appears aligned with your FIB'ing direction, be very cautious that it could be FIB induced.

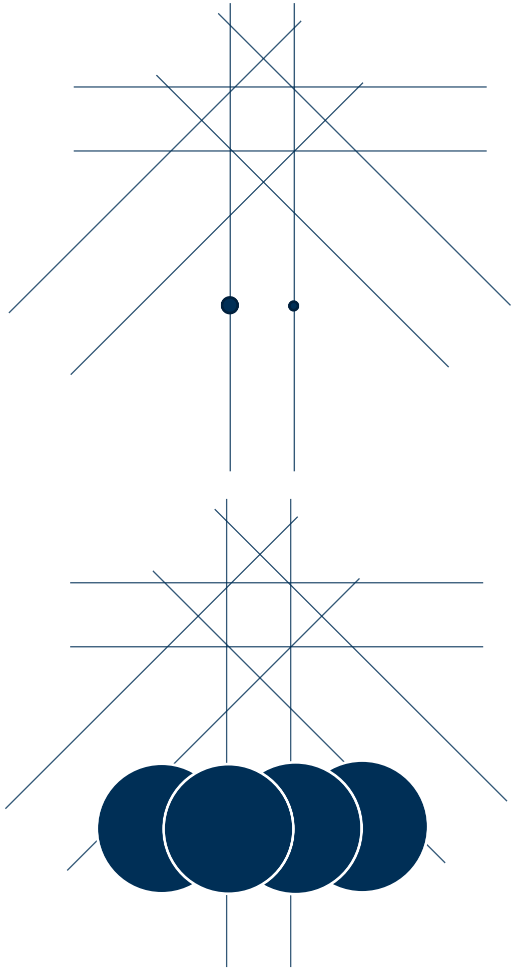
Always examine the unirradiated microstructure carefully, to make sure you have confidence in your FIB prep

Remember – FIB can produce loops! Or nucleate new phases – like hydride

STEM imaging of defects

- In STEM, we typically use a **high convergence angle**
- This is akin to imaging using a range of tilt angles in one go
- This effectively **"smooths" out** artefacts like **bend contours, thickness fringes** etc.
- **Contrast** of dislocations against the background is **vastly improved!**
- **Sacrifices clarity** of small defects, but can give **greater confidence** to a complex microstructure
- **Slower** to capture an image of same pixel resolution
- Not great for in-situ
- A bit more faff to set up an imaging condition
- **Combines well with analytical STEM**, so defects can be easily correlated EDX or EELS measurements of the chemistry

$\underline{g} = 200$



TEM

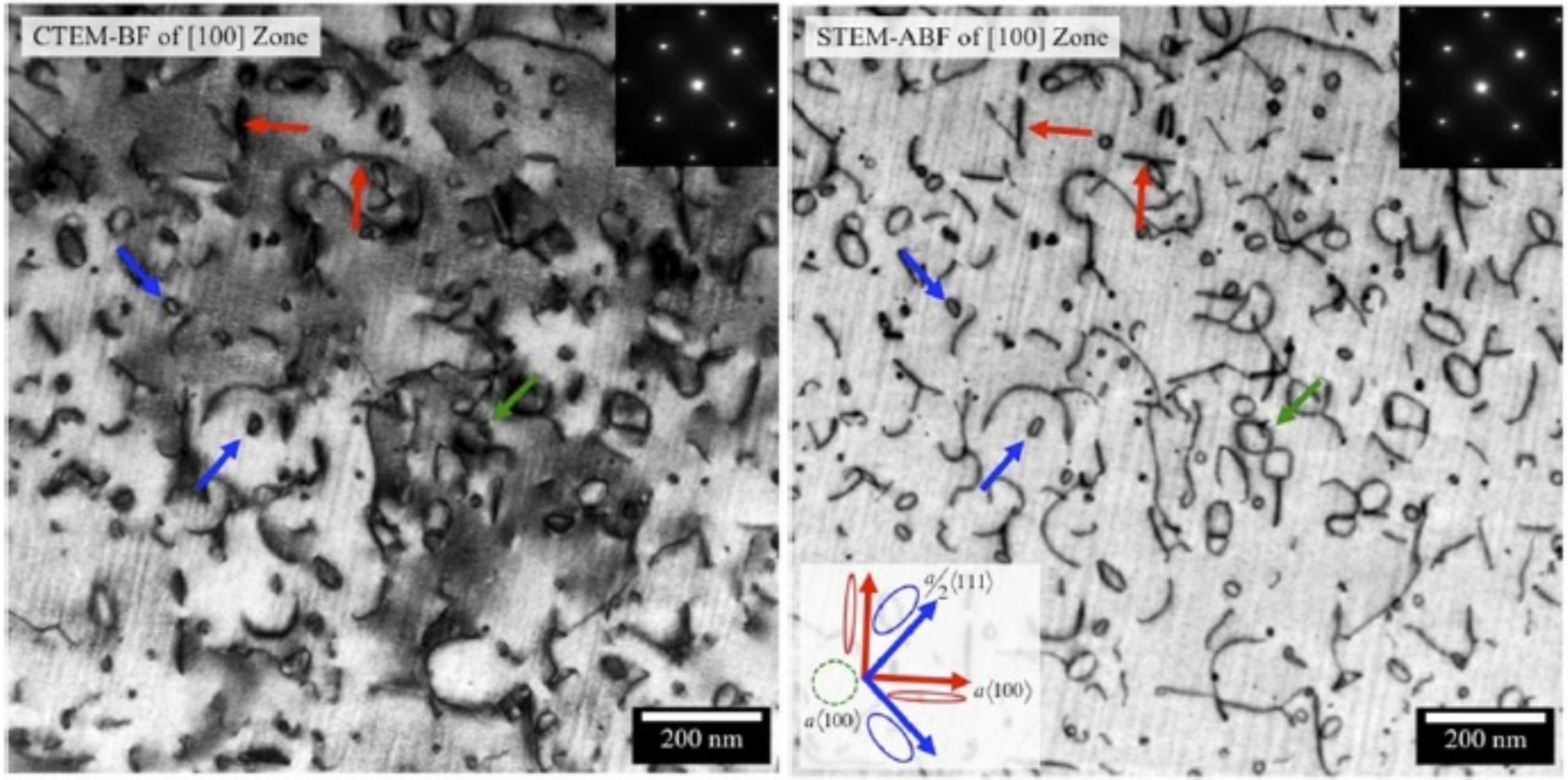
- Parallel beam so diffraction spot is sharp
- Small portion of reciprocal space is sampled
- Little interference from other reflections

STEM

- Converged beam so diffraction spot is fat
- Large portion of reciprocal space is sampled
- Strong interference from other reflections

STEM imaging of defects

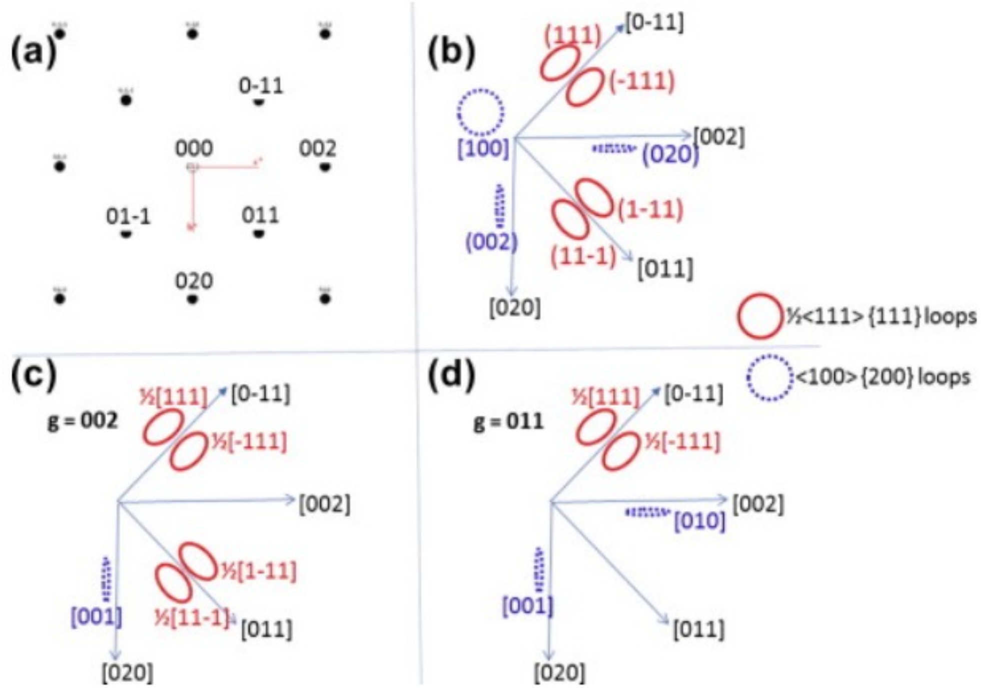
C.M. Parish et al.: Application of STEM characterization for investigating radiation effects in BCC Fe-based alloys



Burgers vector analysis via projection

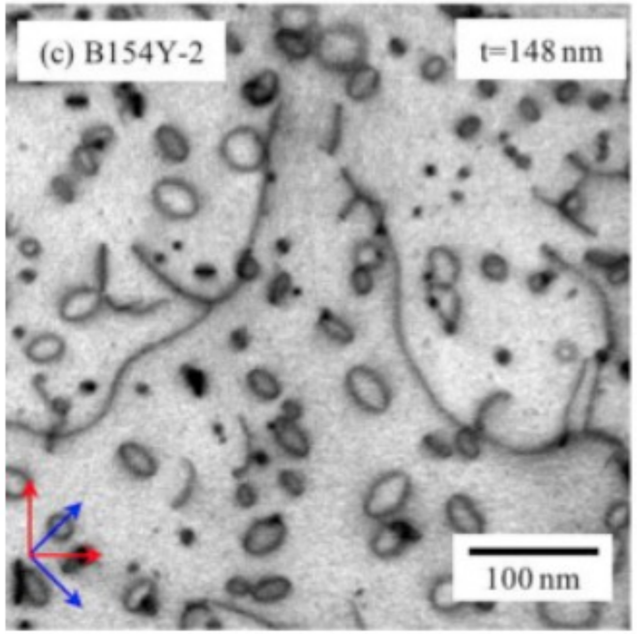
If the morphology of your defects is simple (i.e, and the defects are well defined, then we can deduce the Burgers vector just from how it is projected in the foil

For example:
In BCC, when oriented near an [001] zone axis, we know that loops on {200} planes will appear edge-on (with the **b**=[001] loops having **g**.**b**=0), whereas 111-loops will appear elliptical



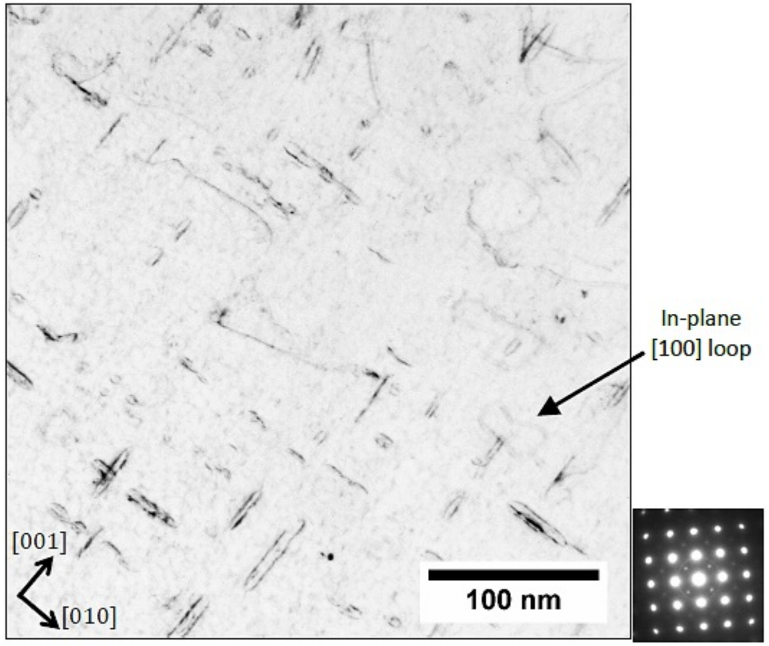
Yao, Edwards and Kurtz, J.NucMat.2012.12.002

111-type loops in FeCrAl



Field et al J.NucMat.2017.07.061

100-type loops in Fe6Cr



Haley 2018 DPhil thesis (composite image from orthogonal g conditions)

Burgers vector analysis via projection

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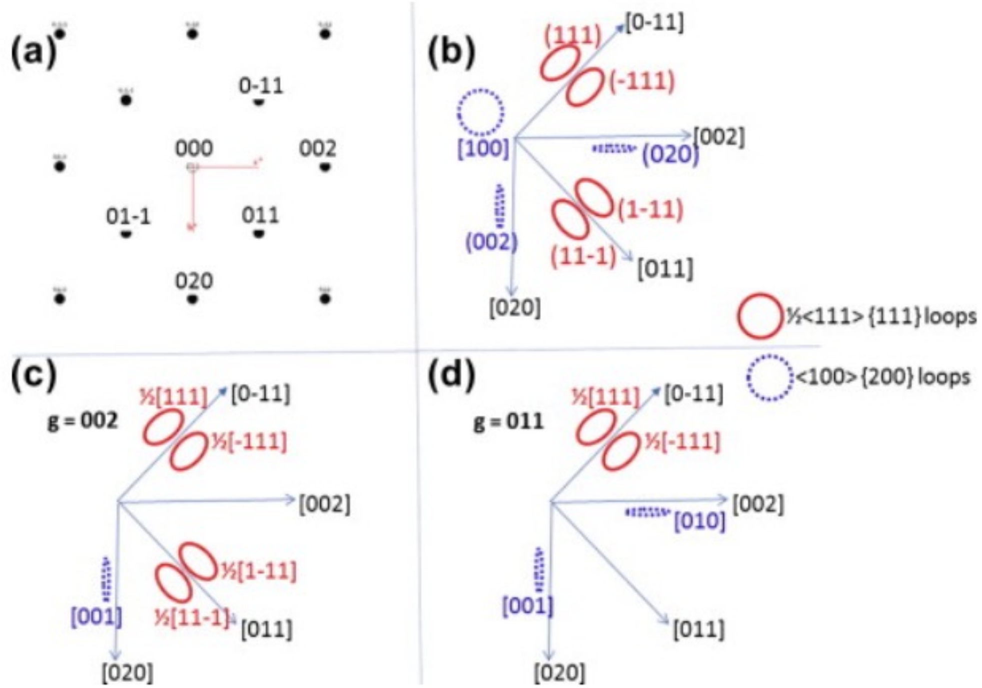
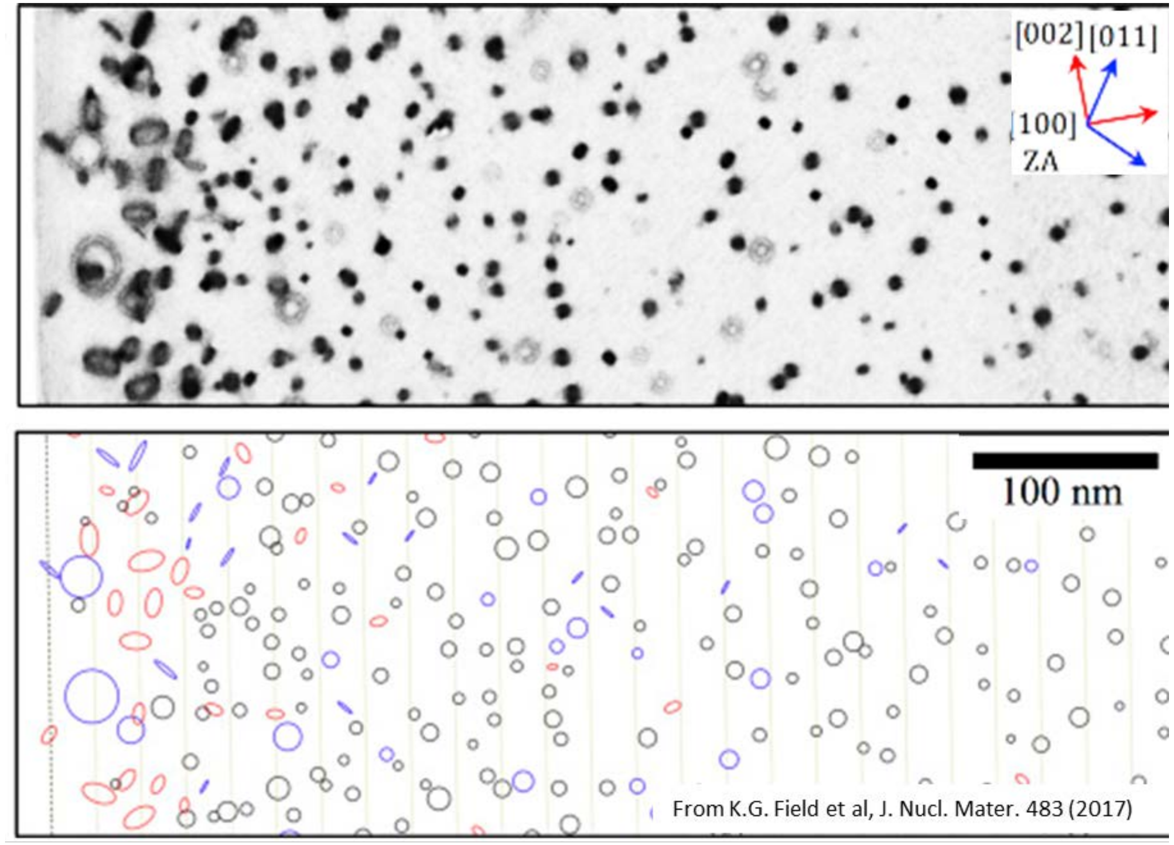


Yao, Edwards and Kurtz, J.NucMat.2012.12.002

For large defects in simple microstructures, counting defects becomes trivial



From K.G. Field et al, J. Nucl. Mater. 483 (2017)

4D STEM – what is it?

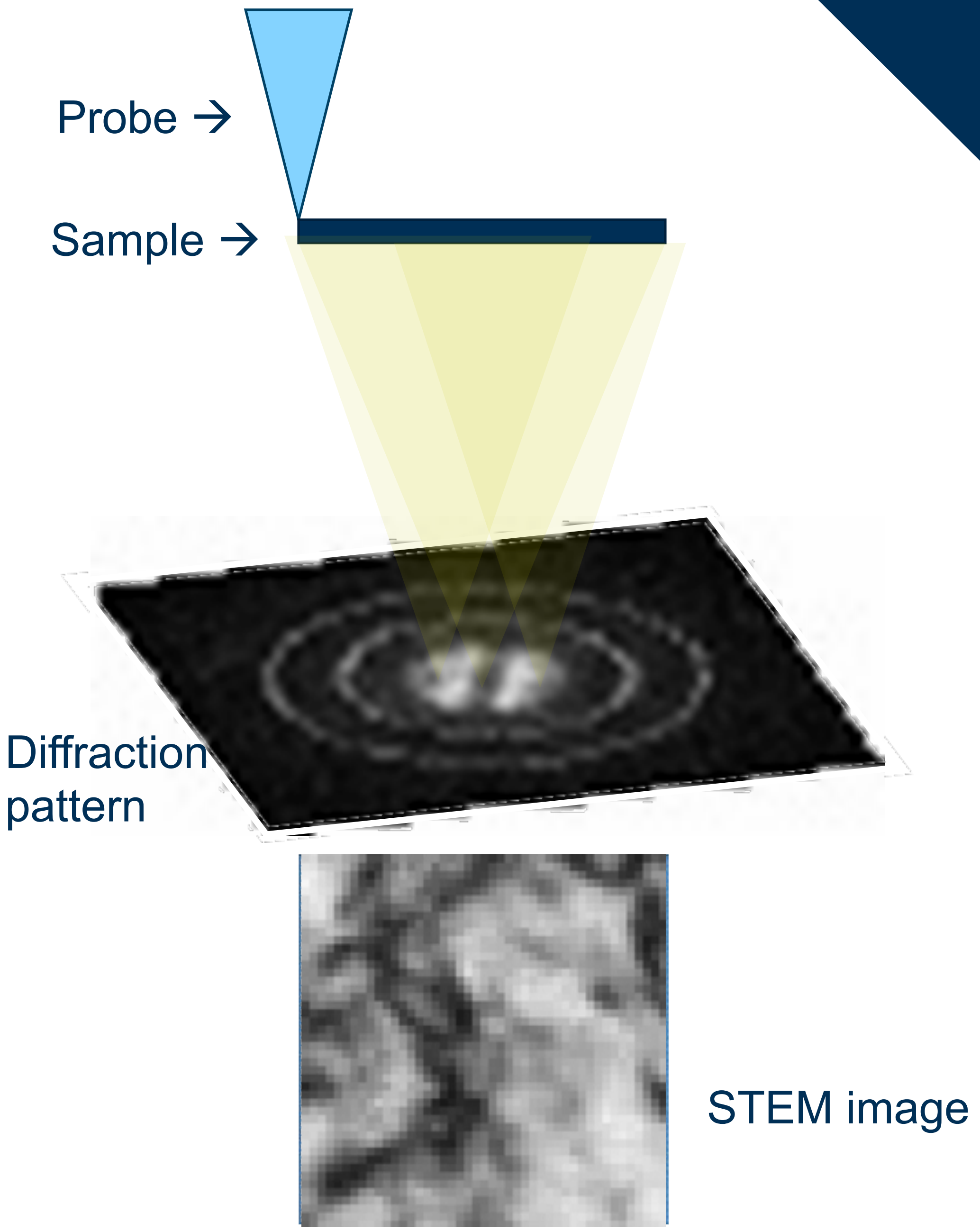
Capture of 2D information at every pixel of a 2D STEM scan > 2x2=4D

At each pixel, we record a diffraction pattern

Allows us to record:

- Rather than an integrated intensity – like a conventional STEM detector, we capture a 2D image of the exit wave
- Contains diffraction information, phase information, and Z contrast

Becoming more widely available due to the adoption of direct-electron cameras, which are very fast



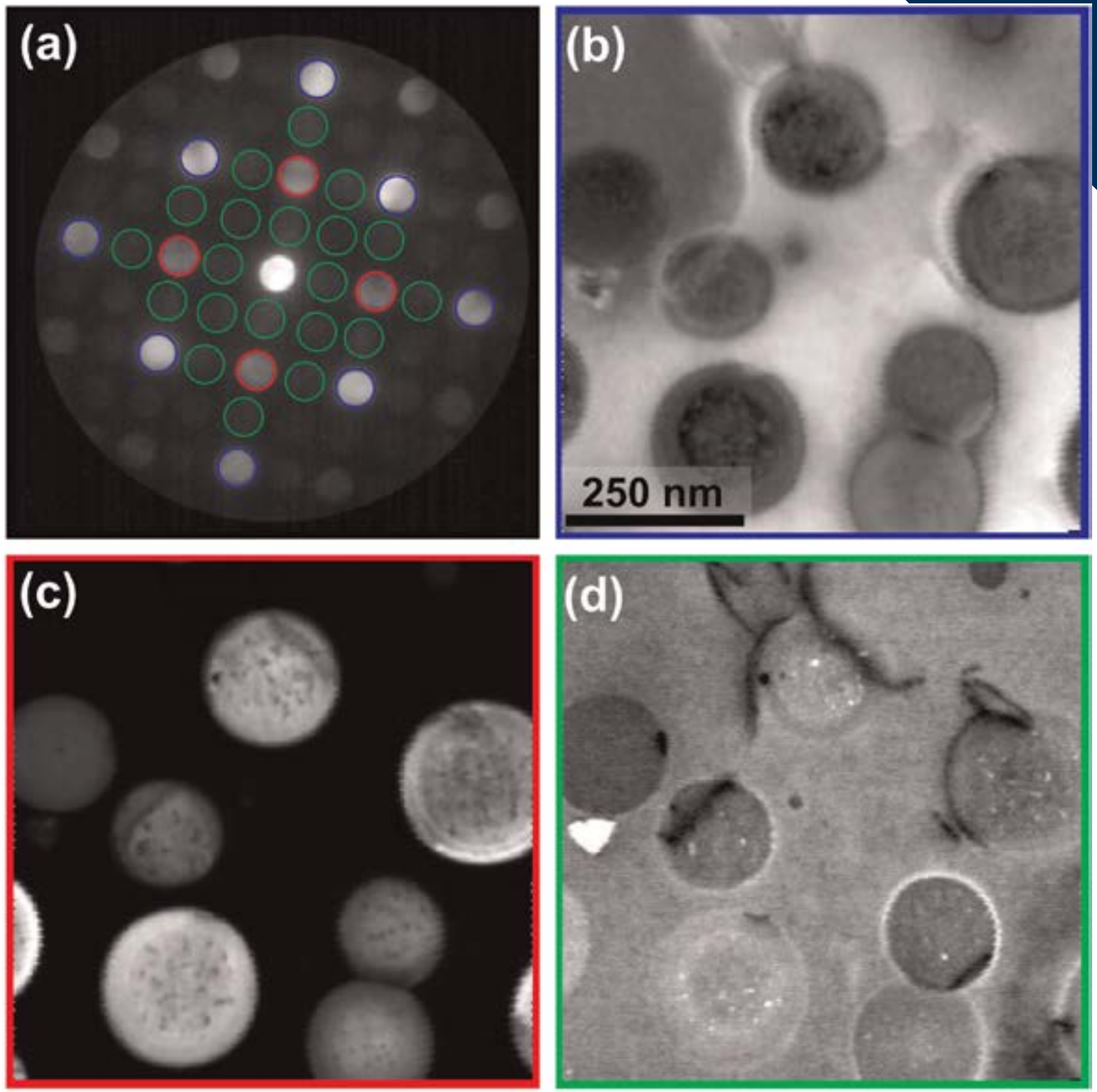
4D STEM - Forming dark-field images, ex-situ!

If we apply a digital mask to the recorded diffraction patterns, and then integrate the intensity for each pixel, then this is akin to inserting an objective aperture

We can form conventional dark field images

We can have multiple “apertures”

We can have apertures of any shape we like



Gammer et al. 10.1016/j.ultramic.2015.03.015

4D STEM - Forming dark-field images, ex-situ!

Tilted to a zone-axis, we have the biggest range of reflections to pick and choose from

Remember though, this is NOT a two-beam condition > we are in a multi-beam condition, and each reflection should not be expected to strictly obey g.b

Using higher order reflections is safer

Consider: is s positive or negative?

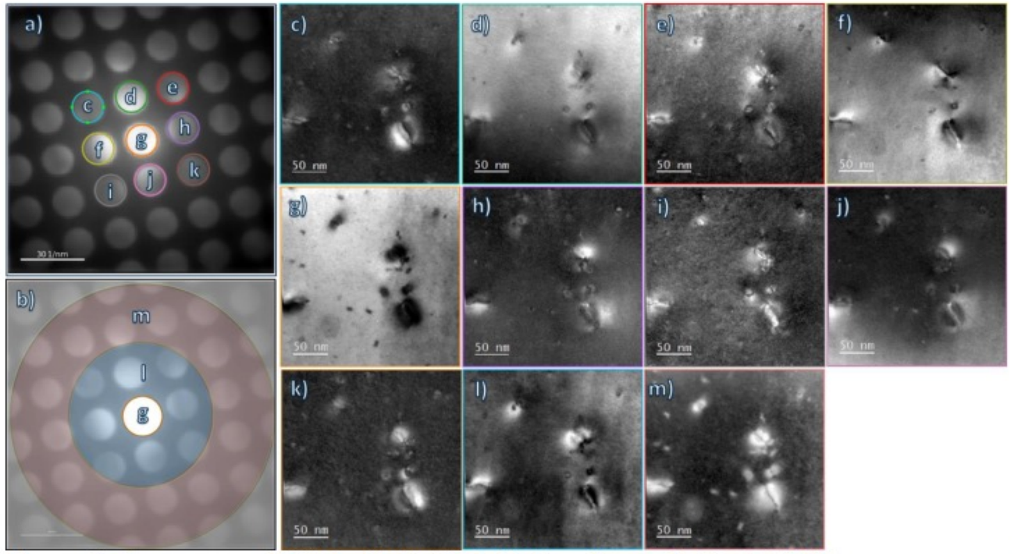


Figure 1. Reconstructed Bright Field, Dark Field and Annular Dark Field images generated using virtual apertures from a 4D-STEM data set that was collected from 150keV self-ion irradiate pure W (99.95%) up to 0.005dpa.

Lim et al. 10.1017/S1431927620014506

4D STEM – Differential Phase Contrast

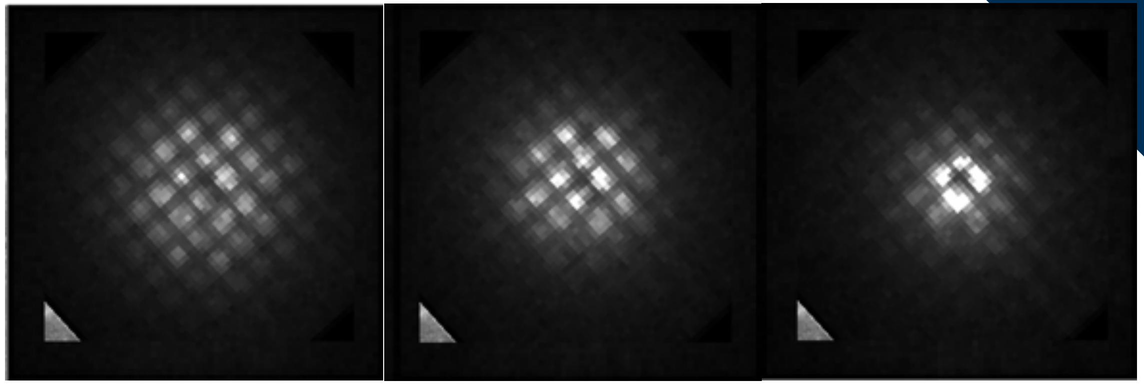
With 4D STEM, we need not limit ourselves to diffraction contrast.

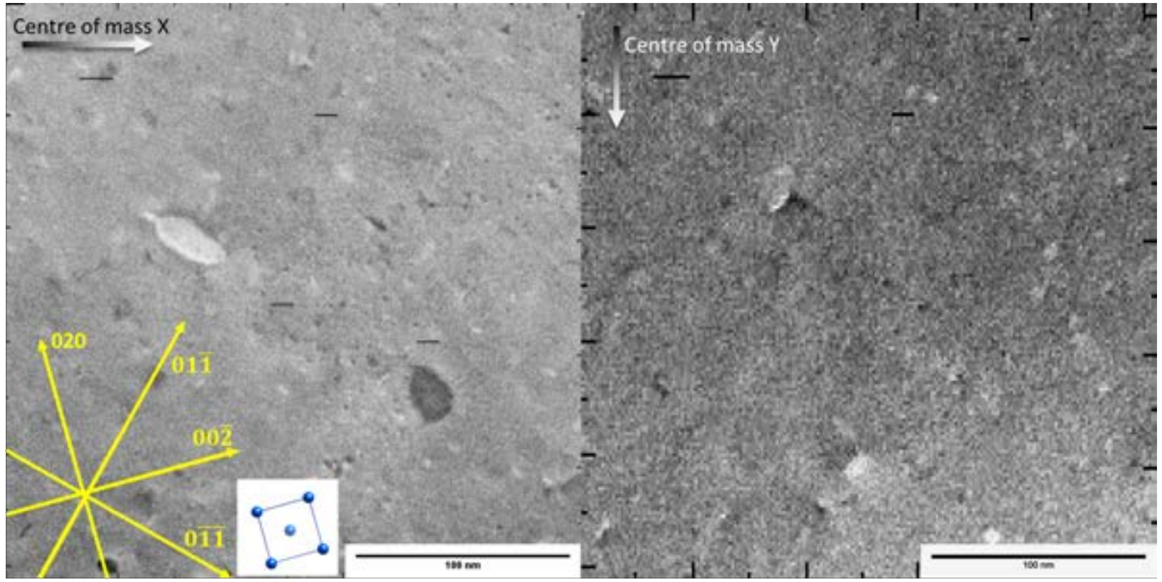
Differential Phase Contrast is a high resolution technique, that produces contrast by measuring the centre-of-mass of the exit wave

Consider a sample tilted to a zone axis, where is the centre of mass?

If the lattice rotates a small amount, what will the centre of mass do?

What if the lattice rotates a lot?





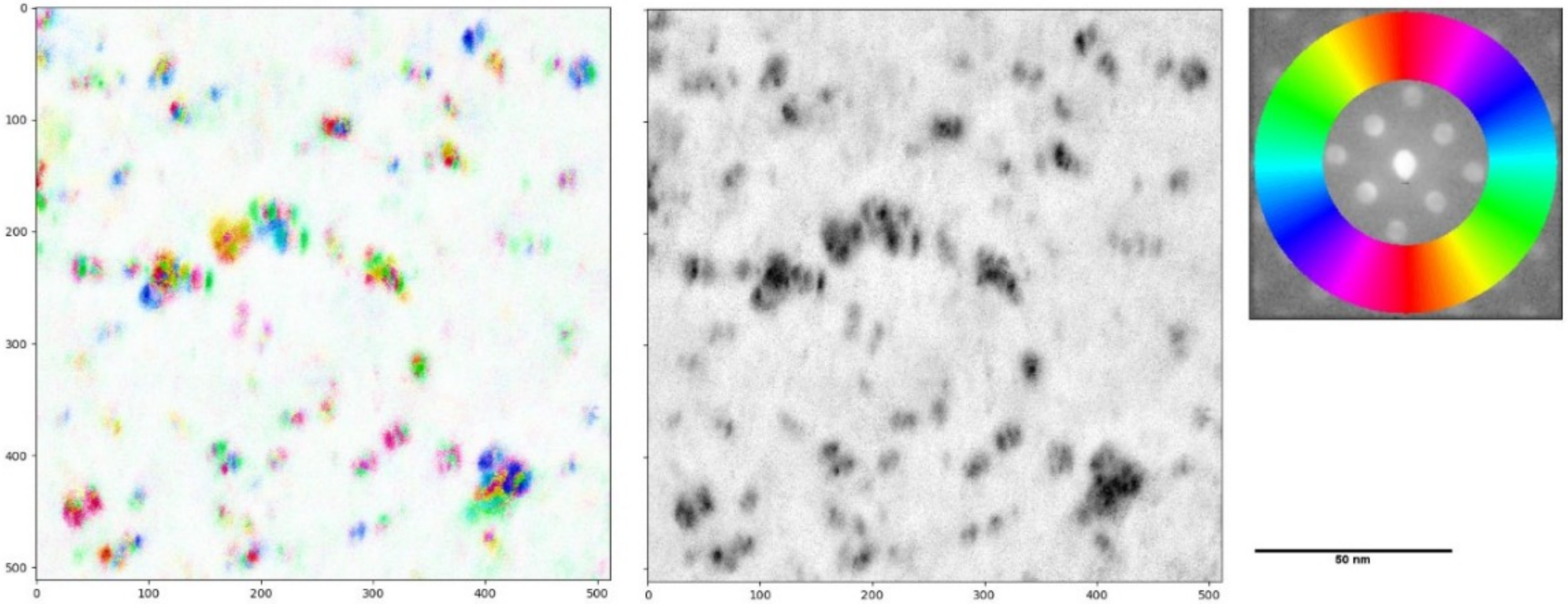
4D STEM – Differential Phase Contrast

Using this principle, we can image dislocations, and dislocation loops, using DPC

We interpret the contrast as the direction in which the lattice is rotating relative to the zone axis

The contrast is semi-quantitative

- The contrast has positive and negative values
- There is a unique direction in which the contrast is zero – this is the same as g.b
- The projected appearance of a dislocation or loop is a good representation of its size
- Can be used to reveal larger dislocation structures



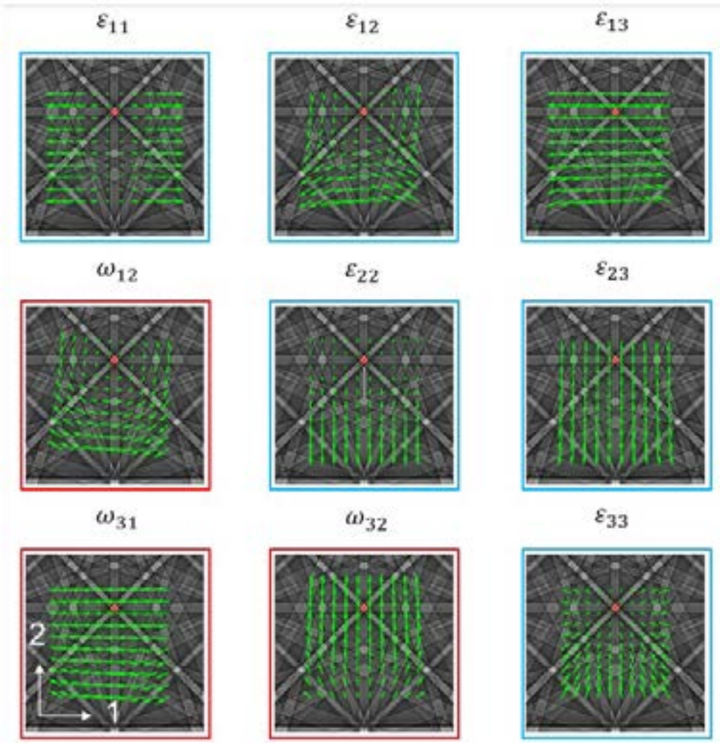
4D STEM – Transmission Kikuchi Diffraction

UK Atomic Energy Authority

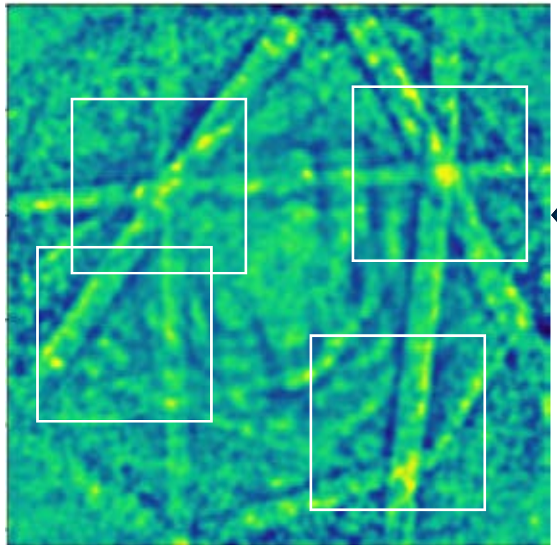
If we wanted to be quantitative with the lattice rotation, we can apply the same techniques used in SEM, by capturing the Kikuchi patterns themselves

Low dependence on grain orientation > genera requirement is that several zone axes are visible

High quality patterns can even be used to quantify the full strain tensor around dislocations



Understanding deformation with high angular resolution electron backscatter diffraction (HR-EBSD) – IOPscience
Britton and Hickey



Filtered Kikuchi pattern captured in STEM

Nature analysis of dislocation loops

Need to know:

Dislocation habit plane AND Burgers vector, and which conditions give inside or outside contrast

OR

Infer inclination from Burgers vector, and which conditions give inside or outside contrast

OR

Infer Burgers vector from habit plane, and which conditions give inside or outside contrast

Nature analysis of dislocation loops

Williams & Carter

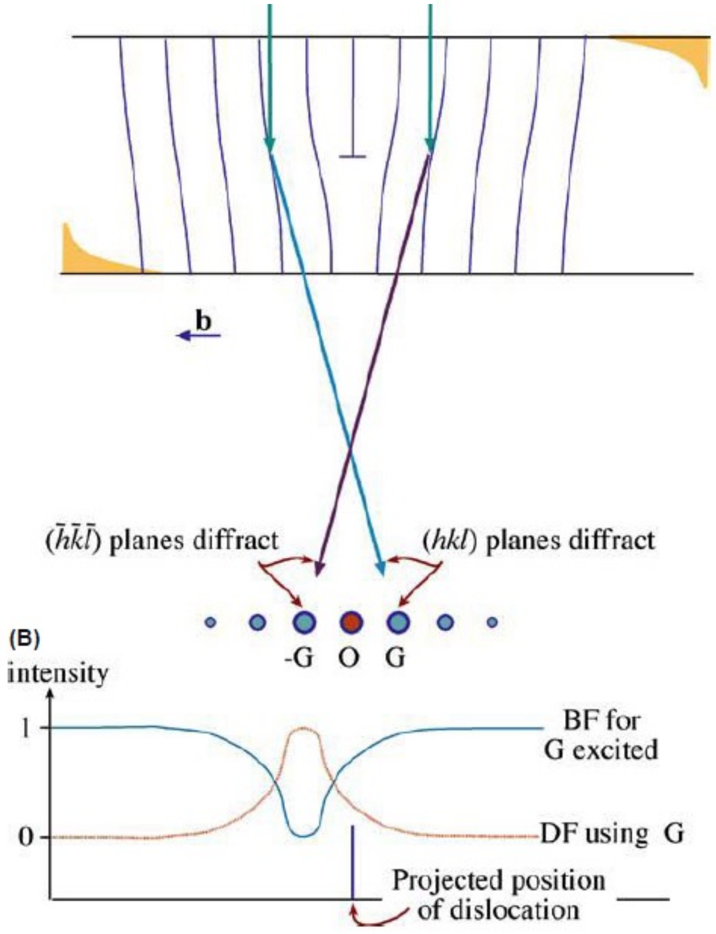


FIGURE 26.1. (A) The specimen is tilted slightly away from the Bragg condition ($s \neq 0$). The distorted planes close to the edge dislocation are bent back into the Bragg-diffracting condition ($s = 0$), diffracting into G and –G as shown. (B) Schematic profiles across the dislocation image showing that the defect contrast is displaced from the projected position of the defect. (As usual for an edge dislocation, **u** points into the paper.)

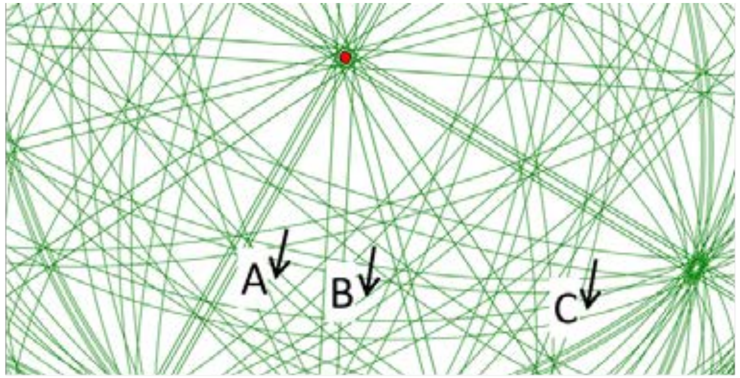
In a kinematical two-beam condition, contrast appears only on one side of the dislocation core

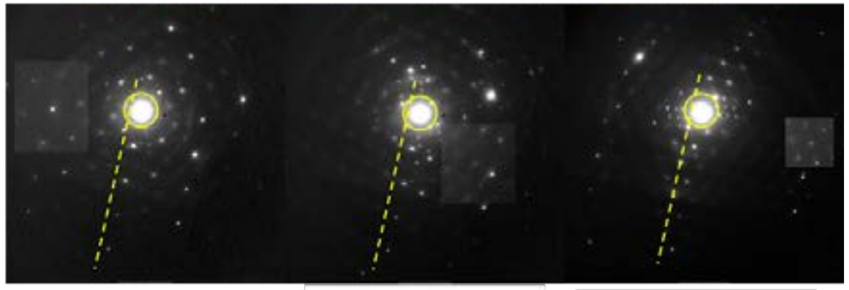
The side depends on the direction of g, the sign of s, and the position of the extra plane of atoms

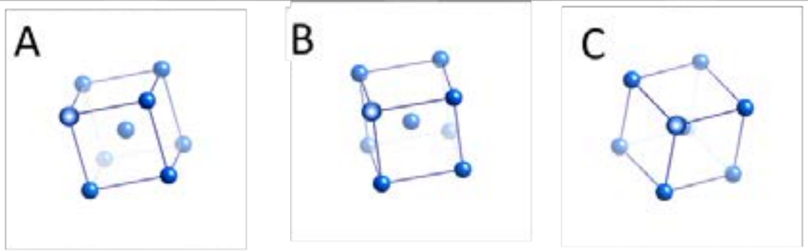
This behaviour is well known for dislocation loops – contrast is either INSIDE the loop, or OUTSIDE the loop

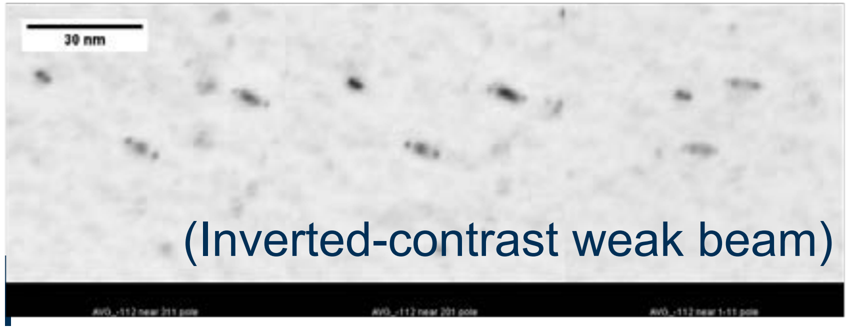
If we know g, s, and the inclination of the loop, we can deduce whether it is interstitial or vacancy type

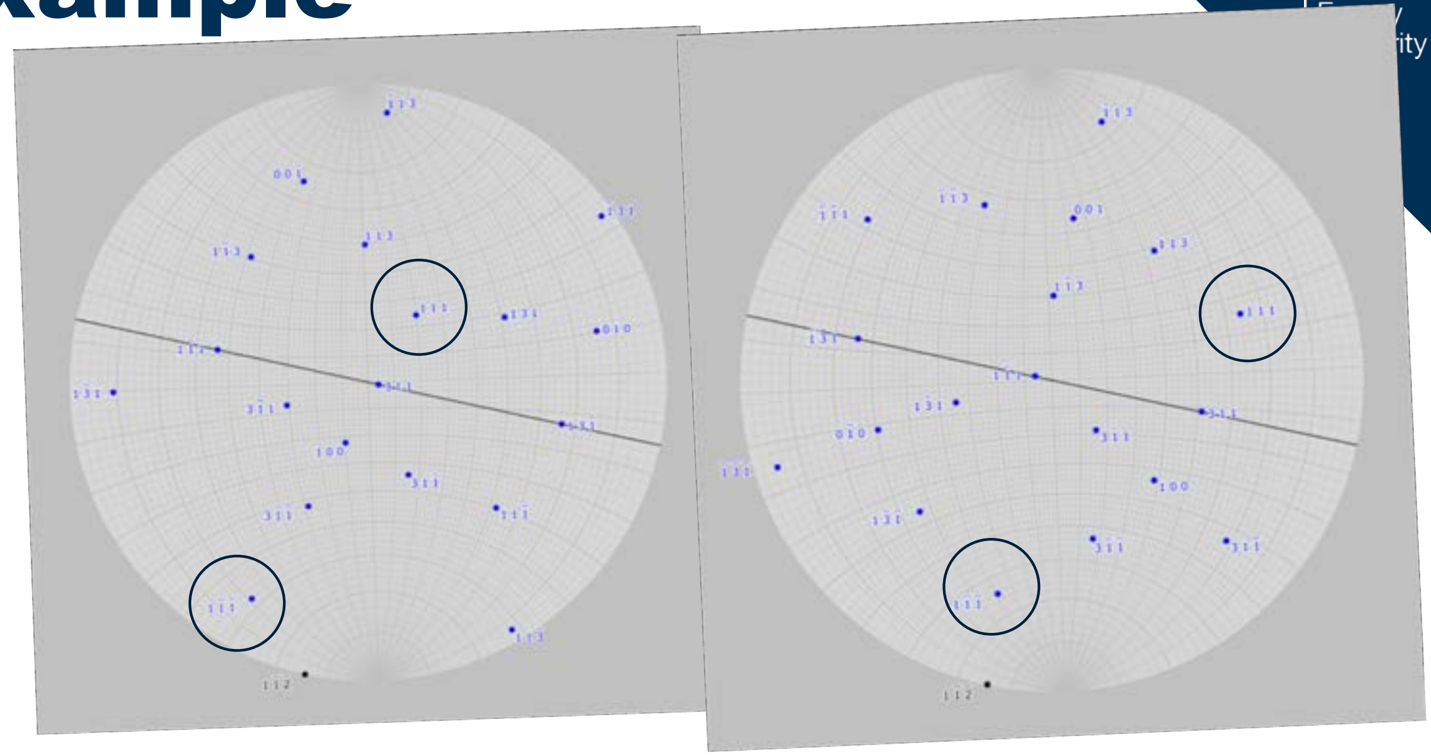
Nature Analysis - example









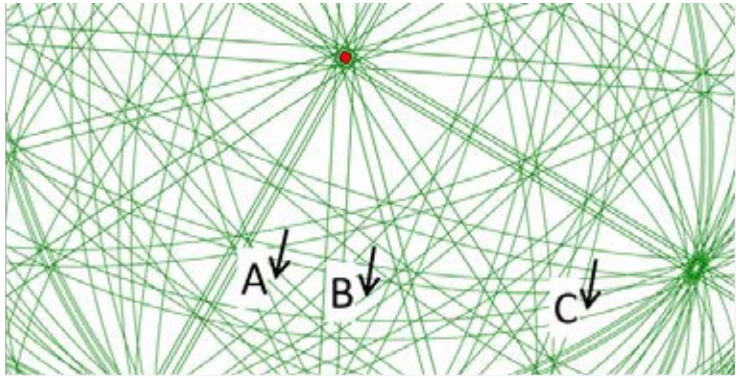


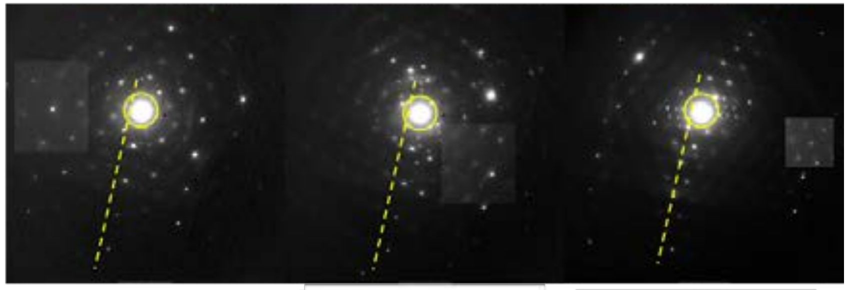
In this example, we have measured the inclination and likely habit plane, and from this, the likely Burgers vector (we do not strictly need the Burgers vector, but you should check to make sure you are not in “unsafe” imaging conditions – explained later).

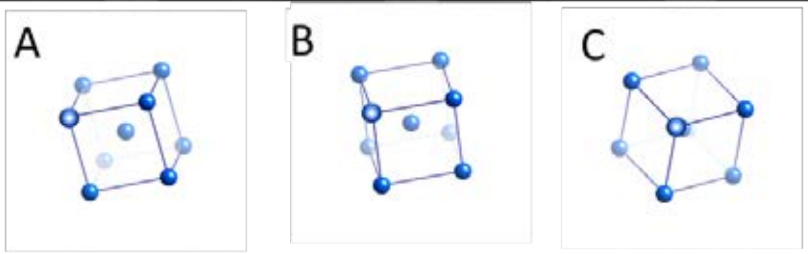
The loops are small, <10nm and some <5nm, so weak-beam was used. Maintaining similar imaging conditions, images were captured at various positions along the Kikuchi band – these have been marked as A, B and C on the Kikuchi map. As we tilt, we see the shape of the defect doesn’t change much, but it does rotate. Using a stereogram can be helpful here, with the possible Burgers vectors/habit planes marked out (CrystalMaker/SingleCrystal are good for this). Based on the image at position A, and knowing the BCC system, the Burgers vector could be either 1-1-1 or 111 or 100. 1-1-1 is most probable of these, since 100 and 111 would be have a much lower inclination than the contrast implies. Tilting to position C reinforces this, as we see large rotation in the positions of 111 and 100, but only a slight anti-clockwise rotation in 1-1-1, which agrees with the rotation of the dislocation loops imaged. Thus we have high confidence the Burgers vector is ±1/2[1-1-1], and it is close to or completely pure edge.

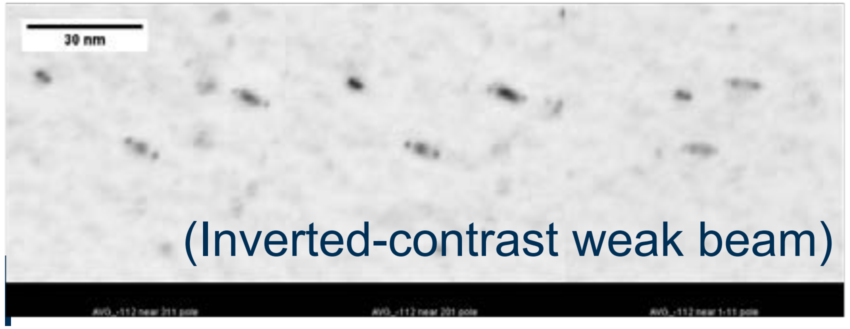
Nature Analysis - example

UK Atomic Energy Authority

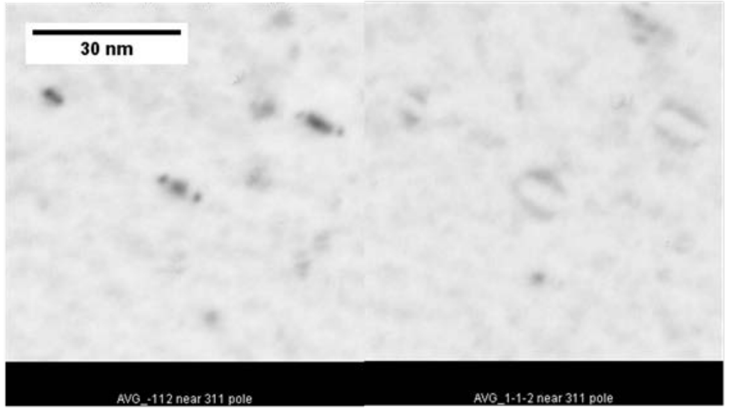


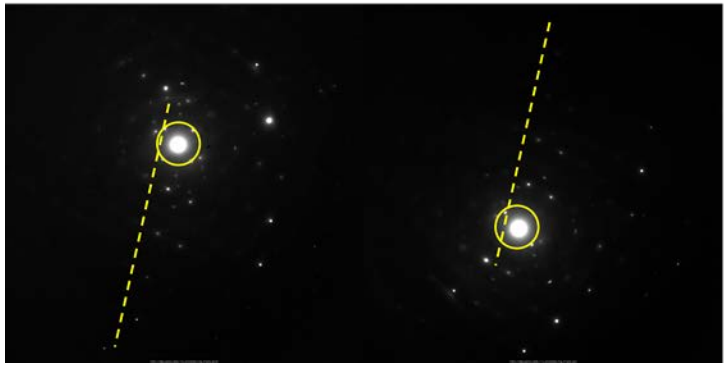


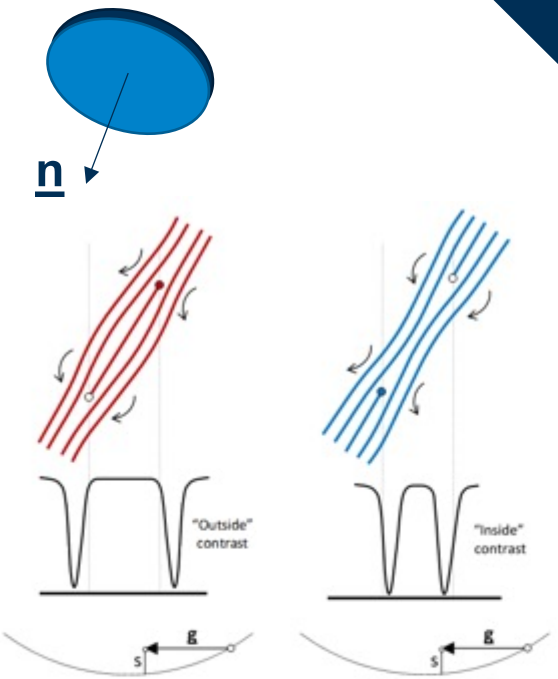




(Inverted-contrast weak beam)







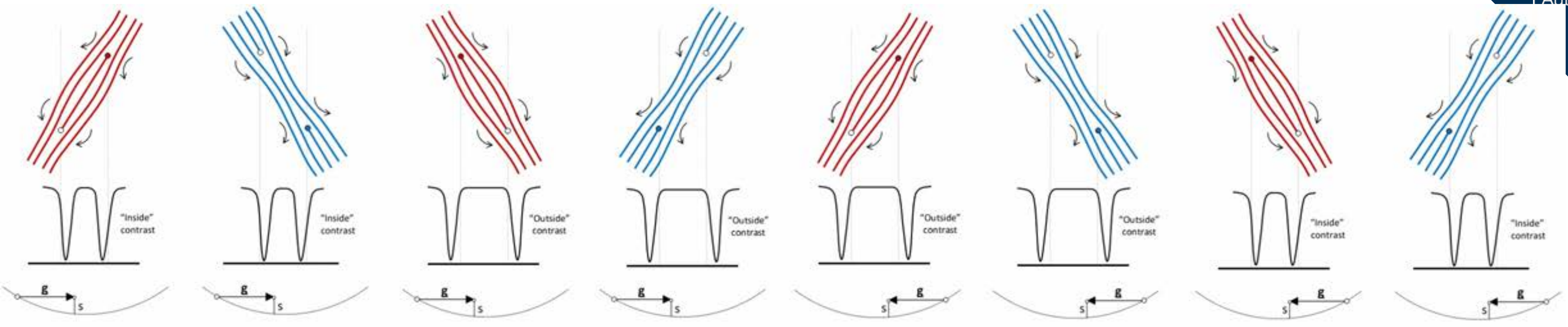
Based on the tilt experiment, we know the loop is oriented as depicted, and thus g points “under” the loop, from our perspective.

Since we have “inside” contrast in this condition, this tells us that these dislocation loops must be vacancy-type.

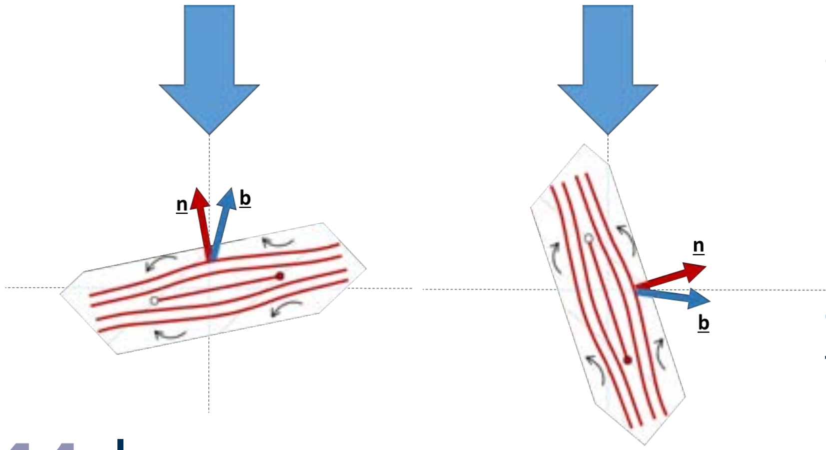
Formally, inside contrast occurs when (<u>**g**</u>.<u>**b**</u>).s<0 and outside when (<u>**g**</u>.<u>**b**</u>).s>0, for the convention of interstitial loops having <u>**b**</u> upward drawn, and vacancies downward drawn, i.e. for beam direction <u>**z**</u>, i.e. “down”, <u>**b**</u>.<u>**z**</u><0 for interstitials and <u>**b**</u>.<u>**z**</u>>0 for vacancies.

In this example then, s is positive, <u>**g**</u>=-112, and contrast is “inside”, therefore, <u>**b**</u>=+1/2[1-1-1] for (<u>**g**</u>.<u>**b**</u>).s<0

Inside-outside contrast for edge loops



Unsafe conditions where non-edge loops show reverse inside-outside contrast



Unsafe conditions are important to rule-out if you think you could have non-edge loops (e.g. for BCC iron, is [111](110) possible? Or in hcp, <a> loops are typically not pure-edge. Are you imaging a loop close to edge-on? If so, how sure are you on its inclination?

Consider what the maximum discrepancy is between the Burgers vector **b** and loop plane normal **n**. Is it possible, given the beam direction **z**, for either **b** and **n** to point in different hemispheres, or for **b** and **n** to be projected in opposite directions with respect to **g**? In general, if you have images over a wide range of conditions and tilts, and you know certain crystallographic constraints to your material, you can be confident in ruling out these effects out.

Nature Analysis - alignments

The image or diffraction pattern will undergo many rotations as it propagates down the microscope column – remember, electrons precess magnetic field lines! Although most modern TEMs are aligned to avoid rotations at the imaging plane, if changing magnification, you might still notice slight shifts in the projected image rotation.

Inversions are also something to account for. This happens when the beam goes through a cross-over point. The ray-diagram from your microscope's manual shows this well, below taken from a Jeol 2100 manual. Watch the direction of the arrow as it propagates down the column. For a Jeol-2100 in image mode, the lenses are such that the image is aligned with the specimen. However, consider the position of reflection **g** when in diffraction mode, the position of the reflection is inverted, so appears 180 degrees from the true orientation. If you get the direction of g wrong, then you will deduce the incorrect loop nature!

Also, think carefully about how you infer the sample orientation – remember that your Kikuchi lines are back projected, so if comparing to a Stereogram with zone axes or poles depicted "upward", the zone-axis in the Kikuchi lines will be projected 180 degrees to this. However, if there is an inversion, then the Kikuchi lines will look upward projected.

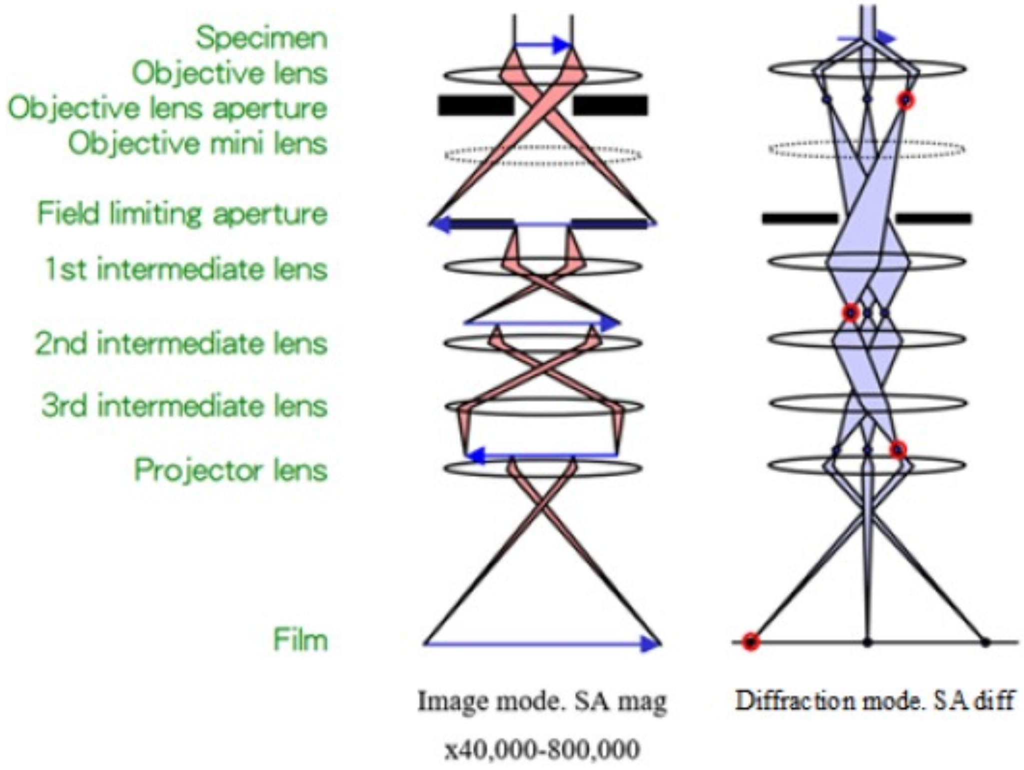


Figure A.1: Ray diagram for a JEOL 2100L microscope, from [204]

Nature Analysis - alignments

To determine if there is an inversion or rotation experimentally, we can conduct a simple experiment. It is often recommended for a TEM alignment sample to be used for this, but this is not strictly necessary. To know the rotation, we need to know the orientation of the crystal. An alignment sample is useful here, e.g. a single crystal sample, containing features of well defined features, e.g. facets of an inclusion, a twin boundary, or screw dislocations. If you have atomic-resolution images with 2-fold symmetry, then you can align to this. Then it is as simple as checking the various magnifications, and noting any difference between the image rotations at each magnification. And noting any difference between image and diffraction pattern.

This will only be able to tell you the rotation within 180 degrees. To see if there is an inversion:

- Capture a two-beam bright-field image containing a prominent feature – this could be a large precipitate, or edge of the sample, hole etc.
- Capture a parallel beam pattern
- Defocus the pattern by reducing the strength of the lens – this will shift the cross over point down, coarsening the spots.
- In the coarsened spots, you will see a bright field (and dark field) image that is oriented correctly with respect to the pattern.
- Is this image aligned with the original bright field image? If not, then there is an inversion.

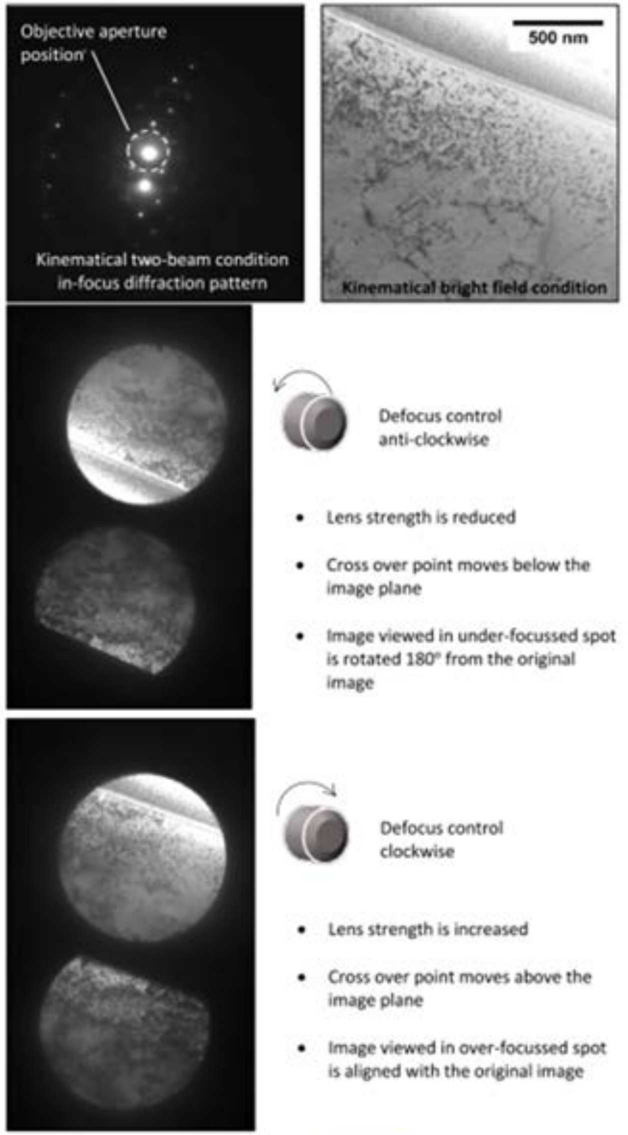


Figure 3.15: Experiment to determine whether an inversion between image and diffraction space exists in the JEOL 2100L.