Magnetic Imaging in the Electron Microscope

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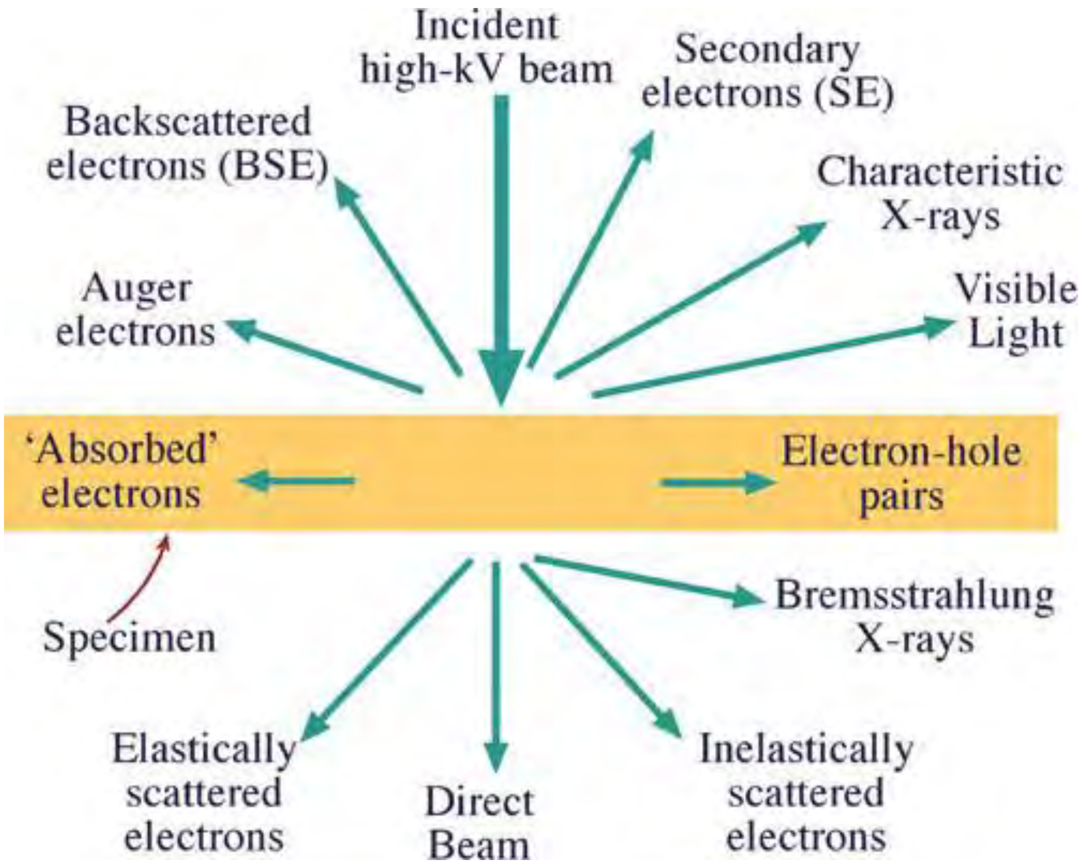
Outline

- Electron interactions and the electron microscope
- Imaging methods in the electron microscope (scanning and transmission – SEM, TEM and STEM)
- Implementation of Lorentz microscopy
- Examples of application of (S)TEM
- Developments

Electron interaction with matter



From Williams and Carter – Transmission Electron Microscopy

Electrons in the electron microscope

Accelerating voltage (kV)	Non-relativistic λ (pm)	Relativistic λ (pm)	Mass ($\times m_o$)	Velocity ($\times 10^8$m/s)
100	3.86	3.70	1.20	1.64
200	2.73	2.51	1.39	2.09
300	2.23	1.97	1.59	2.33
1000	1.22	0.87	2.96	2.82

Non relativistic calculation

$$eV = \frac{p^2}{2m_o} \qquad \lambda = \frac{h}{\sqrt{2m_o eV}}$$

Relativistic calculation

$$E^2 = p^2c^2 + \left(m_o c^2\right)^2 \qquad \lambda = \frac{h}{\sqrt{2m_o eV\left(1 + \frac{eV}{2m_o c^2}\right)}}$$

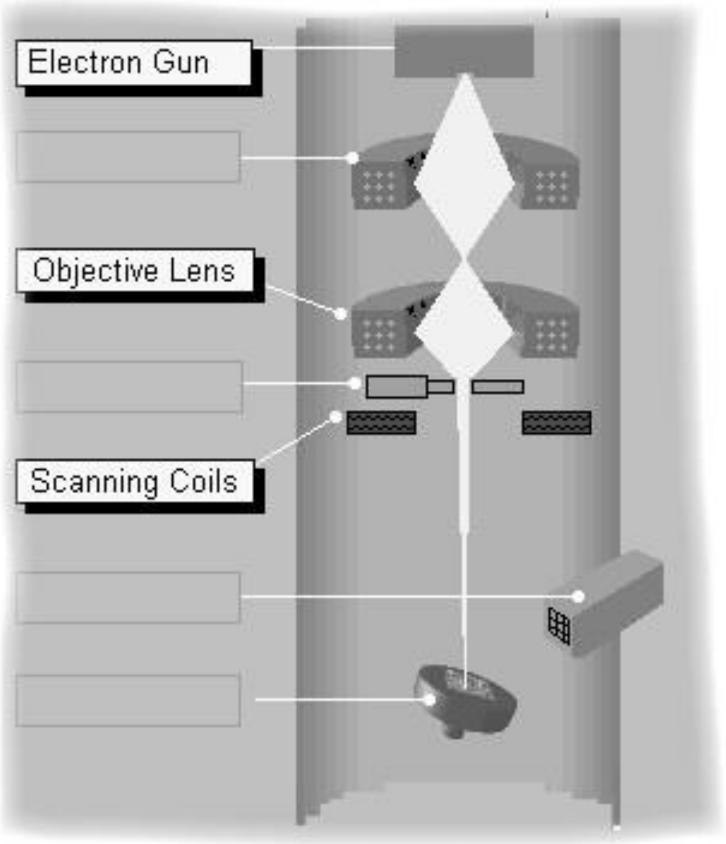
The Scanning Electron Microscope

Scanning electron microscopy (SEM) is a technique for imaging **surfaces**.

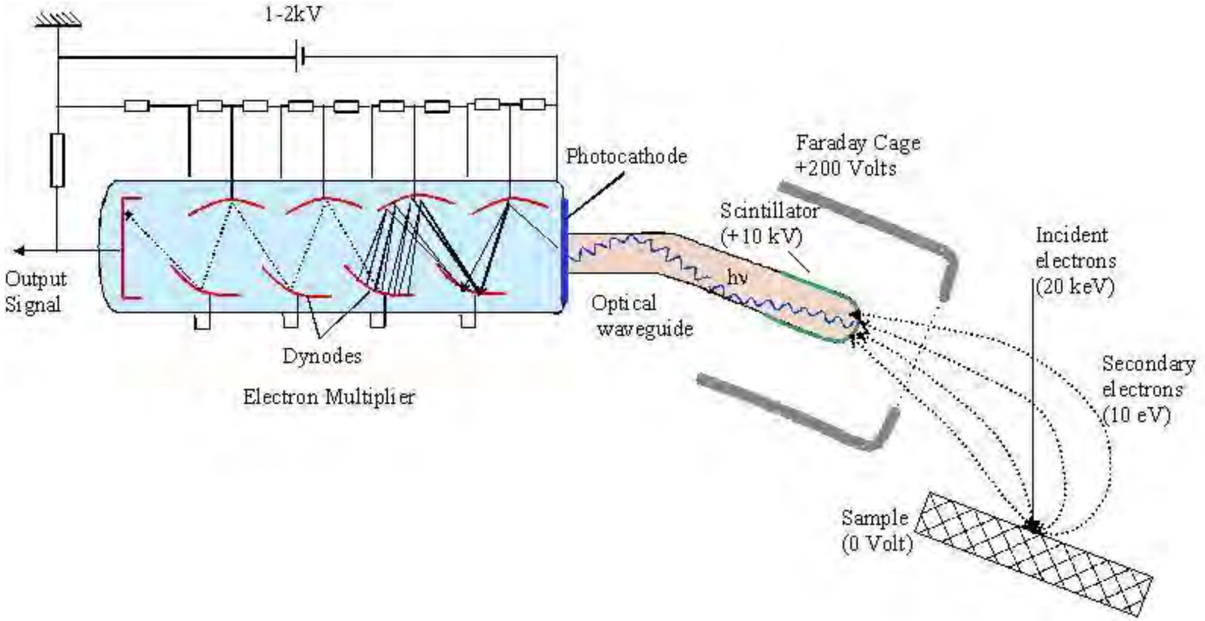
The electrons are emitted from a source called the gun. Usually emits electrons of energy in range 5–40 keV.

Special lenses focus the electrons by using magnetic fields.

Probe size limited by diffraction and aberrations.



SEM Detection



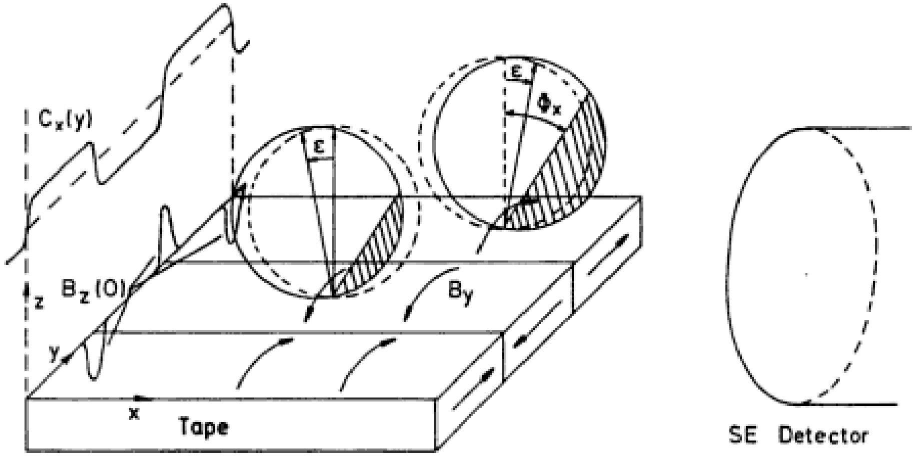
Secondary electrons (SE) have energies up to ~ 50eV.

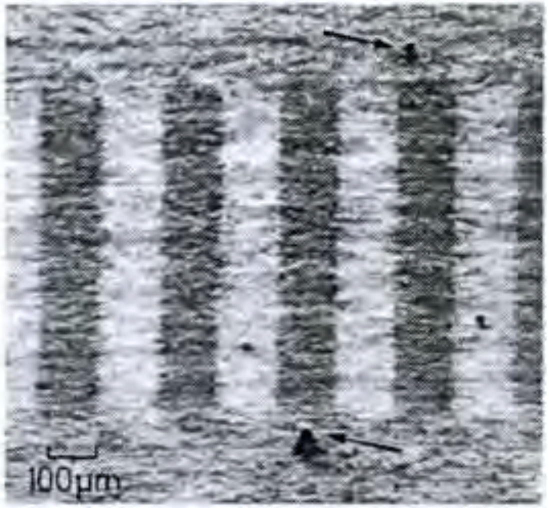
Backscattered electrons (BSE) have energies up to primary beam energy.

Faraday cage voltage means can predominantly image SE or BSE.

SEM Type I contrast

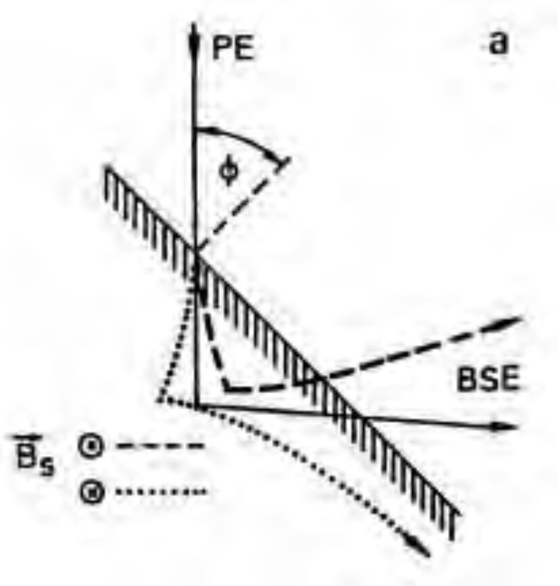
Type I contrast utilises the fact that the secondary electrons are deflected by stray fields from the sample. Orientation of detector can then show contrast from regions with different directions of stray field.

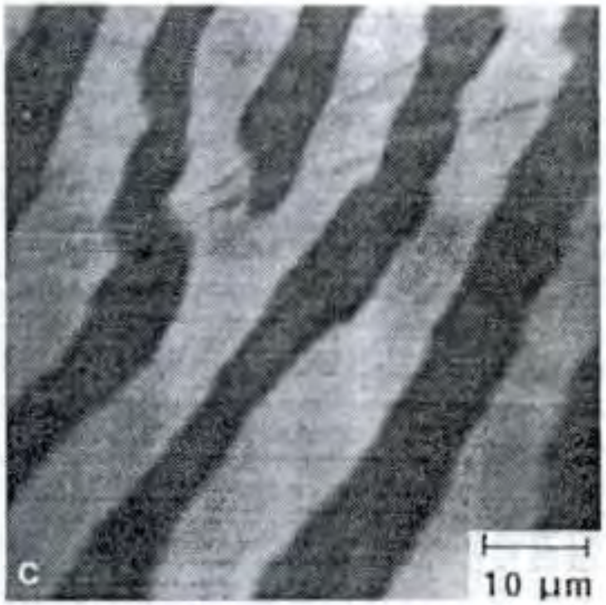




SEM Type II contrast

Type II contrast utilises the fact that the backscattered electrons are deflected by the magnetic induction within the sample. This results in an increased or decreased BSE signal.

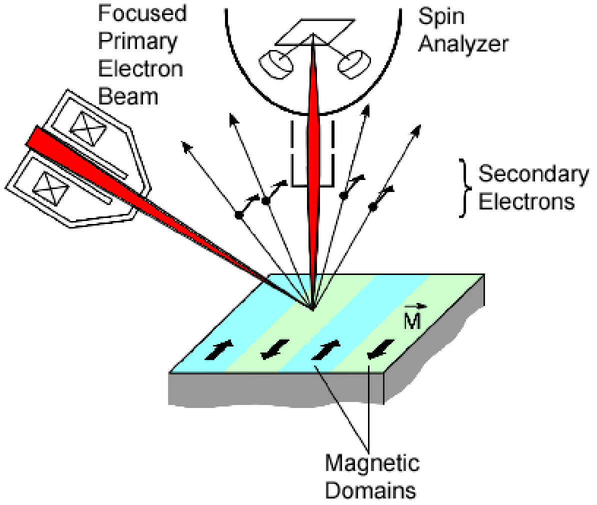




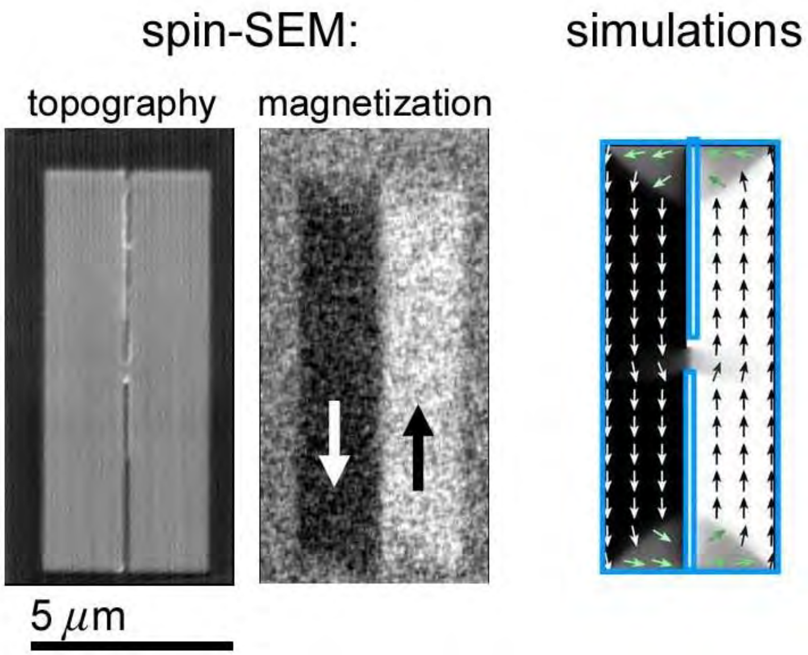
SEMPA

Scanning electron microscope with polarisation analysis:

- polarisation of secondary electrons are detected
- gives magnetisation of local region

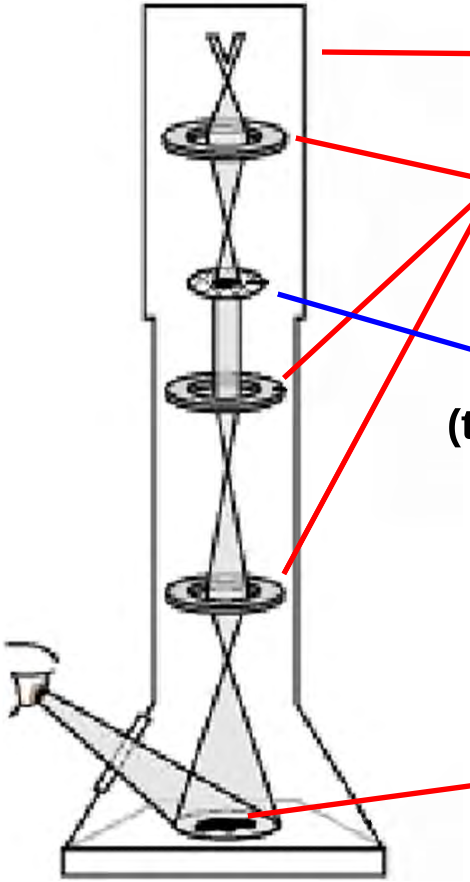


$$P_x = (N_\uparrow - N_\downarrow)/(N_\uparrow + N_\downarrow),$$

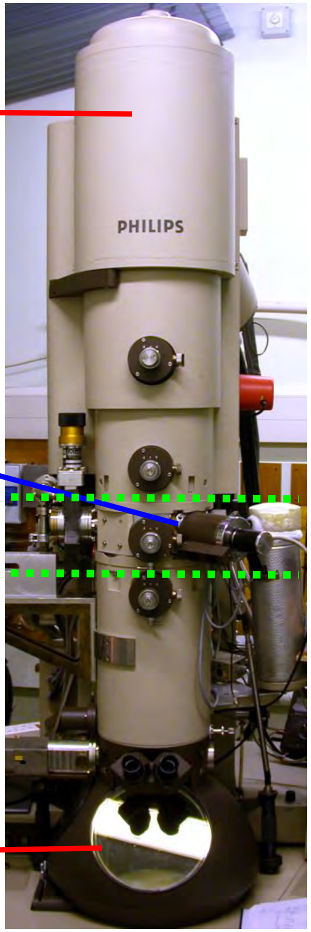


Components of the transmission electron microscope (TEM)

A Cartoon TEM



A real TEM



Electron Source

Electro-magnetic lenses

Specimen (thickness < 100 nm)

Viewing screen

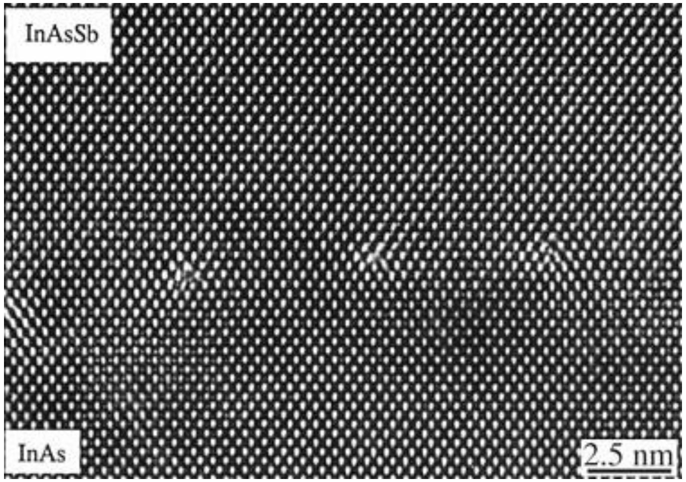
Transmission Electron Microscopy

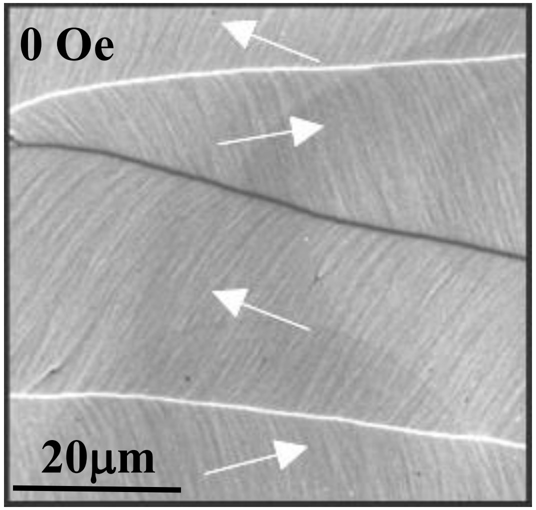
Concentrate now on “regular” TEM.

Samples must be thin (~<100nm).

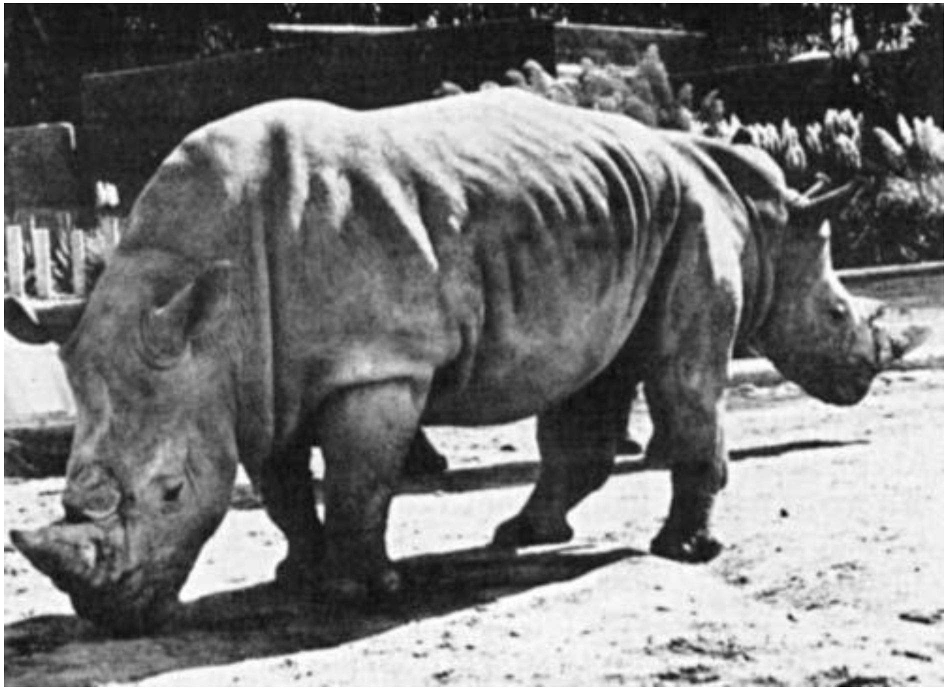
Standard TEM has objective lens on.

Magnetic imaging is normally carried out with objective off or weakly excited – Lorentz microscopy.





TEM – beware!



Boxer Hand Shadow

Say what you see?

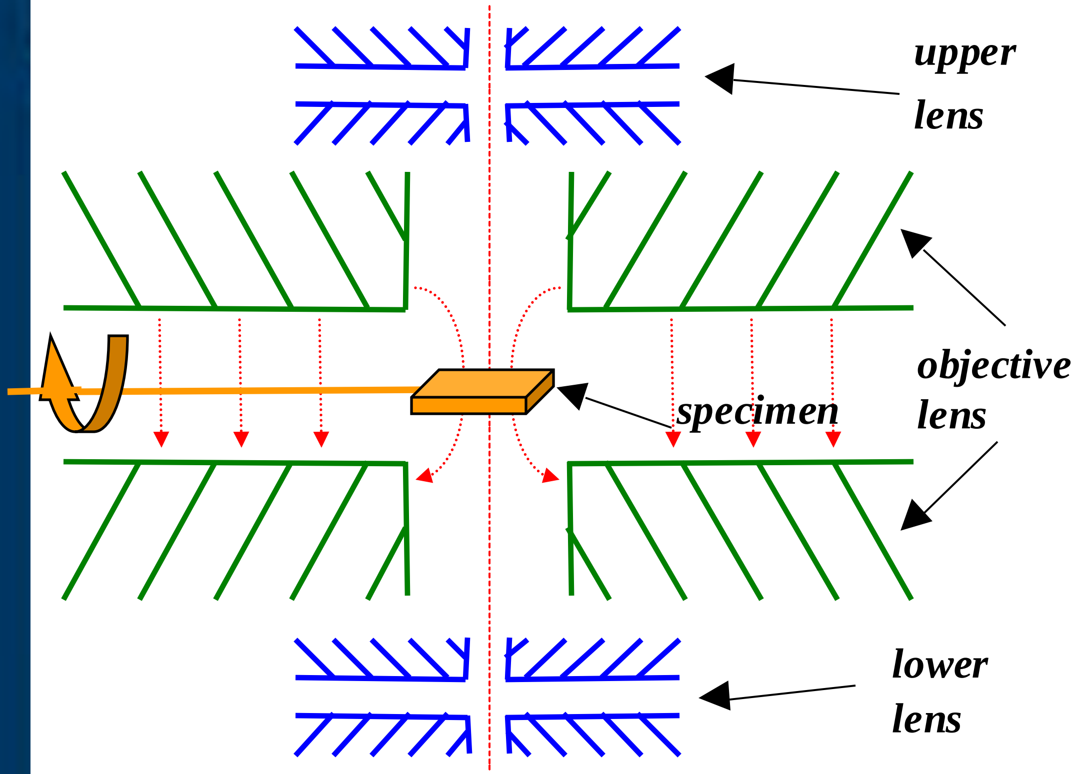
You are seeing projected information – should verify what you are seeing before making detailed interpretation.

Lorentz microscopy

Lorentz microscopy:

- thin films (< 100nm)
- high spatial resolution (<10 nm has been demonstrated)
- information on domain and domain wall structures
- sensitive to induction (contrast arises from specimen **magnetisation and stray fields**)
- quantitative information on spatial distribution of integrated induction components
- suitable for real time studies involving field, currents and temperature variation
- availability of complementary (perfectly registered) nanostructural information

Lorentz TEM operation



Also use custom specimen rods to deliver field, heat and current pulses to samples in-situ

Standard mode:

Objective lens generates high (few Tesla) magnetic fields perpendicular to the sample plane

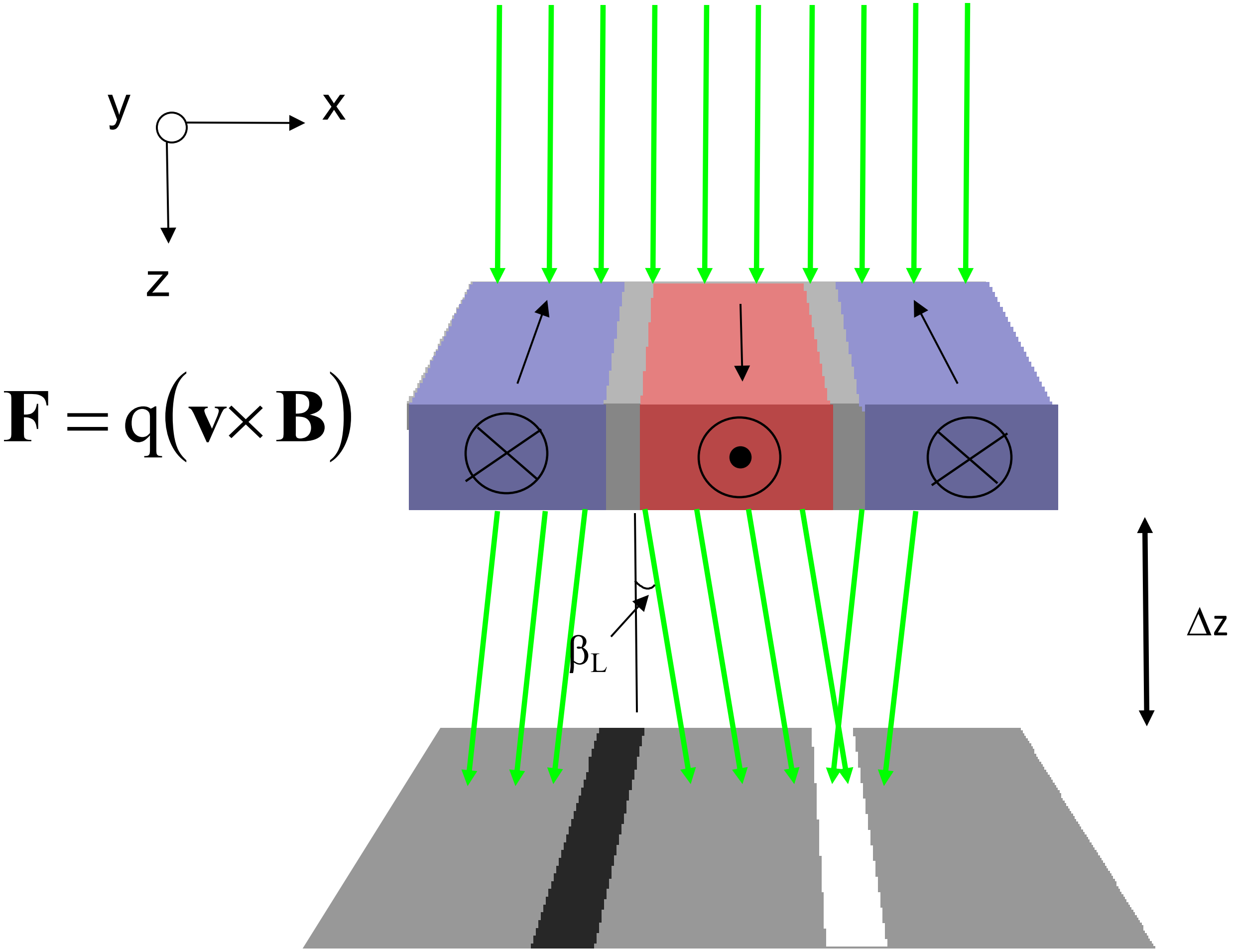
Lorentz:

- **Switch off objective lens!!!**

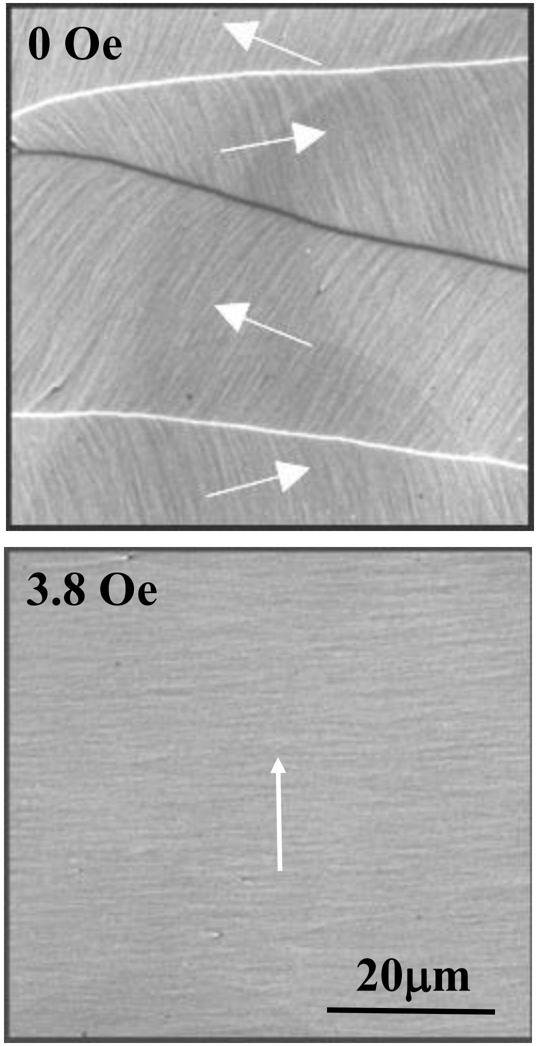
Field-free/low field mode:

- Instead use Lorentz/mini-lenses
- Perform in-situ magnetising experiments by mild excitation of objective lens and tilting the sample

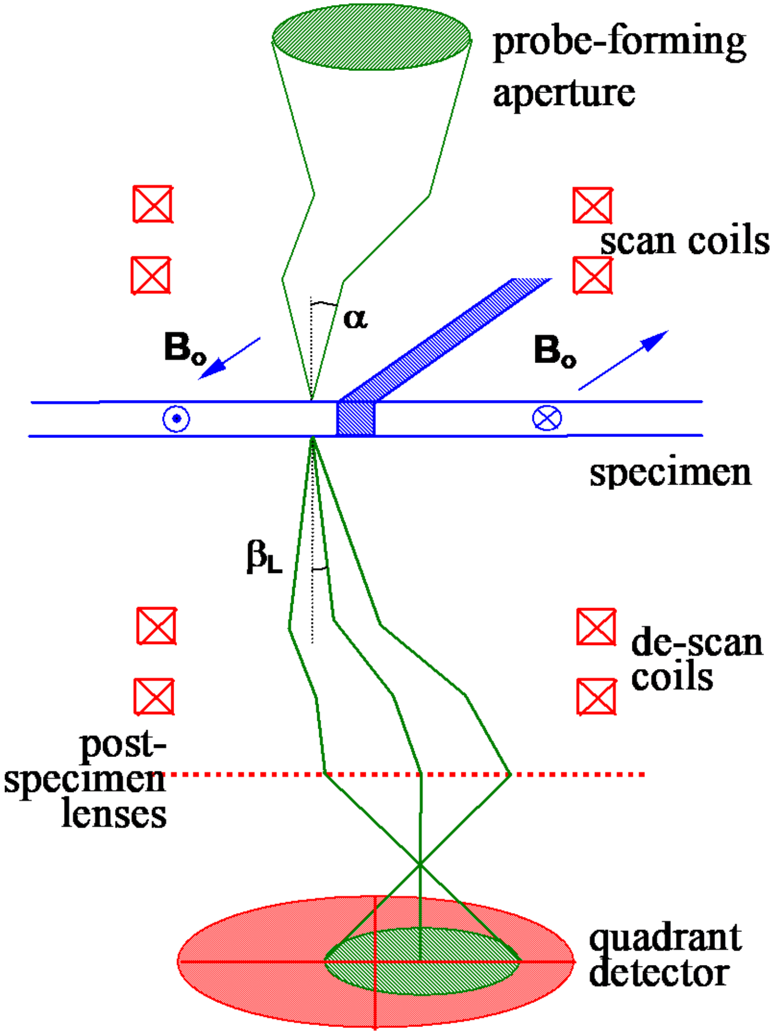
Lorentz TEM imaging: Fresnel mode

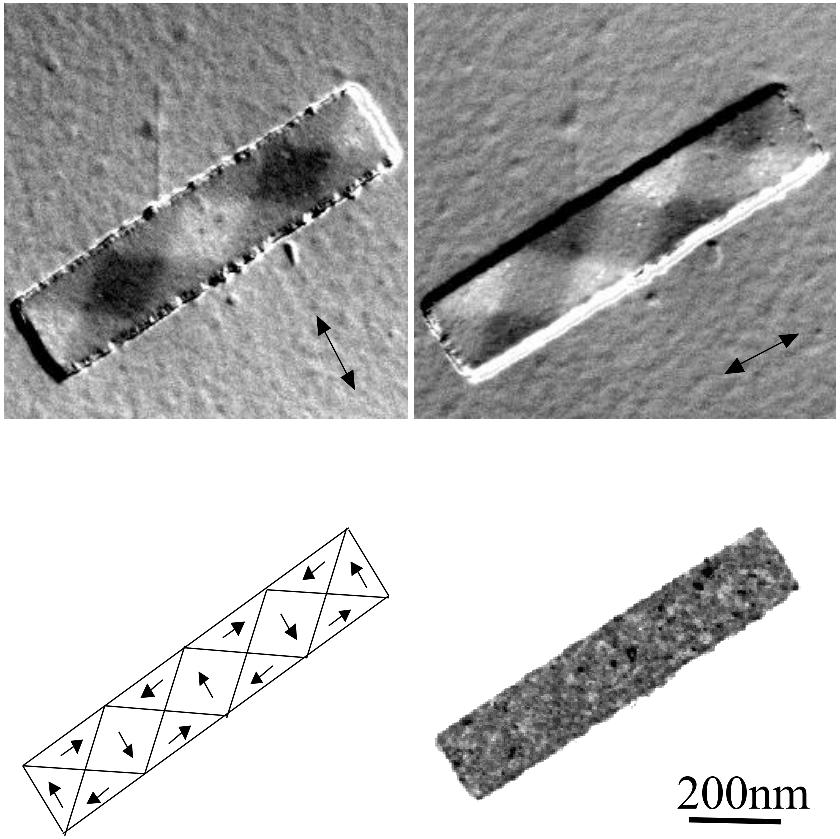


$$\mathbf{F} = q(\mathbf{v} \times \mathbf{B})$$



Lorentz STEM imaging: differential phase contrast





1000 × 200 nm^2 permalloy element,
40 nm thick

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Lorentz microscopy – Key points

Fresnel imaging

- quick and simple method for getting domain geometry
- generally non-linear imaging
- ideal for overview of magnetisation processes

DPC imaging

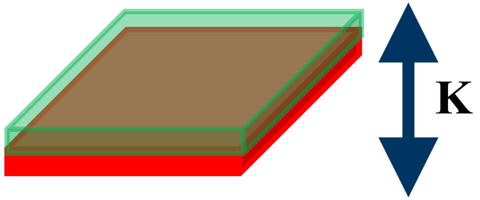
- sensitive to induction (contrast arises from specimen **magnetisation and stray fields**)
- quantitative information on spatial distribution of integrated induction components
- good for detailed induction maps

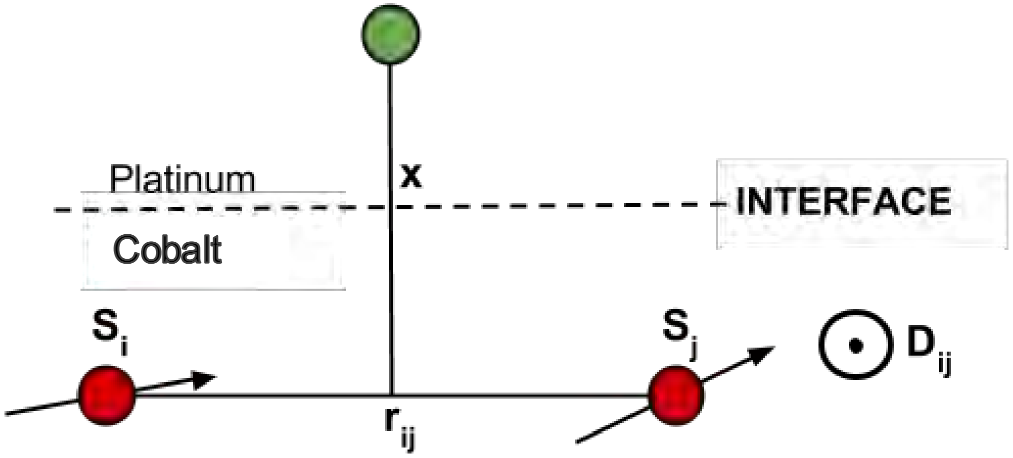
In both cases have to be aware contrast in images arises also from non-magnetic sources.

Interfacial exchange in perpendicular films

Thin films with perpendicular anisotropy in contact with heavy metal – large spin orbit coupling.

Gives rise to Dzyaloshinskii-Moriya interaction at interface.





FM exchange

$- J(S_i.S_j)$

Interfacial exchange

$- D_{ij}.(S_i \times S_j)$

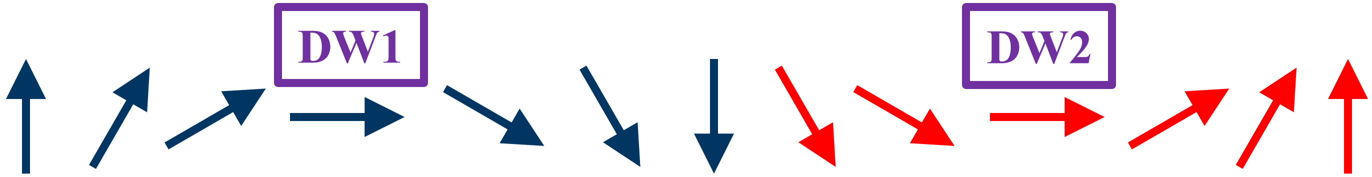
In absence of DM interaction expect divergence free Bloch walls.

DM interaction should promote Néel walls with defined chirality depending on sign of **D**.

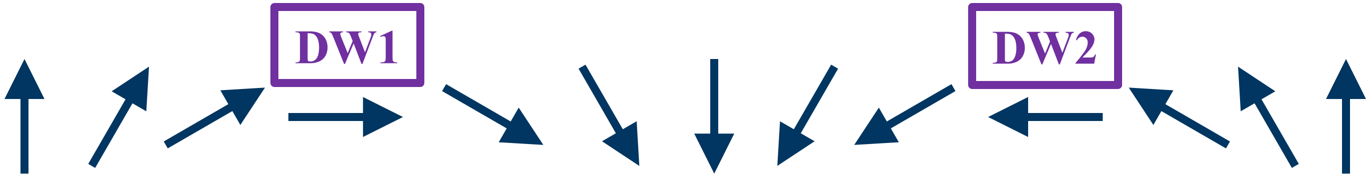
Interfacial exchange – Néel walls

Can we identify
i) if walls are of Néel type?
ii) chirality of wall?

Pair of “unwinding” Néel walls

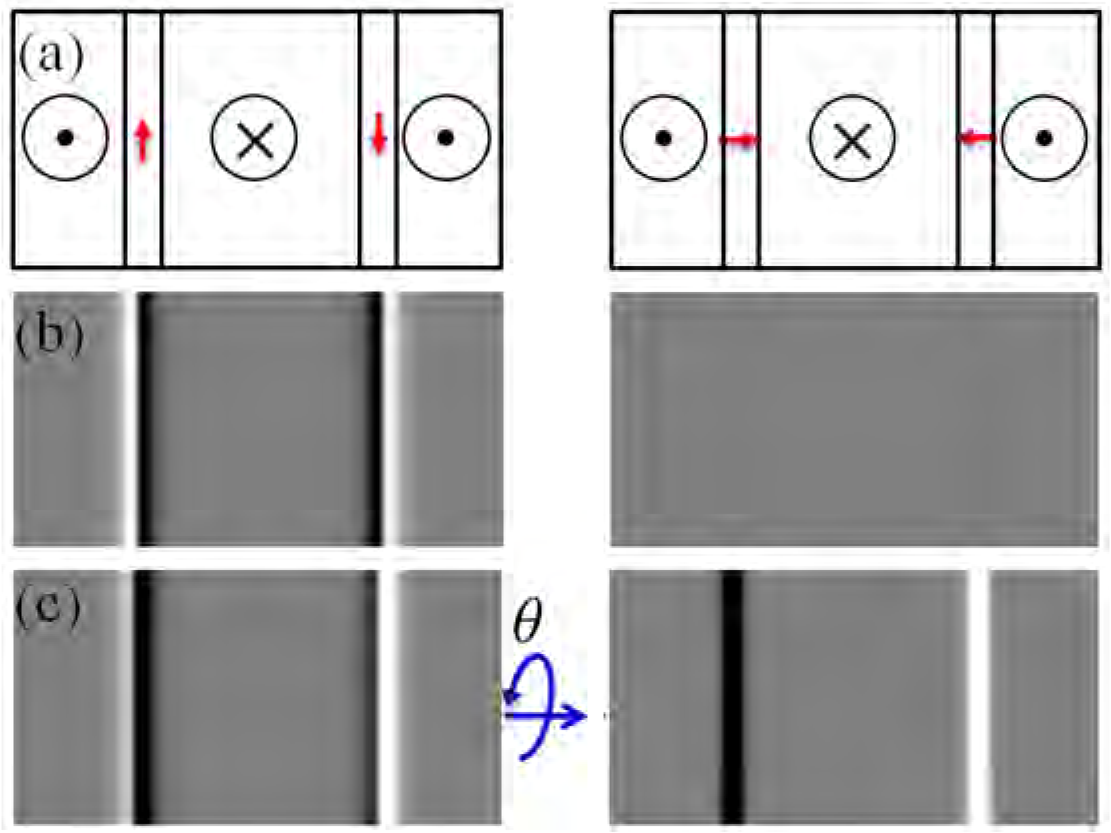


Pair of “winding” Néel walls



Néel and Bloch walls in perpendicular films

Thin films magnetised perpendicular to plane of film.
Need to be able to identify Bloch and Néel walls – use Fresnel imaging.
Electron beam perpendicular to screen.



Bloch **Néel**

Schematic

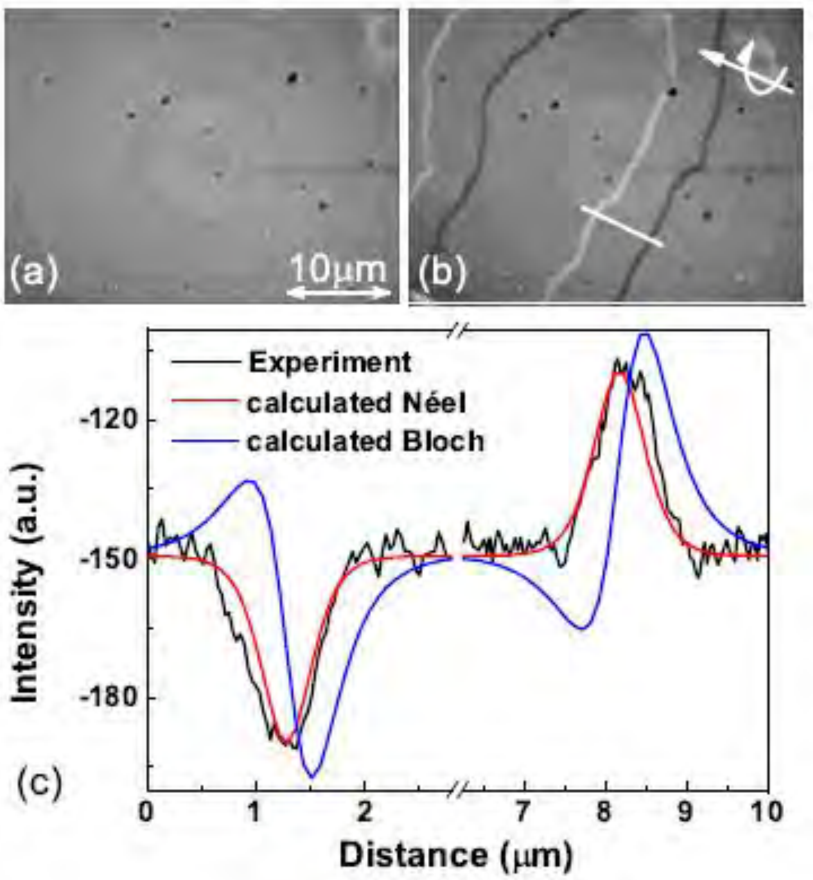
Untilted

Tilted

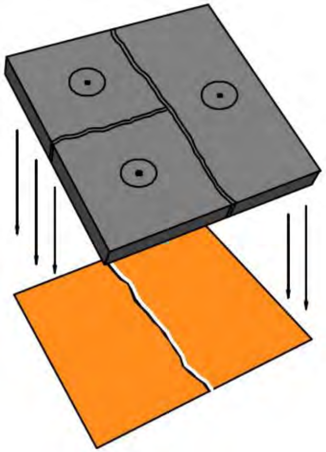
Néel walls in perpendicular films

Ta(3.2 nm)/Pt(3 nm)/Co(0.8 nm)/AlOx(3 nm)
DC sputtering AlOx layer which was deposited by the RF Ar plasma.

Untilted



Tilted



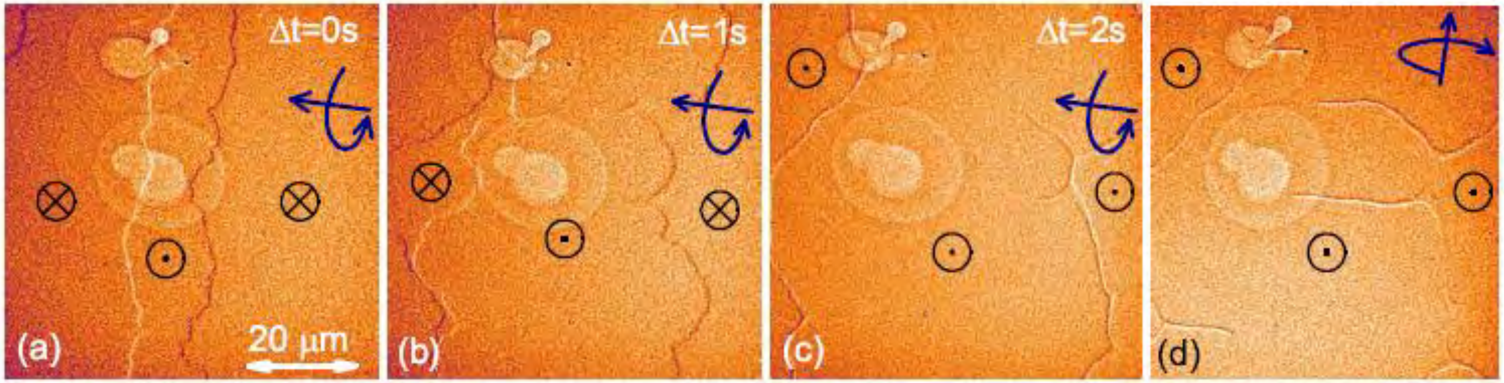
Images proves that the walls are Néel. Does not prove same chirality though.

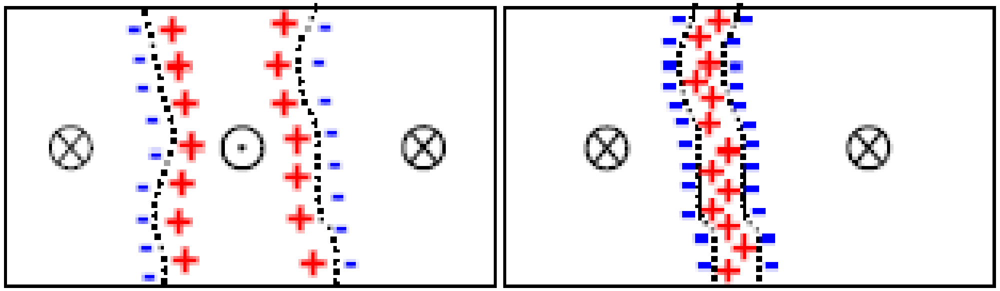
Néel walls in perpendicular films

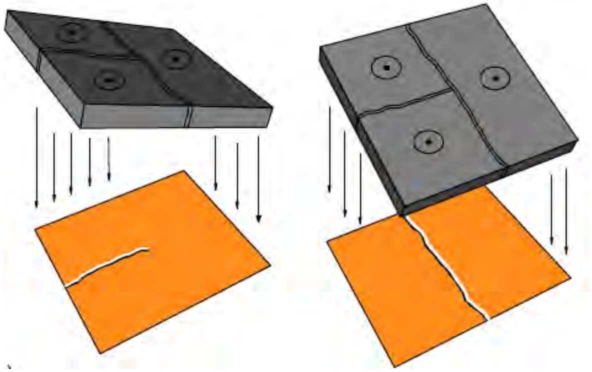
Pushing walls together in field in TEM and MOKE.

No DMI – should get easy annihilation

DMI – divergent walls will repel each other

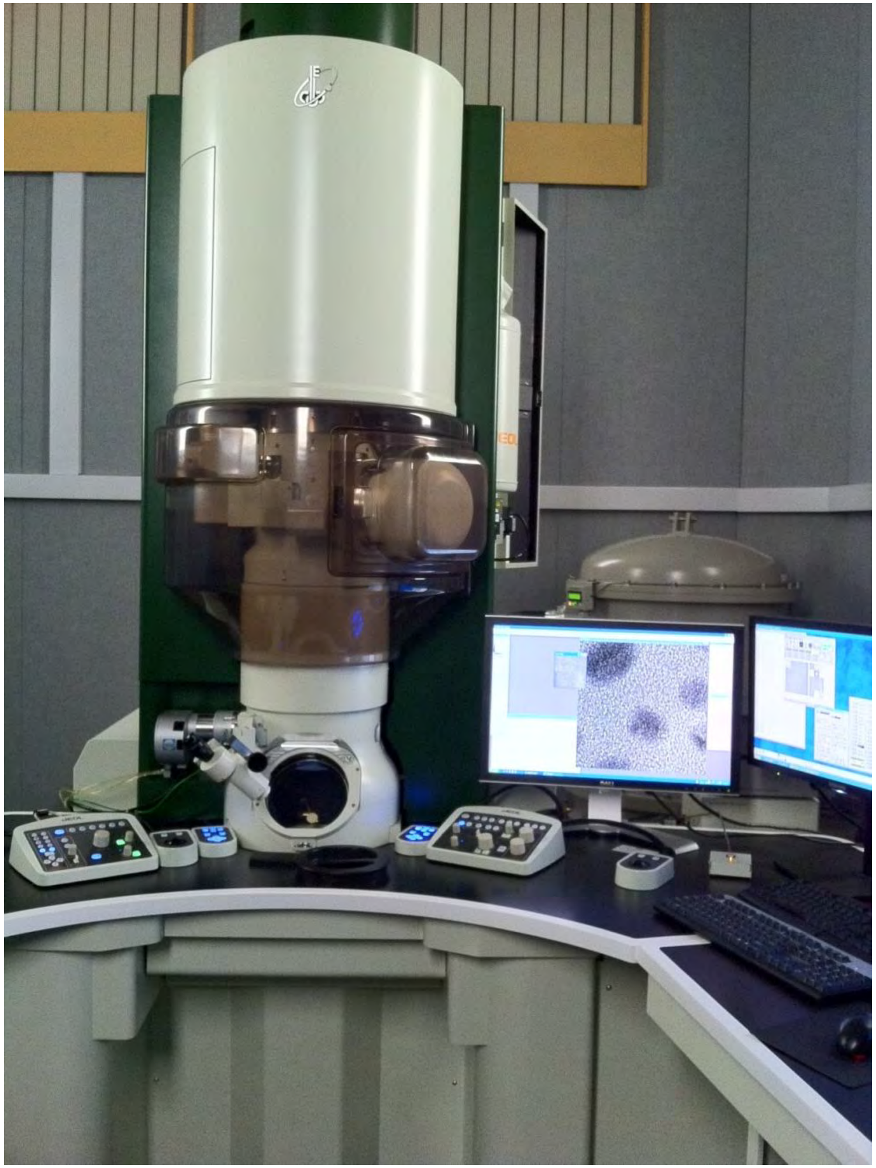






*A Hrabec et al, Nature Communications, **6** 8957 (2015).*

Atomic Resolution Microscope



Scanning transmission electron microscope with aberration corrector for probe.

STEM resolution specification:

Standard mode (objective lens on)

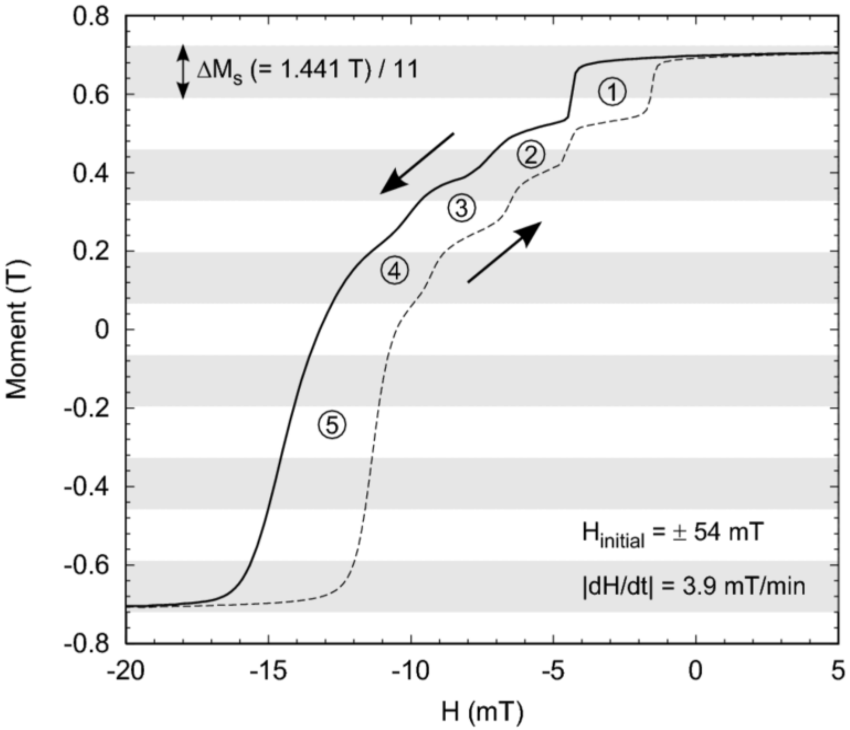
<100pm

Lorentz mode (field free)

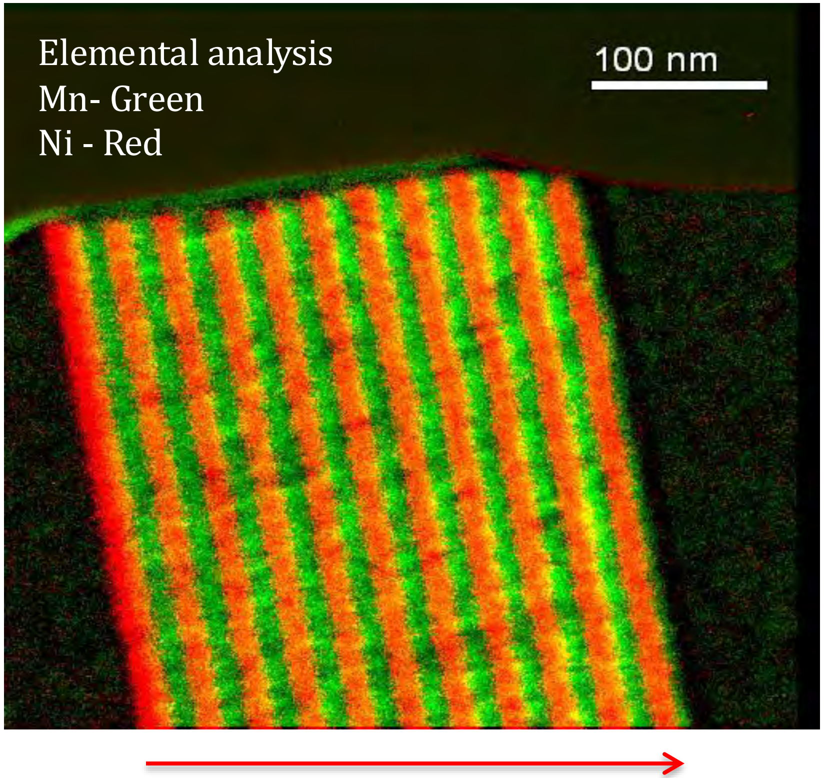
~1nm

Exchange bias in a multi-layered system

Si / SiO / NiFe(20nm)/[FeMn(15nm)/NiFe(20nm)] X 10/Ta



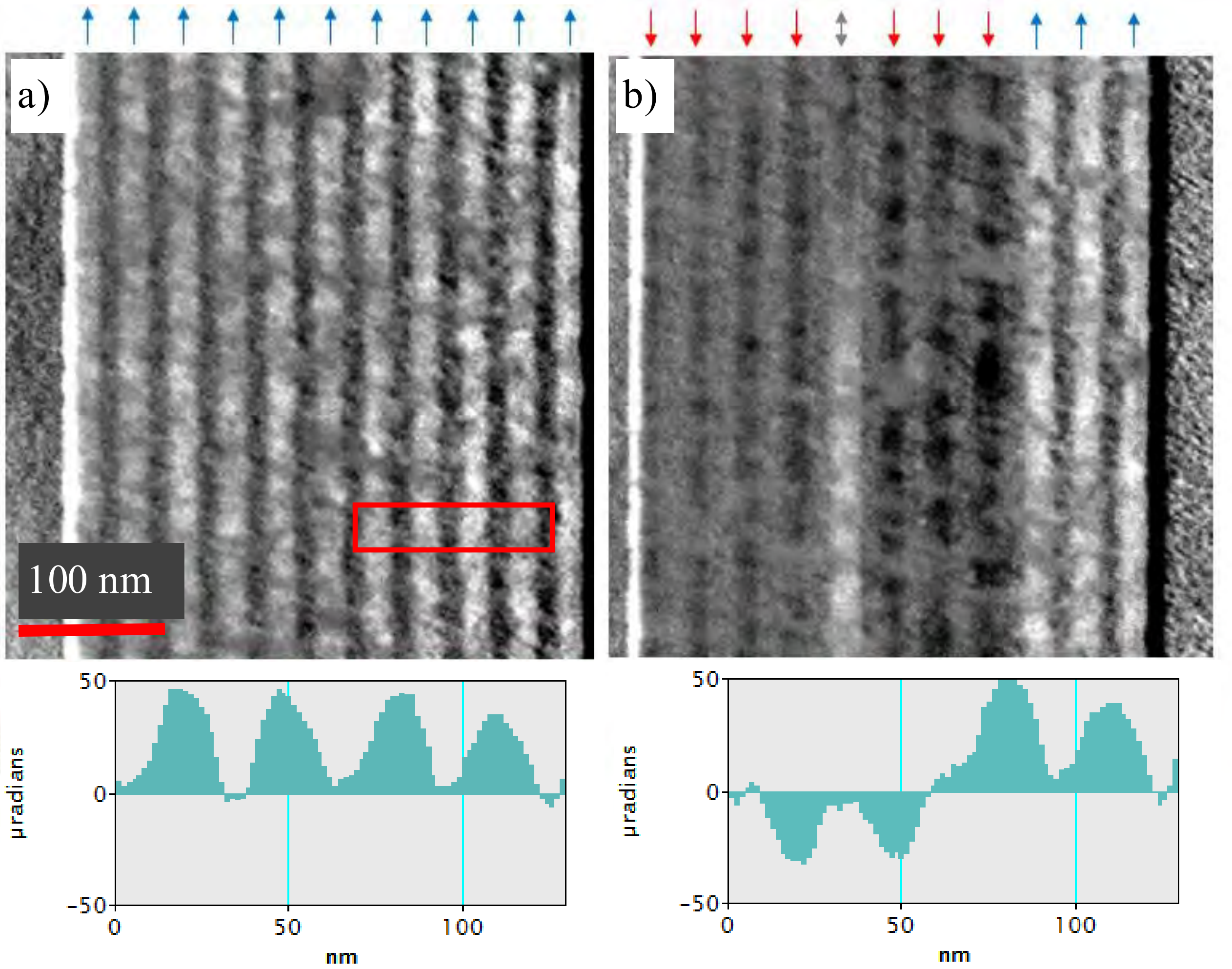
VSM



Cross-section

Exchange bias in a multi-layered system

Si/SiO/NiFe(20nm)/[FeMn(15nm)/NiFe(20nm)]×10/Ta



McVitie et al, Ultramicroscopy. **152**, 57, 2015

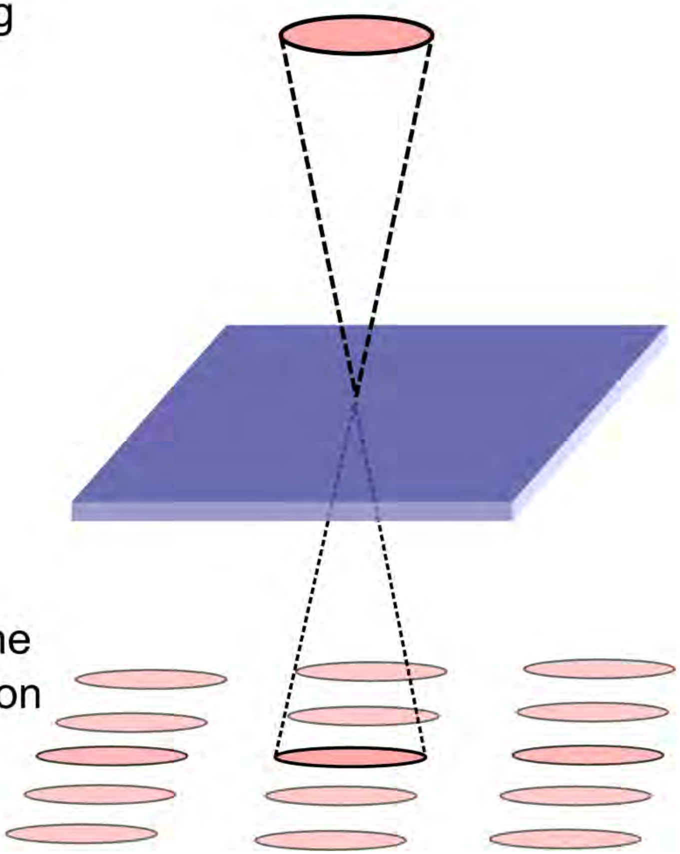
Challenges for magnetic DPC imaging

Differential Phase Contrast imaging becomes very difficult when dealing with:

- perpendicularly magnetised very thin films (<1 nm thick → **very small** Lorentz deflection)
- weak moment materials
- strong diffraction contrast from polycrystalline structure
- bend contours in single crystals

Diffraction effects

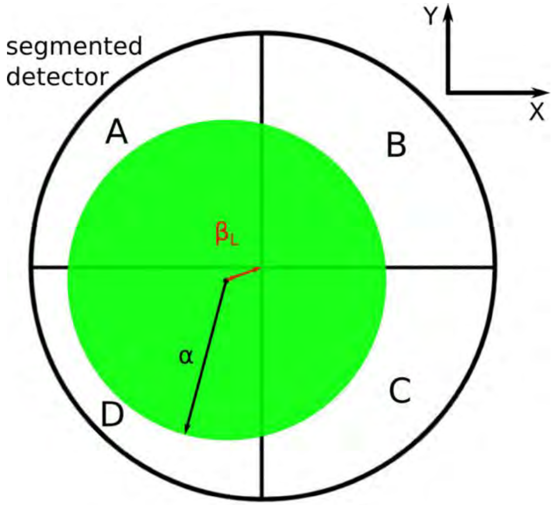
Differential scattering from randomly oriented grains

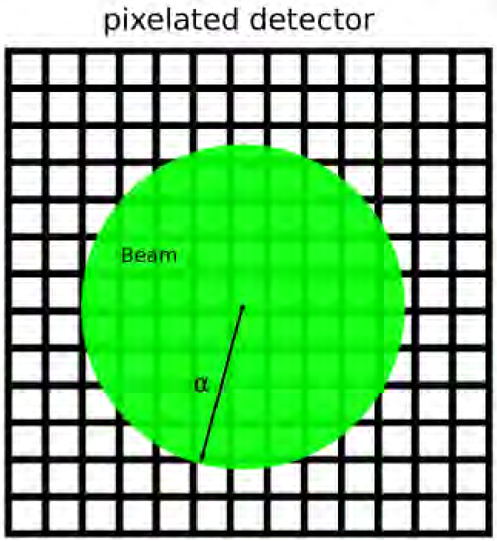


beam forming aperture

specimen

detection plane
Bragg diffraction



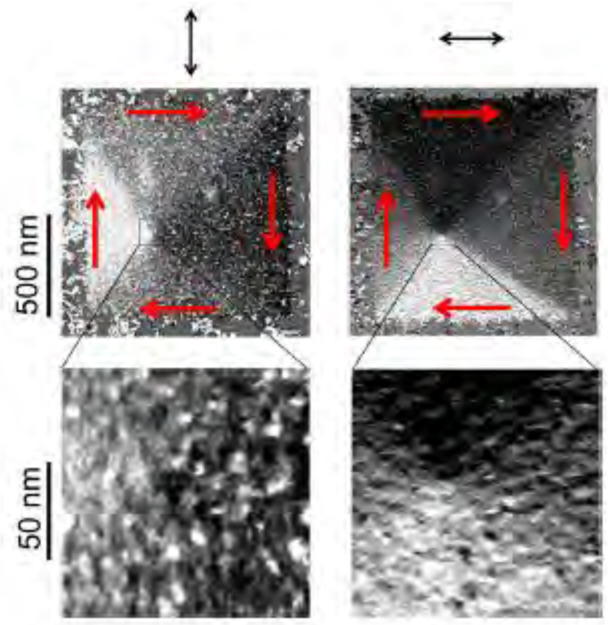


Highlighting the problem

Example, 30 nm thick permalloy square (1 µm)

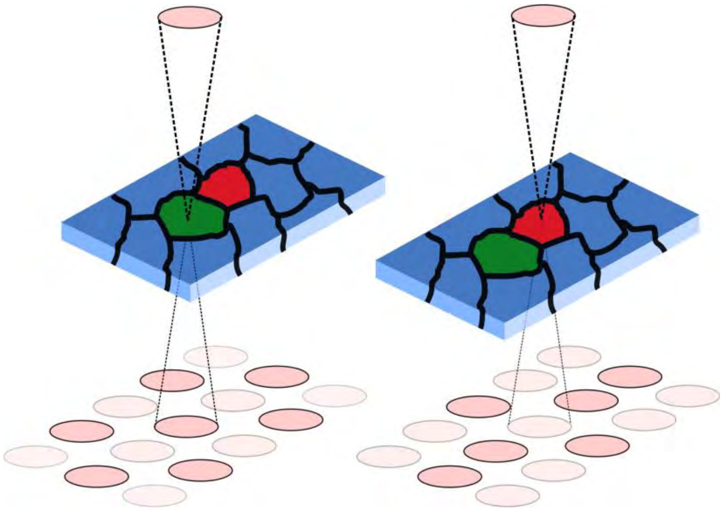
Low mag looks OK

Higher mag – diffraction contrast dominating (grains 5–10 nm).



Differently oriented grains:

- each has different scattering
- simple normalisation does not work

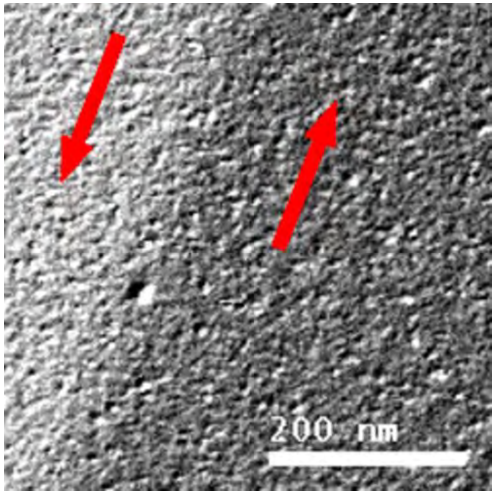


Diffraction recording

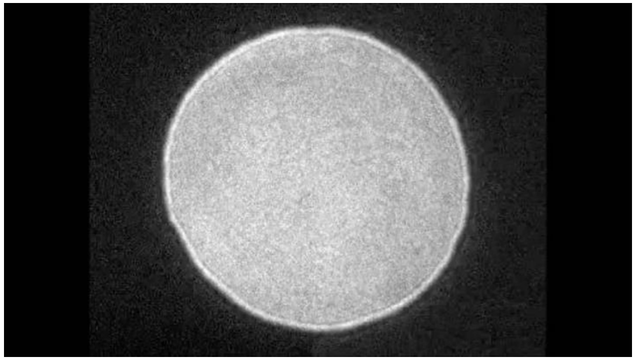
Look at 20nm thick permalloy film (doped with Pt) containing 180° domain wall.

Resolution around 3 nm.

Regular DPC image shown.



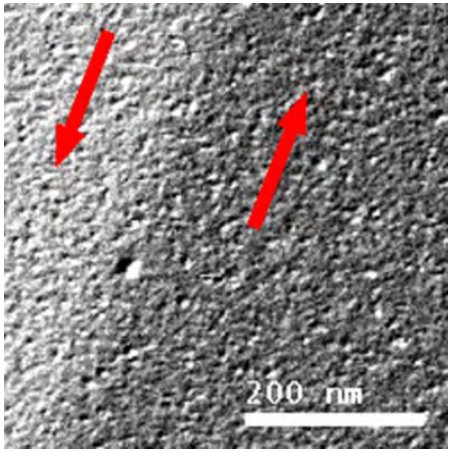
Pixelated diffraction pattern from this sample. Use Orius CCD camera. (~12 mins to acquire 100×100 pixel set)



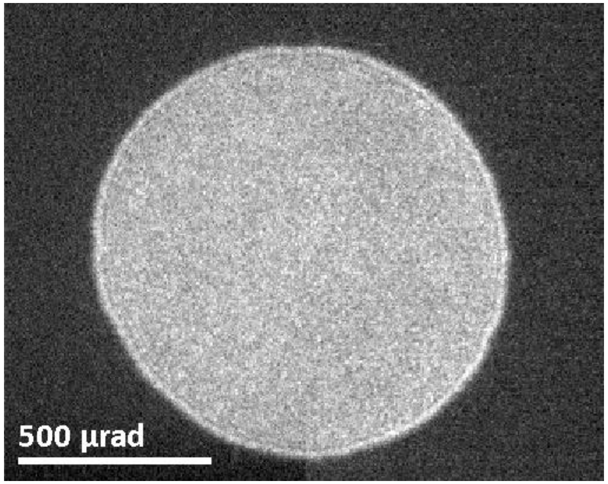
Processing of data

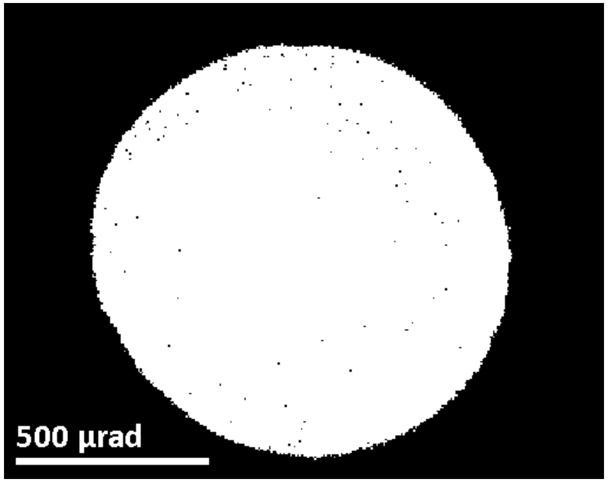
For the diffraction data set use two approaches to filter out high spatial frequency information:

- Thresholding and centre of mass
- Edge detection

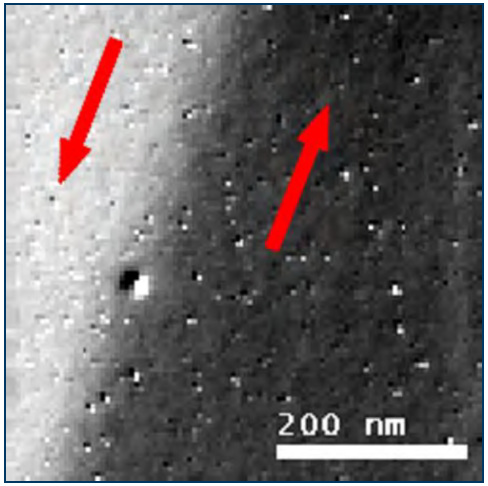


Threshold





Better



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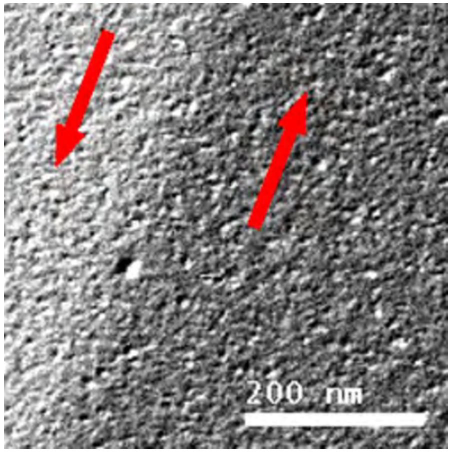
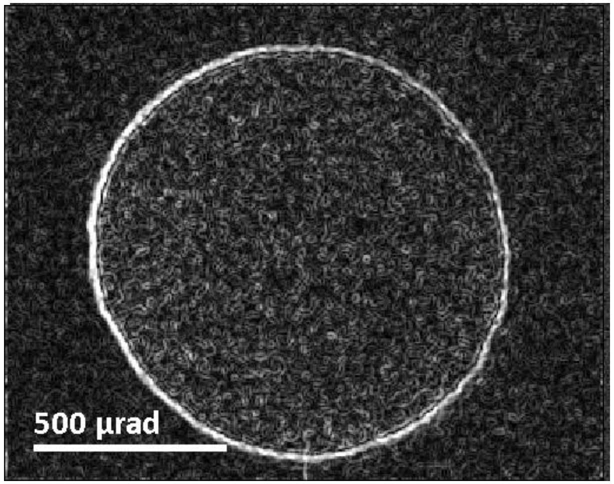
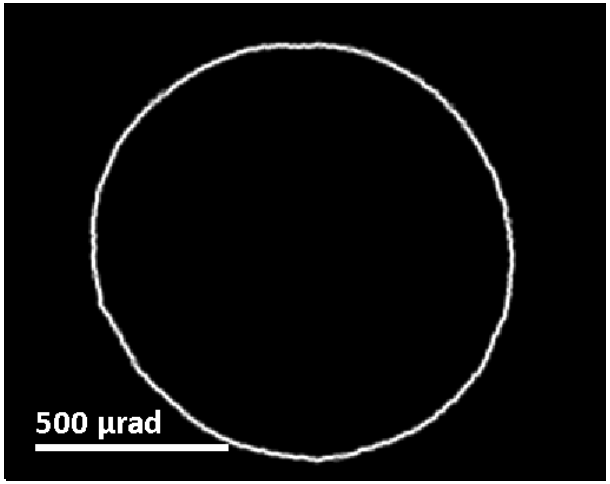
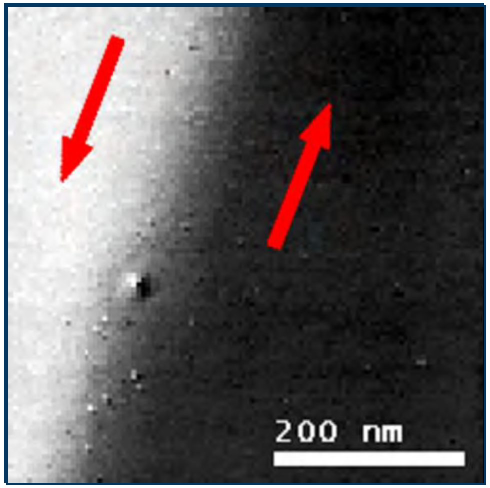


Cross correlation

Better

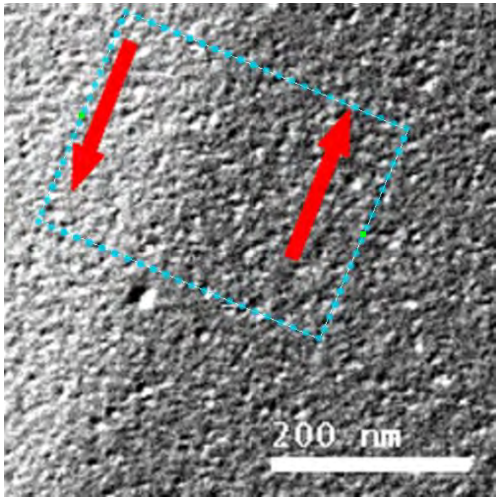


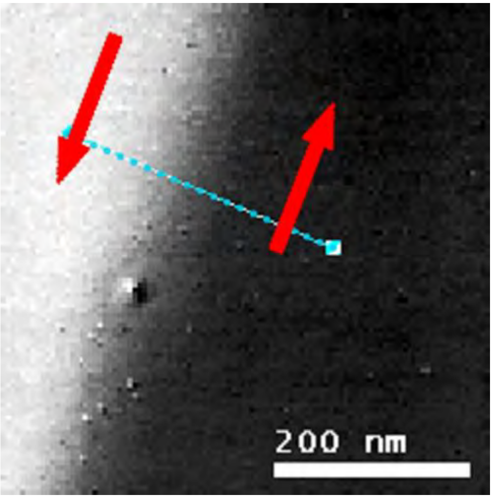


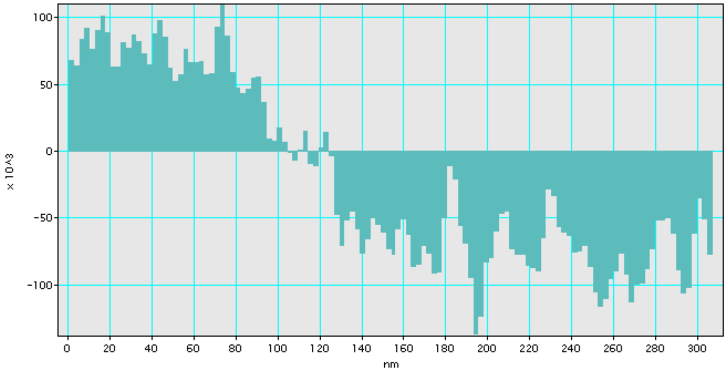


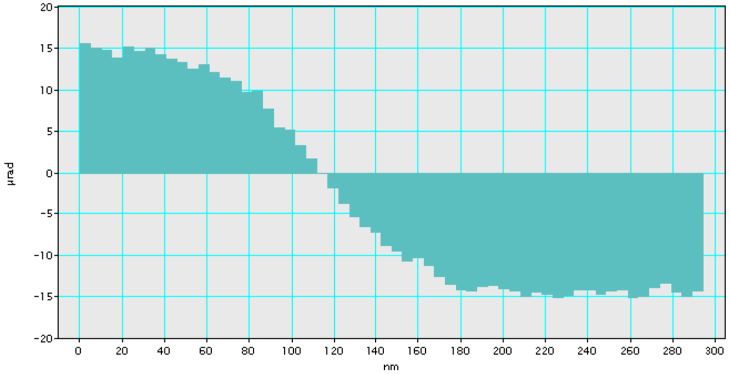
Quantitative comparison

Domain wall profile, DPC vs Pix DPC









Krajnak et al, Ultramicroscopy. **165**, 42, 2016

Summary

•Lorentz TEM allows imaging of domain structures with high resolution

•Simple (non-linear) domain geometry imaging and quantitative induction maps possible

•In-situ experiments allow response to an external field or applied current to be observed

•Often correlate magnetic structure with physical and chemical structure

•Developments in imaging allow weak signals to be measured

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