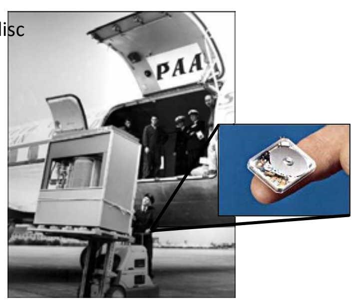
Frithjof Nolting :: Head of LSC :: Paul Scherrer Institut

X-ray Microscopy

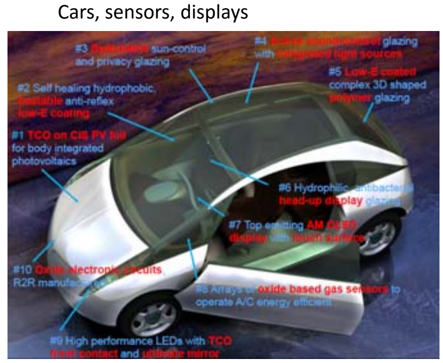
PSI Master School 2017

Basic research – electronic devices

Hard disc



Cars, sensors, displays



Modern communication devices are full of fascinating physics and advanced materials

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Aim of the lecture

Basic concepts of X-ray Microscopy, mainly soft X-rays
- General considerations
- Scanning Transmission X-ray Microscope (STXM)
- Photoemission Electron Microscope (PEEM)
- Contrast mechanism using XAS
- Be careful
- Hard X-ray techniques

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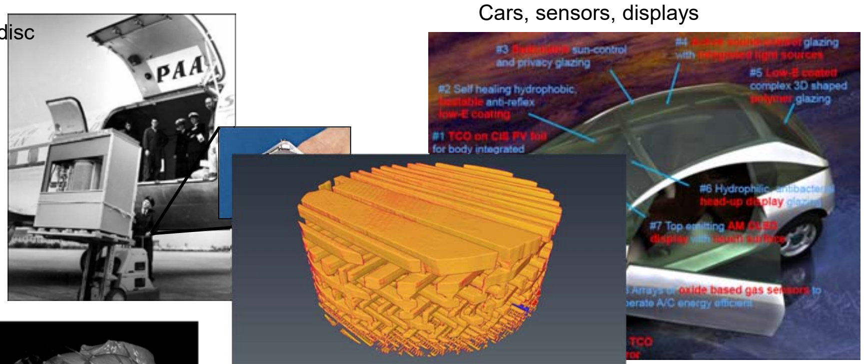
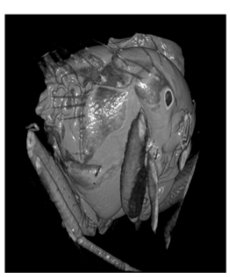
Basic research – electronic devices

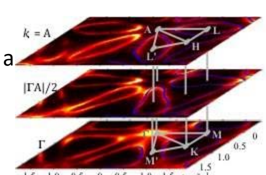
Hard disc

Cars, sensors, displays

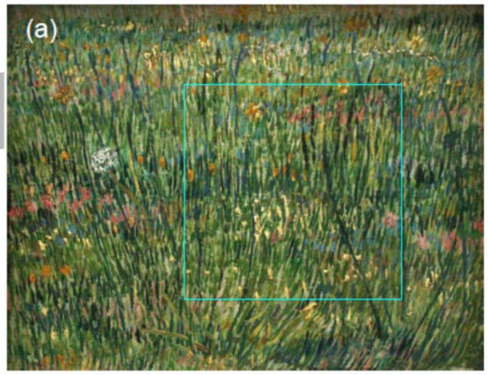


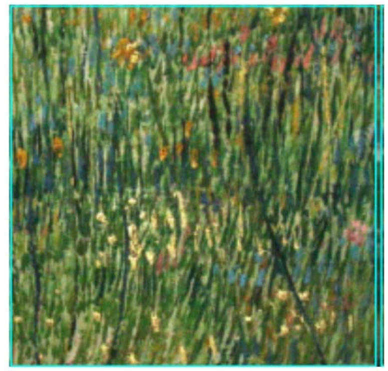


Modern communication devices a
physics and advanced materials



"Seeing the invisible"





The 1887 floral painting by van Gogh, "Patch of Grass".

Dik et al., Anal. Chem. 80 6436 (2008).

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How?

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X-ray microscope

source

X-ray tube

Synchrotron

- Bending magnet
- Insertion device

optics

Mirrors

Refractive elements

Diffractive elements

Electron optic

sample

detector

Photodiode

Phosphorscreen

…

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Source, Optics, and Detectors

Source, optics and detectors see lecture from Laura Heyderman on Monday

Insertion Device

Beam defining aperture

Soft x-ray filter/window

Beam-defining slits

Focussing & monochromator optics

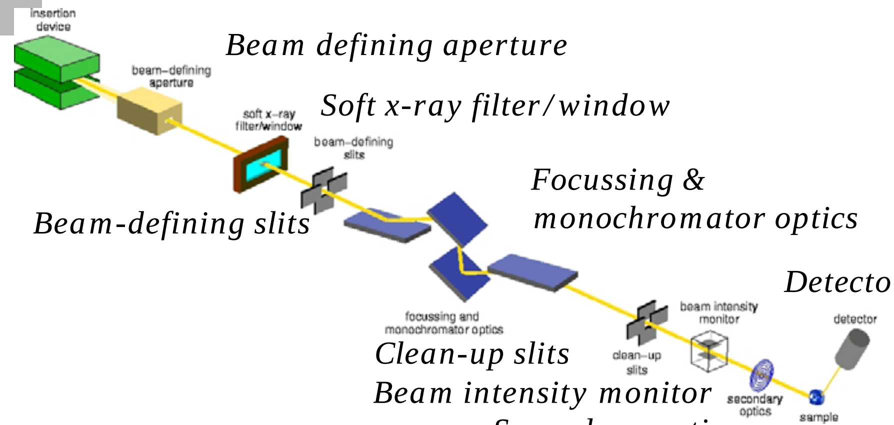
Detector

Clean-up slits

Beam intensity monitor

Secondary optics

Sample



P. Willmott
Intro to Synchr. Rad.

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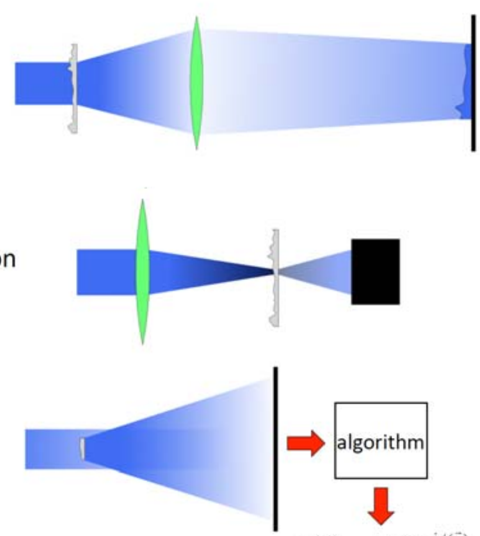
Mircoscopy modes

full field microscopy

scanning transmission microscopy

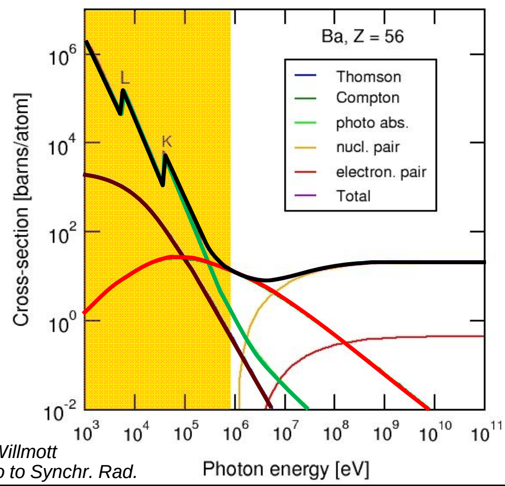
diffraction microscopy



$$\psi(\vec{r}) = \rho(\vec{r})e^{i\phi(\vec{r})}$$

Which contrast can we use?

- Cross-sections for various processes involving interaction of x-rays with matter - primary scatterer is the **electron**
- Plot for Ba; orange area highlights upper energy range covered by synchrotron sources



P. Willmott
Intro to Synchr. Rad.

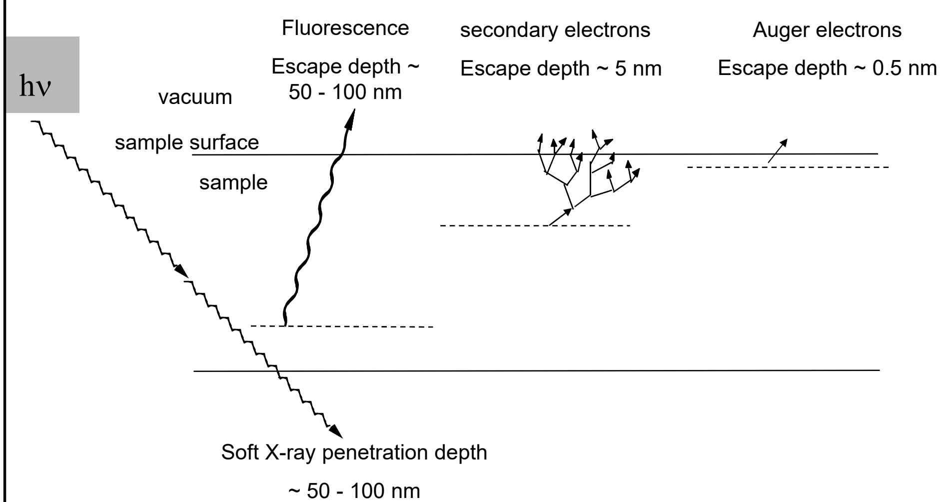
Key scattering processes:

- **Photoelectric Absorption**
- **Thomson Scattering (elastic)**
- **Compton Scattering (inelastic)**

➔ Total !

1 barn = 10^{-24} cm^2
= 10^{-28} m^2

X-ray microscope types



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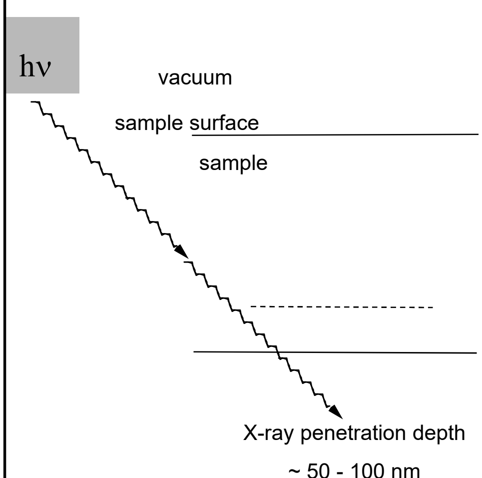
Photon in / Photon out

Photon in / Photon out

You need an optic for the (incoming) photons



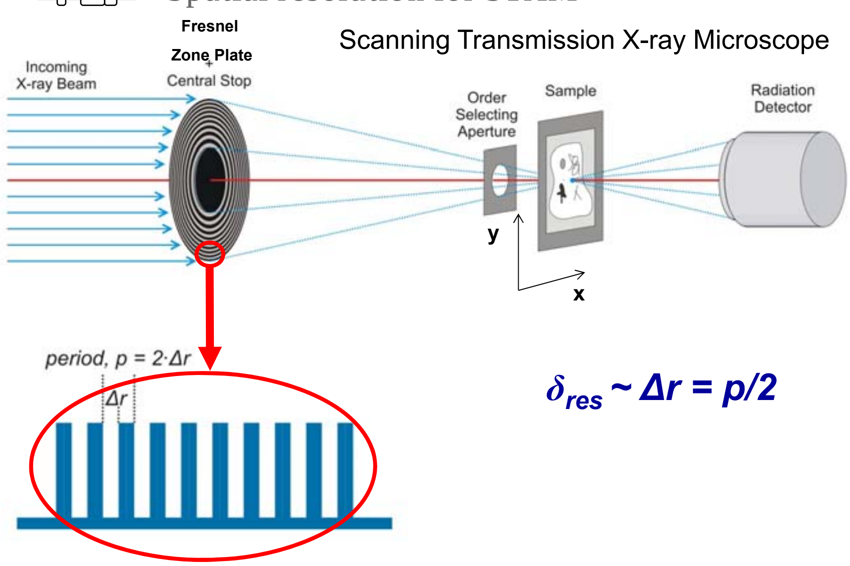
Scanning Transmission X-ray microscope

Focus X-rays

Scan sample

Detect X-rays

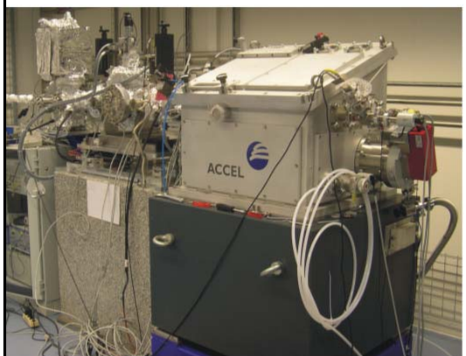
Spatial resolution for STXM

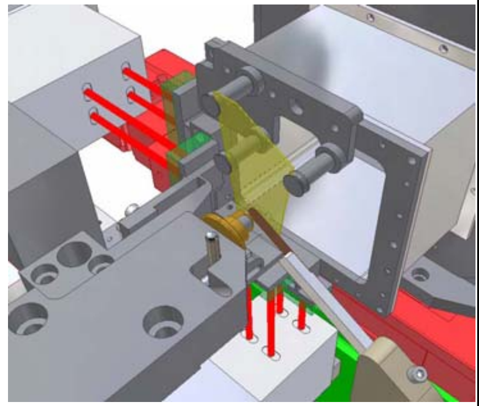


$\delta_{res} \sim \Delta r = p/2$

Courtesy J. Vila-Comamala (PSI)

STXM at the PolLux beamline at the SLS

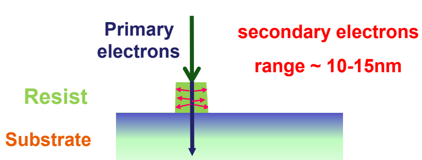


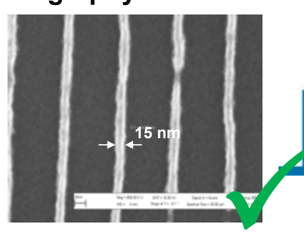


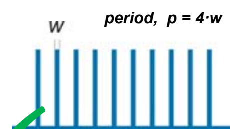
What limits the spatial resolution in STXM

Limits of e-beam lithography FZP Fabrication

Isolated line exposure

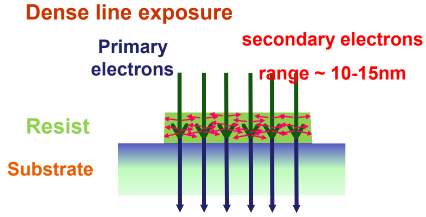


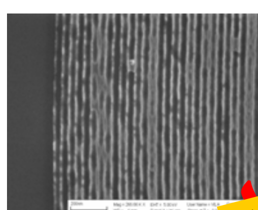


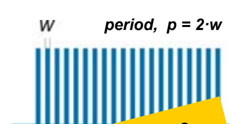


Isolated 10 — 20 nm lines

Dense line exposure







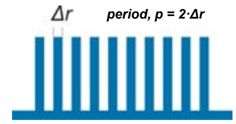
Dense 10 — 20 nm lin

Limits resolution in x-ray microscopy to about 25 nm

Courtesy J. Vila-Comamala (PSI)

Zone-doubling for high resolution

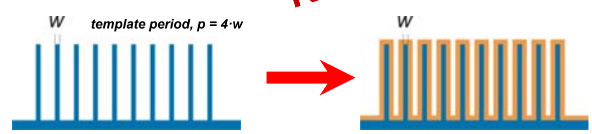
Ordinary FZP



$\delta_{res} \sim \Delta r = p/2$

Resolution 2x

Zone-doubled FZP



Low refractive index material
High refractive index material

$\delta_{res} \sim w = p/4$

Manufacturing advantage: ✓ **One single EBL exposure: No alignment required**

FZP pattern generation is simple and reproducible!

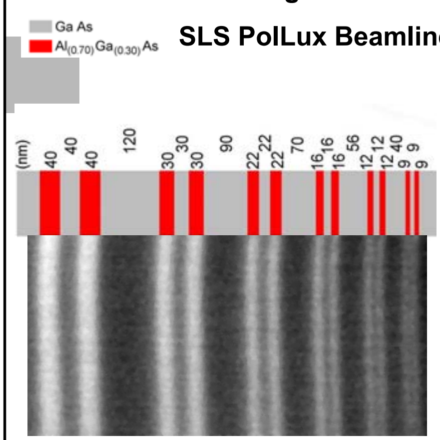
K. Jefimovs, J. Vila-Comamala, T. Pilvi, J. Raabe, M. Ritala, C. David. *Physical Review Letters* **99** (2007) 264801

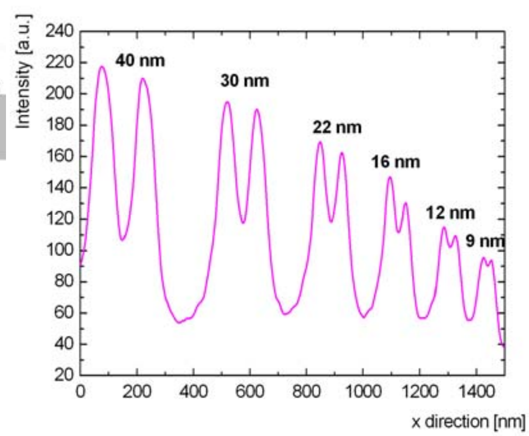
Courtesy J. Vila-Comamala (PSI)

Resolution test

STXM images of GaAs / AlGaAs heterostructures

SLS PolLux Beamline @ 22th September 2008





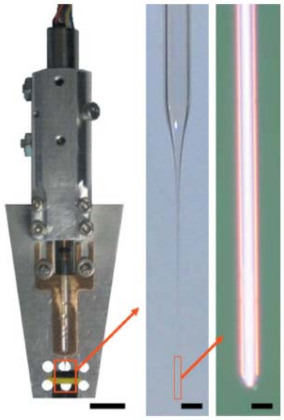
Courtesy J. Vila-Comamala (PSI)

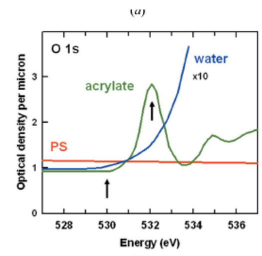
3D chemical imaging

Three-dimensional chemical mapping by scanning transmission X-ray spectromicroscopy

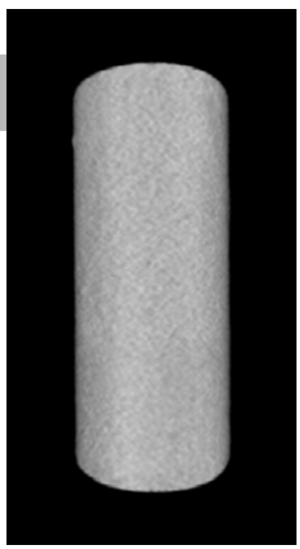
Göran A. Johansson,[a*] Tolek Tyliszczak,[b] Gary E. Mitchell,[c] Melinda H. Keefe[d] and Adam P. Hitchcock[a]

J. Synchrotron Rad. (2007). **14**, 395–402





3D chemical imaging



The technique is illustrated using mapping of a lowdensity acrylate polyelectrolyte in and outside of polystyrene microspheres dispersed in water in a 4 μm-diameter microcapillary. The 3-d chemical visualization provides information about the microstructure that had not previously been observed.

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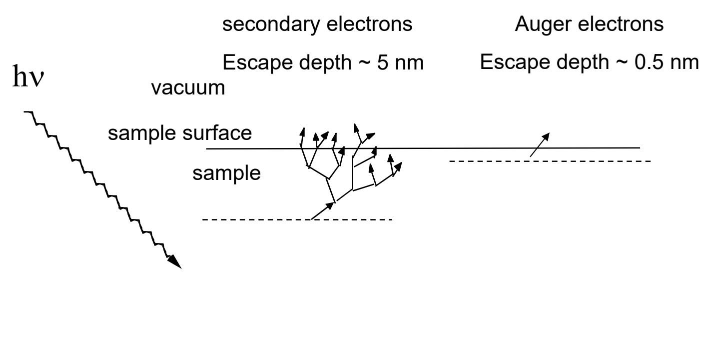
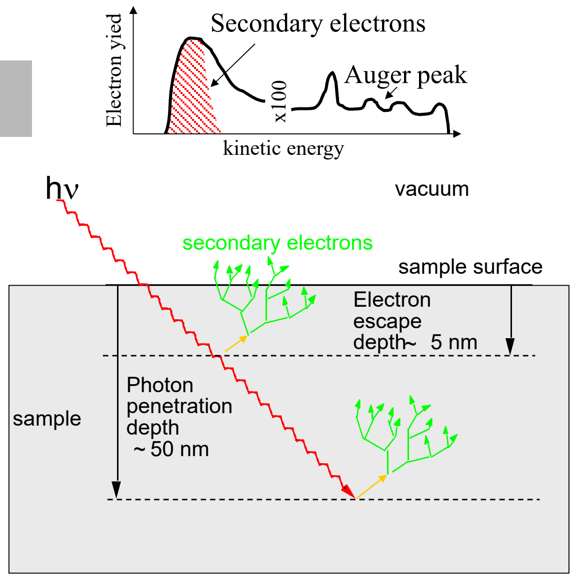
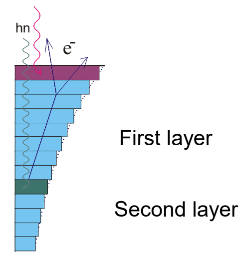
Photon in / Electron out



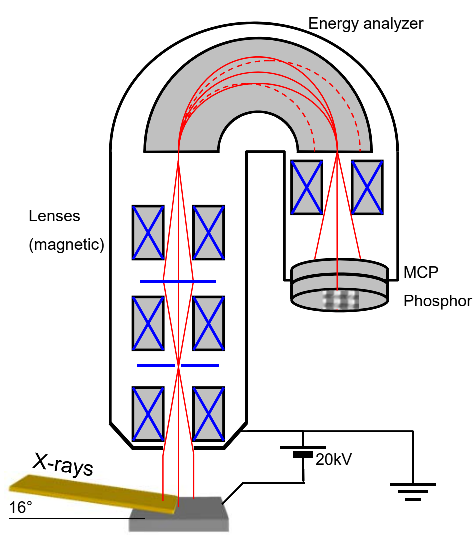
You need an optic for the electrons



Probing surface/interface



Photoemission Electron Microscope - PEEM

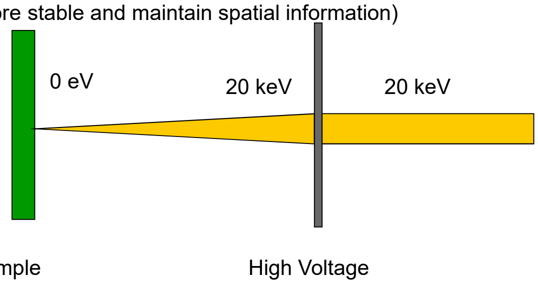


Slow electrons

Probe : slow electrons

Imaging : high energy electrons

(more stable and maintain spatial information)

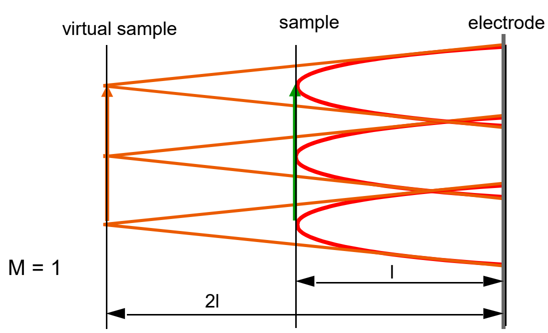


Immersion lens: electrons have before and after the lens different velocity (different wavelength)

Cathode lens: Sample is cathode

electron microscope is anode

Why is the accelerating field a lens?



Accelerated electrons form parabolic trajectory

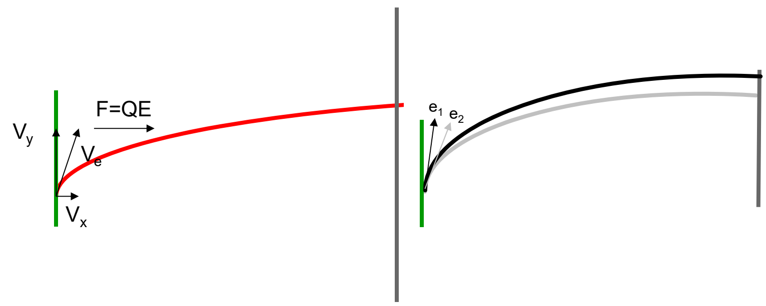
Tangents to parabolas are the incident rays

Extrapolated backwards form a virtual image at unit lateral magnification

Just another lens?

Classical: electron in homogenous electric field

calculate electron trajectory



Trajectory depends on emission angle and velocity

No, it is a very important lens in a PEEM, dominating the spatial resolution due to its spherical and chromatic aberrations.

Spatial resolution PEEM – make an estimate

The spatial resolution (r) in PEEM can be approximated by

$r \approx (d\ \Delta E) / (eU)$

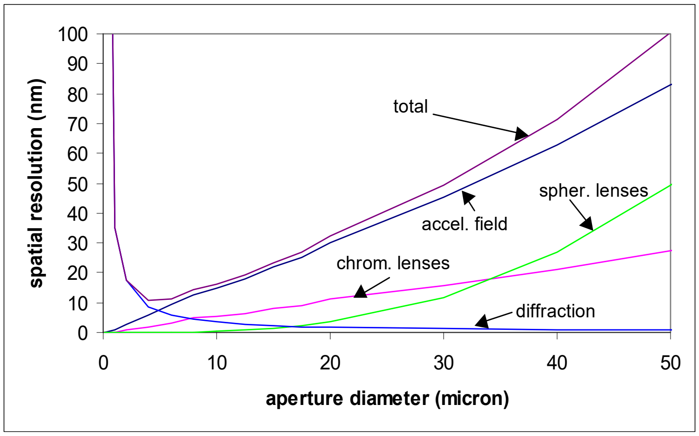
d:	distance sample, objective lens
ΔE:	energy spread of electrons
U:	acceleration voltage

The spatial resolution is given by the chromatic and spherical aberrations.

Chromatic aberrations are due to the energy distribution of the electron, with increasing energy spread is the spatial resolution decreased.

Spherical aberrations are due to the angular distribution of the electrons, which is reduced by the acceleration field (U/d).

Calculated Spatial Resolution



PEEM 2 at the ALS, Simone Anders

Work function 4 eV, sample voltage 30 kV, X-rays

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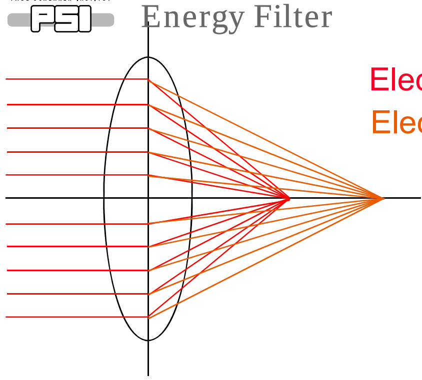
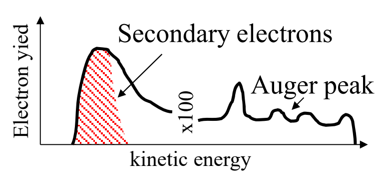
Energy Filter



Electron energy E

Electron energy E+ΔE



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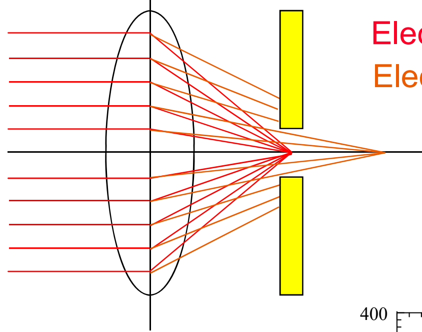
Energy Filter



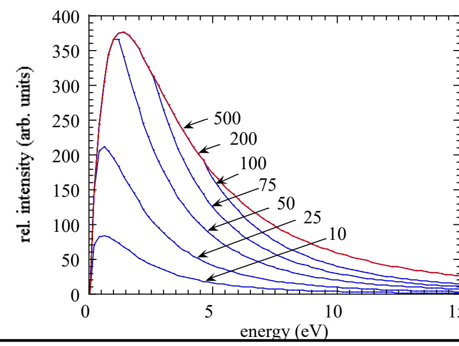
Electron energy E

Electron energy E+ΔE

Aperture cuts off transmission of electrons

with higher energy

Energy distribution is narrowed but transmission is reduced

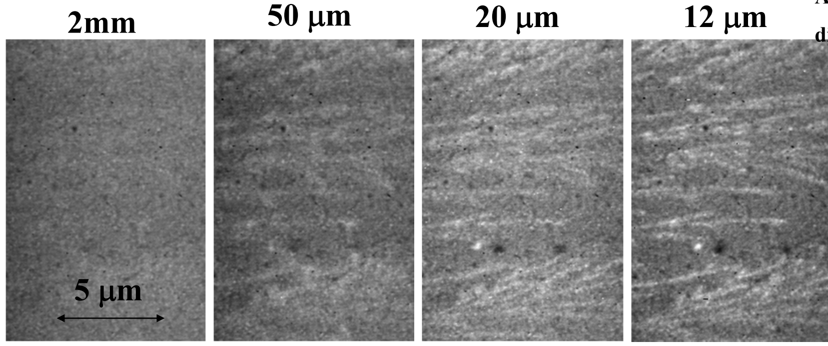


Effect of Aperture Size on Resolution

Spatial resolution depends on aperture size - limits pencil angle of transmitted electrons and transmission

Highest resolution is achieved with 12 mm aperture for PEEM2, ALS

Aperture diameter	2mm	50 μm	20 μm	12 μm



Exposure time	0.4 s	1s	4.2 s	10 s
Transmission	100 %	39 %	9 %	4 %

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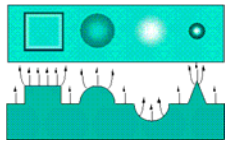
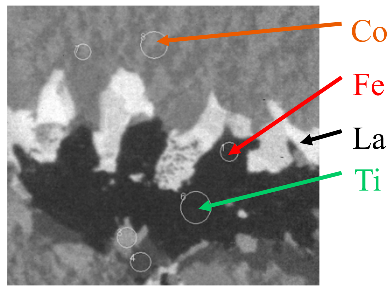
Photoemission Electron Microscope

Topographical Contrast



Elemental Contrast



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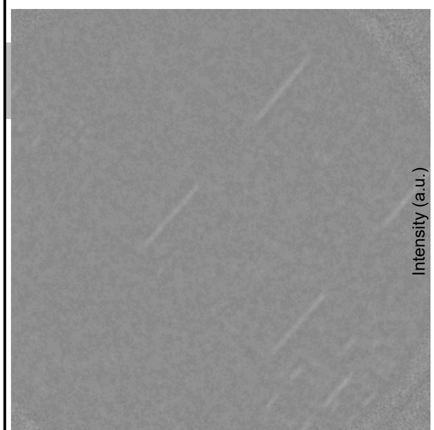
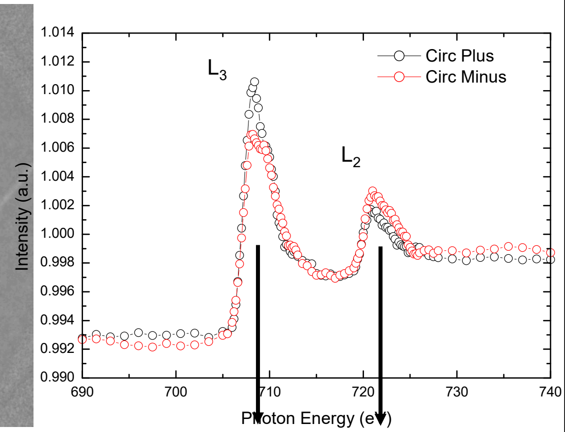
Spectra of individual Fe nanoparticles

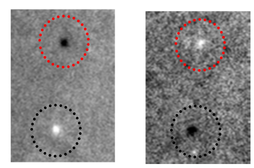
Sample: Fe nanoparticles with diameter = 9 nm





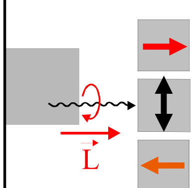
Images with increasing Photon Energy

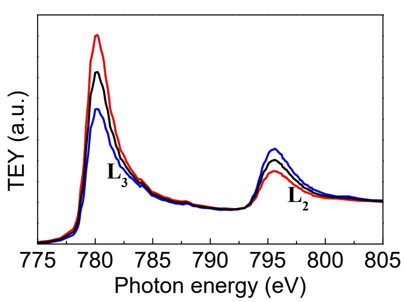
XMCD image



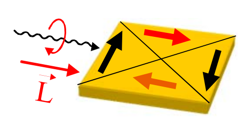
A. Fraile Rodríguez et al. PRL 104, 127201 (2010)

X-ray Magnetic Circular Dichroism (XMCD)



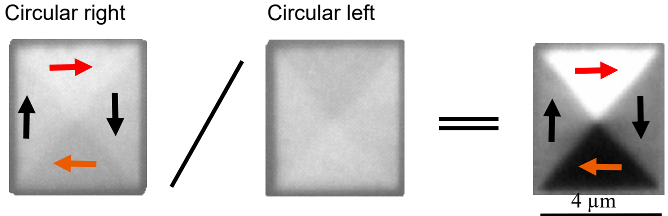


XMCD ~ <**M**> cos(**M**,**L**)



Circular right

Circular left

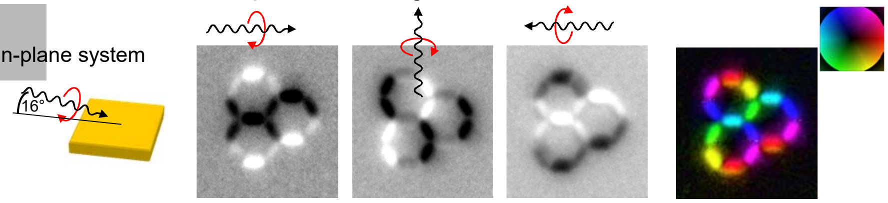


e.g. J. Stohr et al Surface Rev. Lett. **5**, 1297 (1998).

X-ray Magnetic Circular Dichroism (XMCD)

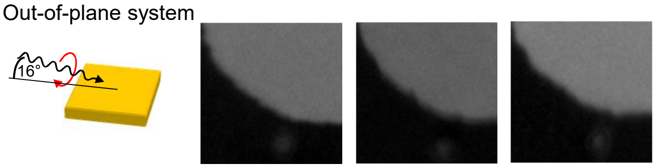
0 degree 90 degree 180 degree 2D magnetization map

In-plane system



Co structures (600 nm)

Out-of-plane system



20nm GdFeCo film

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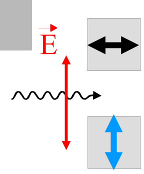
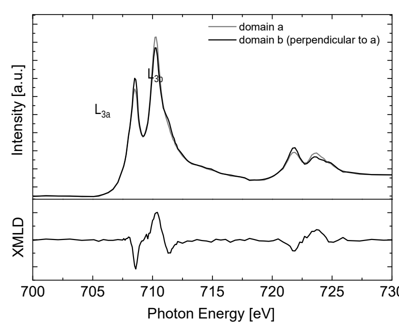
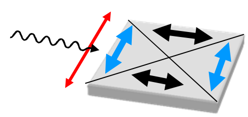
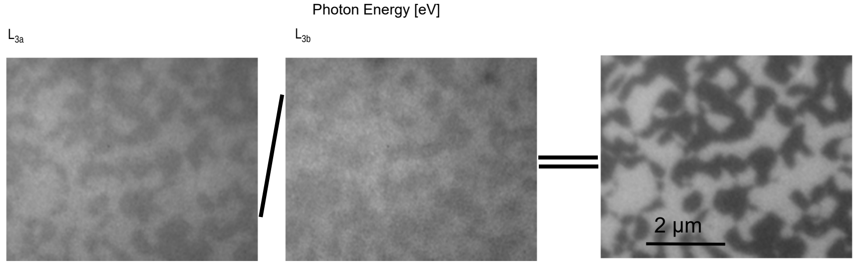
X-ray Magnetic Linear Dichroism (XMLD)





XMLD ~ <**M²**>





e.g. A. Scholl et al Science **287**, 1014 (2000)

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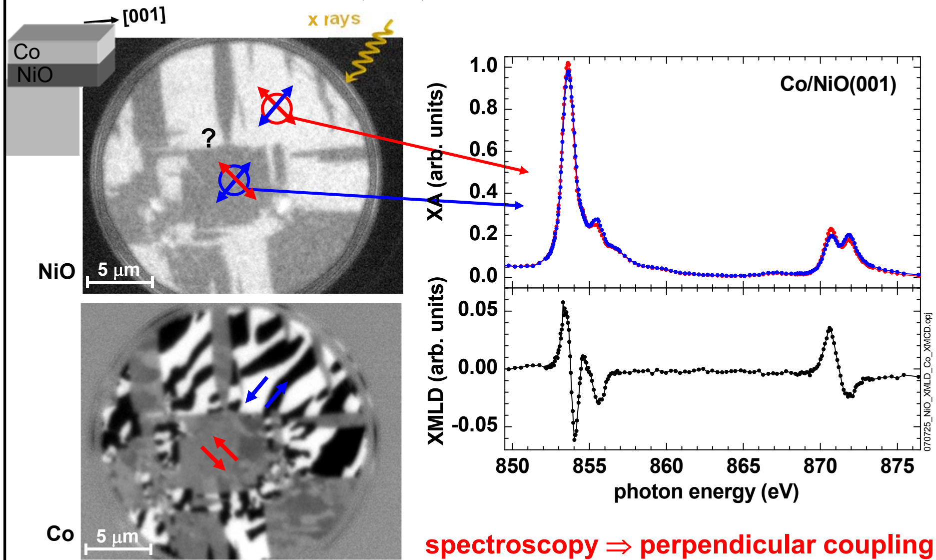
Co / NiO(001)

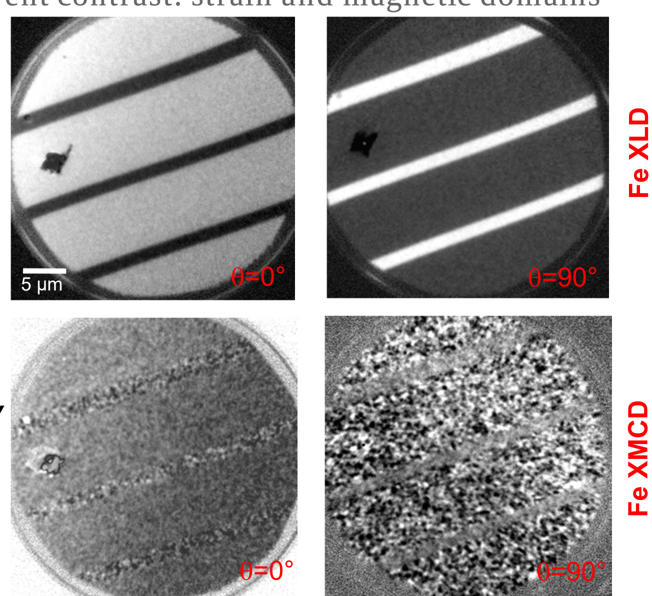


spectroscopy ⇒ perpendicular coupling

Different contrast: strain and magnetic domains

- XMCD image is sensitive to one axis of magnetization
- XLD image is sensitive to an axis of anisotropy

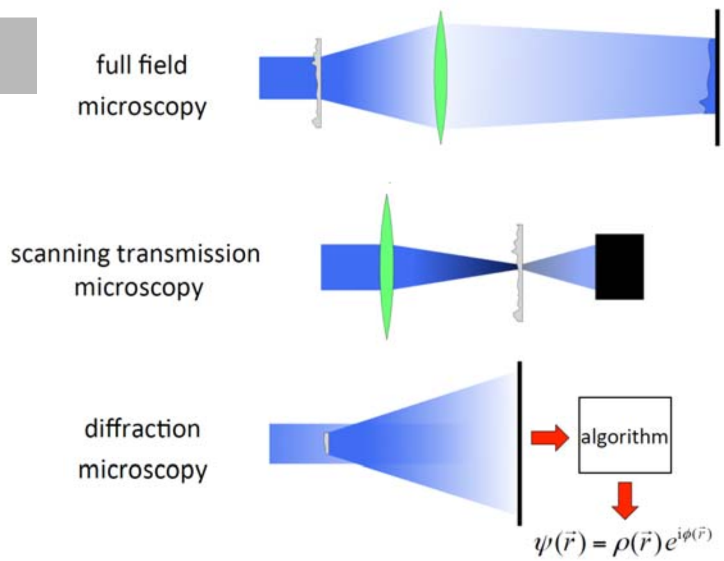
Strong modification of anisotropy directly probed with XLD!



⟷ Sensitivity axis for magnetization ↕

R.V. Chopdekar et al. Phys. Rev. B 86, 014408 (2012).

Non resonant absorption



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Aim of the lecture

Basic concepts of X-ray Microscopy, mainly soft X-rays

- General considerations
- Scanning Transmission X-ray Microscope (STXM)
- Photoemission Electron Microscope (PEEM)
- Contrast mechanism using XAS
- Be careful
- Hard X-ray techniques

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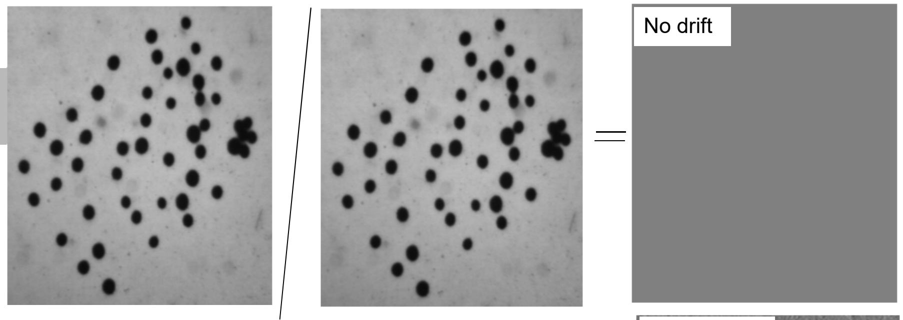
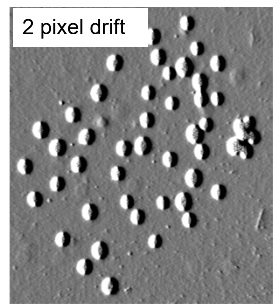
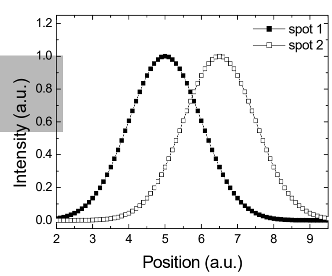
Be critical: Image drift!

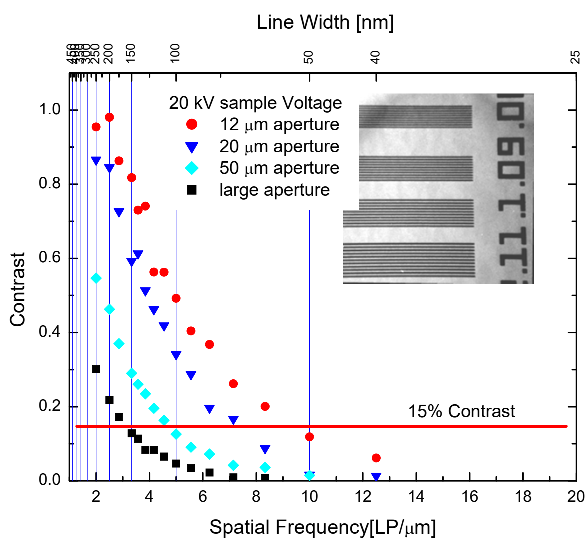


Don‘t forget to get pictures which one can publish



Be critical: spatial resolution





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Aim of the lecture

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Wilhelm Röntgen – hard X-rays

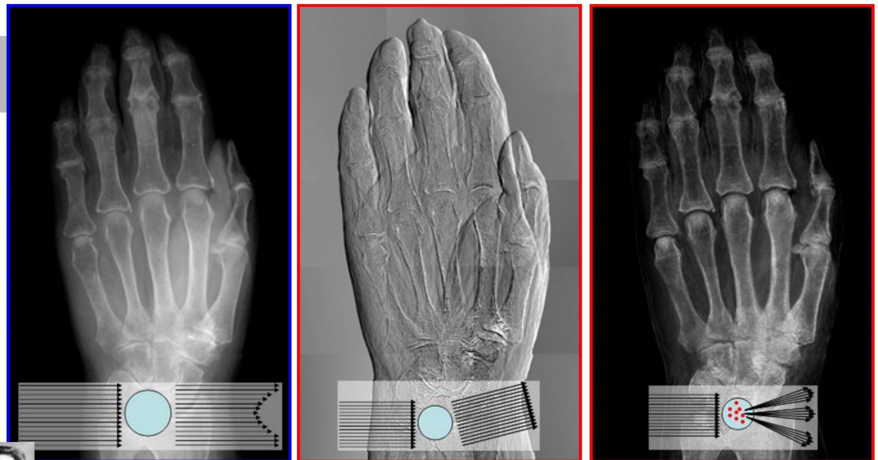
1895 Discovery of X-rays by Wilhelm Röntgen

1901 Nobel prize in physics

Image of hand of Albert von Kölliker

this is the second image, the first one, very similar is said to be the hand of his wife

Grating interferometry



Absorption image

Conventional radiography

(since 1895…)

“Differential Phase image”

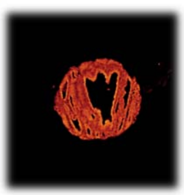
“Scatter image”

Index of refraction: n = 1 - δ + iβ

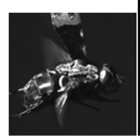
$\Phi = 2\pi\delta(\lambda)/\lambda$ Phase Absorption $\mu = 4\pi\beta(\lambda)\lambda$

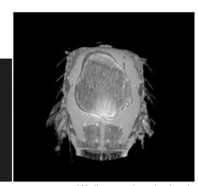
Courtesy Federica Marone

From static to dynamical, quantitative imaging



P. Donoghue *et al.*, Nature 442, Aug. 2006



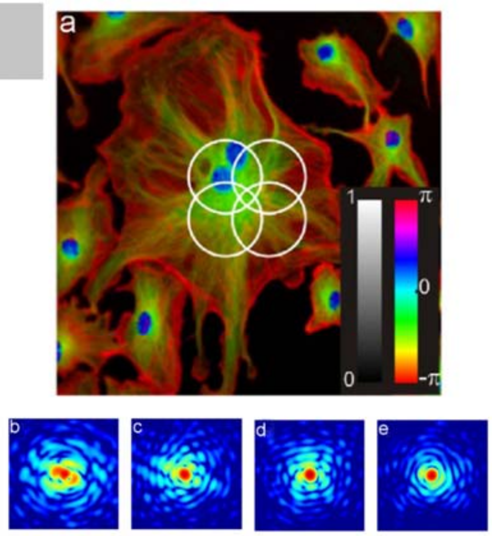


Walker *et al.*, submitted

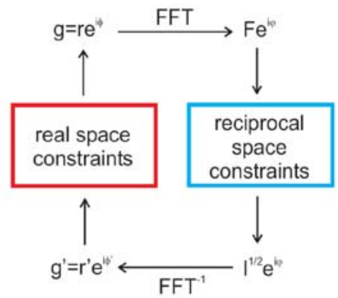
3D to 4D microscopy

Ptychography

Coherent diffraction patterns from overlapping regions of the specimen



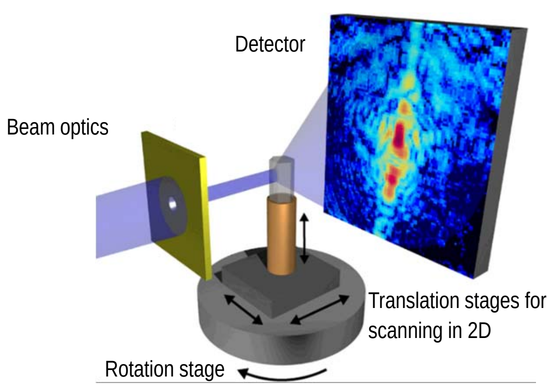
Phase retrieval algorithms to reconstruct complex-valued transmissivity



H. M. L. Faulkner & J. M. Rodenburg, Phys. Rev. Lett. **93** (2004) 023903

- Extended objects
- No limit on object size
- Robust convergence

Scanning X-ray diffraction microscopy - Tomography



OMNY project

→ explore limits of X-ray tomography

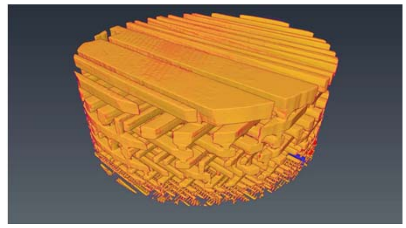
→ goal: sub-10 nm 3D resolution

→ require sub-10nm positioning accuracy!

Nano Tomography

3-D X-ray imaging makes the finest details of a computer chip visible

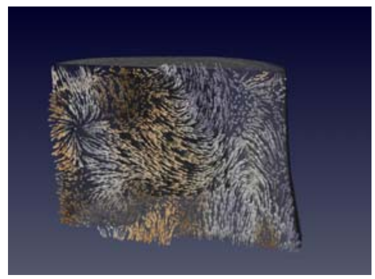
Commercially available computer chip (about 10 micrometres)



Mirko Holler, Manuel Guizar-Sicairos, Esther H. R. Tsai, Roberto Dinapoli, Elisabeth Müller, Oliver Bunk, Jörg Raabe, Gabriel Aeppli, Nature 16 March 2017, DOI: 10.1038/nature21698

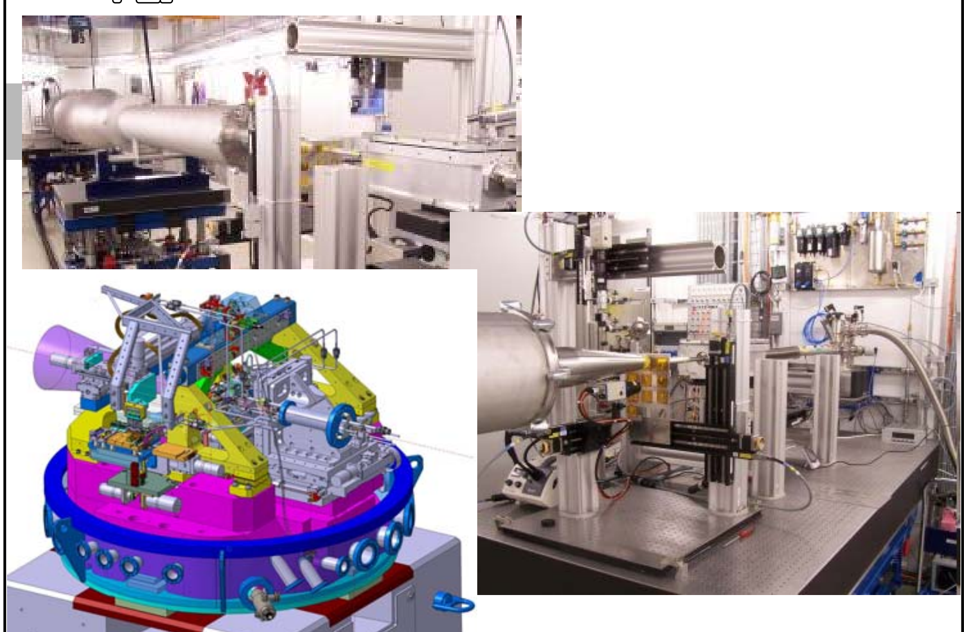
First-time 3D imaging of internal magnetic patterns

Micrometre-sized pillar (about 1 micrometer) made of gadolinium-cobalt



Claire Donnelly, Manuel Guizar-Sicairos, Valerio Scagnoli, Sebastian Gliga, Mirko Holler, Jörg Raabe, Laura J. Heyderman, *Nature* 20 July 2017, DOI: 10.1038/nature23006

CSAXS beamline at the SLS



Conclusion: Scanning Transmission X-ray microscope

Focus X-rays

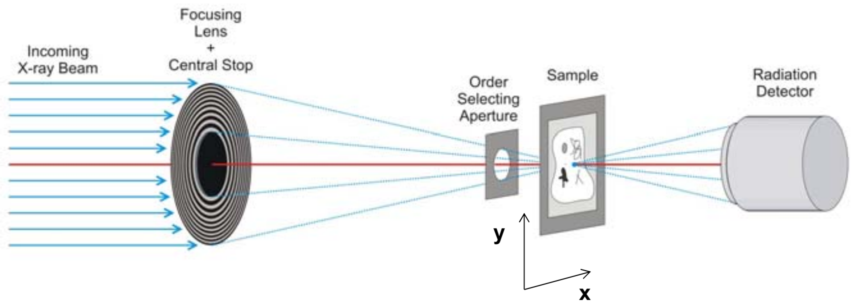
determine the spatial resolution (determined by the outer zone width)

Scan sample

precision of scanning also important for the resolution

Detect X-rays

can be large detector



Conclusion: Photoemission Electron Microscope – PEEM

Components:

X-ray (moderated focused)

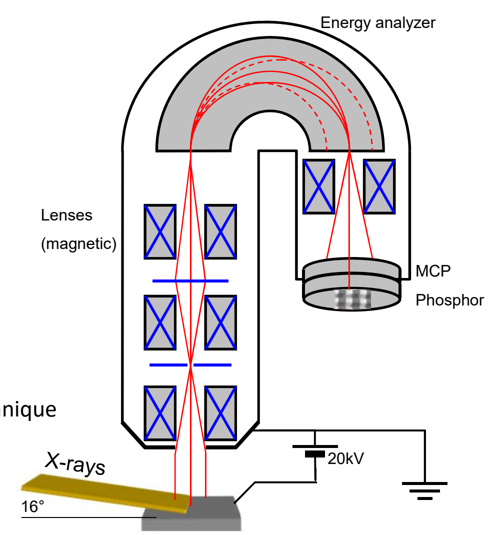
Accelerating field

Electron optic

Maybe energy filter

Detector

Focus of X-ray not determining
the spatial resolution, full field technique



The spatial resolution (approximation)

$$r \approx (d\ \Delta E) / (eU)$$

d: distance sample, objective lens

ΔE: energy spread of electrons

U: acceleration voltage

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Conclusion: Scanning X-ray diffraction microscopy - Tomography