

# X-Ray Microscopy Techniques



PAUL SCHERRER INSTITUT



The Swiss Light Source, Paul Scherrer Institut

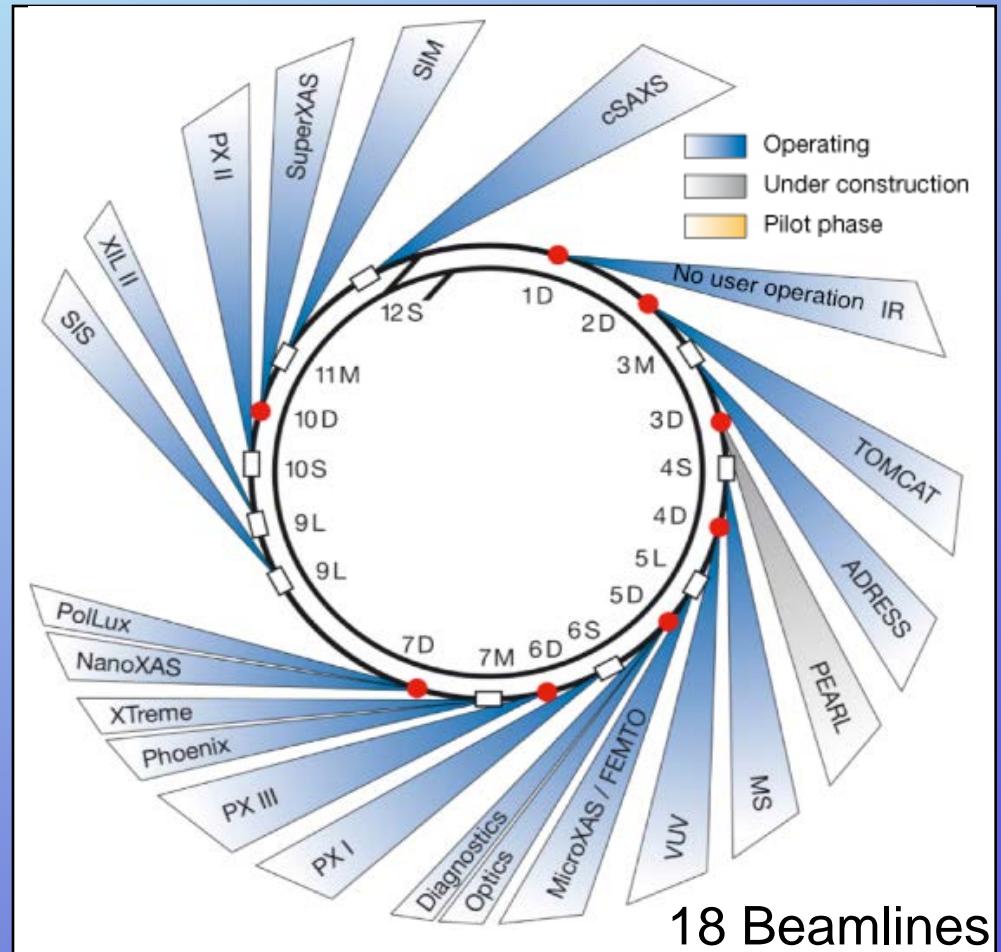
- The electrons are accelerated close to the speed of light in a linear accelerator and injected into the storage ring
- Bending magnets or insertion devices (wigglers or undulators) cause electrons to bend or wobble through the section and emit light.

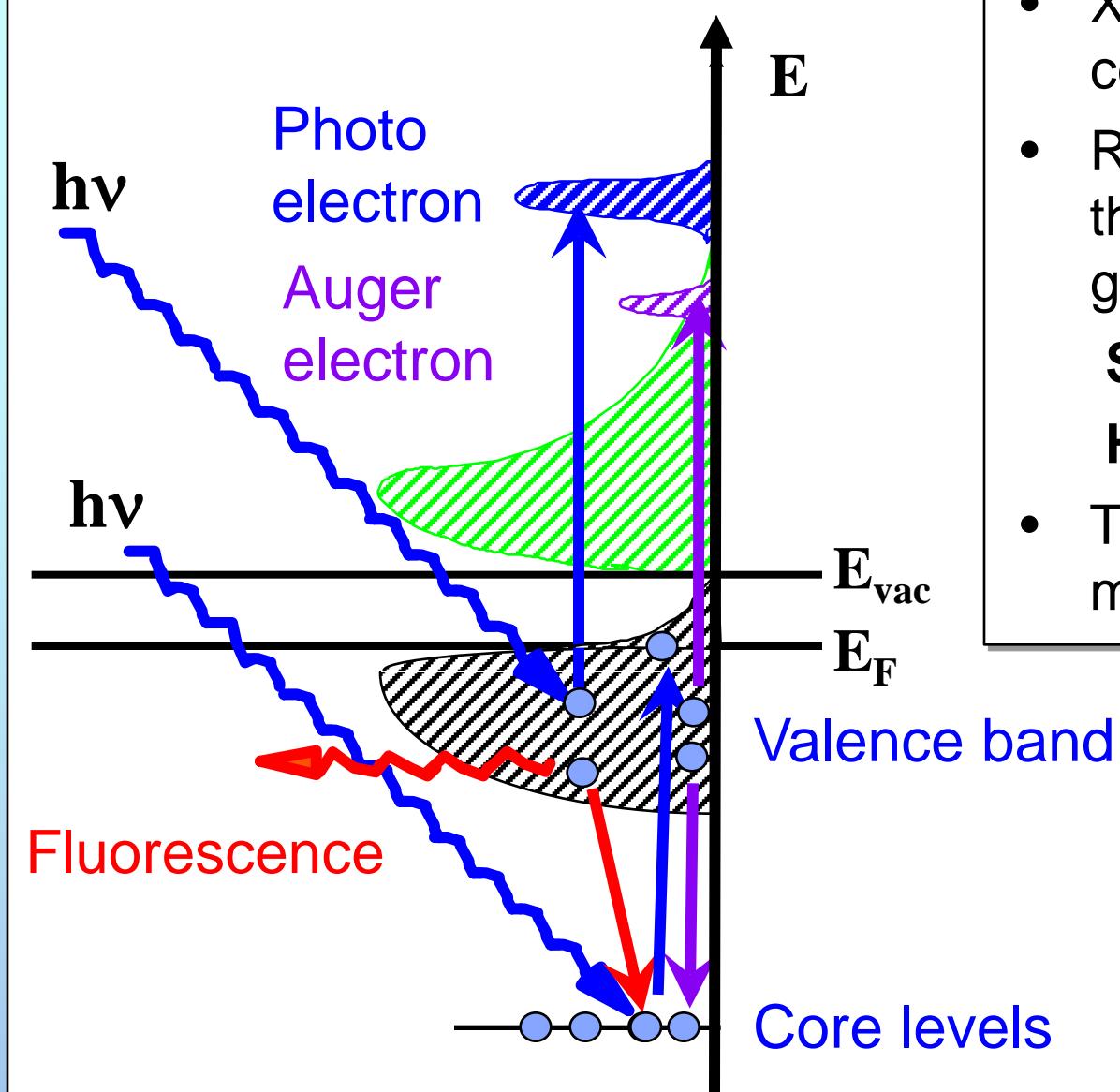


**Reference energy: 2.4 GeV**

**Circumference: 288 m**

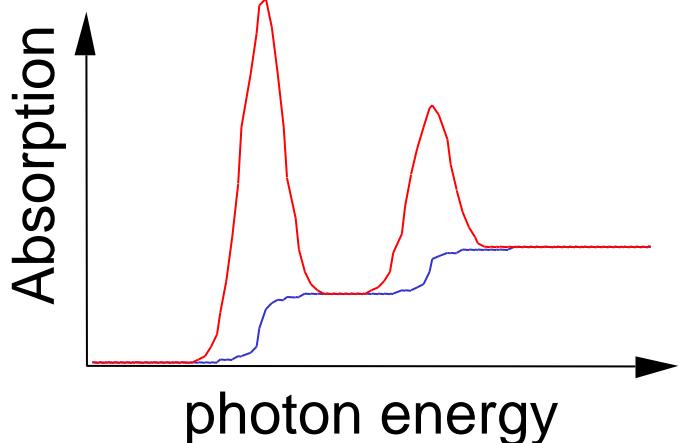
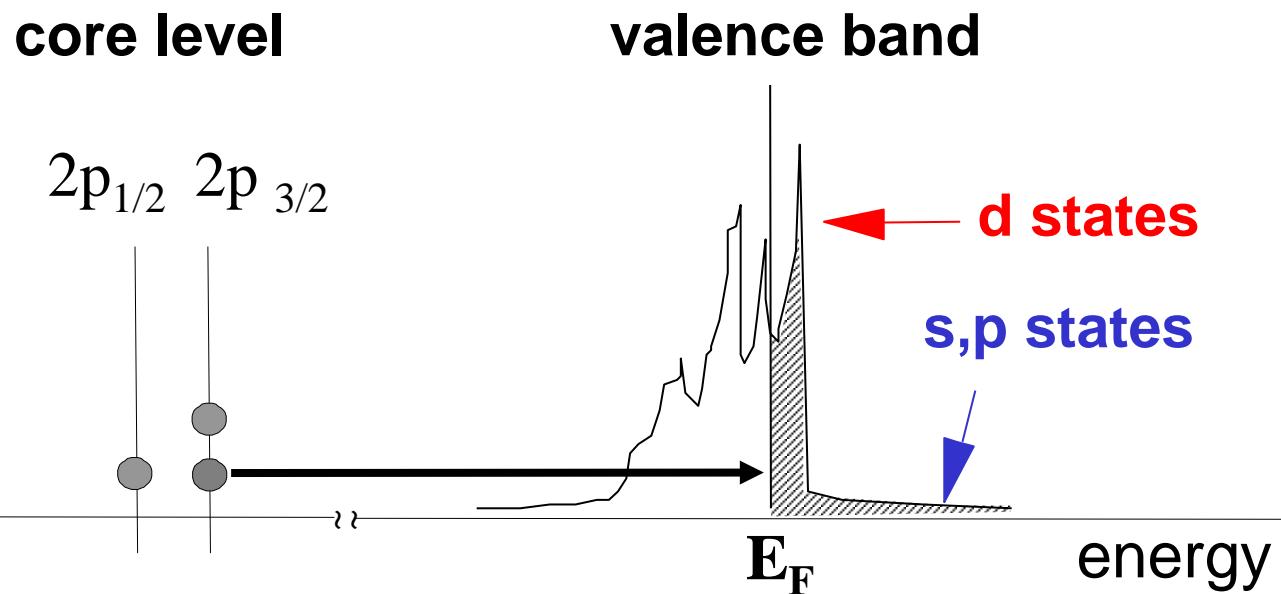
**Current: 350 mA (400 mA)**





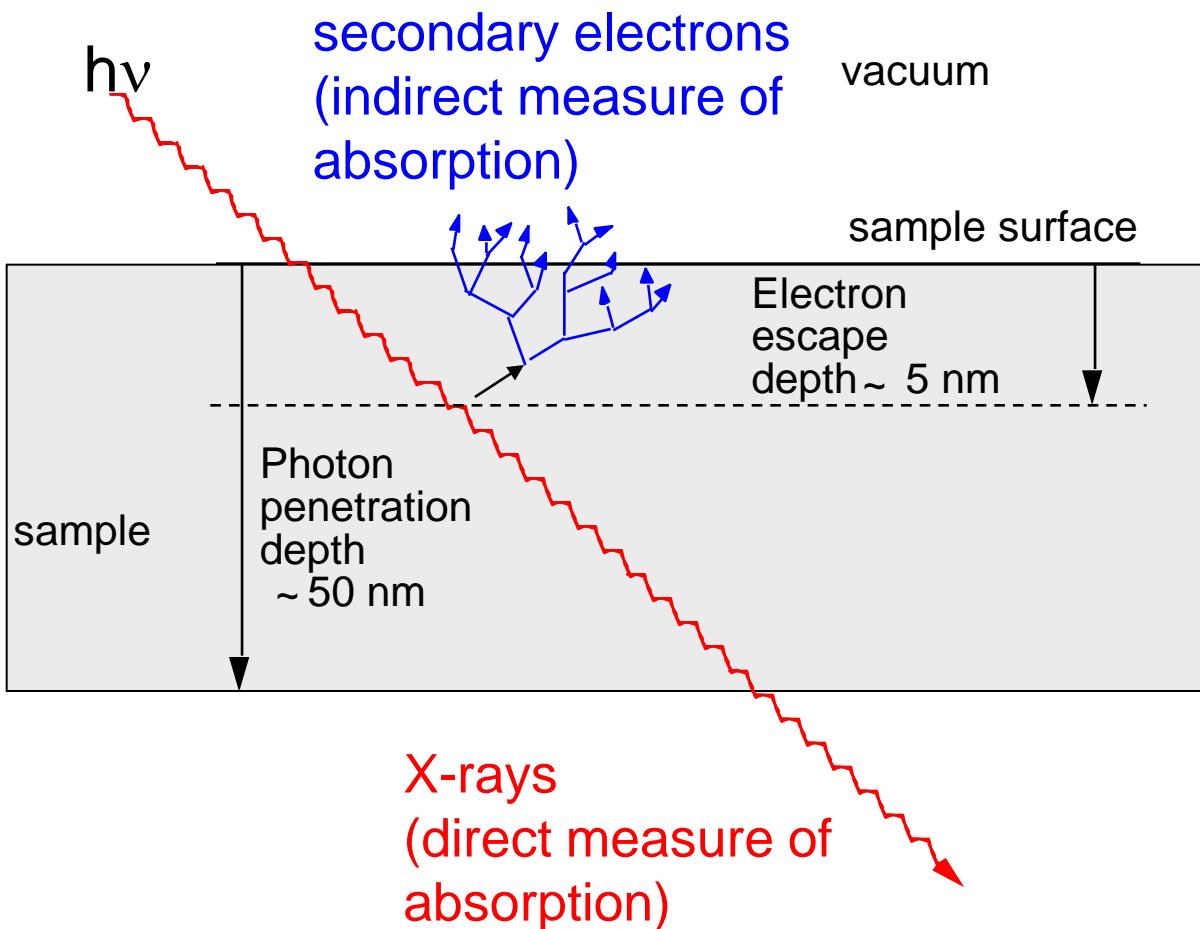
- X-rays excite an electron from the core to the valence band
- Relaxation of the electron from the valence to the core level gives:
  - Soft x-rays:** more Auger es
  - Hard x-rays:** more fluorescence
- Therefore different interactions: more than just imaging

**Spectrum:**  
*change energy &  
observe absorption:*



- Peak corresponds to (set of) transition(s) from core level to valence band
- Density of unoccupied states above Fermi level
- Each element: own characteristic peaks

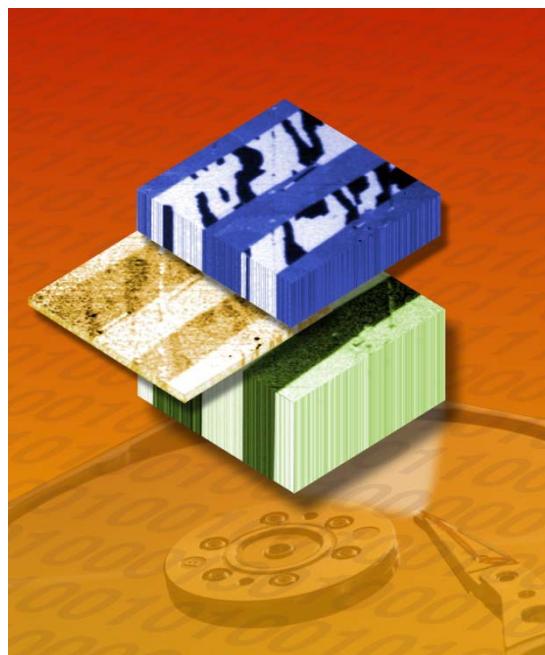
# PEEM & TXM



**Photoemission  
Electron  
Microscopy  
(PEEM) to probe  
surface / interfaces**

**Transmission  
X-ray Microscopy  
(TXM)**

# Photoemission Electron Microscopy

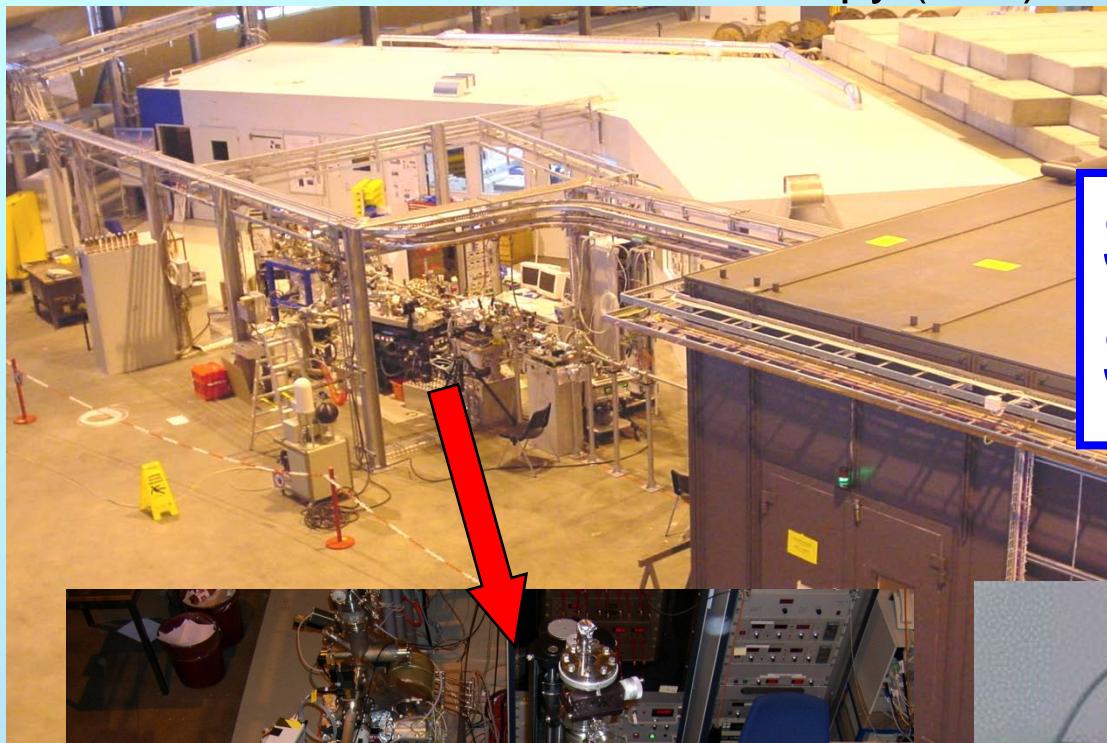


Slow electrons: mean free path is submono to several monolayers (few nm's)

Surfaces, thin films and interfaces

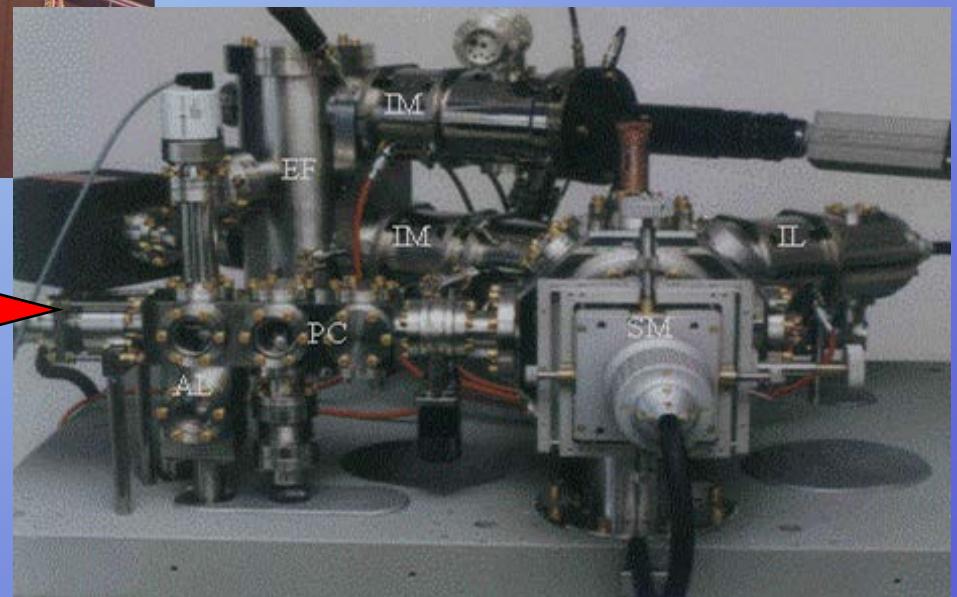
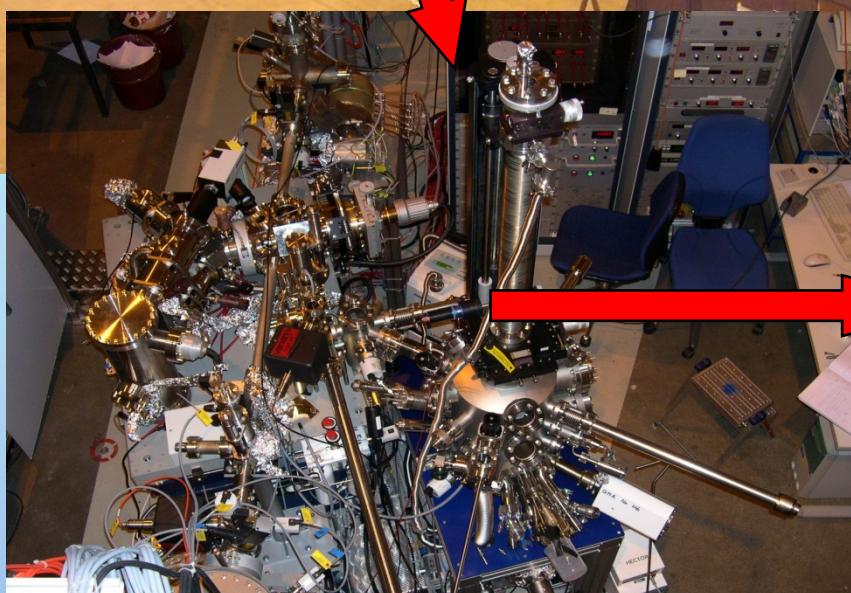
....consequences for electron optics.

## The Surface and Interface Microscopy (SIM) Beamlne



**SIM Beamlne,  
Swiss Light Source**

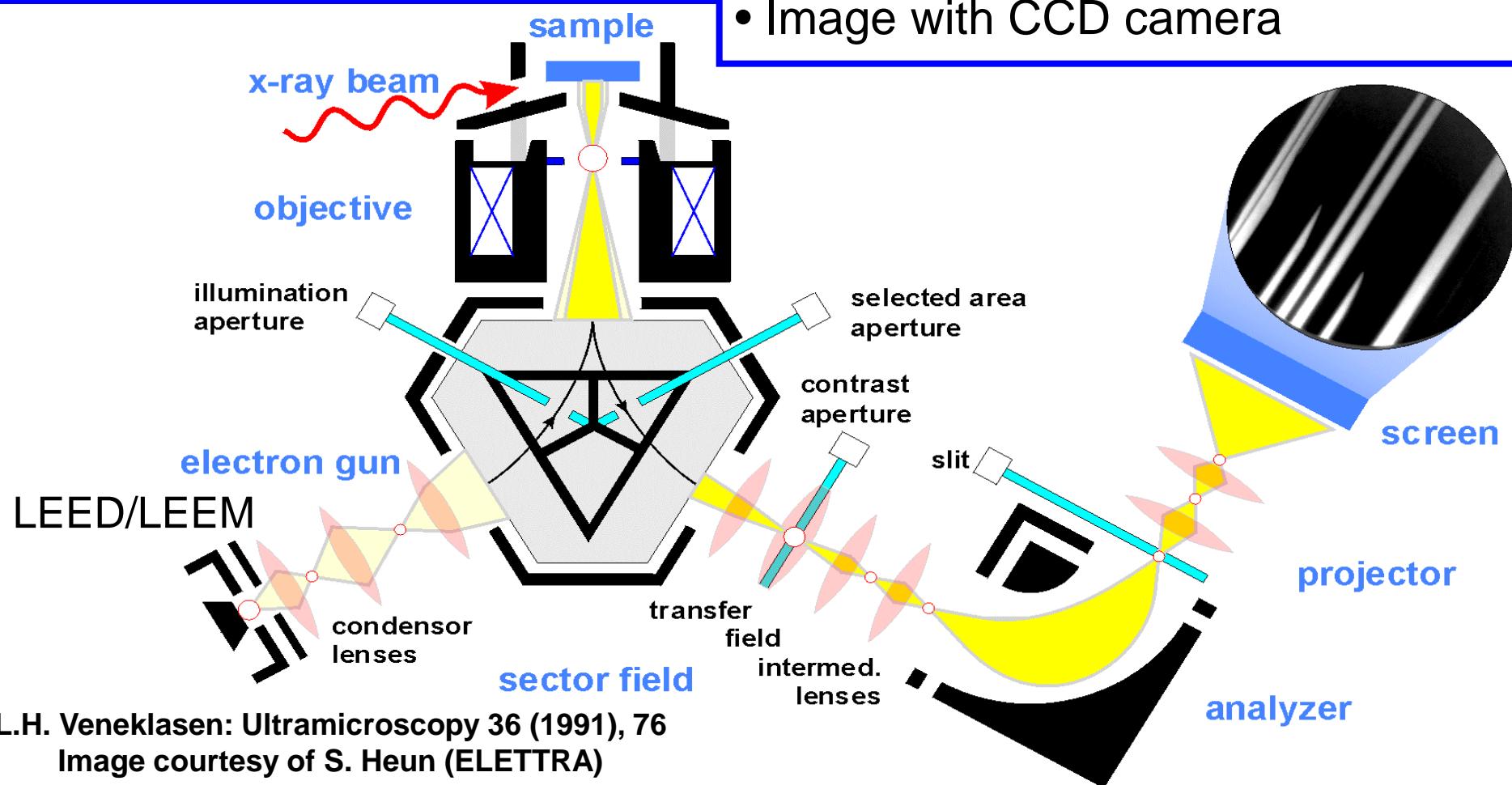
Close-up of the PEEM



The Photoemission Electron Microscope (PEEM)

# Spectromicroscope

- Channel plate: amplifies electrons
- Phosphor screen: converts to light
- Image with CCD camera



L.H. Veneklasen: Ultramicroscopy 36 (1991), 76  
Image courtesy of S. Heun (ELETTRA)

Elmitec Elektronenmikroskopie GmbH

Clausthal-Zellerfeld, Germany

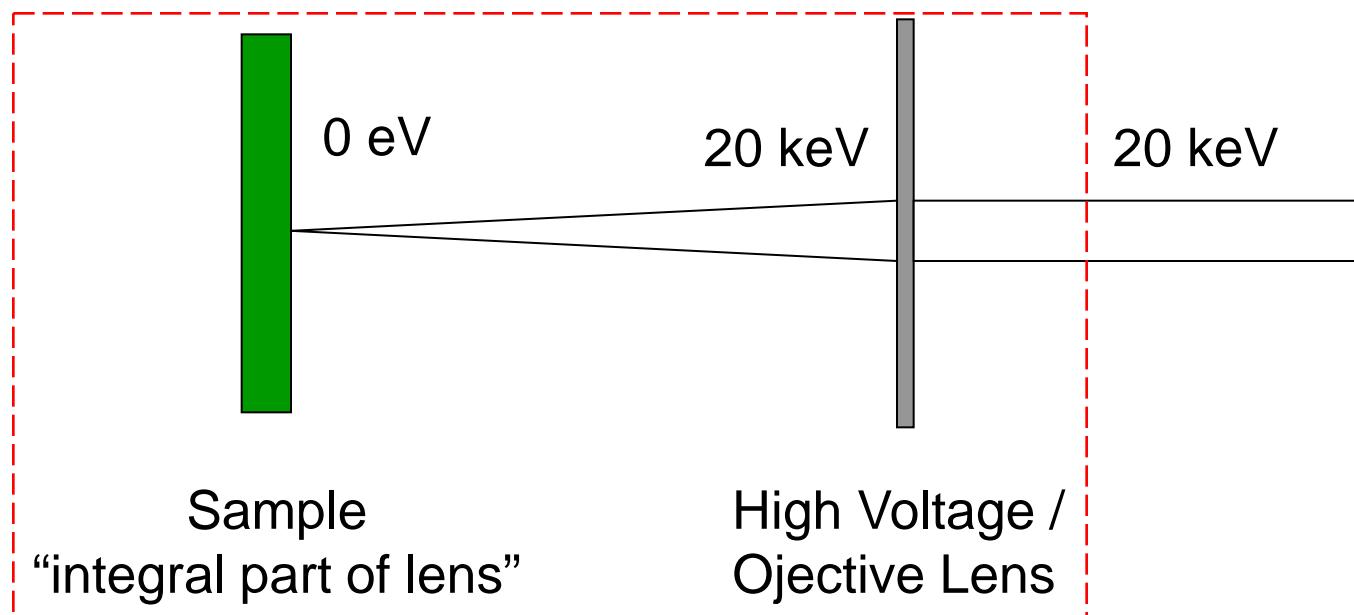
# Slow Electrons

Probe : slow electrons

Imaging: high energy electrons

(more stable and maintain spatial information)

Lens Equivalent has two functions: accelerating field due to potential & focussing function



## High voltage:

- reduced sensitivity to external magnetic fields
- reduced energy spread and smaller electron beam diameters

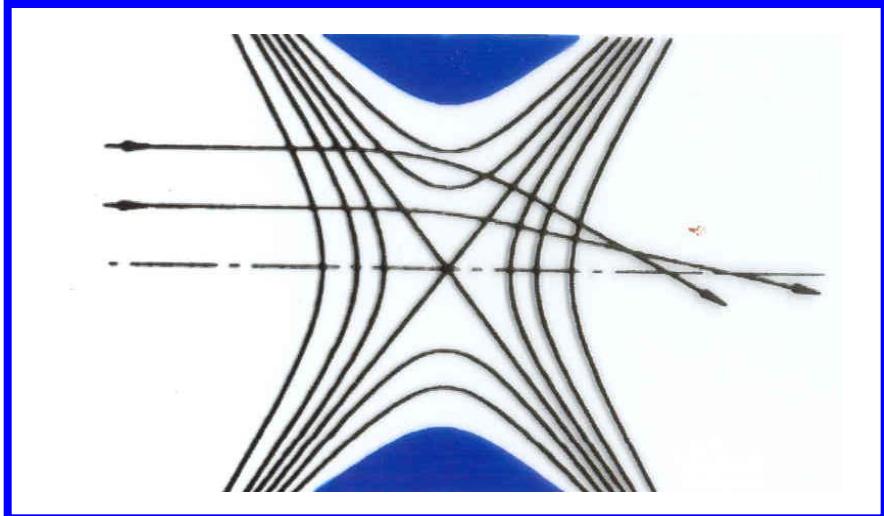
Immersion lens: electrons have before and after the lens different velocity (different wavelength)

Cathode lens: Sample is cathode

electron microscope is anode

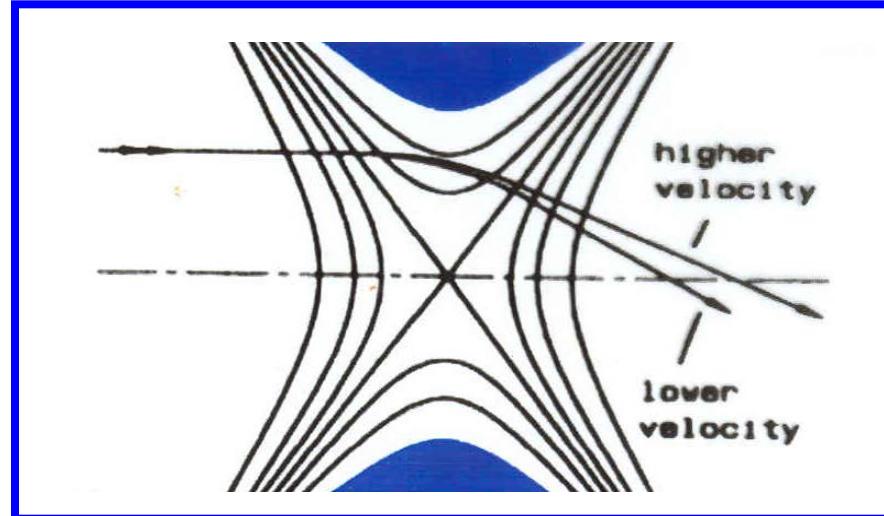
## Two Kinds of Aberrations

### Spherical



Beams parallel away from the lens axis are focused in a slightly different place than beams close to the axis and therefore a blurring of the image.

### Chromatic

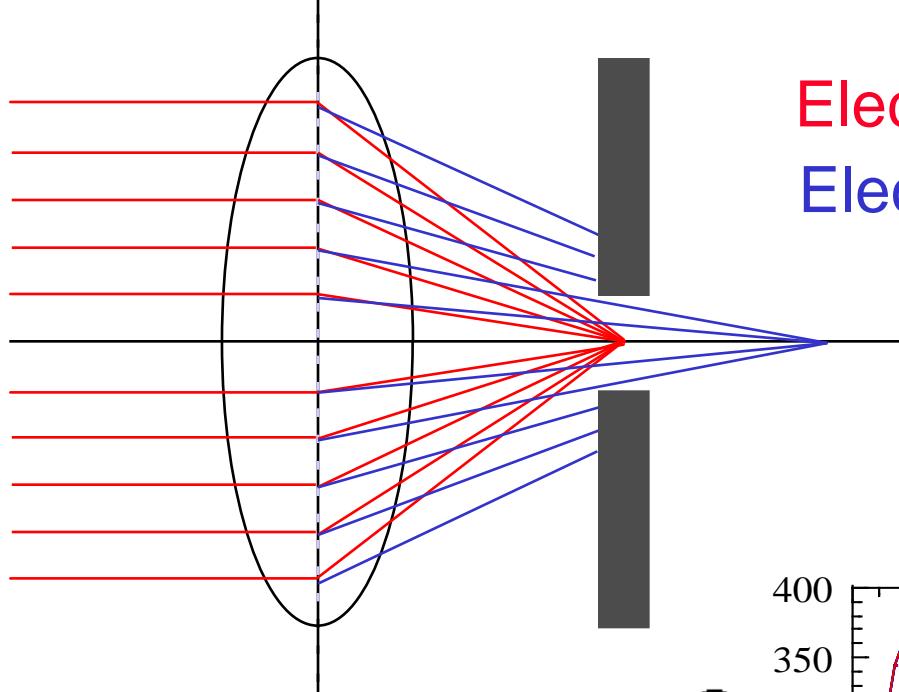


Different wavelengths of light are focused to different positions.

Light → electrons  
Glass → electrostatic/magnetic lenses

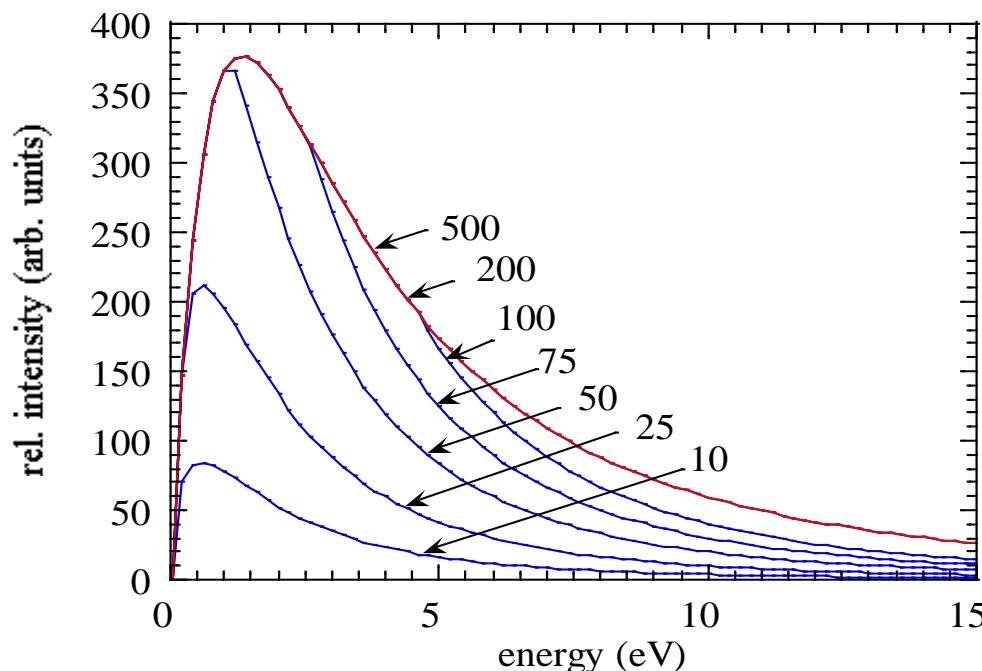
# Energy Filter

To remove chromatic aberrations:



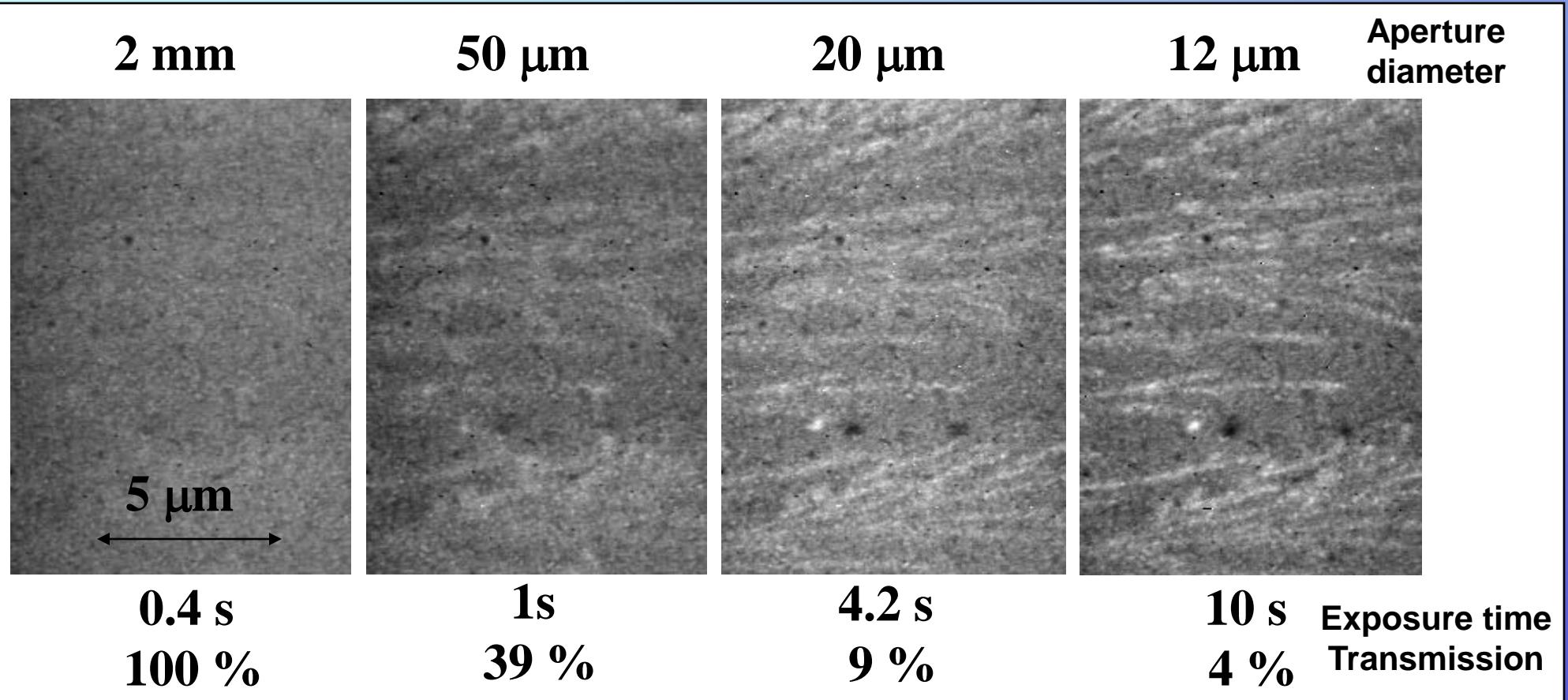
Energy distribution is narrowed but transmission (intensity) is reduced. Therefore need to find compromise.

Electron energy  $E$   
Electron energy  $E + \Delta E$   
Aperture cuts off transmission of electrons with higher energy



## 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  $\mu\text{m}$  aperture for PEEM2



# Spatial Resolution for Magnetic Imaging

PEEM with X-rays: **50-20 nm** spatial resolution

**Aberration-corrected instruments using an electron mirror:**

SMART (spectromicroscope for all relevant techniques)

- at BESSY II, Berlin, Germany
- collaboration of seven Universities in Germany

PEEM III

- at ALS, Berkeley, USA
- mainly ALS

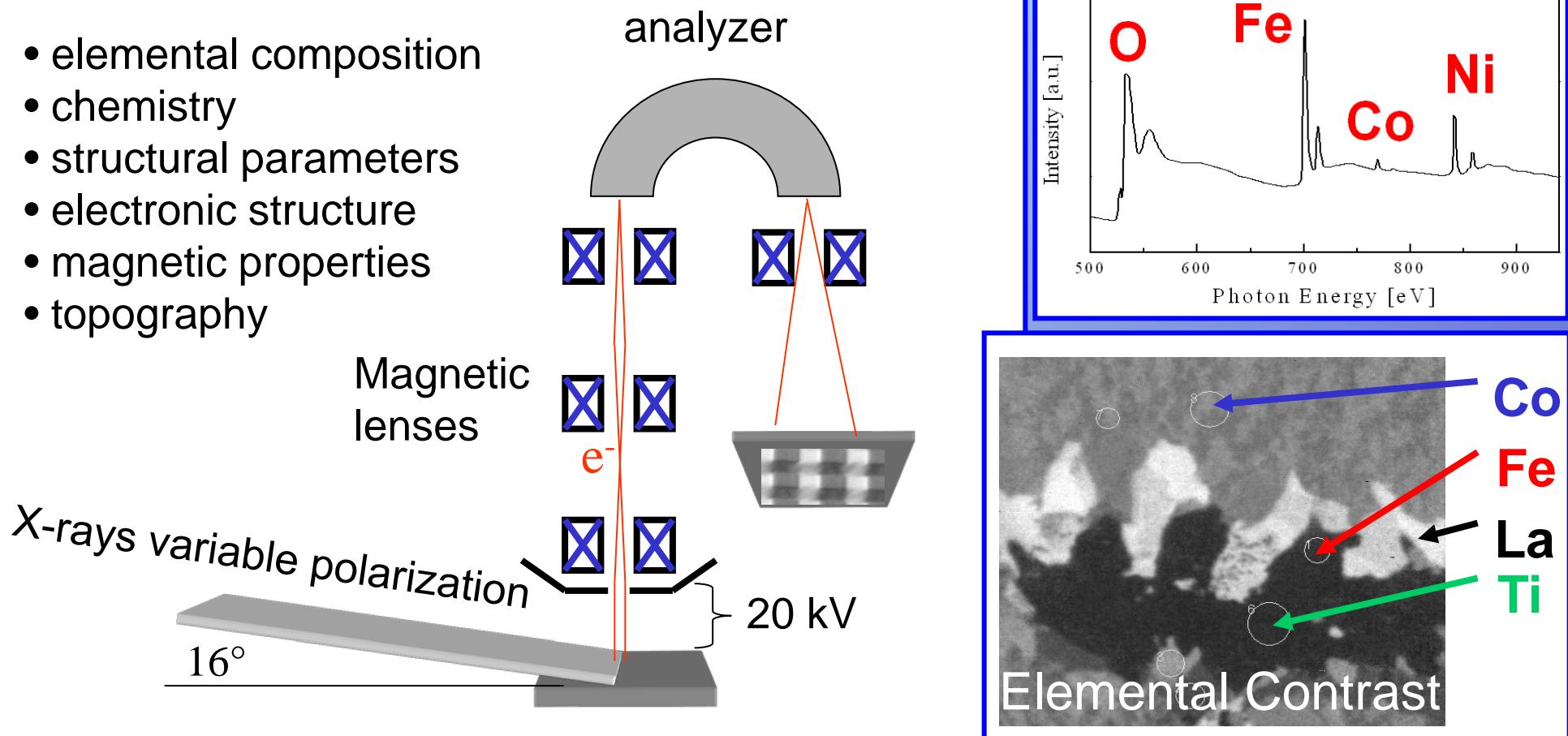
**down to a few nm** spatial resolution

# Photoemission Electron Microscope

## SIM beamline (SLS)

Armin Kleibert  
Carlos Vaz

- elemental composition
- chemistry
- structural parameters
- electronic structure
- magnetic properties
- topography

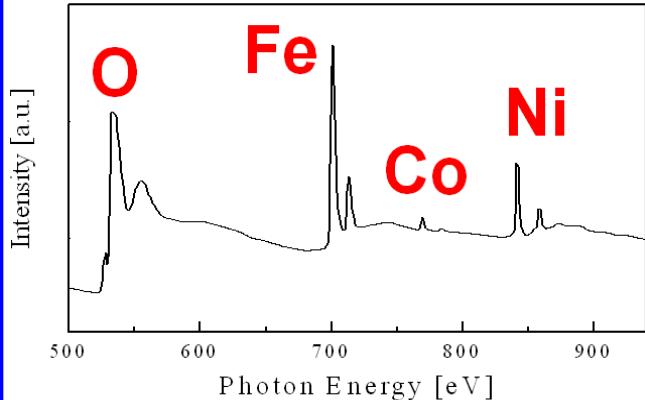
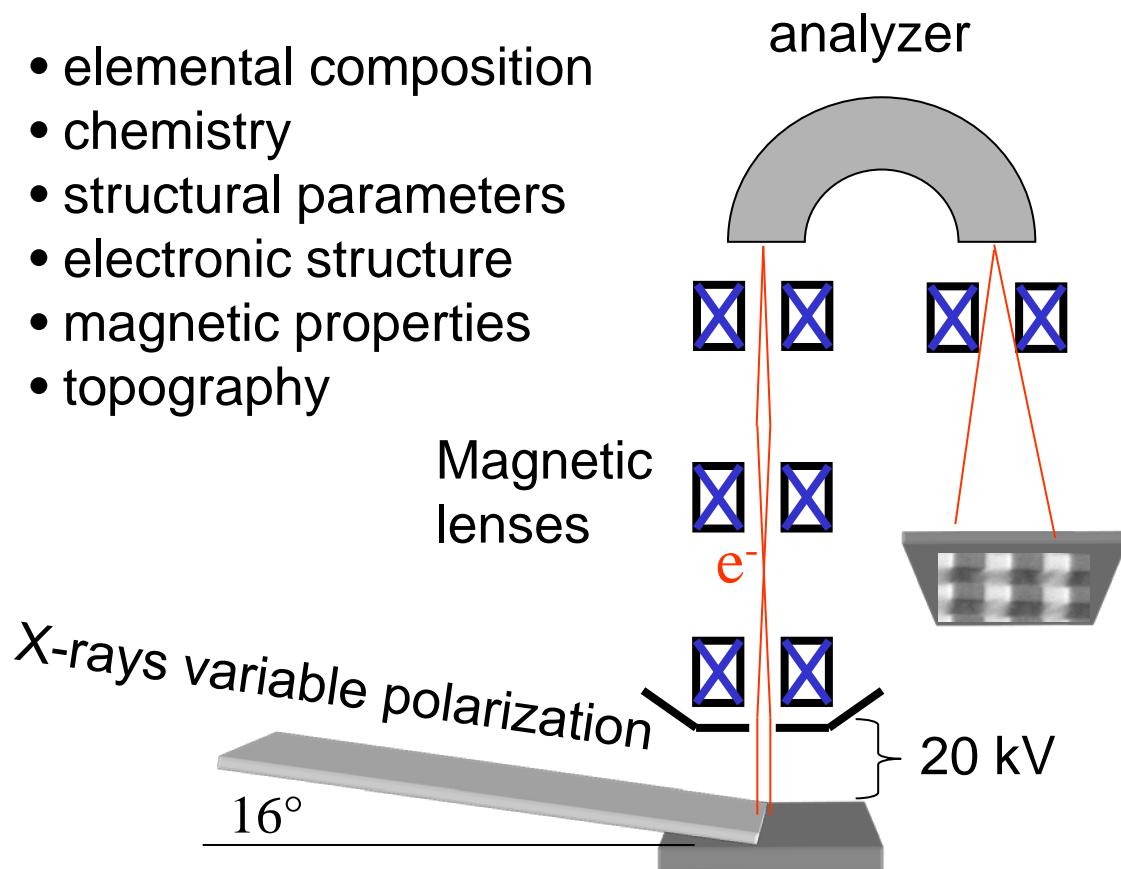


# Photoemission Electron Microscope

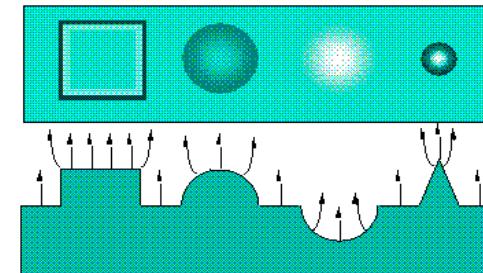
## SIM beamline (SLS)

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### Topographical Contrast



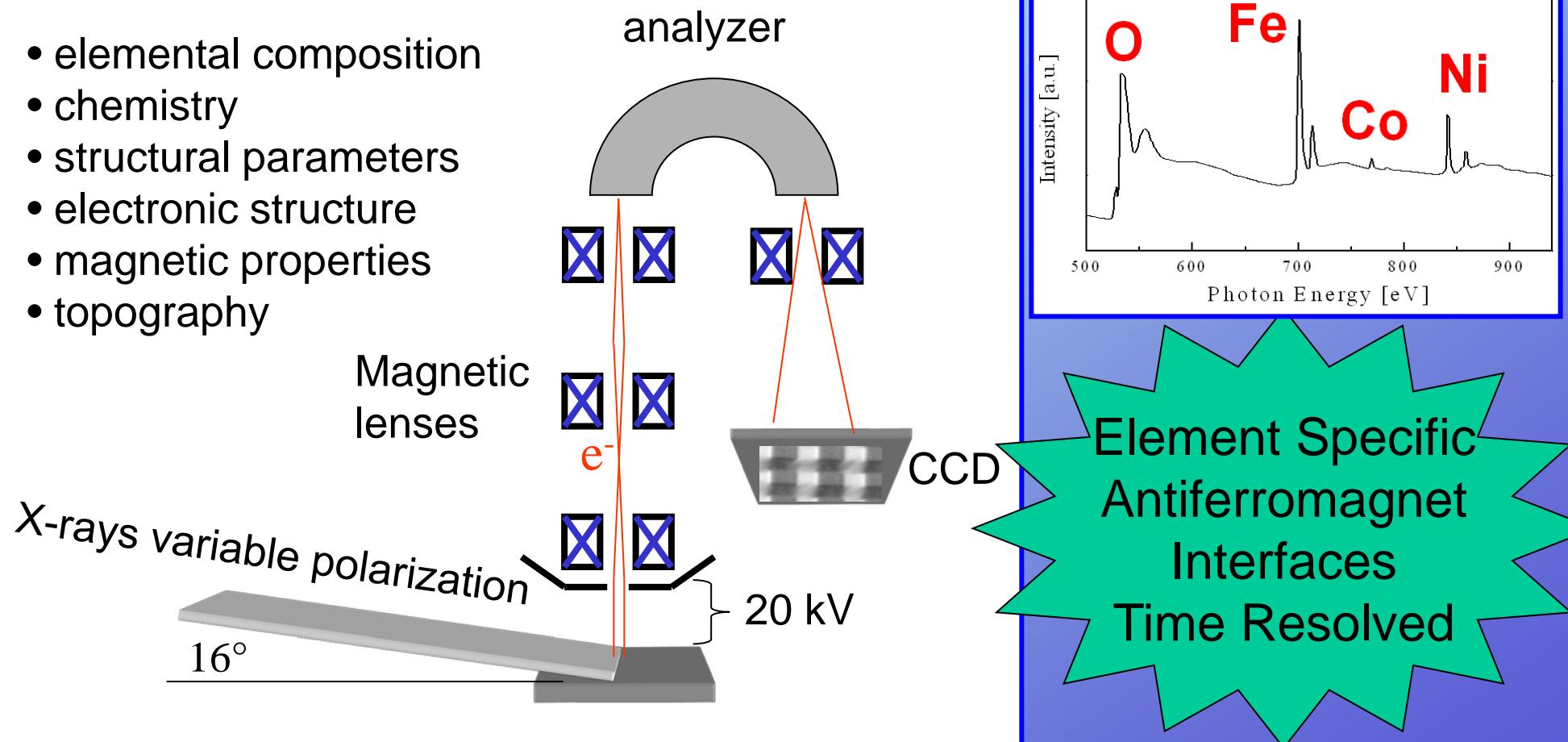
Microfocussing due to distortion  
of the local electric fields

# Photoemission Electron Microscope

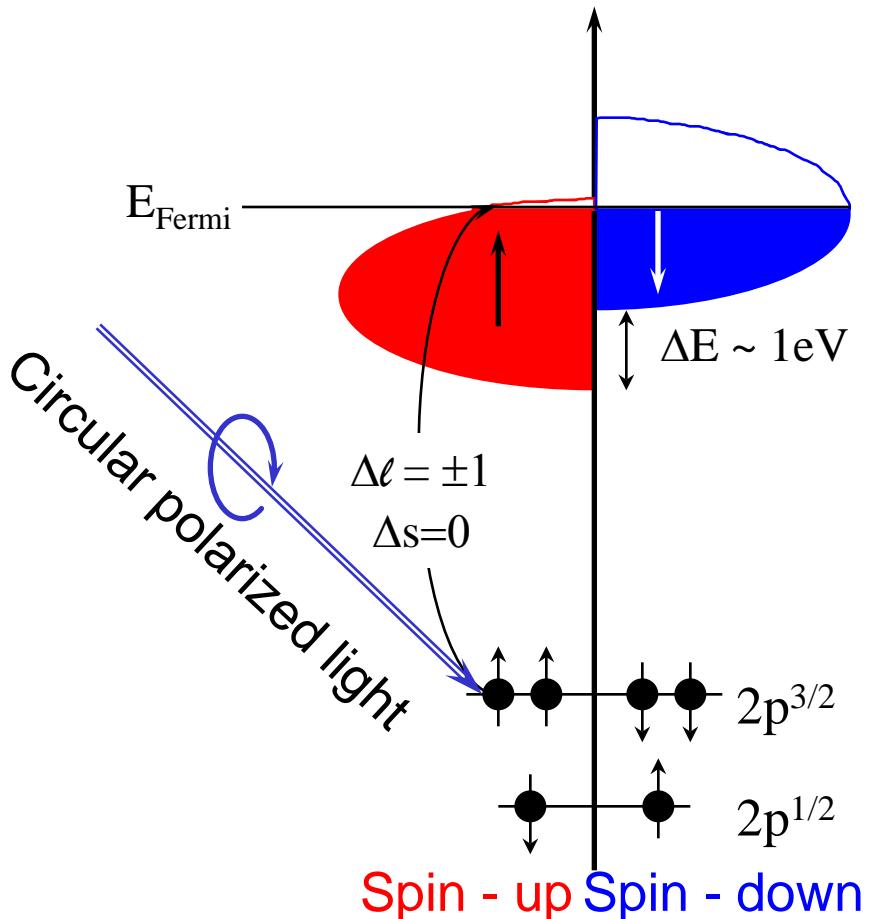
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- magnetic properties
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# X-Ray Magnetic Circular Dichroism (XMCD)

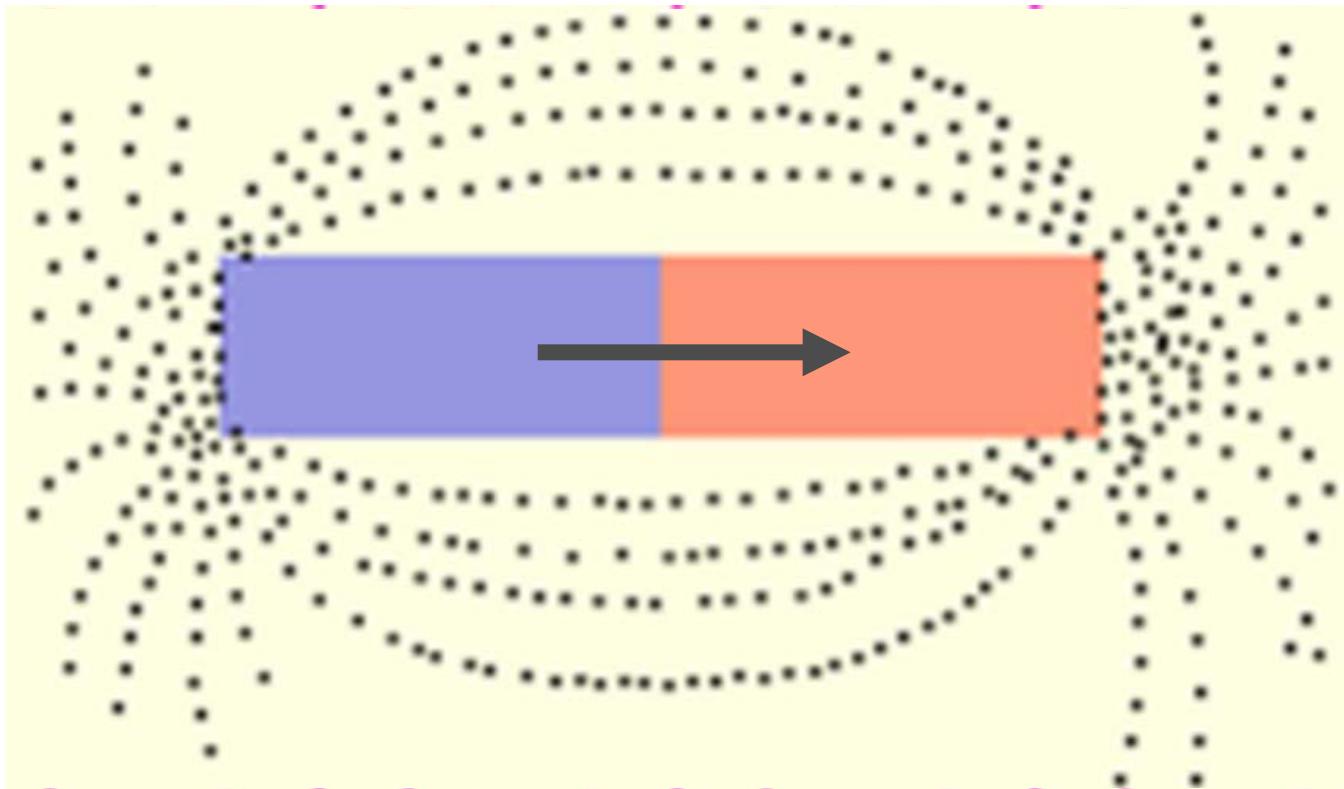


G. Schütz et al. PRL (1987)

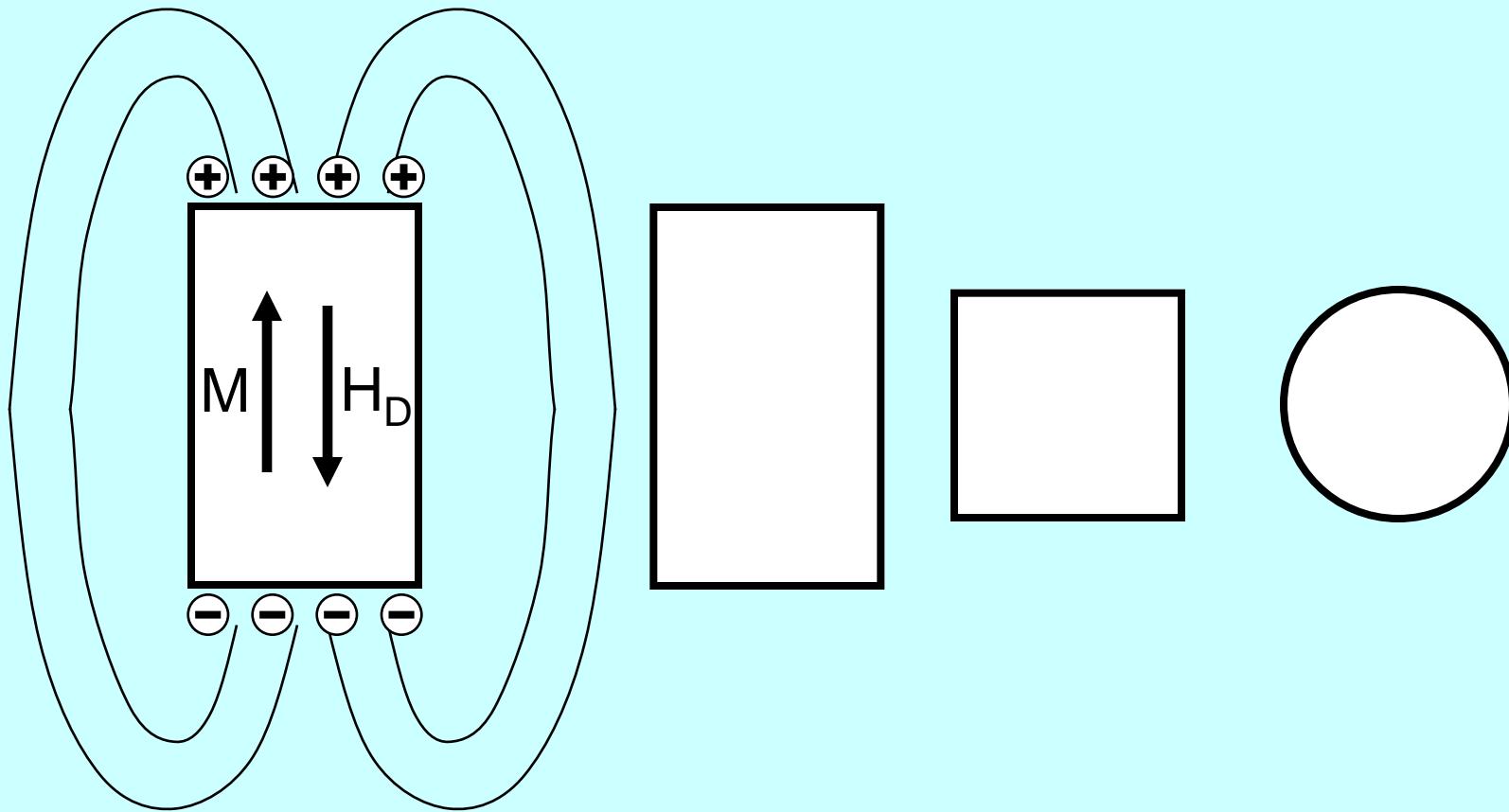
- L-edge absorption in d band transition metal
- Magnetic metal: d valence band split into spin-up and spin-down with different occupation
- Absorption of right/left circular polarisation: light mainly excites spin-up/down photoelectrons
- Spin flips forbidden: measured resonance intensity reflects number of empty d-band states of a given spin
- Can determine sizes and directions of atomic magnetic moment

Time for a  
game. . . .

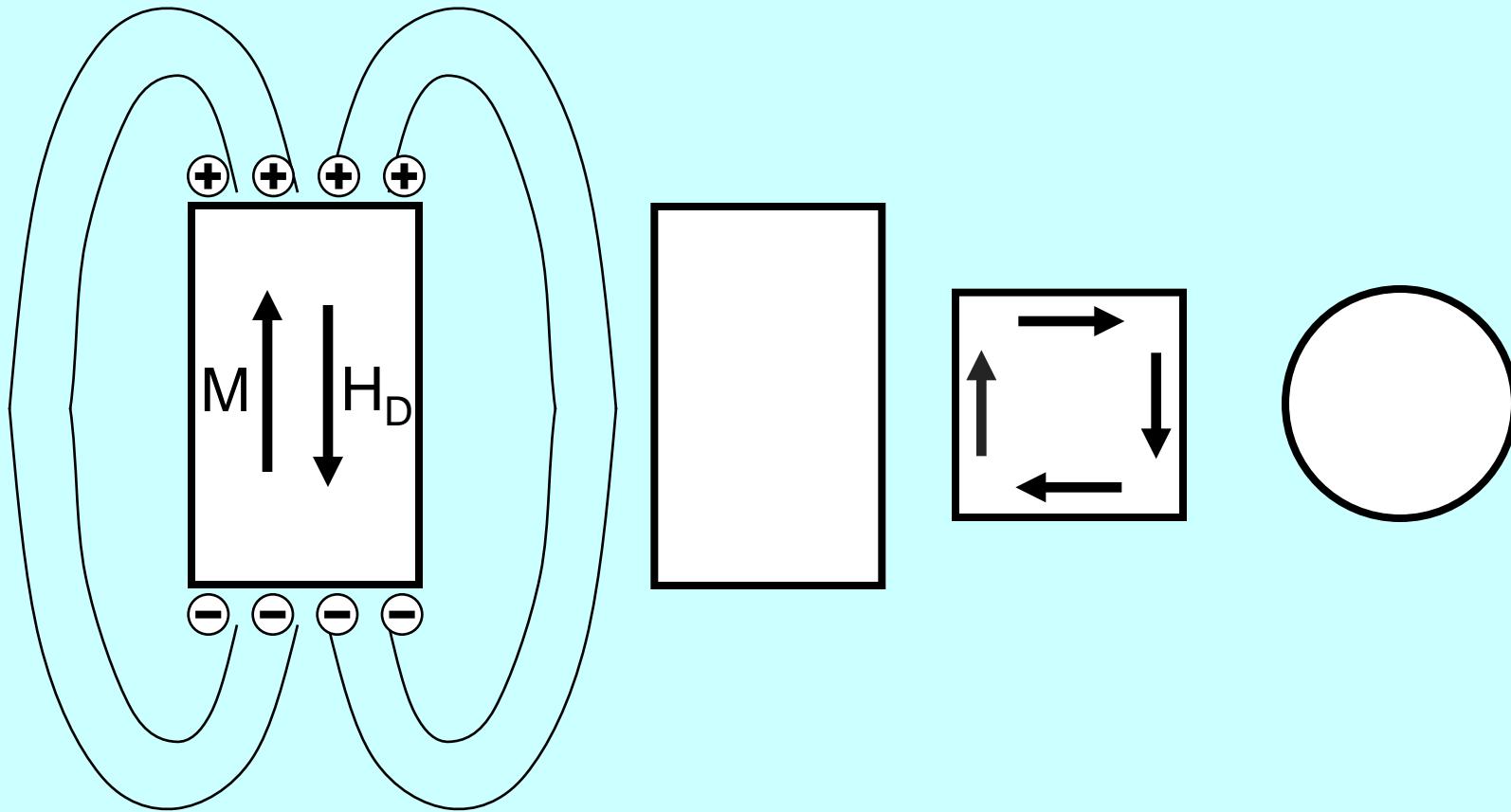
# Magnetostatic or Stray Field Energy



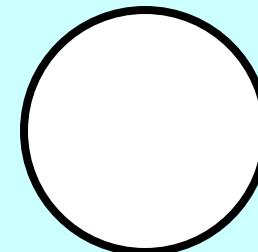
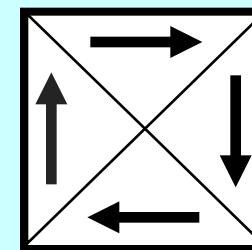
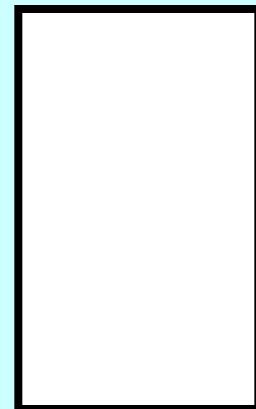
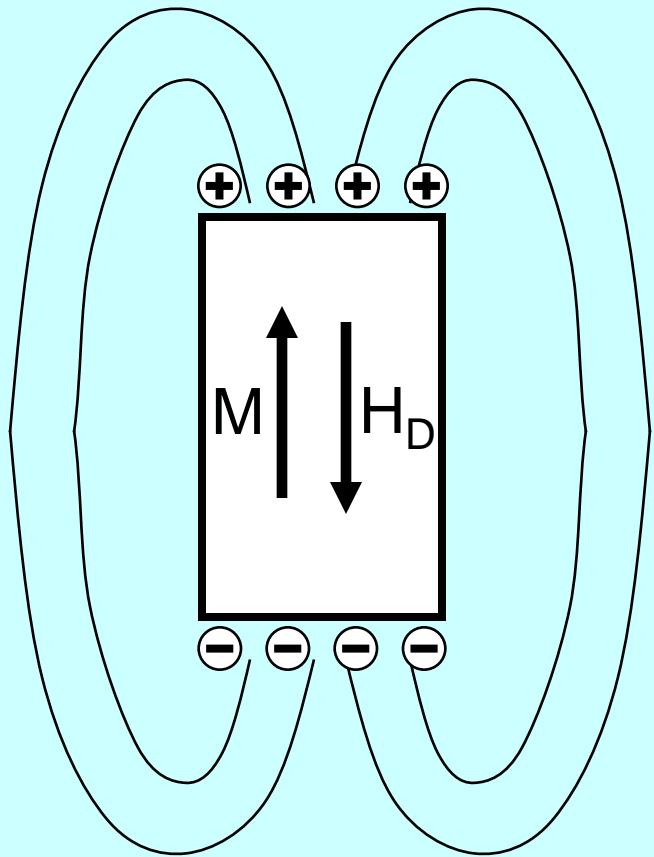
# Rectangle, Square, Disk



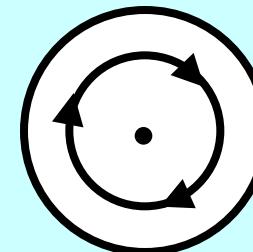
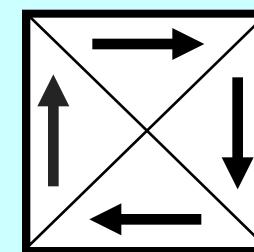
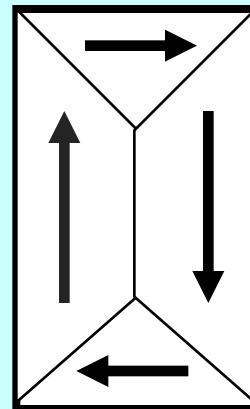
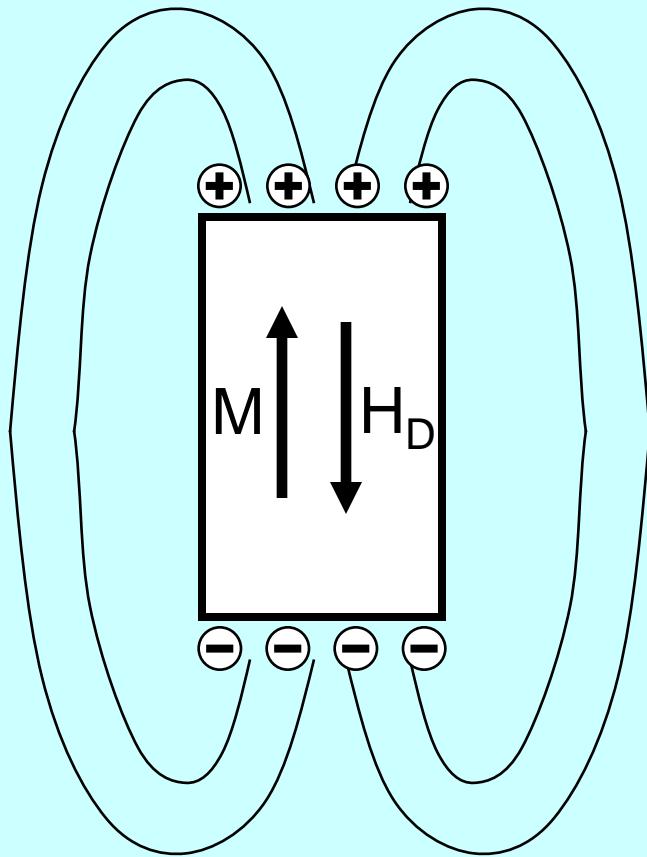
# Rectangle, Square, Disk



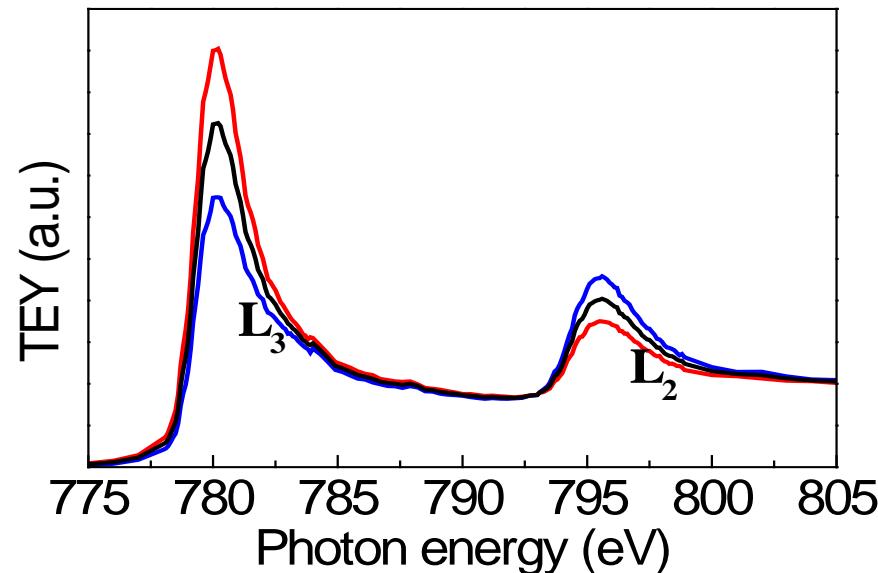
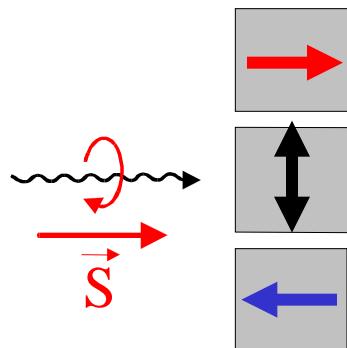
# Rectangle, Square, Disk



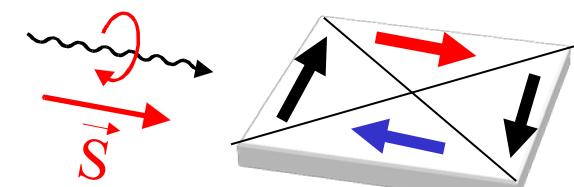
# Rectangle, Square, Disk



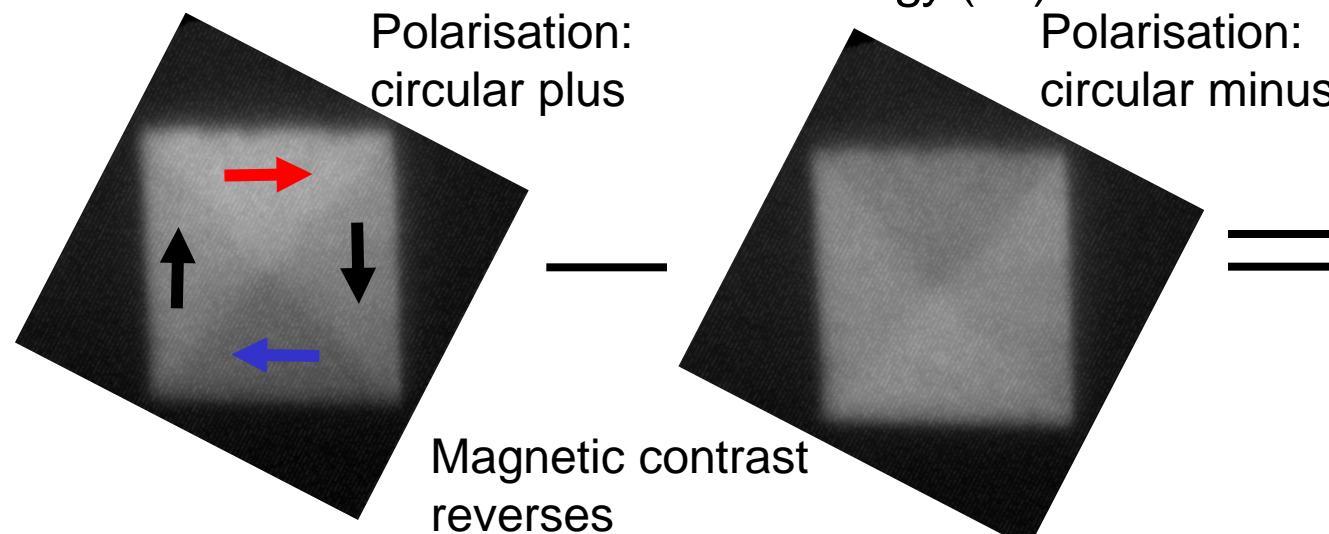
# X-ray Magnetic Circular Dichroism (XMCD)



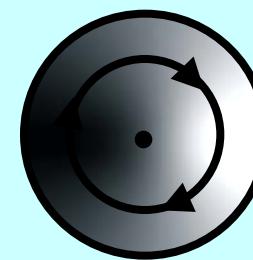
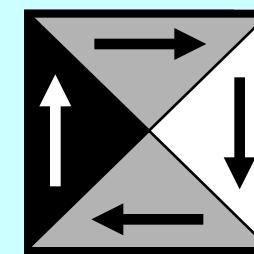
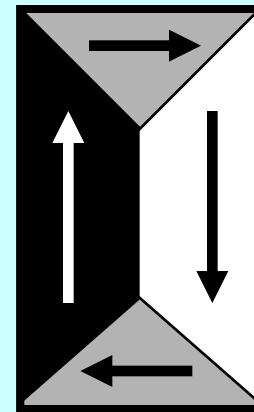
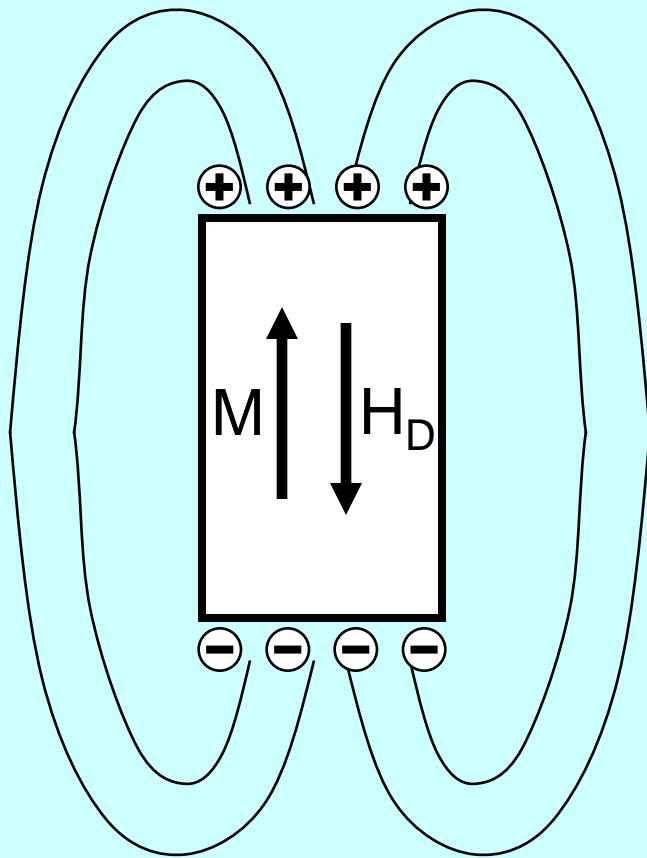
$$\text{XMCD} \sim \mathbf{M} \cos(\mathbf{M}, \mathbf{S})$$



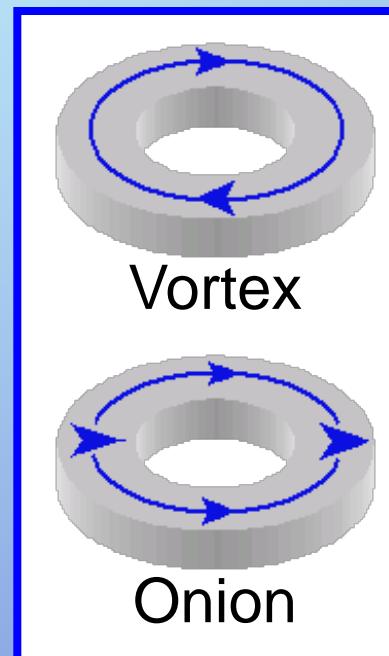
Square Ferromagnetic Element: Landau Domain Pattern



# Rectangle, Square, Disk



# Ring



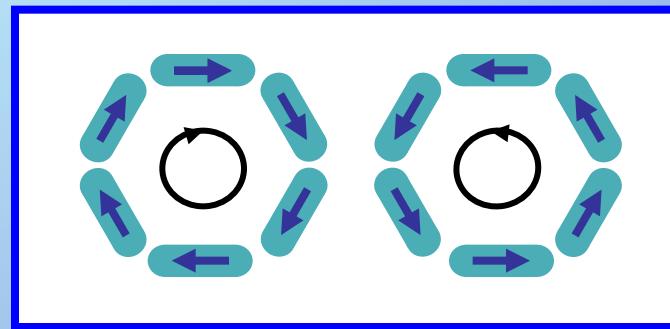
Interacting Magnets.....

....with the help of some frogs....!

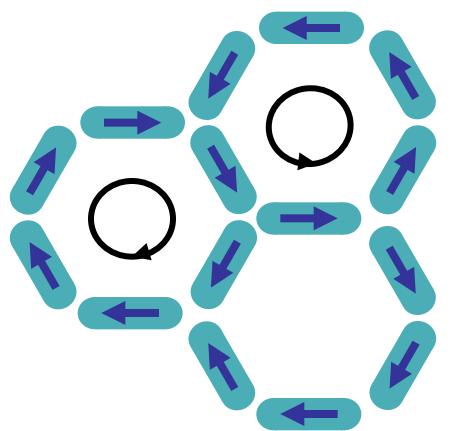
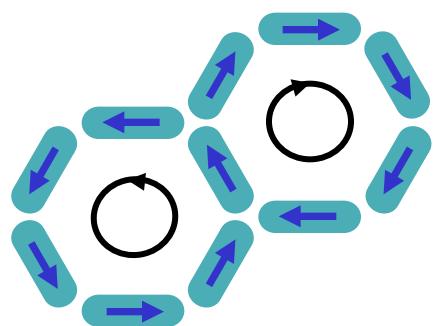
# Interacting Magnetic Frogs



# Ring of Nanomagnets



# Rings of Nanomagnets

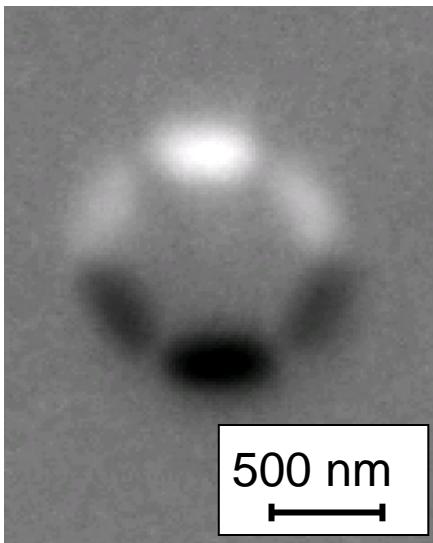


?

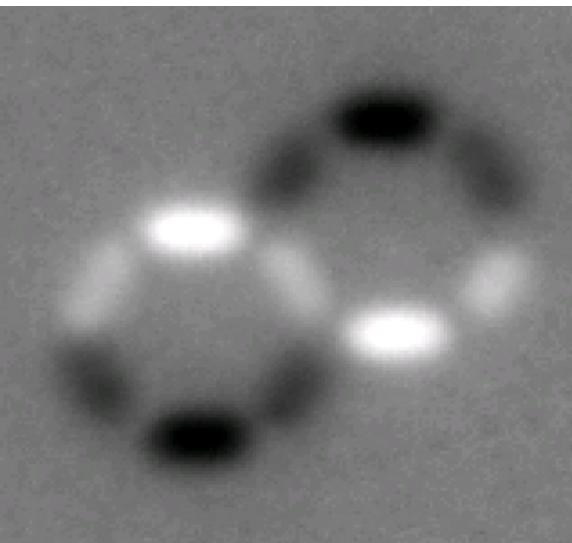


# Artificial Spin Ice in PEEM

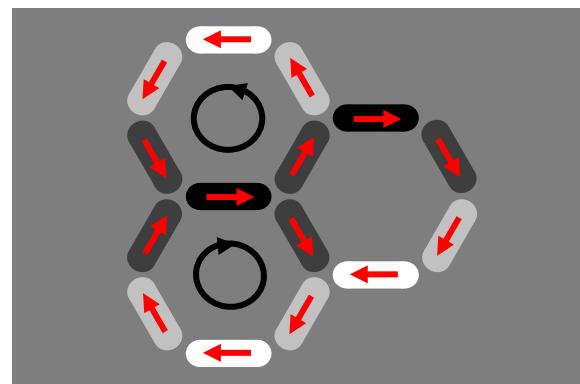
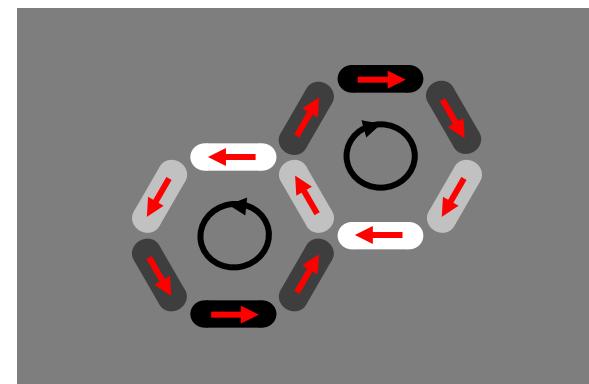
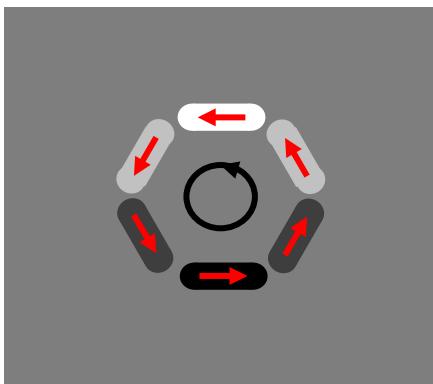
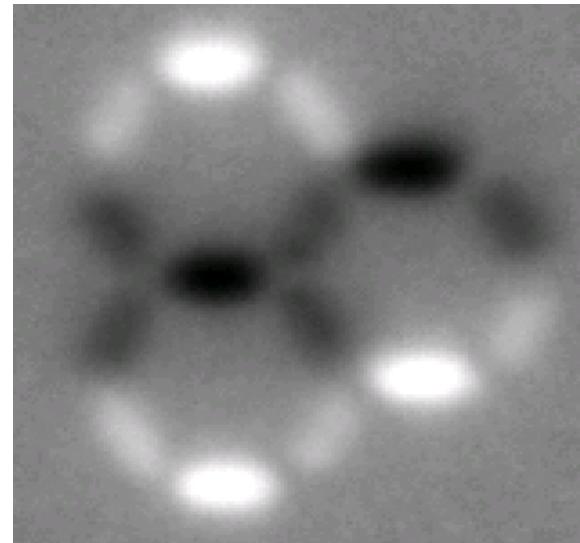
One ring



Two rings



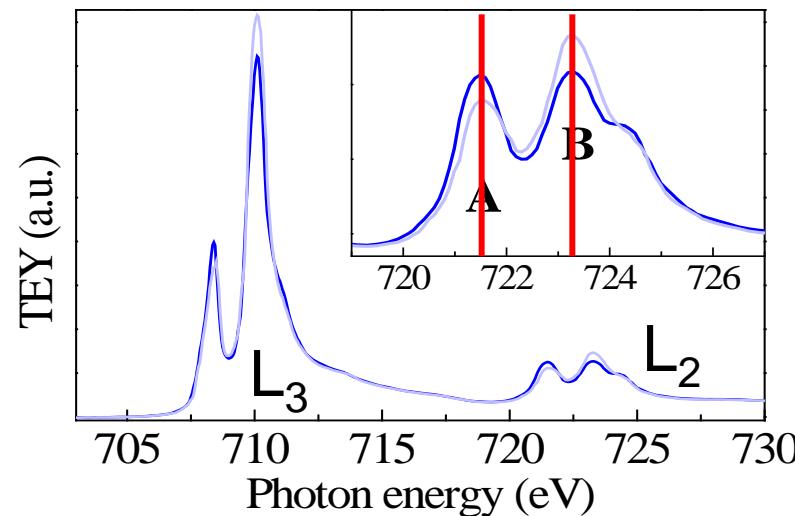
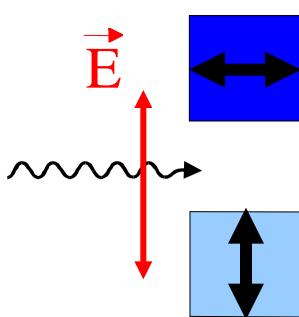
Three rings



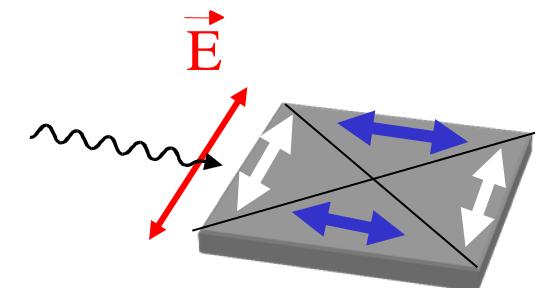
X-ray direction



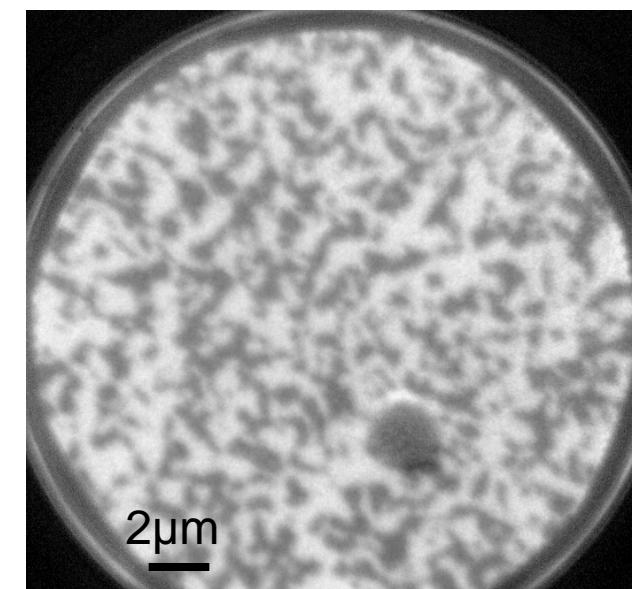
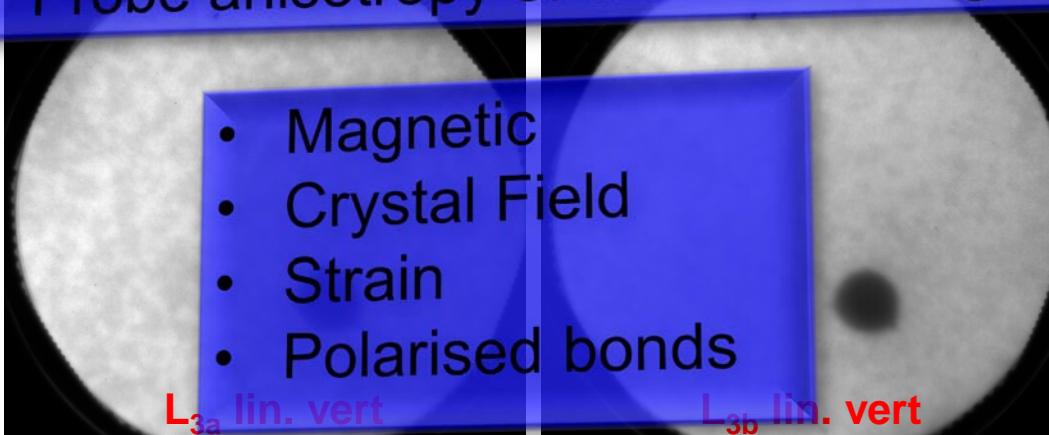
# X-ray Magnetic Linear Dichroism (XMLD)



$$\text{XMLD} \sim \langle \mathbf{M}^2 \rangle \cos^2(\mathbf{M}, \mathbf{E})$$



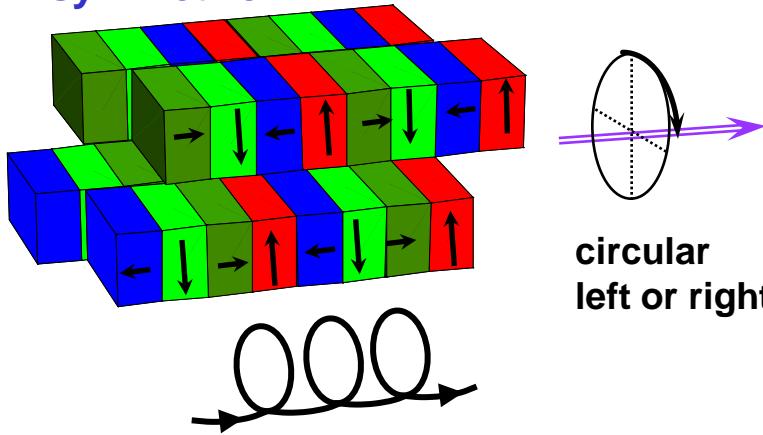
"Probe anisotropy of electron charge"



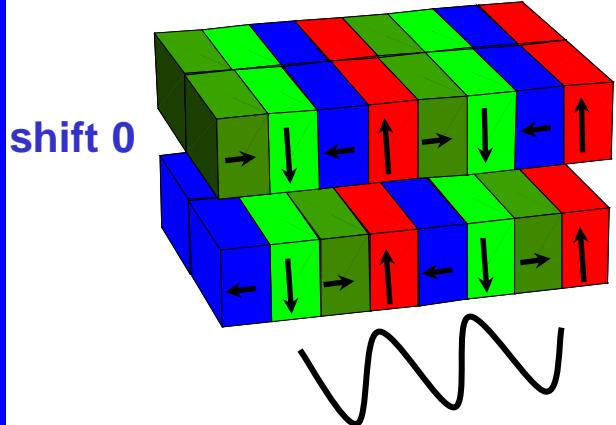
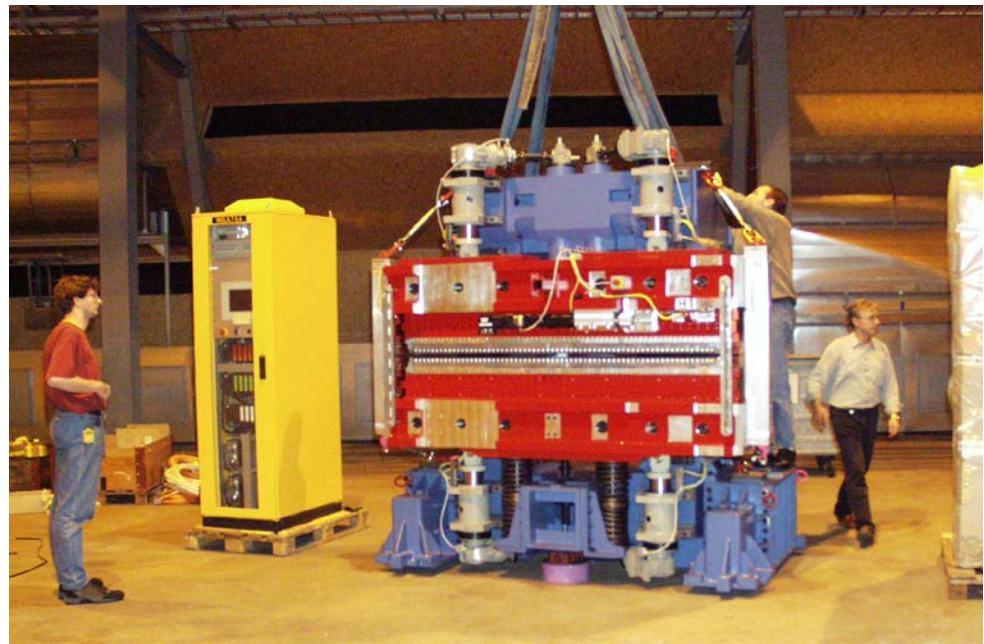
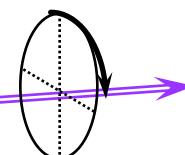
# Undulator

Magnetic Structure:  
changing phase, changes  
polarisation

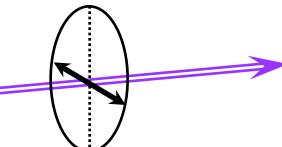
symmetric



circular  
left or right

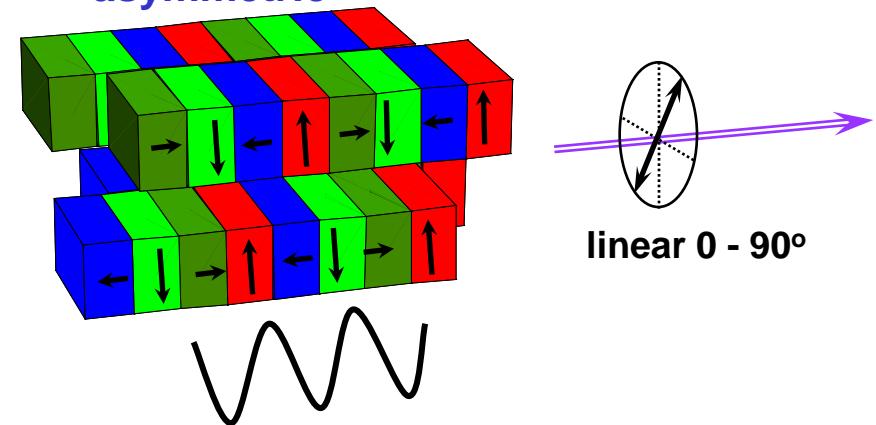


shift 0

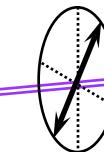


horizontal

shift  $\pi/2$   
asymmetric

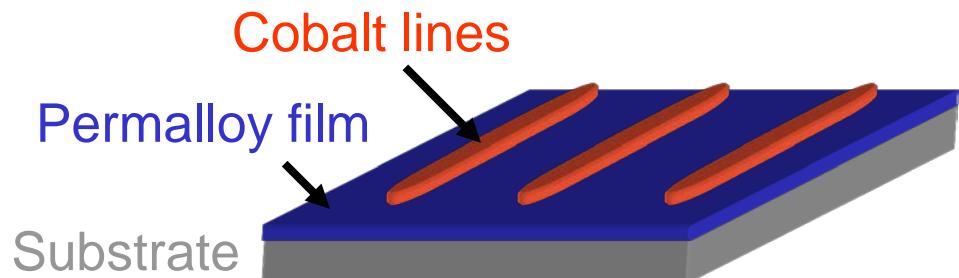
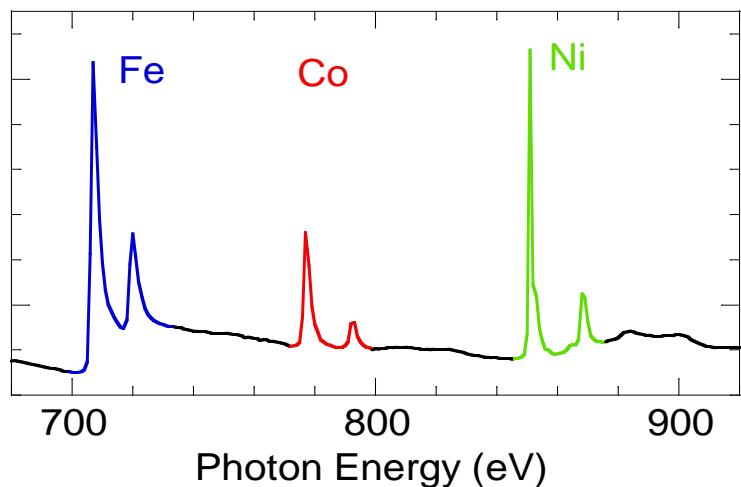


linear 0 - 90°

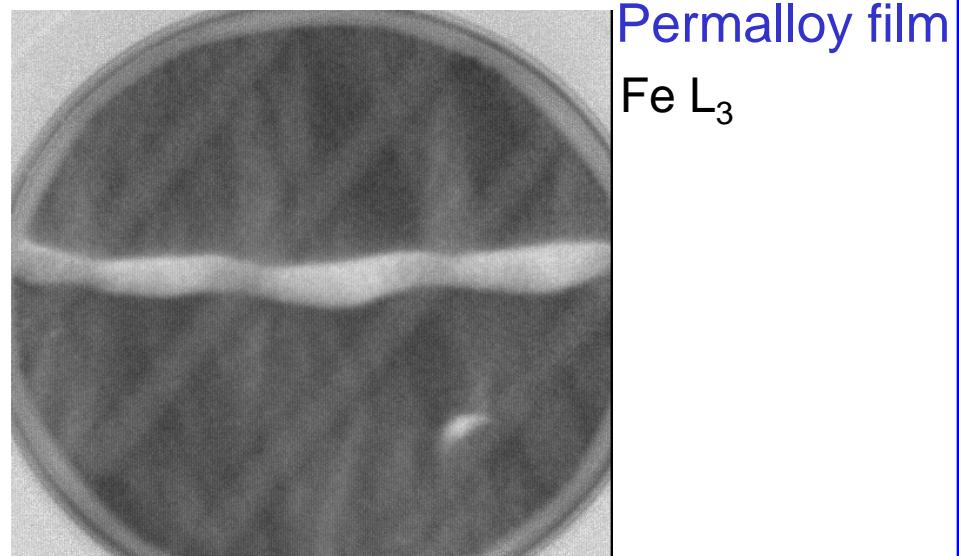
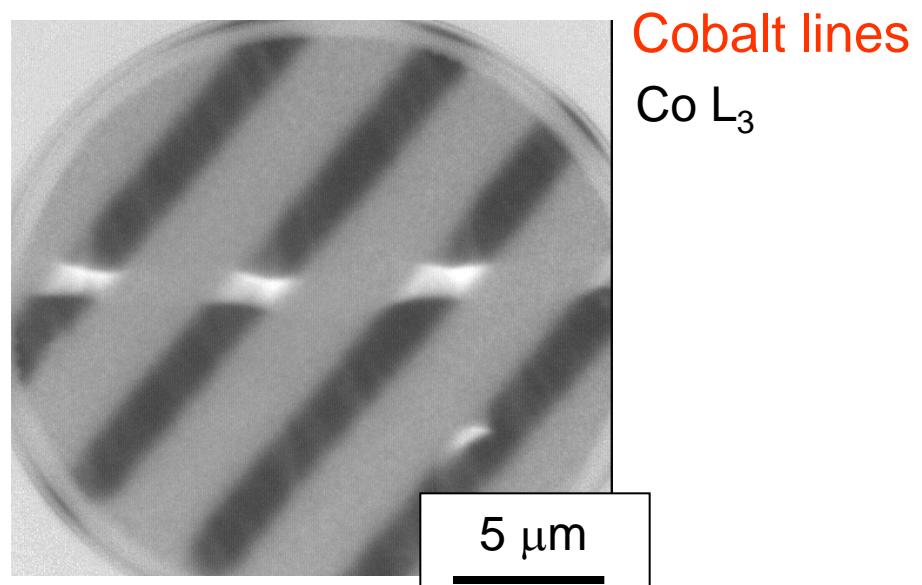


# *Brief Examples*

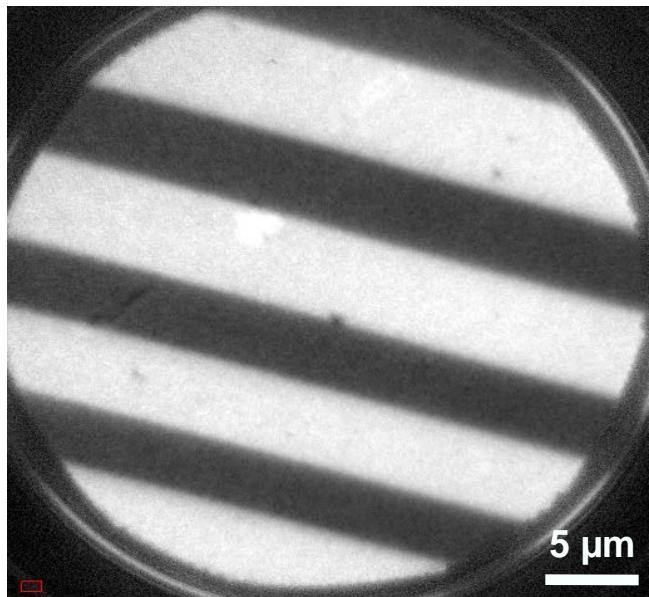
# Element specific contrast



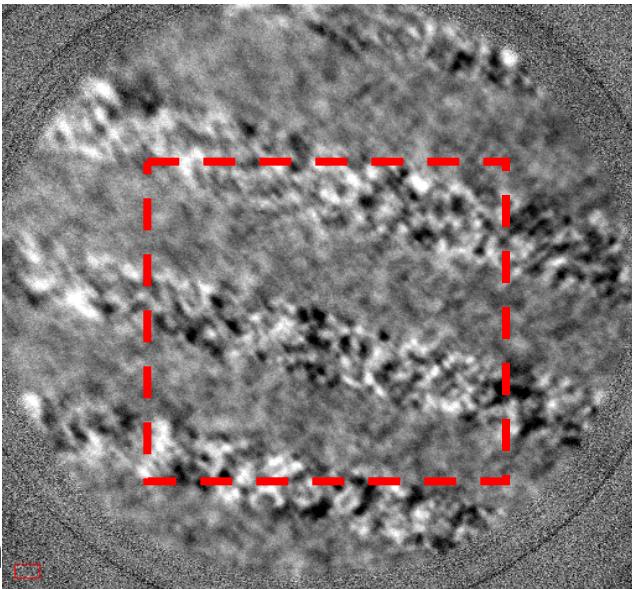
Coupling of hard and soft magnetic layer:  
L. Heyderman, A. Fraile-Rodriguez, A. Hoffmann



# Strain and Magnetic Domains



**Fe X-ray Linear Dichroism**

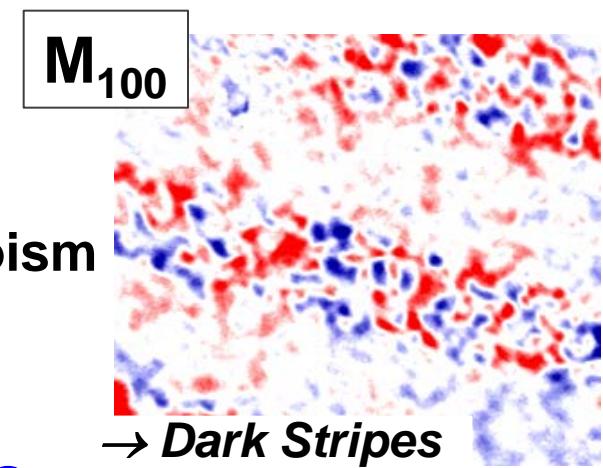
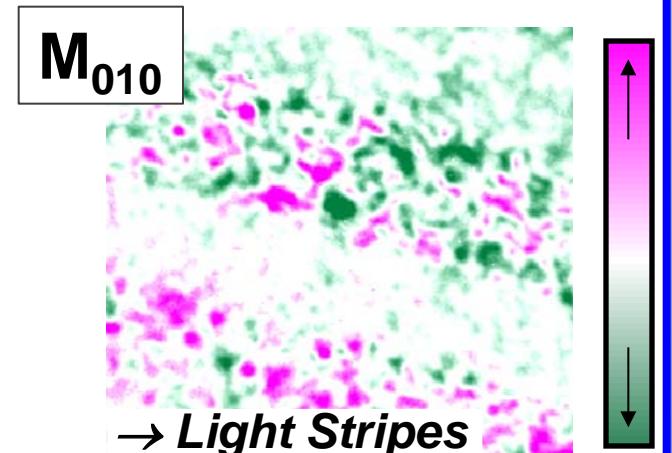


**Fe X-ray Magnetic Circular Dichroism**



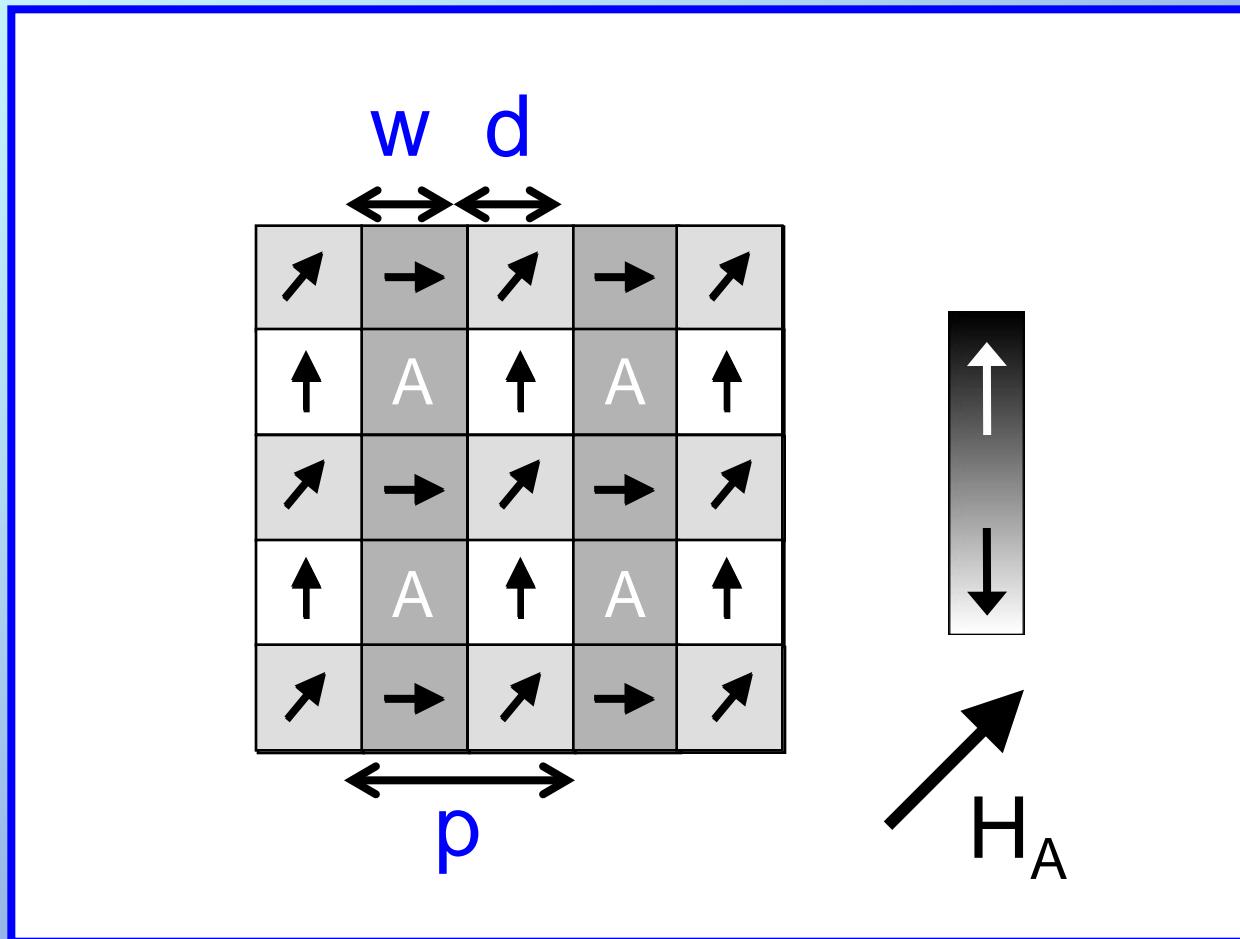
- **Fe XLD: strain domains**
- **Fe XMCD: magnetic domains**
- **Substrate-induced strain strongly modifies magnetic anisotropy**

RV Chopdekar et al. PRB (2012)



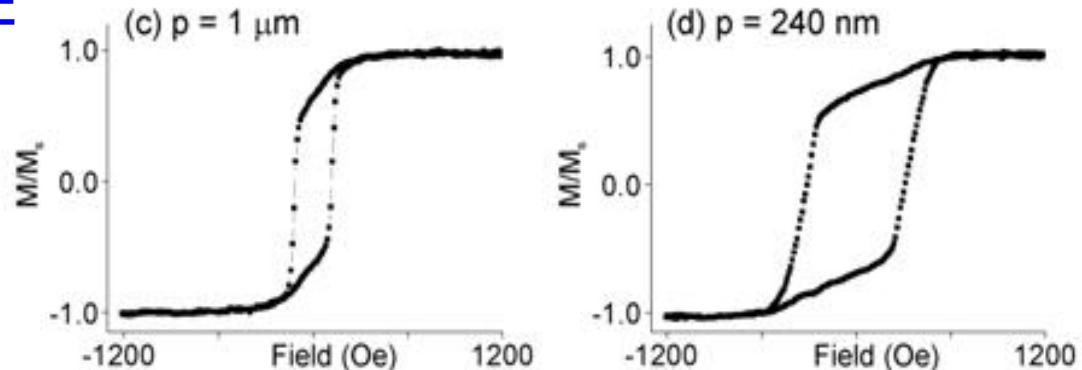
$\mathbf{M}_{001} \sim 0$

# Antidot Arrays – Basic Domain Configuration

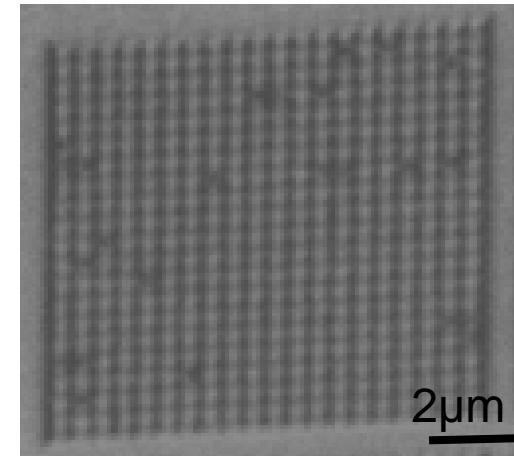
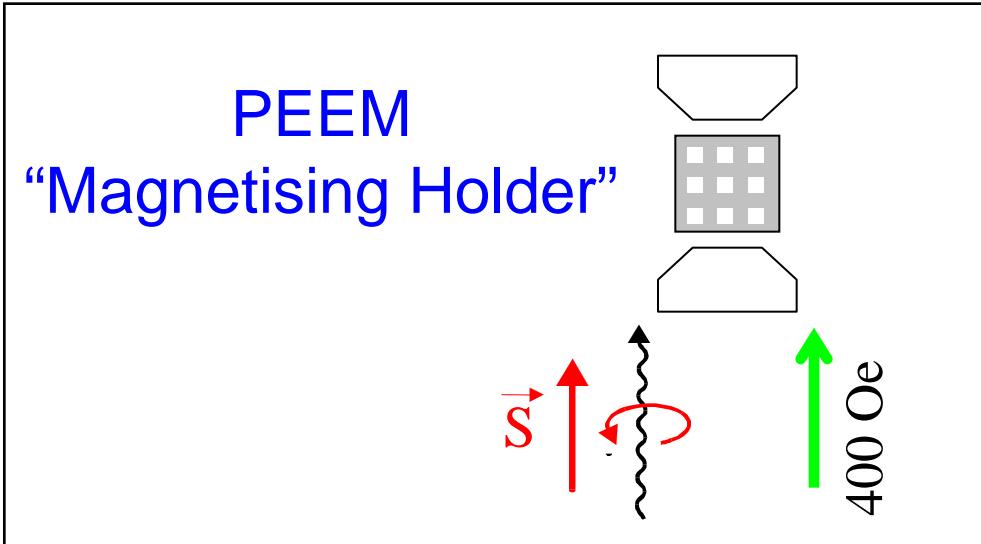


# Remanent Hysteresis Loop in Antidot Arrays

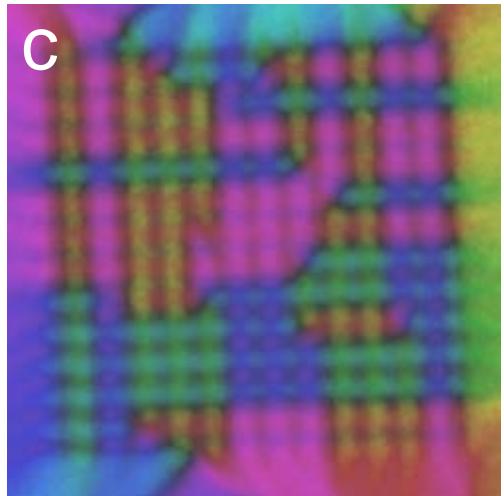
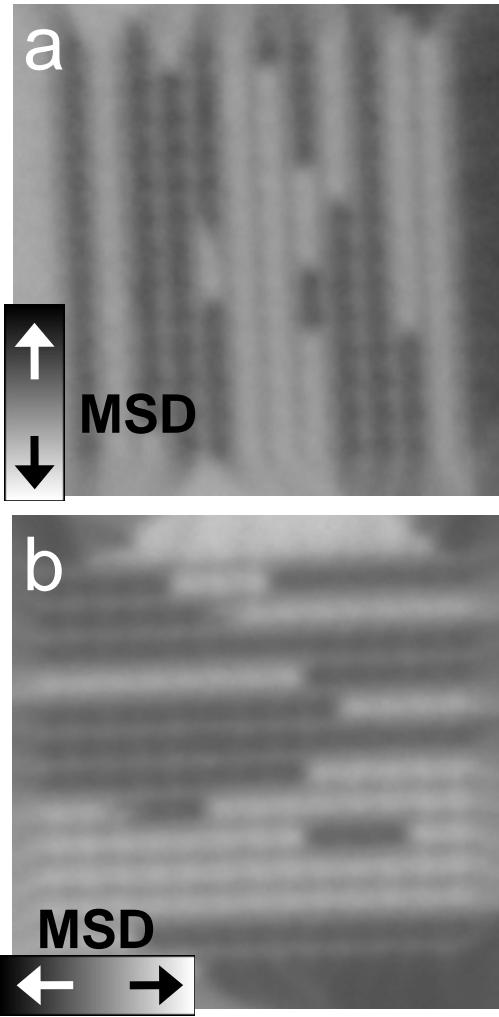
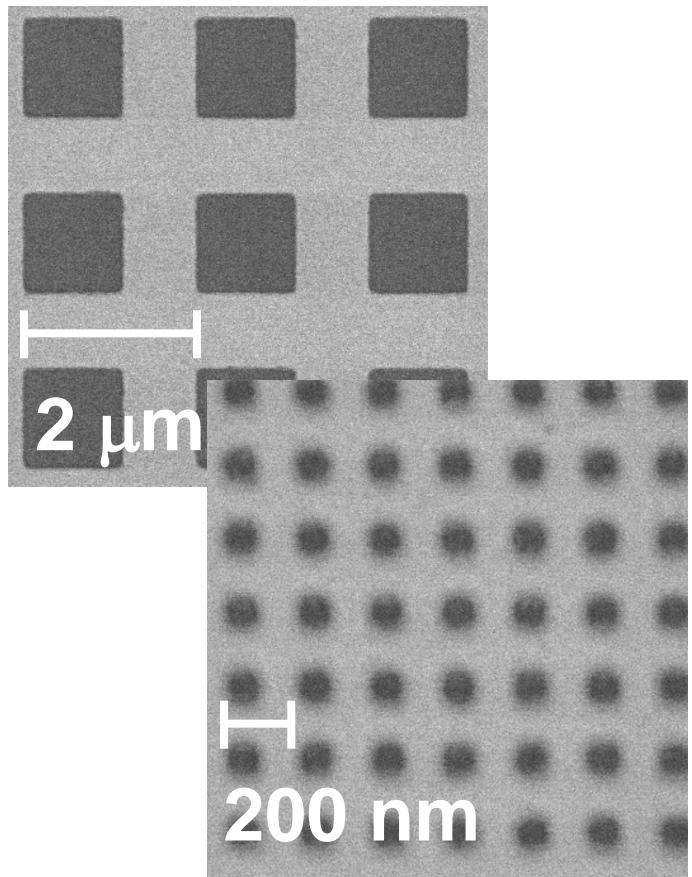
MOKE



Observe magnetisation reversal in applied magnetic field:

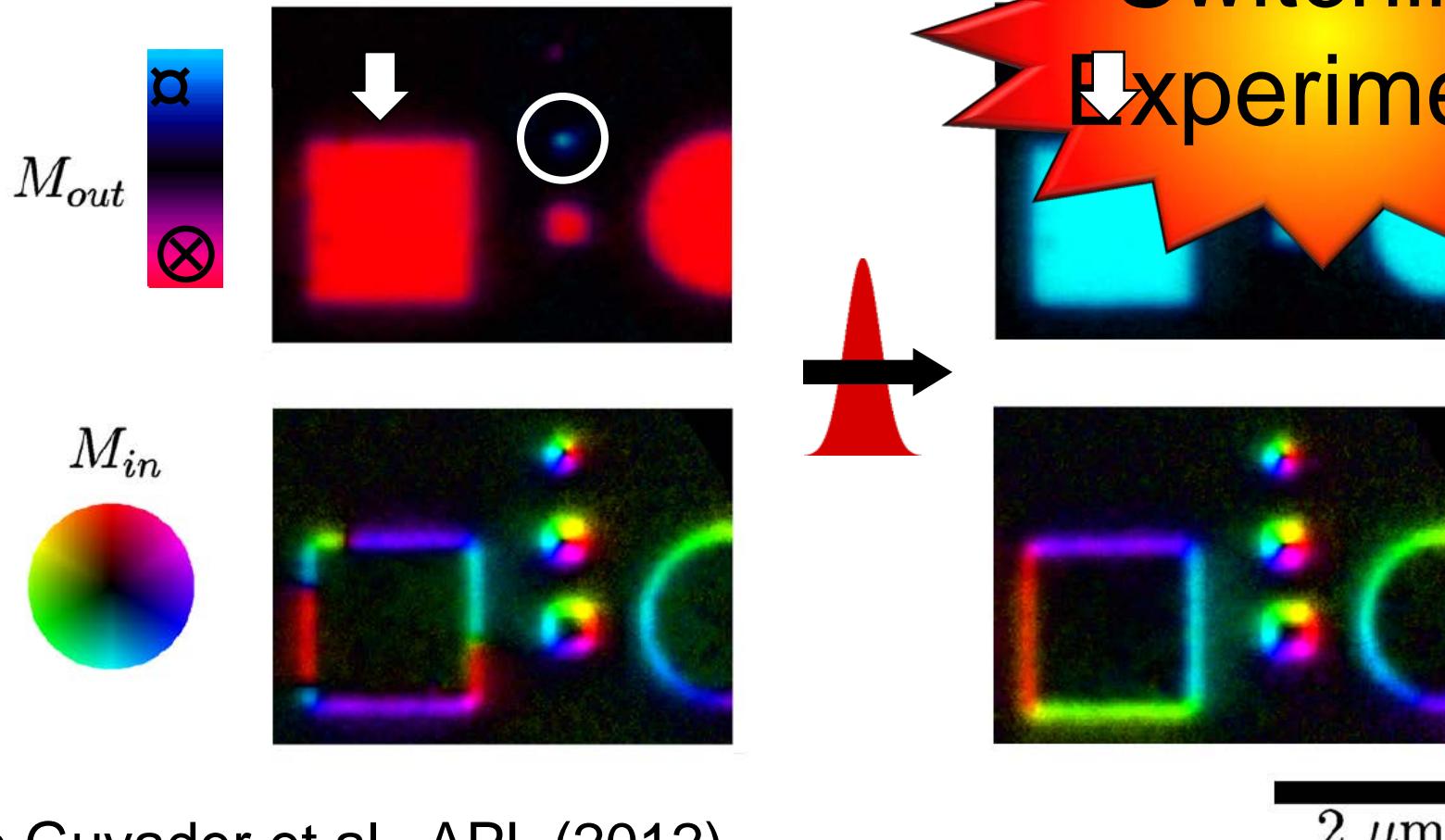


# Cobalt Antidot Arrays



L. J. Heyderman et al., APL (2003), JAP (2004), PRB (2006), JMMM (2007)  
Mengotti et al., JAP (2007)

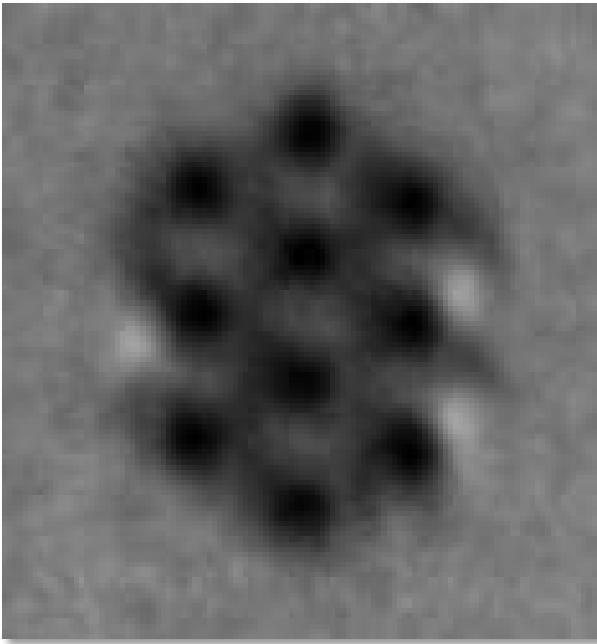
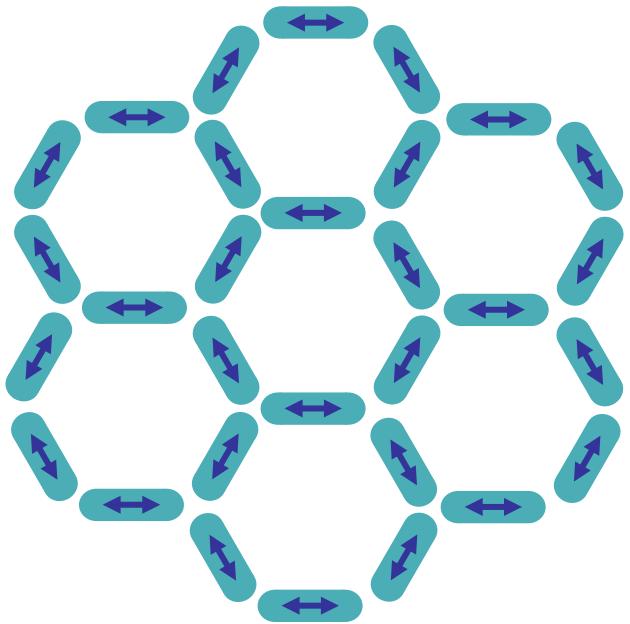
- ❖ 200 nm domains in 400 nm GdFeCo nano



Switching  
Experiments

L. Le Guyader et al., APL (2012)

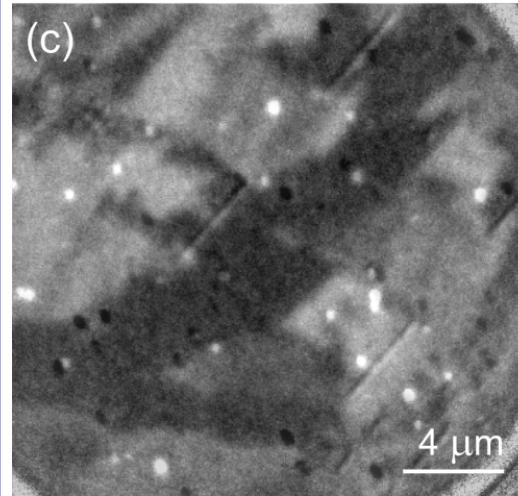
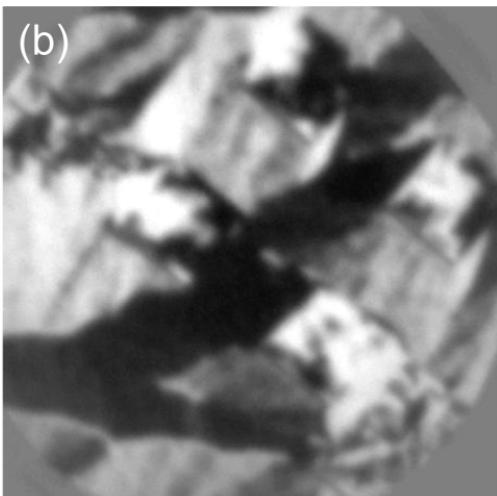
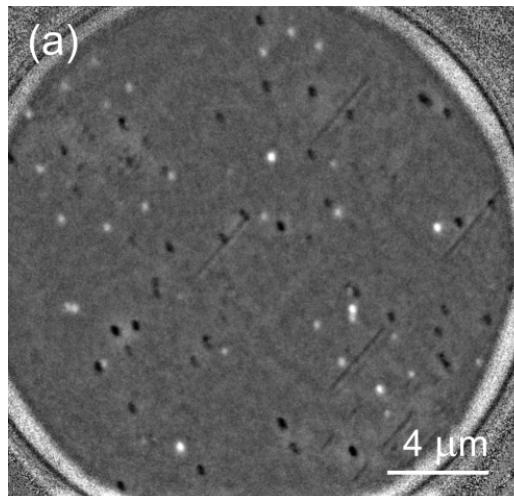
T. A. Ostler et al., Nature Communications (2012)



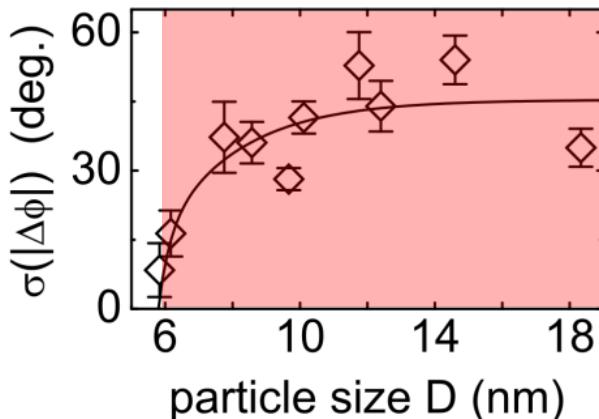
A. Farhan et al. Nature Physics (2013), PRL (2013) & PRB (2014)

# Iron Nanoparticles Coupled to Cobalt Thin Film

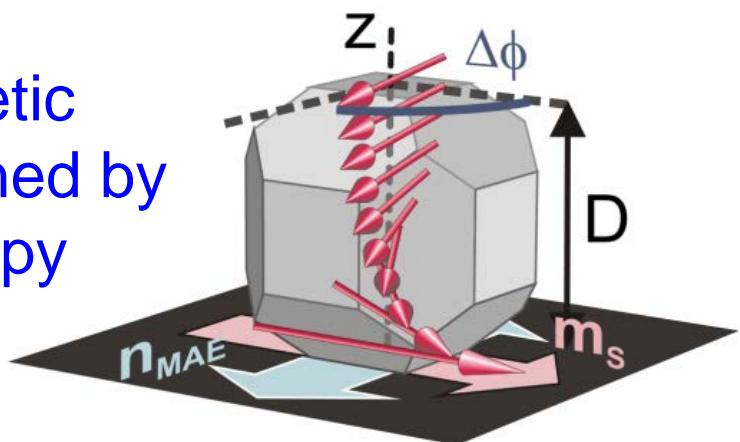
5-25 nm Fe particles/Co thin Film



Noncollinear alignment  
for particles  $> 6$  nm

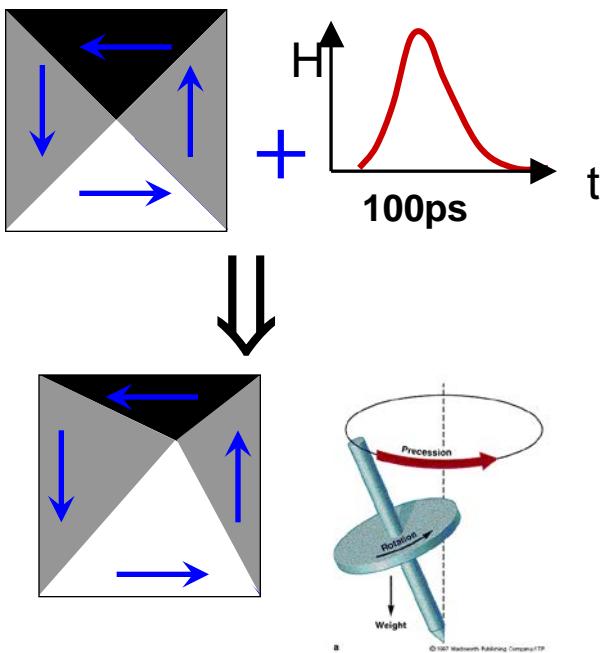


Spin-spiral magnetic  
structure determined by  
magnetic anisotropy  
energy



A. Fraile Rodríguez, A. Kleibert, J. Bansmann, A. Voitkans, L. J. Heyderman,  
and F. Nolting, PRL (2010)

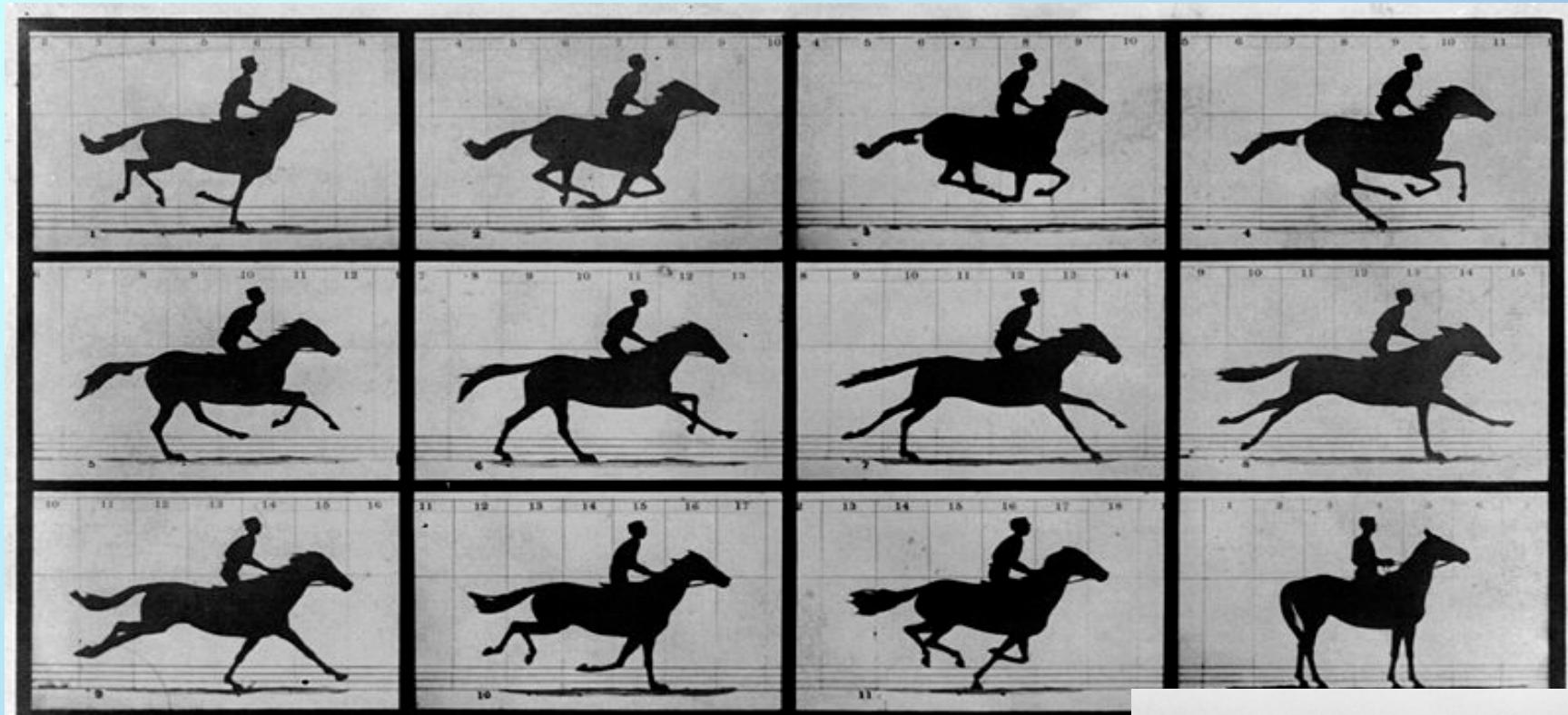
# Time Resolved Imaging



- Image excitations in magnetic nanostructures
- Precession frequency & damping
- Pump-probe experiment
- SLS: X-ray stroboscope

J. Raabe et al., Phys. Rev. Lett. 94, 217204 (2005)

# Why perform time-resolved imaging?



Copyright, 1878, by MUYBRIDGE.

## THE HORSE IN MOTION.

Illustrated by  
MUYBRIDGE.

"SALLIE GARDNER," owned by LELAND STANFORD; running at a 1.40 gait over the Pa-

The negatives of these photographs were made at intervals of twenty-seven inches of distance, and about the twenty-fifth part of a second of time was assumed in each twenty-seven inches of progress during a single stride of the mare. The vertical lines were twenty-seven inches apart, and these lines represent elevations of four inches each. The exposure of each negative was less than the two-thousandth part of a second.

Are all four feet of a horse off the ground at the same time during a gallop.

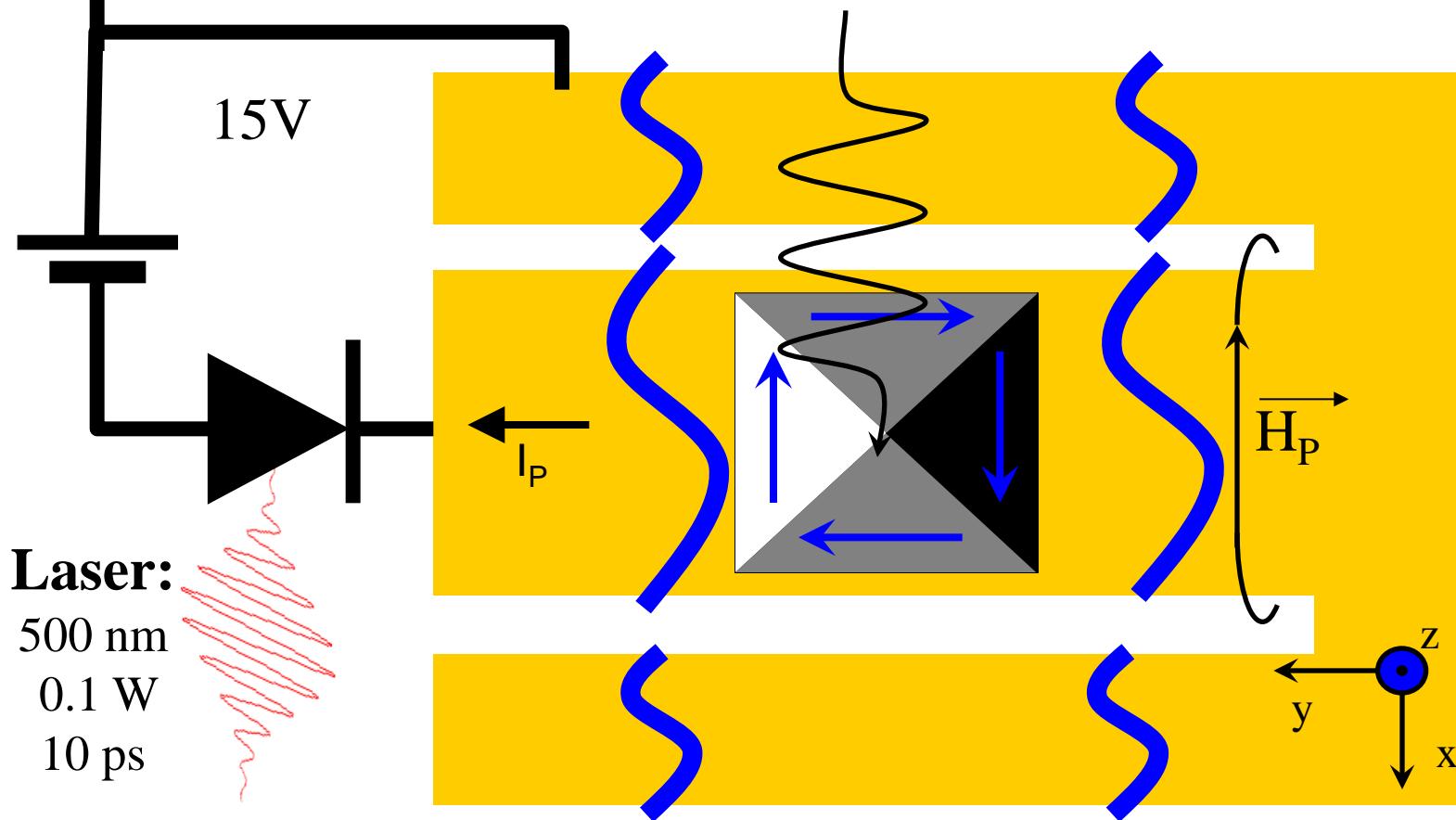


*The Horse in Motion* 1878, animated in 2006, using photos by Eadward Muybridge, Wikipedia

# Sample Layout

-20kV

15V



X-Rays: ~1 keV, C+ / C-



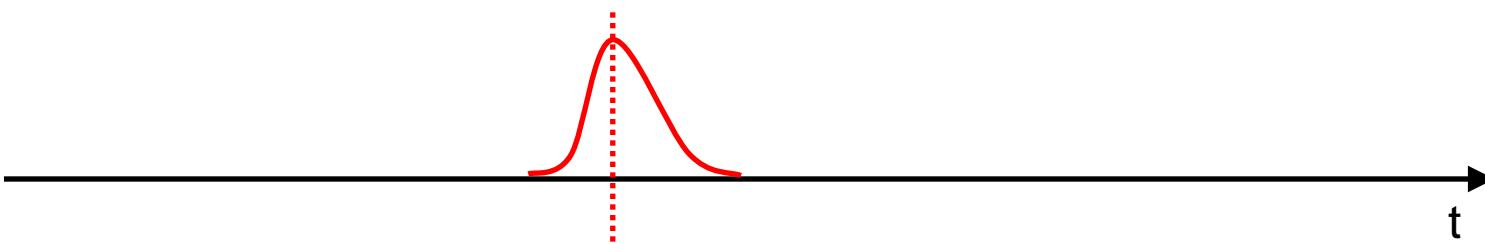
## Magnetic Coil + Fast Optical Switch

Pulsed laser illuminates photodiode to give a current pulse.  
This creates a magnetic field pulse exciting the magnetization.

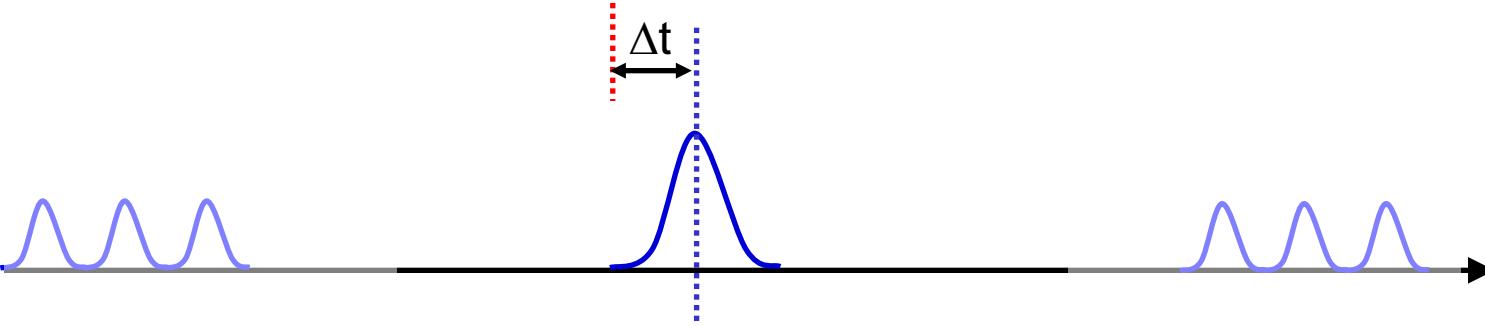
- Excite system with magnetic pulse
- Time later: measure with an x-ray pulse

**Pump**

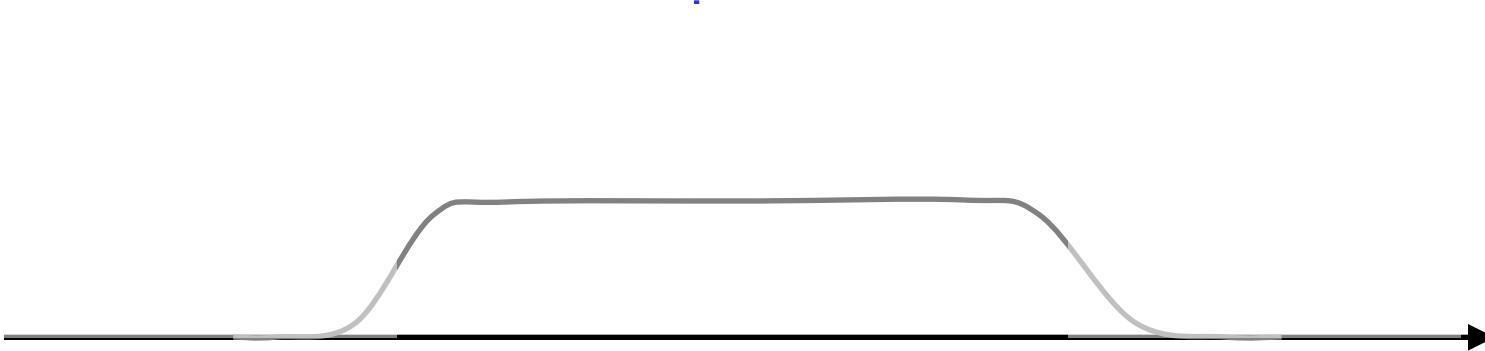
Magnetic  
pulse,  
laser  
pulse etc

**Probe**

X-ray  
pulse

**Gate**

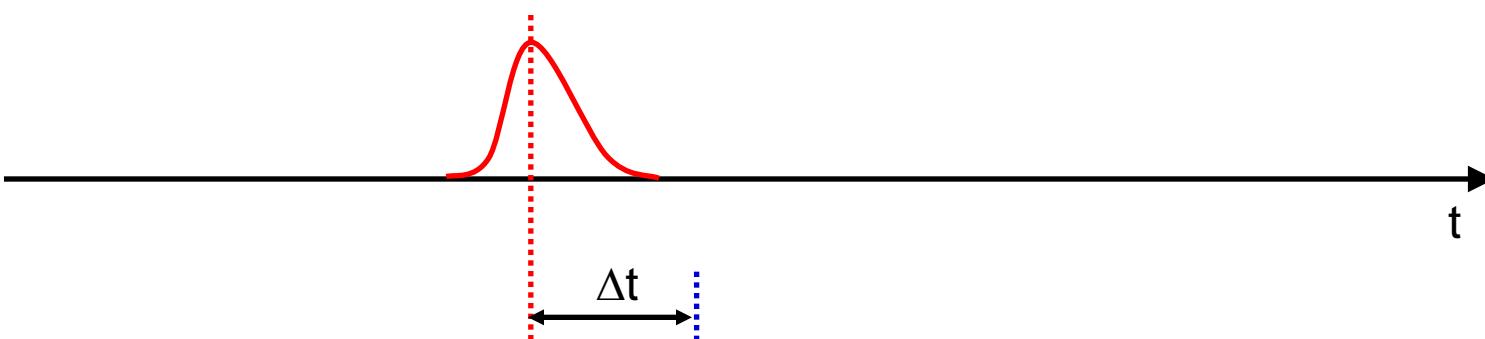
detector  
voltage



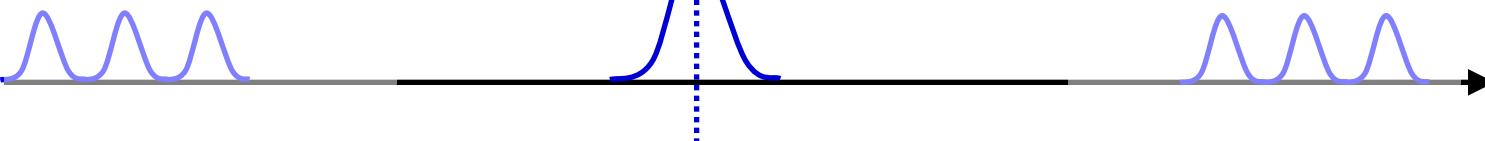
- Excite system with magnetic pulse
- Time later: measure with an x-ray pulse

**Pump**

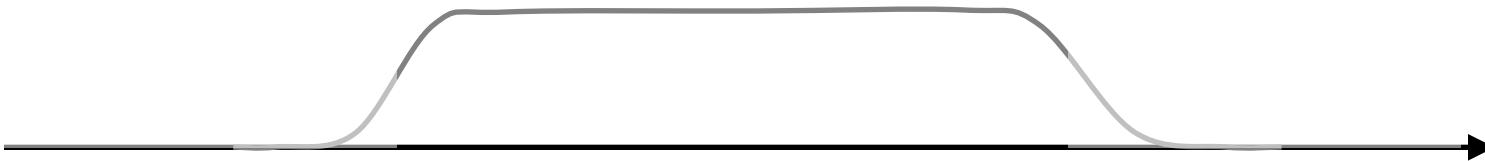
Magnetic  
pulse,  
laser  
pulse etc

**Probe**

X-ray  
pulse

**Gate**

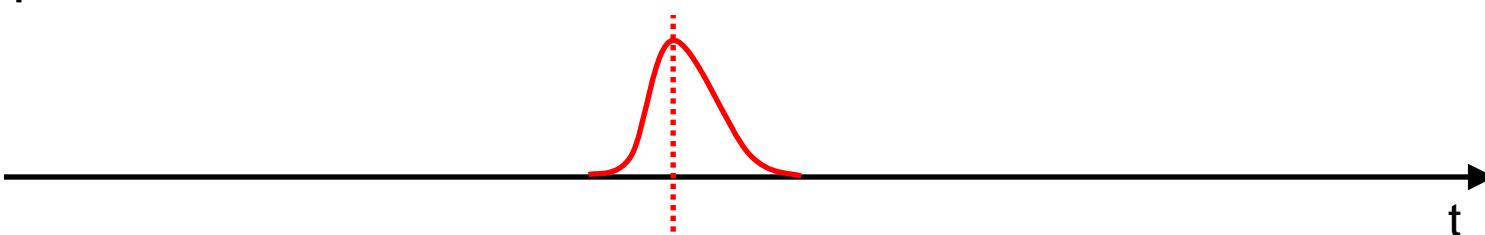
detector  
voltage



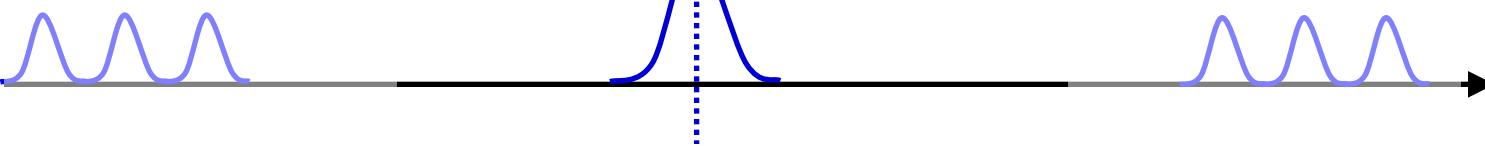
- Not enough intensity in each shot so repeat several times:  
pump-probe

**Pump**

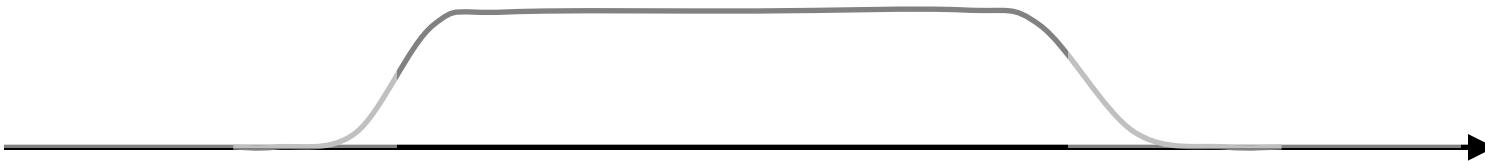
Magnetic  
pulse,  
laser  
pulse etc

**Probe**

X-ray  
pulse

**Gate**

detector  
voltage



# Summary

## Pump:

stripline / coil

Pulse:  $H < 100 \text{ G}$ ,  $10^2 \text{ ps}$

## Detect:

gated PEEM

$\Delta x \sim 100 \text{ nm}$ ,  $\sim 1 \text{ ML}$

## Probe:

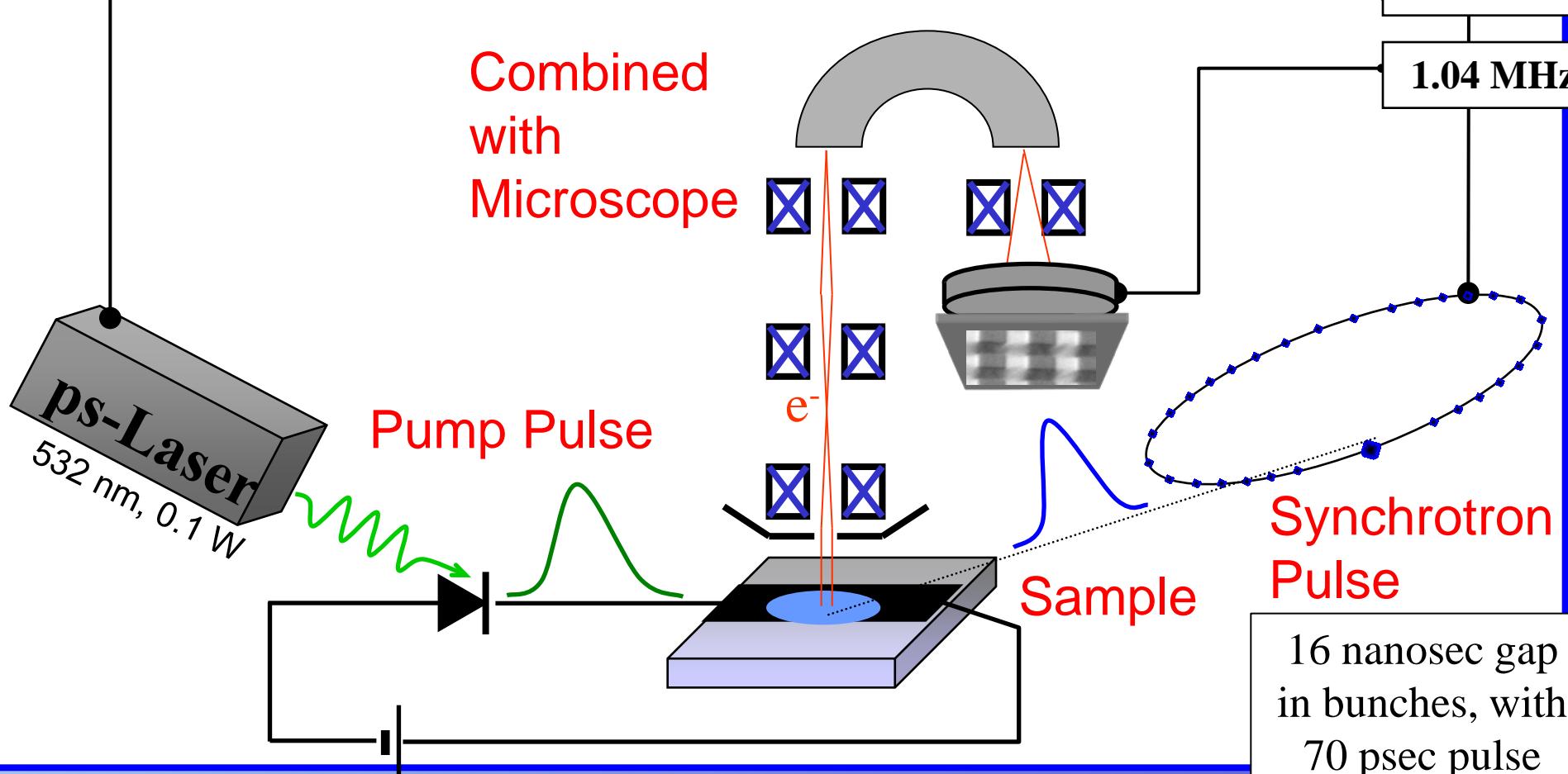
X-ray stroboscope

$\sim 1 \text{ keV}$ ,  $\Delta t = 70 \text{ ps}$

500 MHz

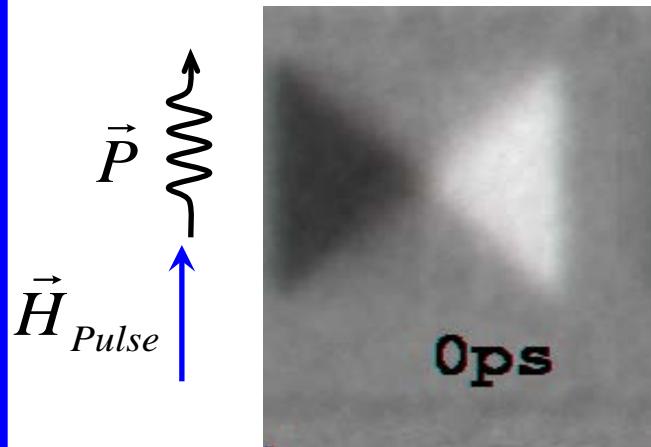
1.04 MHz

Combined  
with  
Microscope



16 nanosec gap  
in bunches, with  
70 psec pulse

# Py Square: Excitation



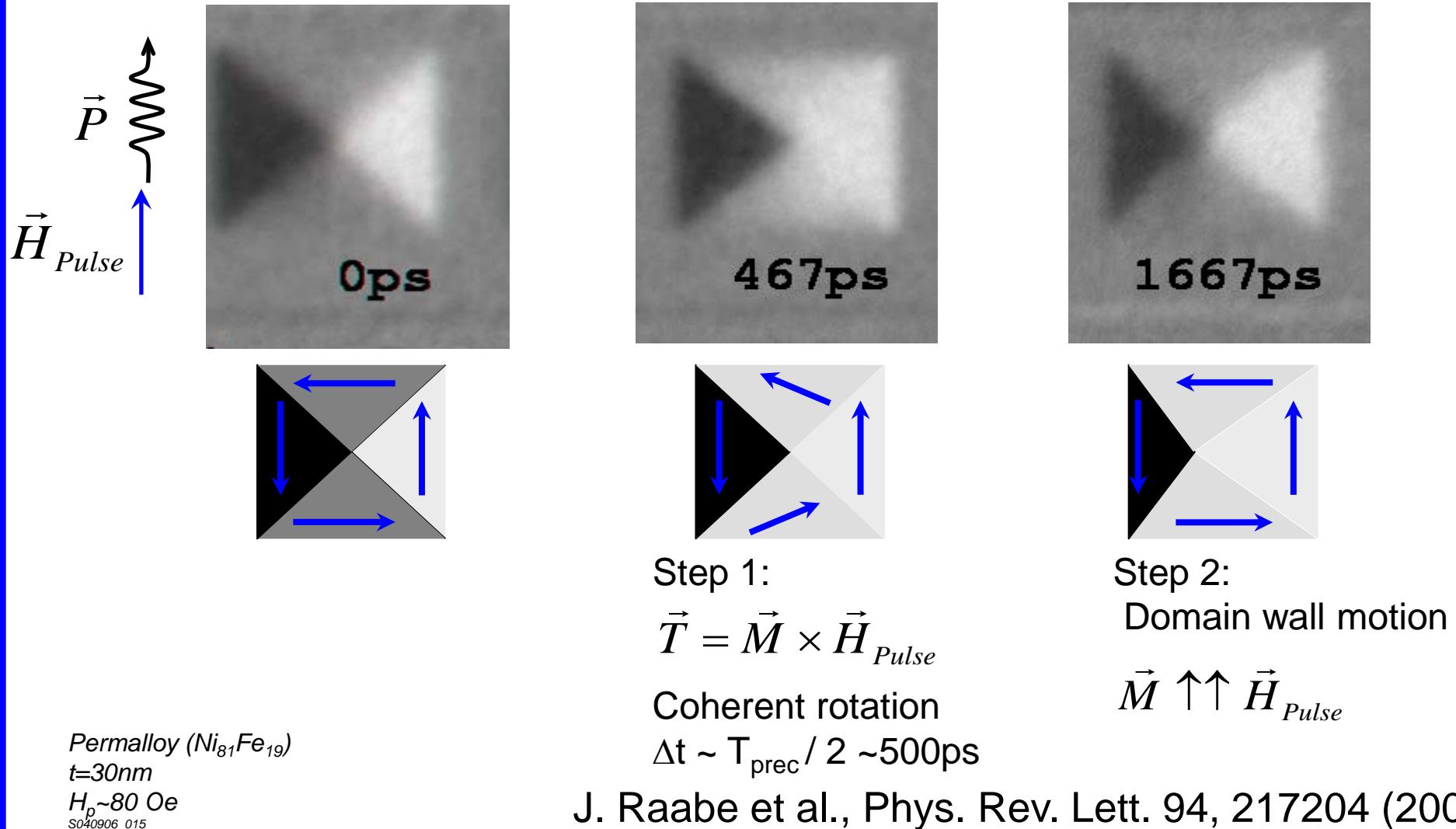
Permalloy ( $Ni_{81}Fe_{19}$ )

$t=30nm$

$H_p \sim 80$  Oe  
S040906\_015

J. Raabe et al., Phys. Rev. Lett. 94, 217204 (2005)

# Py Square: Excitation



# Summary

- Element selective (multilayer, coupled systems)
- Surface/interface sensitive (sampling depth a few nm)
- Antiferromagnetic and Ferromagnetic domains
- Spatial resolution: 50-20 nm, future aberration corrected: few nm's
- Time resolved measurements
- Temperature 120 K – 1000 K
- Submonolayer sensitivity
- Combination with other analytical techniques: LEEM & LEED
- In-situ and ex-situ sample preparation
- Sample size 3 to 15 mm diameter, 0.2 mm - 2 mm thick

## Challenges (limitations):

- UHV compatible ( $<10^{-7}$  mbar)
- Smooth surface ( $< 1 \mu\text{m}$ , hard to say)
- X-ray damage
- Image in applied magnetic field below 50 Oe
- High voltage often leads to discharges (20 keV, at 2 mm distance)
- Charging effects due to electrical insulating sample (can get around this)

# Conclusion

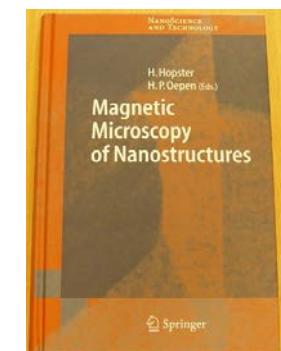
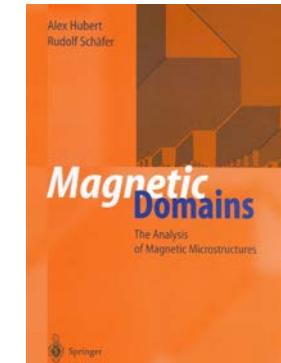
# Information on Different Methods

A. Hubert and R. Schäfer, Magnetic Domains  
The Analysis of Magnetic Microstructures

Magnetic Microscopy of Nanostructures

An overview of techniques to image the magnetic structure on the nano-scale

H. Hopster and H. P. Oepen



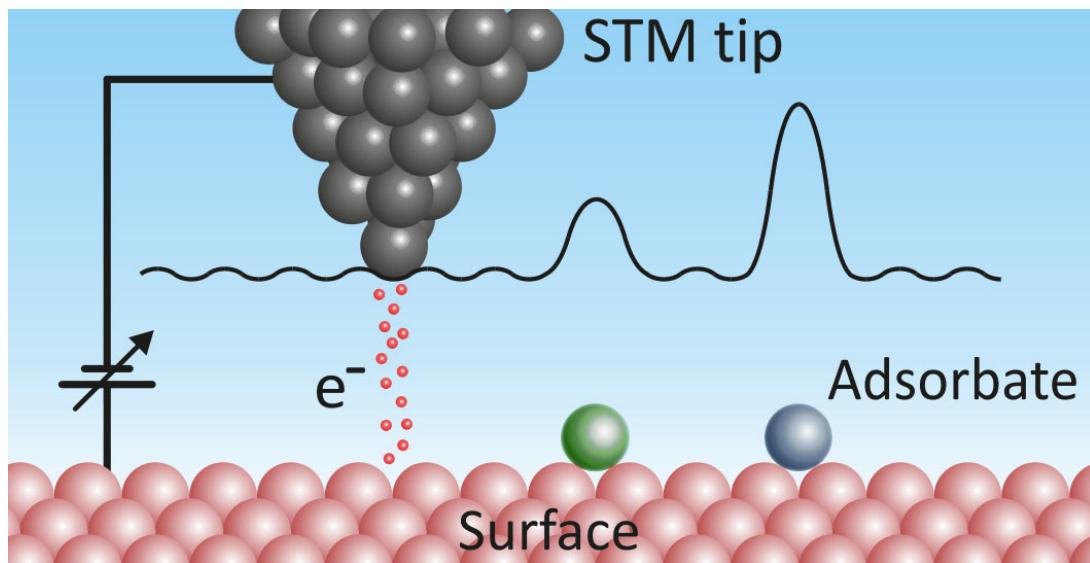
Internet, for example:

Techniques to Measure Magnetic Domain Structures, R.J. Celotta, J. Unguris, M.H. Kelley, and D.T. Pierce, Methods in Materials Research (2000)

# Comparison Between Different Techniques

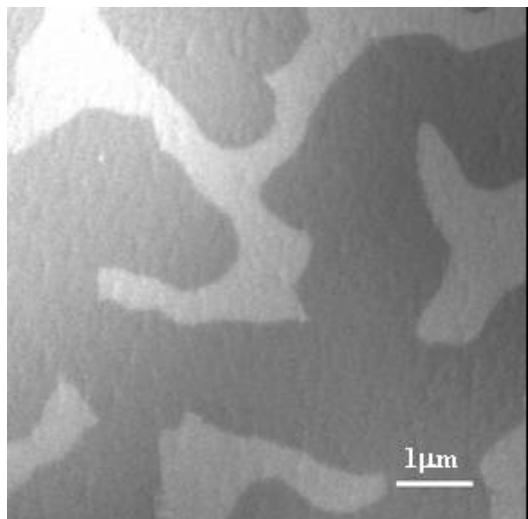
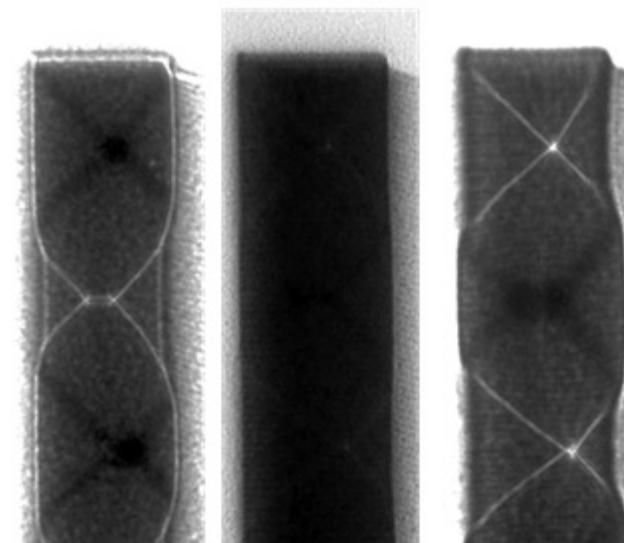
- Contrast Origin: B, M,  $H_{ext}$
- In Plane or Out-of-Plane components
- Quantitative or Qualitative
- Best Resolution, but better Typical Resolution
- Information depth
- Sensitivity, Acquisition Time
- Vacuum Equipment: none, HV, UHV
- Sample requirements: thickness, surface roughness, clean surface, insulators ?
- In-situ experiments: maximum field, heating, stress
- Additional information: crystallography, topography, chemical, electronic
- Commercial Availability, Cost & Complexity - Manpower

# Further Techniques



<http://www.fhi-berlin.mpg.de/>

Overfocus   In Focus   Underfocus

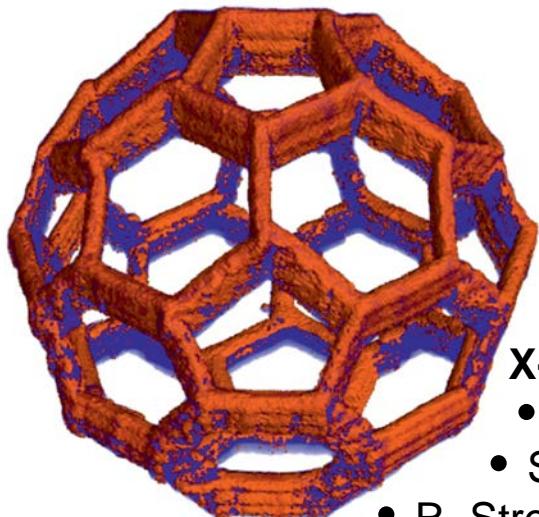


B.-S. Kang et al.  
J Appl. Phys 98 (2005) 093907

<http://lma.unizar.es>

- Scanning Tunnelling Microscopy
- Lorentz Microscopy
- Transmission X-ray Microscopy
- X-ray & Neutron Tomography
- X-ray & Neutron Scattering
- Low Energy Muons

# Further Techniques

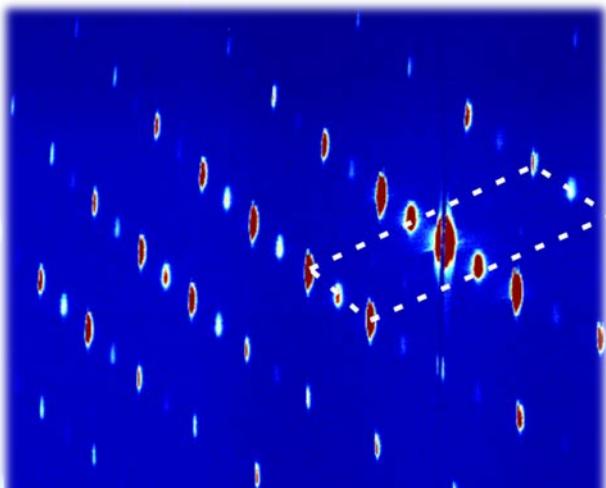


- **Muons:** L. Anghinolfi et al.  
Nat. Comm. (Accepted 2015)

**X-rays:**

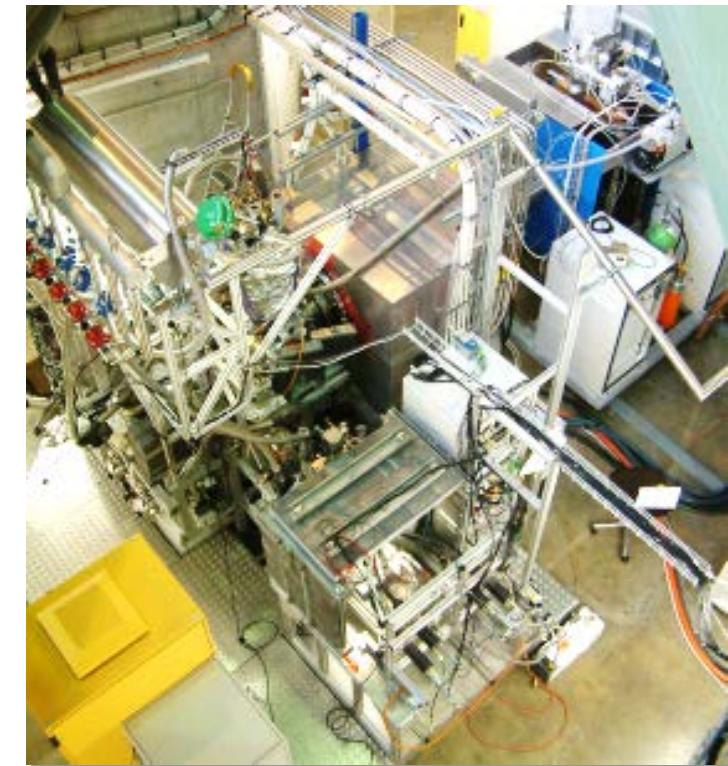
- C. Donnelly et al. PRL (2015)
- S. Da Col et al. PRB(R) (2014)
- R. Streubel et al. Nat. Comm. (2015)

**Neutrons:** Manke et al. Nat. Comm. (2010)



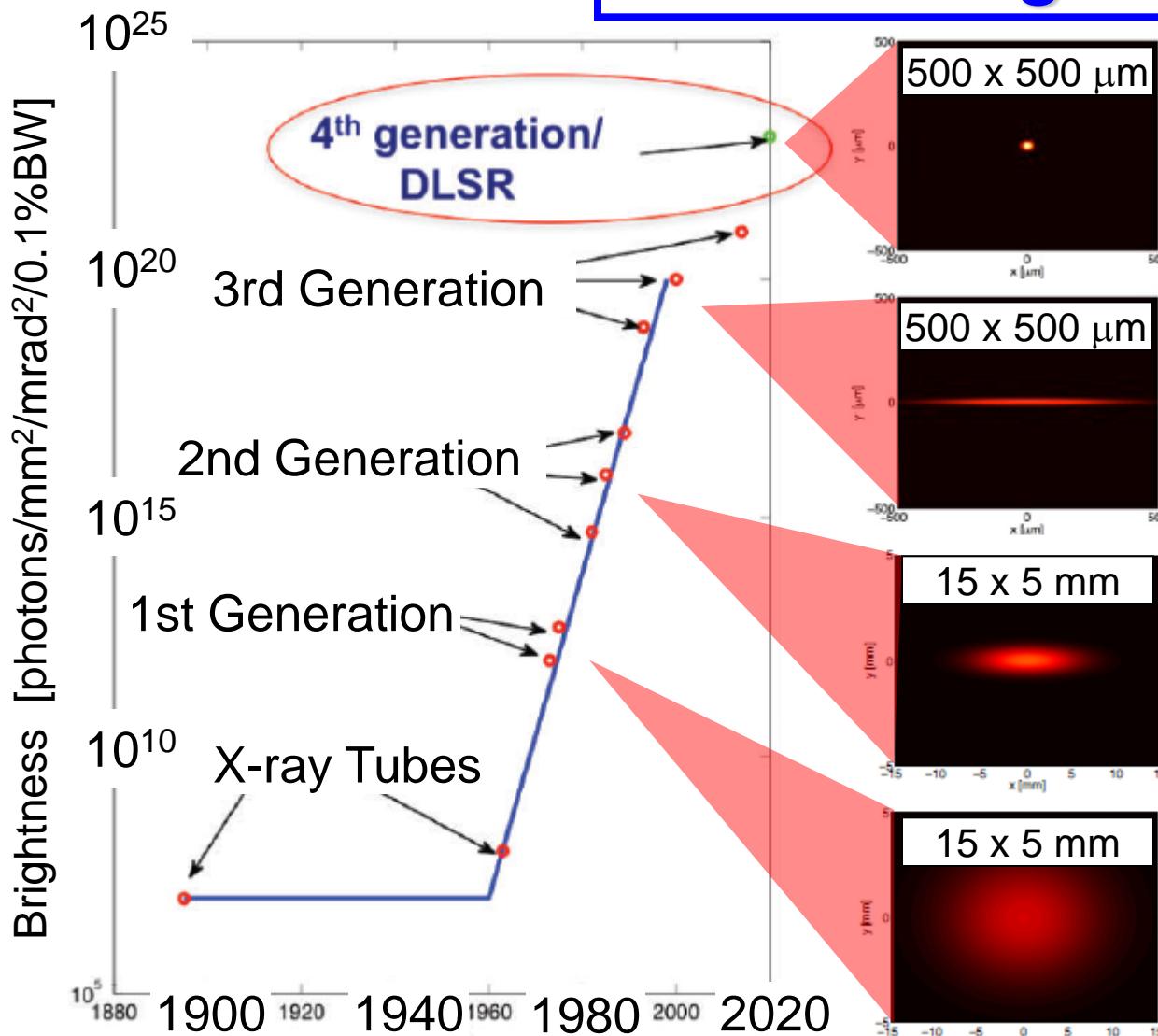
**X-rays:** J. Perron et al. PRB (2013)

**Neutrons:** T. Maurer et al. PRB (2014)



- Scanning Tunnelling Microscopy
- Lorentz Microscopy
- Transmission X-ray Microscopy
- X-ray & Neutron Tomography
- X-ray & Neutron Scattering
- Low Energy Muons

# Future Light Sources



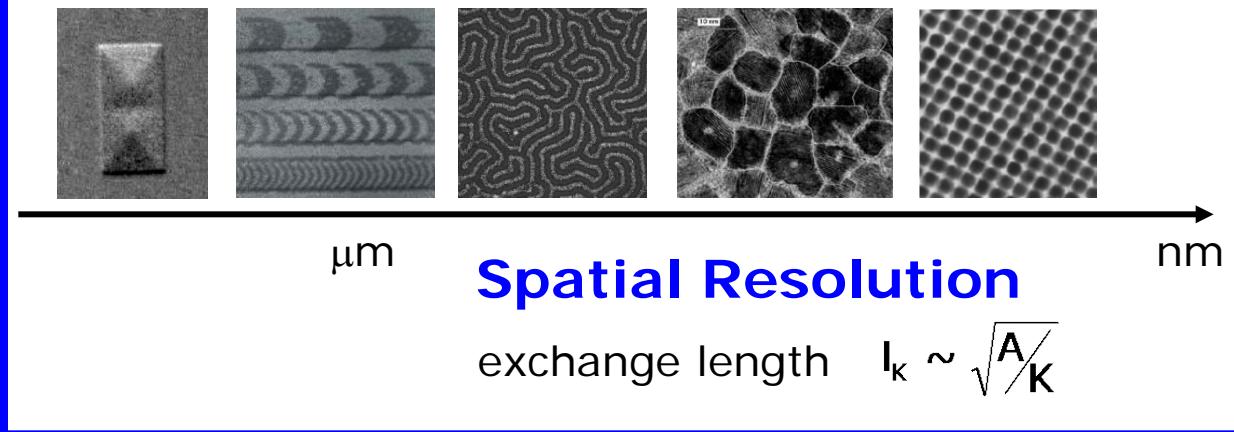
- 4th Generation/DLSR**  
(diffraction limited storage ring)
- Multibend Achromat (MBA) accelerator lattices
  - Large increase in brightness
  - Several soft bend magnets in each storage ring sector replace 2-3 hard bend magnets
  - smaller horizontal beam dispersion corrected by stronger focusing magnets
  - elliptical profile replaced by compact and nearly circular profiles, with horizontal spatial & angular widths of source decreased by ~ factor of 10 relative to existing sources

**Soft X-ray Science Opportunities Using Diffraction-Limited Storage Rings, ALS 2014 (Scale → Rectangle Size)**

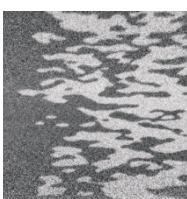
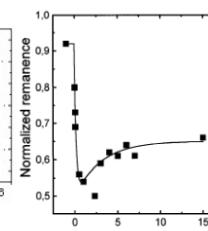
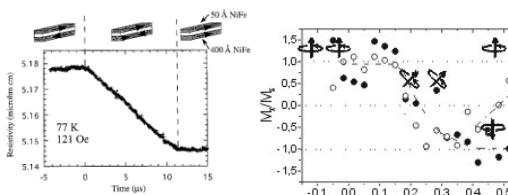
# Future Challenges

# Time Resolution

- Currently sub100ps ( $\approx$ 10ps): precession relaxation dynamics (LLG).
  - Limited flux of photons: repeatable phenomena (stroboscopic pump-probe).



ns                    ps                    fs



exchange interactions     $t(\text{fs}) \sim \frac{4}{E(\text{eV})}$

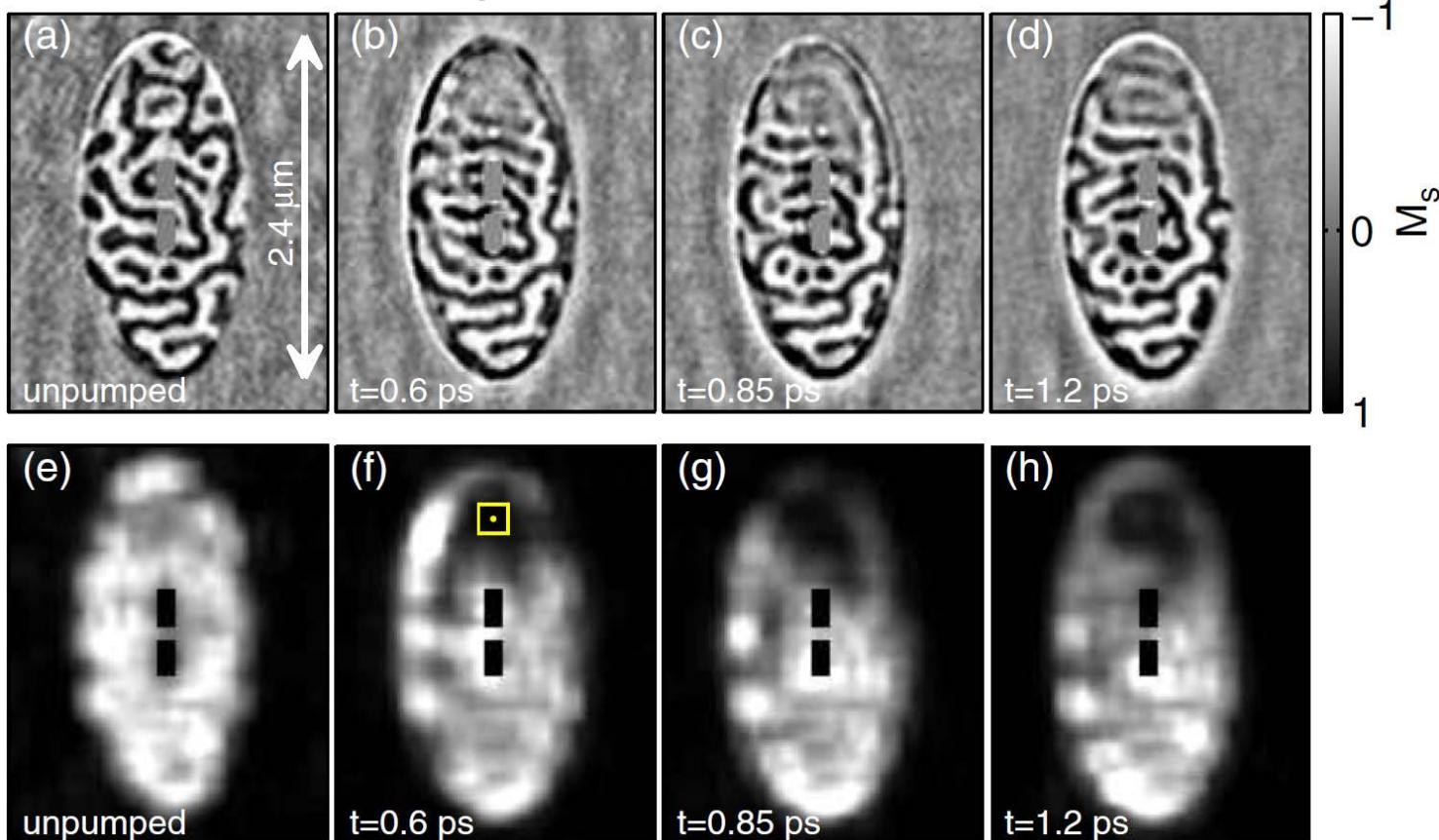
- Future challenge: fs time scale (exchange interaction time, spin fluctuation time)
  - nm spatial resolution in single shot experiment.
  - Need high flux ( $10^{12}$ ph/s ) X-ray source
  - Lensless imaging and Full-field X-ray microscopy

# Elemental Sensitivity

Cu 30nm  
Ta 3,5nm  
Ni<sub>80</sub>Fe<sub>20</sub> 4nm  
AlO<sub>X</sub> 1..2 nm  
Co<sub>70</sub>Fe<sub>30</sub> 3nm  
Ir<sub>17</sub>Mn<sub>83</sub> 12nm  
Cu 30nm

## magnetic tunnel junction

Peter Fischer, ALS

***Ultrafast Optical Demagnetisation (100 fs,  $\lambda=780$  nm, 0.2  $\mu$ J)***

C. von Korff Schmising PRL (2014)

**In their conclusions:** Ultrafast transport of spin-polarized electrons  
→ Domain size controls time scales & spatial extent.  
However, exact mechanisms still to be determined.....

# Test your understanding.....

Magnetic Force Microscopy, Kerr Microscopy & PEEM

Which technique:

- is sensitive to the stray/external magnetic field?
- are sensitive samples with out-of-plane M?
- gives a value of spin and orbital moment?
- has the best spatial resolution?
- can be used to look at back surface of sample?
- requires UHV?
- is difficult for measuring insulators?
- can provide chemical and electronic information?

Can you name any other techniques for imaging magnetic domains?