PAUL SCHERRER INSTITUT PSI

Paul Scherrer Institut, Swiss Light Source

Frithjof Nolting
X-ray microscopy (and PEEM)
Surface Science, FS 2012

Surface Science lecture

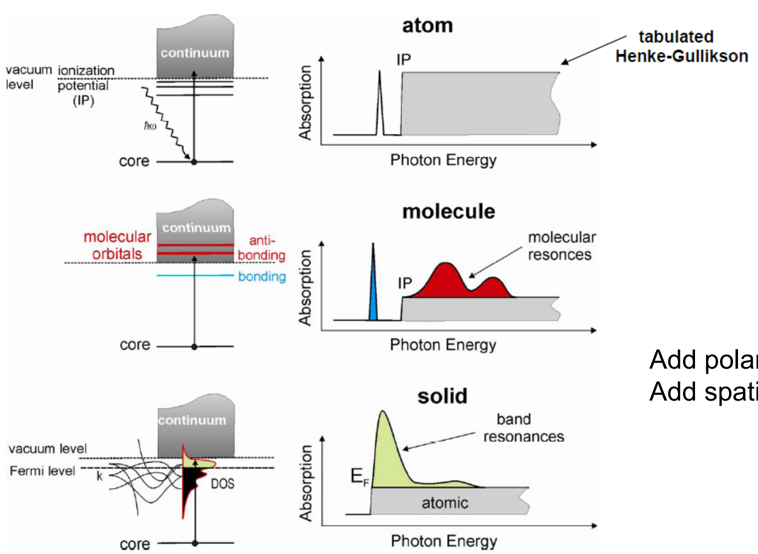
Di, 21.02.2012 Fixing Dates, Intro to Vacuum Technology, Labvisits Basel (Toni Ivas, Sylwia Nowakowska, Roland Steiner)
Di, 28.02.2012 Introduction, Concepts Samples and Structure (Thomas Jung)
Di, 06.03.2012 Adsorption / Desorption (Thomas Jung)
Di, 13.03.2012 Electron Diffraction Methods, in particular RHEED, LEED (Bert Müller)
Di, 20.03.2012 Electronic Properties and Surface Electron Spectroscopies: XPS/UPS, Auger, ARPES (Andrij Romaniyuk)
Di, 27.03.2012 FASNACHT
Di, 03.04.2012 Diffusion and Growth (Thomas Jung)
Di, 10.04.2012 Local Probes and Experiments I, STM, Inelastic tunneling and STS (Thomas Jung)
Di, 17.04.2012 X-ray Absorption Spectroscopy (Frithjof Nolting)
Di, 24.04.2012 Surface Magnetism XMCD / PEEM (Frithjof Nolting)
Di, 01.05.2012 Surface Optics, Kelvin Probe (Thilo Glatzel)
Di, 08.05.2012 Local Probes and Experiments II, AFM FIM (Thomas Jung)
Di, 15.05.2012 Applications of Surface Science in Industry (M. de Wild)
Di, 22.05.2012 Schlussprüfung (Jan Girovsky, Thomas Jung)
Di, 29.05.2012 Excursion (Thomas Jung)

Di, 17.04. 2011 X-ray Absorption Spectroscopy (F. Nolting)
Di, 24.04. 2011 PEEM and X-ray Microscopy (F. Nolting)
Both with an emphasis of magnetism

Repetition VIII

- •Near Edge X-ray Absorption Fine Structure
  reflects density of unoccupied states
- •Absorption processes and decay (soft X-rays)
- •Sampling depths (total electron yield, secondary, Auger, Fluorescence)
- •Multiplet structure (chemical, electronic sensitivity)
- •XMCD (sum rules)
- •XMLD
- •Magnetism (spin and orbital moment, magnetocrystalline anisotropy)

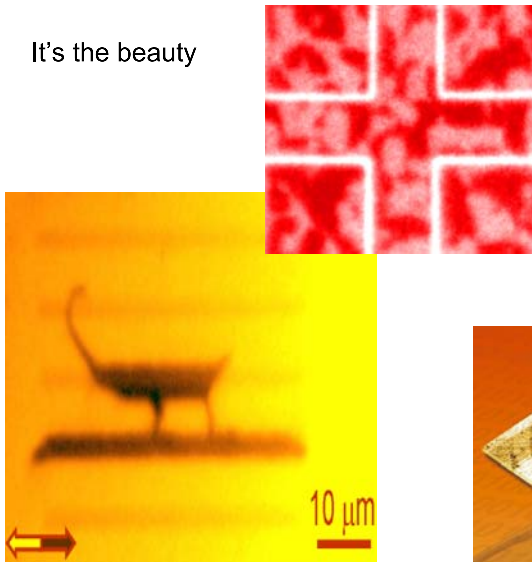
X-ray Absorption Spectra in a Nutshell

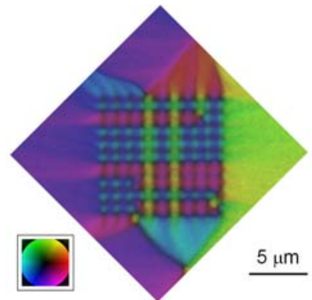


Add polarization
Add spatial resolution

Why do we care

It’s the beauty



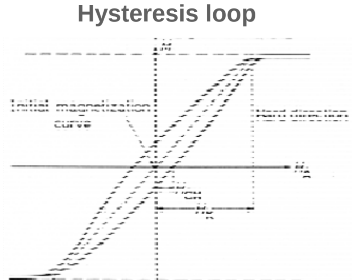


Why do we care

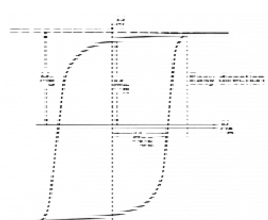
It’s the function

Hysteresis loop

hard axis

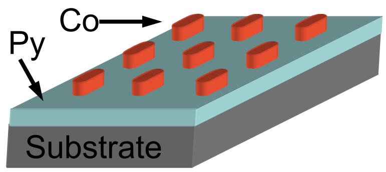


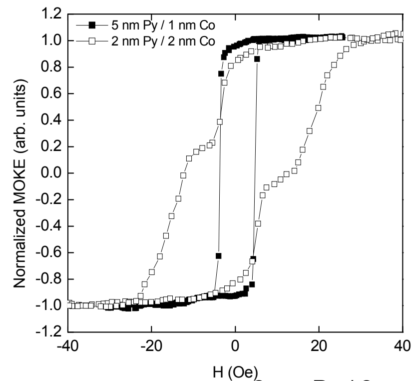
easy axis



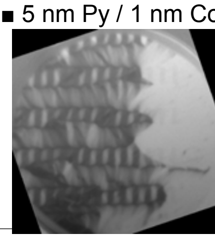
Why do we care

It's the function

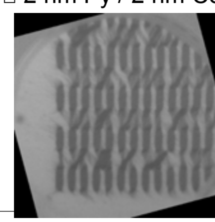




■ 5 nm Py / 1 nm Co



□ 2 nm Py / 2 nm Co



Magnetic domain configuration in the Py film

10 µm

A. Fraile Rodríguez, L. J. Heyderman, F. Nolting, A. Hoffmann, J. E. Pearson, L. M. Doeswijk, M. A. F. van den Boogaart, and J. Brugger, Appl. Phys. Lett. **89**, 142508 (2006).

Outline

The magnetic domain (crash class II)

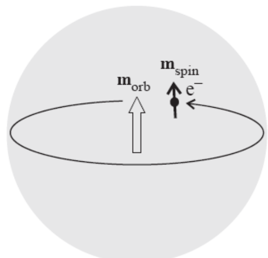
Photoemission Electron Microscope (PEEM)
- Electron optic
- XMCD/XMLD image
- Aberration correction

The X-ray source
- Röntgen
- Storage ring
- Polarized X-rays

Research example
- Nanocrystals

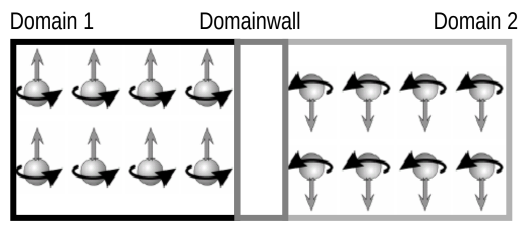
PEEM without X-rays

Switching on the interaction



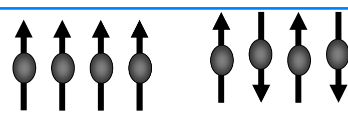
Atoms have an magnetic moment

With interaction they can align to each other and can create macroscopic magnetic field

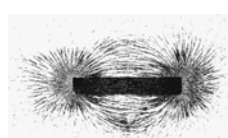


Domains - Energy Minimization

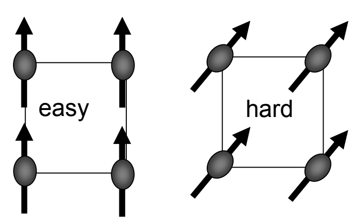
Exchange energy:
ferromagnet parallel spins
antiferromagnet antiparallel spins



Magnetostatic energy
Closure

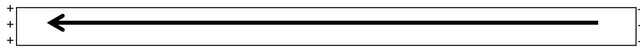


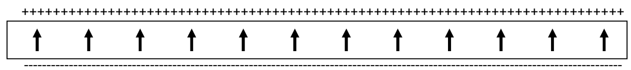
Magnetic Anisotropy
preferential magnetization along axes
easy / hard axis



Zeeman :
spin alignment in the external magnetic field

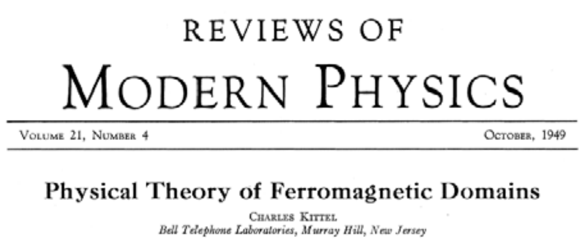
Magnetostatic energy





Costs more energy

Domains - Energy Minimization


REVIEWS OF

MODERN PHYSICS

VOLUME 21, NUMBER 4 OCTOBER, 1949

Physical Theory of Ferromagnetic Domains

CHARLES KITTEL

Bell Telephone Laboratories, Murray Hill, New Jersey

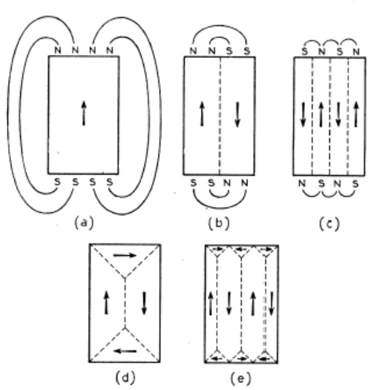


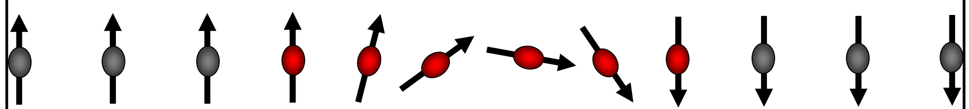
FIG. 9. The origin of domains.

1907 Weiss
1931 Sixtus and Tonks
1932 Bitter
1935 Landau and Lifshitz

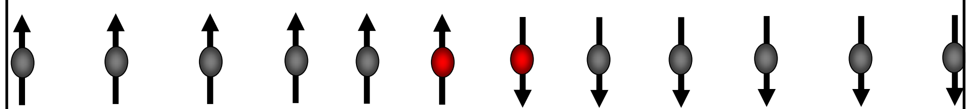
Reviews
C. Kittel Rev. Mod. Phys **21** (1949) 541
A. Hubert and R. Schäfer "Magnetic Domains" (Berlin: Springer) 1998

Domain Walls

Exchange energy favors wide walls:



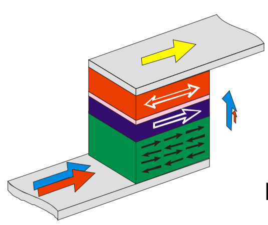
Anisotropy energy favors narrow walls:



Domain wall width

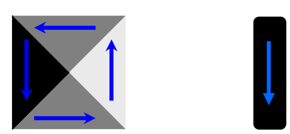
$$\lambda \sim a \left(\frac{E_{exchange}}{E_{anisotropy}} \right)^{1/2}$$

one dimension below critical length scale

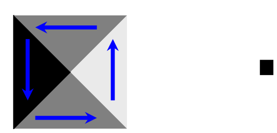


Domain size, domain wall width, Exchange length
Spin diffusion length, Spin precession length

Effect of shape



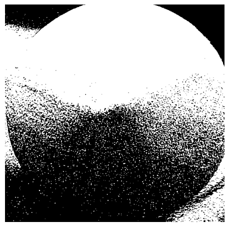
Effect of size



Effect of composition, coupling …

Dealing with multielements, ferromagnetic, antiferromagnetic

What do we need to take an image?



X-ray microscope

source

X-ray tube
Synchrotron
- Bending magnet
- Insertion device

optics

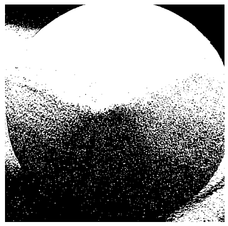
Mirrors
Refractive elements
Diffractive elements
Electron optic

detector

Photodiode
Phosphorscreen
…

sample

What do we need to take an image?



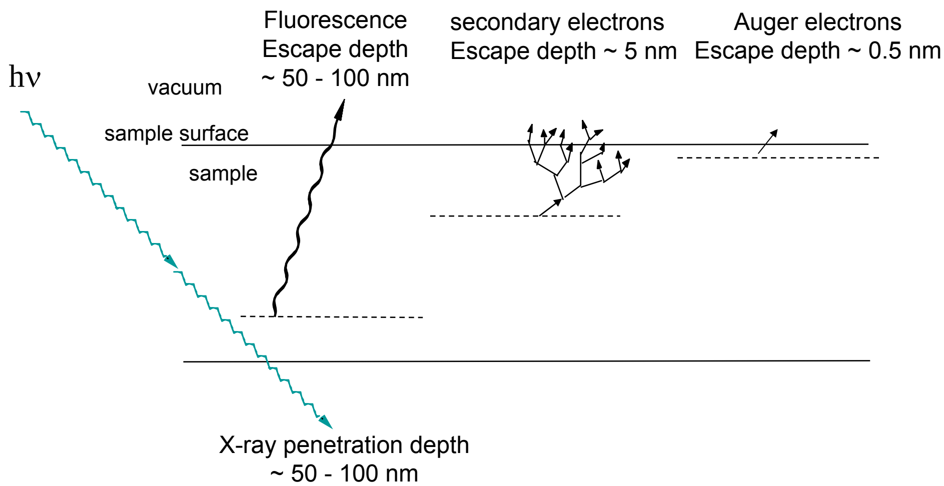
F. Nolting Surface Science FS 2012

http://perso.neel.cnrs.fr/olivier.fruchart/

F. Nolting Surface Science FS 2012

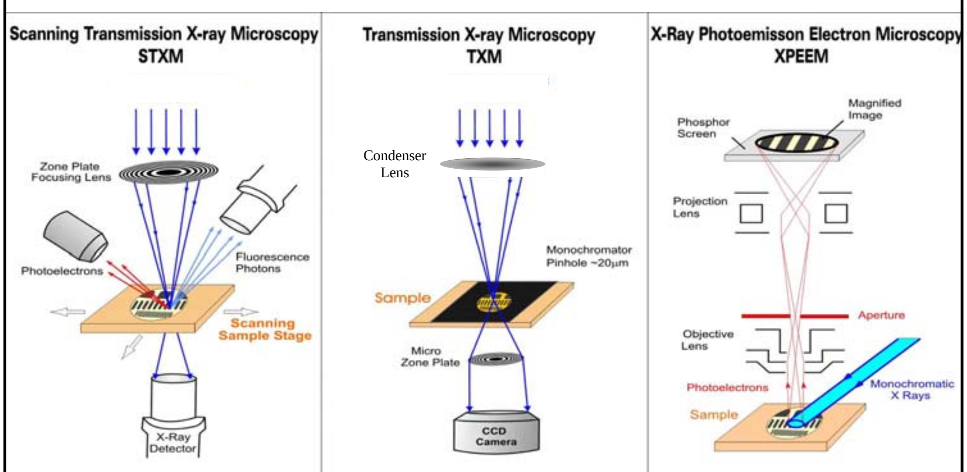
Detecting methods

Photon in / Photon out — **Photon in / Electron out**



How?

Different ways of measuring X-ray absorption
=
Different ways of magnetic imaging with X-rays



Courtesy J. Stöhr

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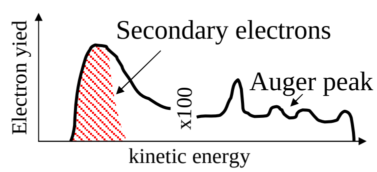
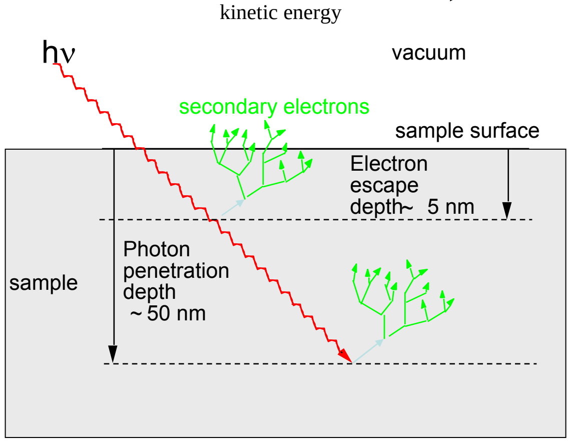
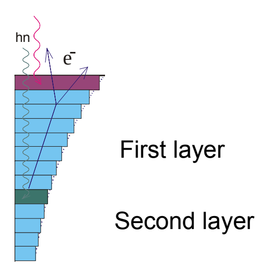
PEEM without X-rays

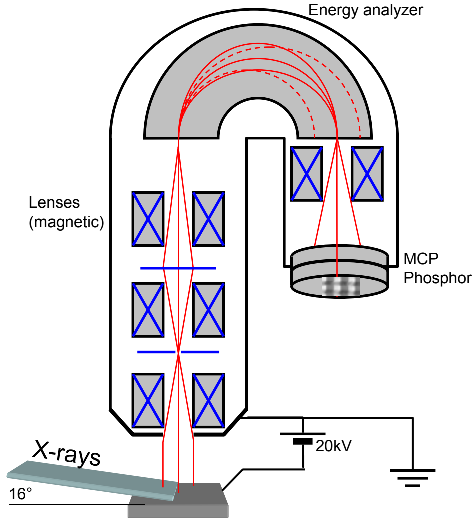


Probing surface/interface





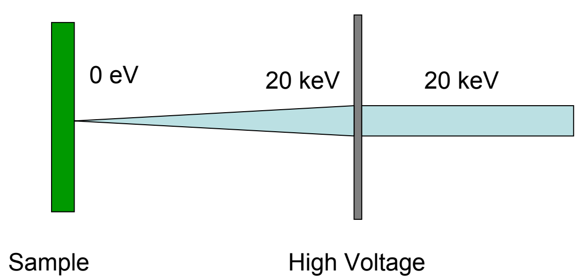
Photoemission Electron Microscope - PEEM



Slow electrons

Probe : slow electrons

Imaging : high energy electrons
(more stable and maintain spatial information)

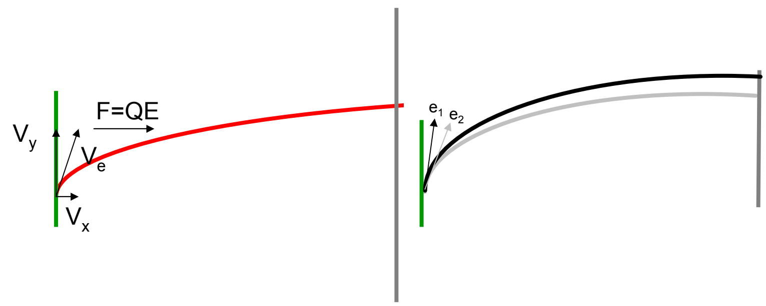


Immersion lens: electrons have before and after the lens different velocity (different wavelength)

Cathode lens: Sample is cathode
electron microscope is anode

Just another lens?

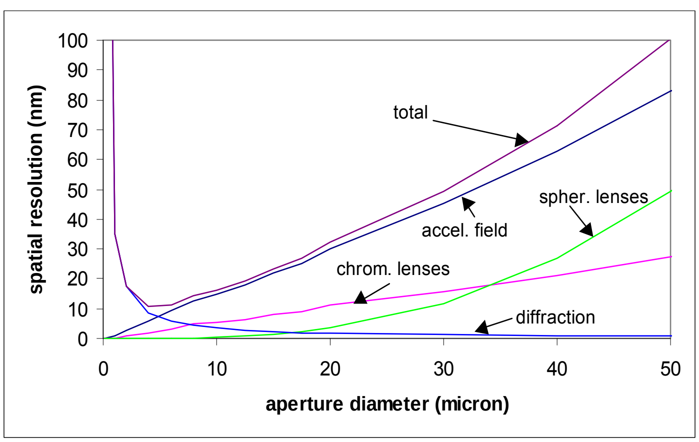
Classical: electron in homogenous electric field
calculate electron trajectory



Trajectory depends on emission angle and velocity

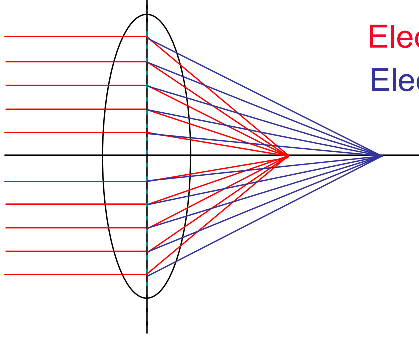
No, it is a very important lens in a PEEM, dominating the spatial resolution due to its spherical and chromatic aberrations.

Calculated Spatial Resolution



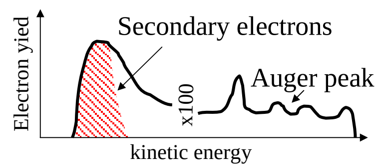
PEEM 2 at the ALS, Simone Anders
Work function 4 eV, sample voltage 30 kV, X-rays

Energy Filter

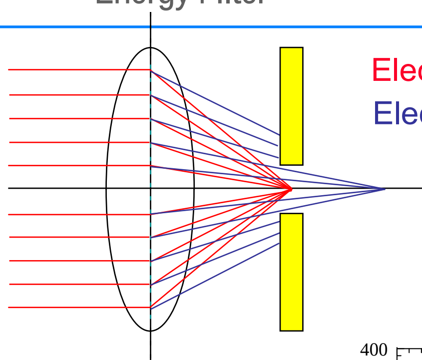


Electron energy E

Electron energy E+ΔE



Energy Filter

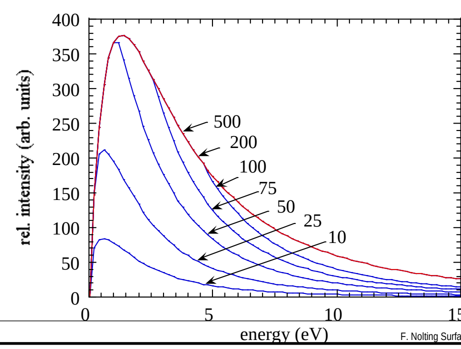


Electron energy E

Electron energy E+ΔE

Aperture cuts off transmission of electrons with higher energy

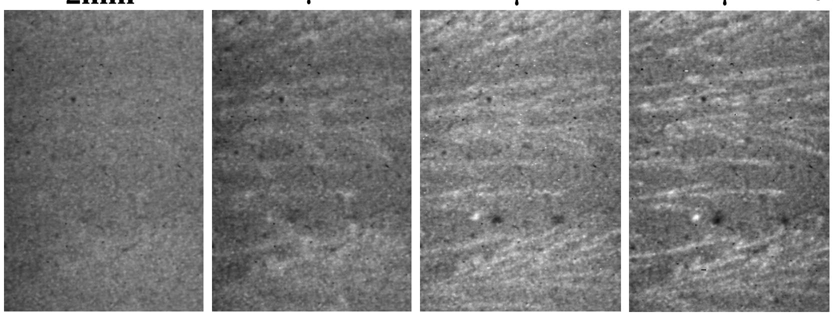
Energy distribution is narrowed but transmission is reduced



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 μm aperture for PEEM2, ALS

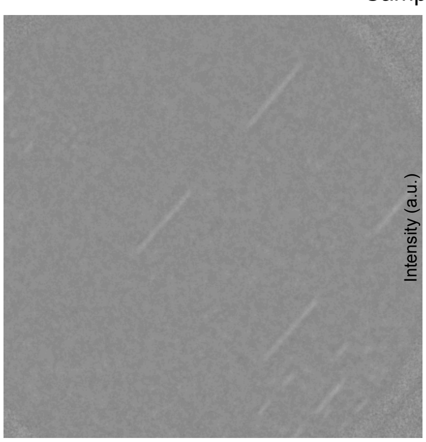
2mm	50 μm	20 μm	12 μm	Aperture diameter

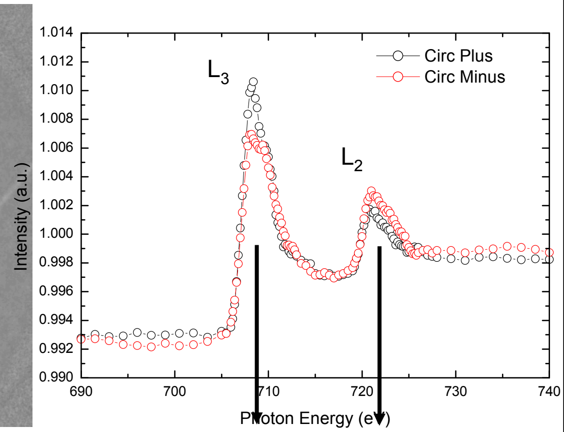


0.4 s	1s	4.2 s	10 s	Exposure time
100 %	39 %	9 %	4 %	Transmission

Spectra of individual Fe nanoparticles

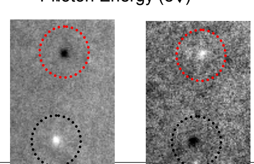
Sample: Fe nanoparticles with diameter = 9 nm





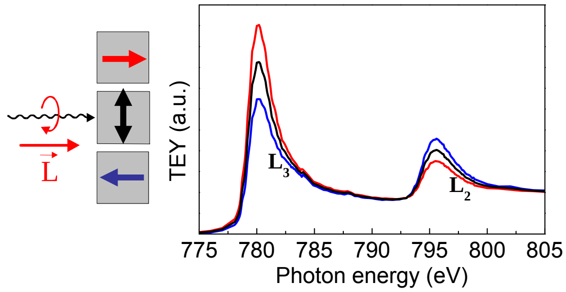
Images with increasing Photon Energy

XMCD image

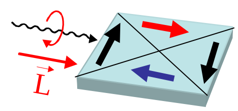


A. Fraile Rodríguez et al. PRL 104, 127201 (2010)

X-ray Magnetic Circular Dichroism (XMCD)

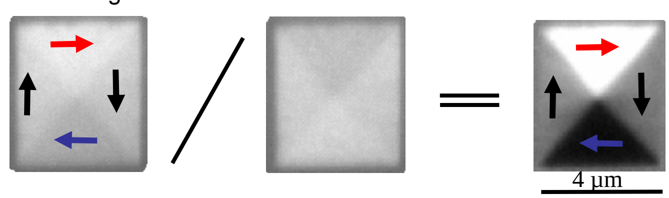


XMCD ~ <**M**> cos(**M**,**L**)



A/B ≈ (A-B)/(A+B)
magnetization direction
Domain shape

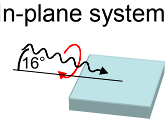
Circular right   Circular left



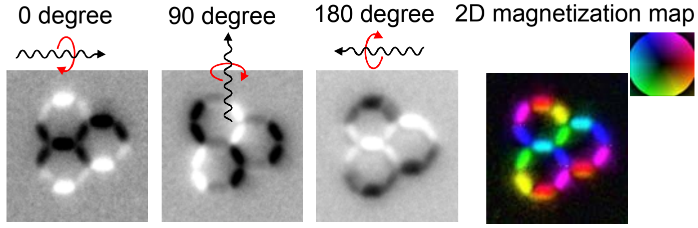
e.g. J. Stohr et al Surface Rev. Lett. **5**, 1297 (1998).

X-ray Magnetic Circular Dichroism (XMCD)

In-plane system

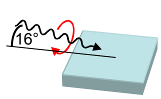


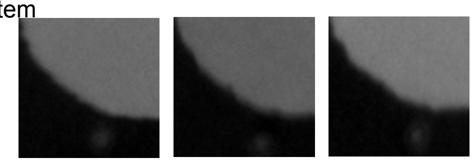
0 degree   90 degree   180 degree   2D magnetization map



Co structures (600 nm)

Out-of-plane system

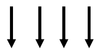




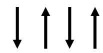
20nm GdFeCo film

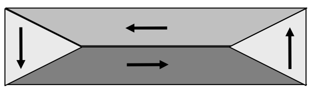
Antiferromagnet

Ferromagnet (FM)
Net magnetic moment

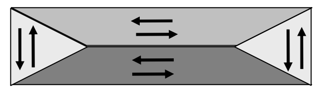


Antiferromagnet (AFM)
No net magnetic moment



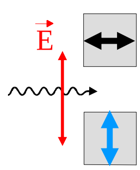


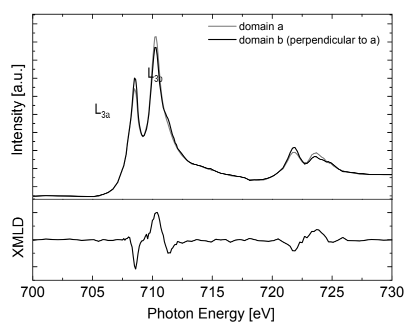
Magnetostatic energy
Exchange energy
Anisotropy energy

?  ?

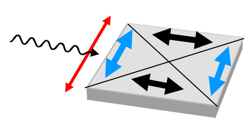
Exchange energy
Anisotropy energy
(magnetoelastic)

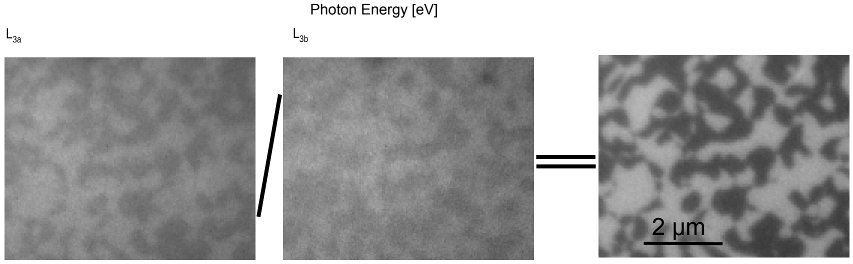
X-ray Magnetic Linear Dichroism (XMLD)





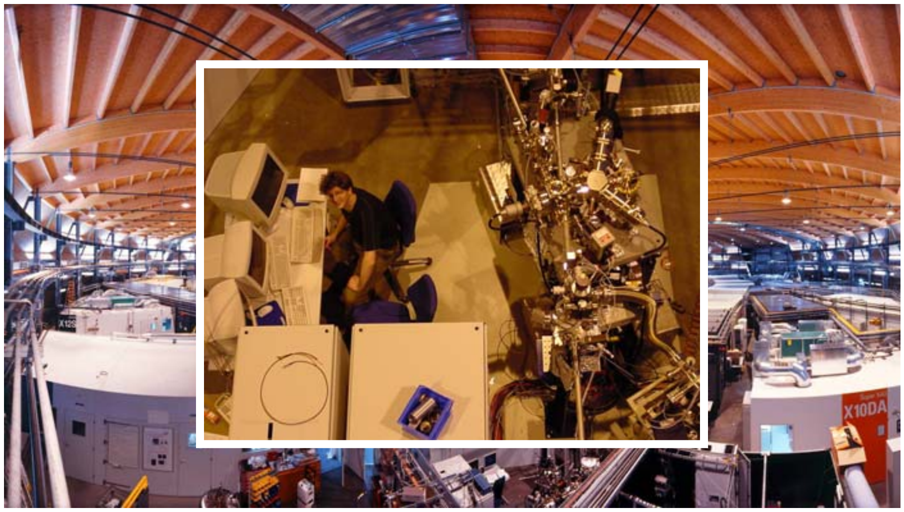
XMLD ~ <**M²**>



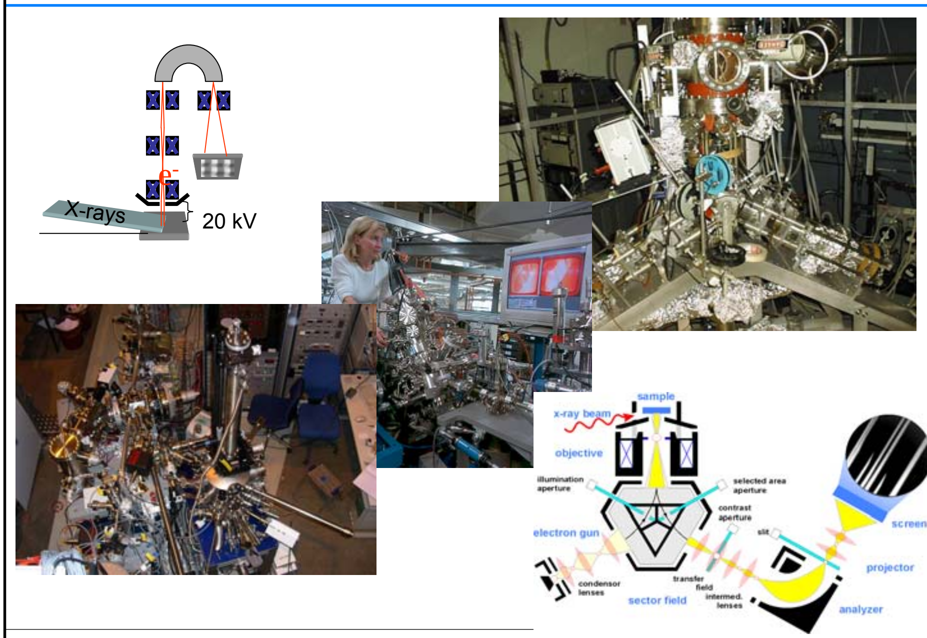


e.g. A. Scholl et al Science **287**, 1014 (2000)

Microscopy with synchrotron light



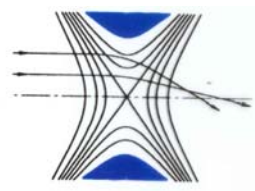
Instruments

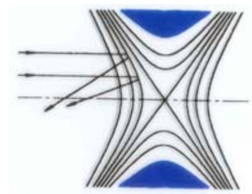


Aberrations and their correction

Spherical aberrations

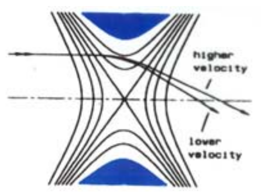
Lens

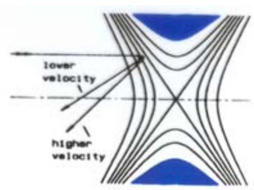




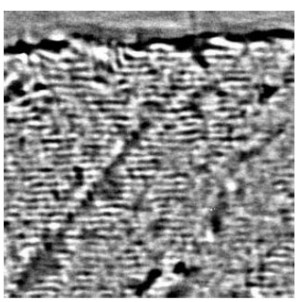
Mirror

Chromatic aberrations





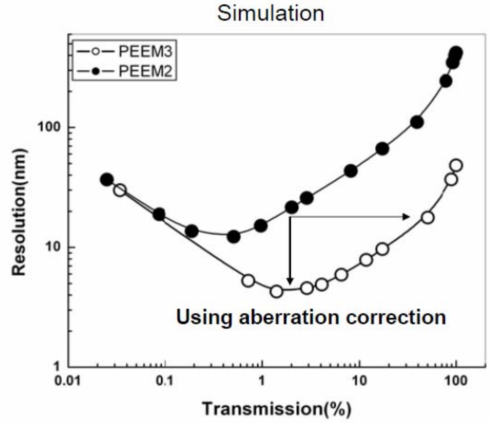
Aberration corrected PEEM



Without aberration correction

Element-resolved X-PEEM image of 25 nm thick Cr/Si layers.

Simulation



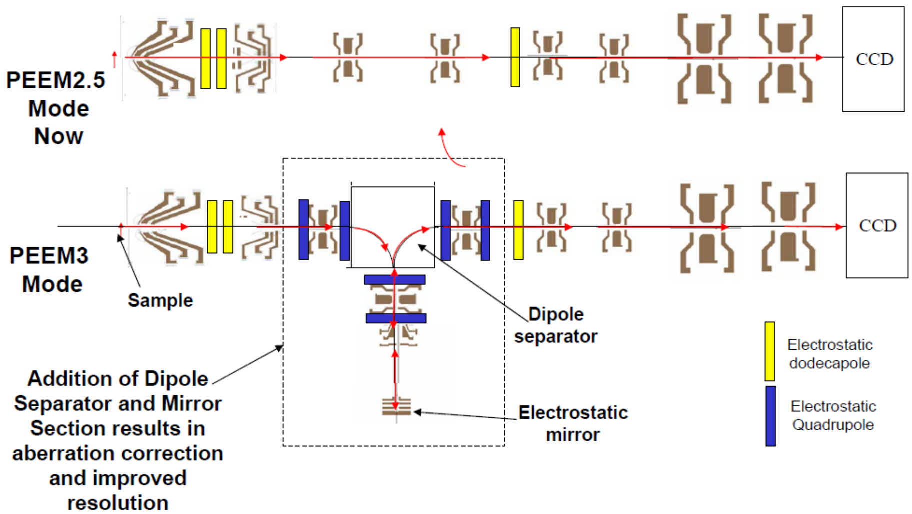
Aberration correction:

4-fold improvement of spatial resolution at a few % transmission (goal 10 nm)

10-fold improvement of transmission at several 10 nanometer spatial resolution.

Courtesy A. Scholl (ALS)

Aberration corrected PEEM



Courtesy A. Scholl (ALS)

Aberration-corrected instruments

SMART (SpectroMicroscope for All Relevant Techniques)
at BESSY II, Berlin, Germany
collaboration of seven Universities in Germany
status: LEEM 3 nm, X-PEEM about 20 nm

PEEM III
at ALS, Berkeley, USA
aberration corrected not yet build

Companies with aberration correction:
ELMITEC
SPECS

Outline

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- Röntgen
- Storage ring
- Polarized X-rays

Research example
- Nanocrystals

PEEM without 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

Wilhelm Röntgen

27. März 1845 in Lennep geboren.

1861 bis 1863 Technische Schule in Utrecht. Aus disziplinarischen Gründen, weil er irrtümlich für den Urheber einer Karikatur seines Klassenlehrers gehalten wurde, verwies man ihn ohne Abitur von der Schule.

1864 - 1868 Eidgenössischen Technischen Hochschule Zürich (ETH Zürich) Maschinenbauingenieur
1869 promovierte Röntgen an der Universität Zürich in Physik mit „Studien über Gase".

1870 begleitete er August Kundt als Assistent nach Würzburg.
1874 Habilitation Universität Straßburg die ihm die Universität Würzburg zuvor wegen seines fehlenden Abiturs verweigert hatte.

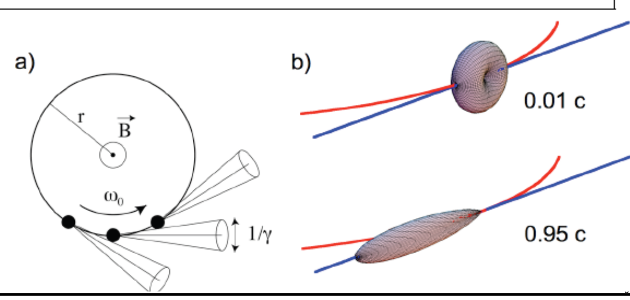
1875 außerordentlicher Professor für Physik und Mathematik an der Landwirtschaftlichen Akademie Hohenheim.
1876 eine Stelle als außerordentlicher Professor für Physik in Straßburg.
1879 ordentliche Professur in Gießen
1888 Professor der Experimentalphysik Würzburg.
1900 Professor an der Universität München
1923 verstorben

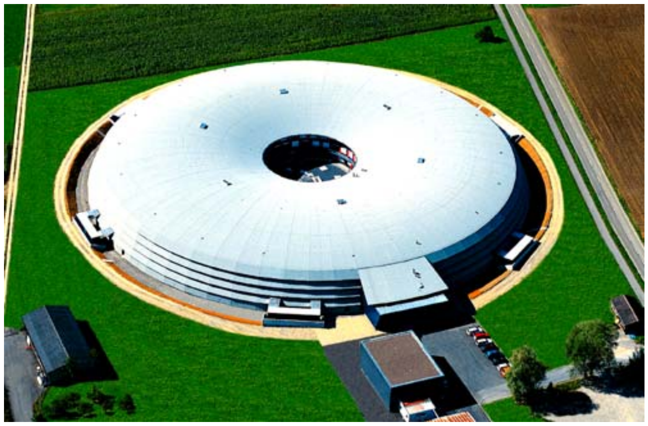
Creation of electromagnetic radiation

The Liénard–Wiechert field $\boldsymbol{E}(t)$ of a point charge q detected by an observer at a time t is determined by the distance r^*, the velocity v^*, and acceleration a^* of the charge at the emission or retarded time $t^* = t - r^*/c$. Defining $\boldsymbol{\beta}^* = \boldsymbol{v}^*/c$ we have

$$\boldsymbol{E}(t) = \frac{q}{4\pi\epsilon_0} \underbrace{\frac{1-(\beta^*)^2}{(r^*)^2\,(1-\boldsymbol{n}^*\!\cdot\boldsymbol{\beta}^*)^3}\,[\boldsymbol{n}^* - \boldsymbol{\beta}^*]}_{\text{velocity field}} + \frac{q}{4\pi\epsilon_0} \underbrace{\frac{1}{c^2\,r^*\,(1-\boldsymbol{n}^*\!\cdot\boldsymbol{\beta}^*)^3}\,\{\boldsymbol{n}^* \times ([\boldsymbol{n}^* - \boldsymbol{\beta}^*] \times \boldsymbol{a}^*)\}}_{\text{acceleration field}}. \quad (4.58)$$

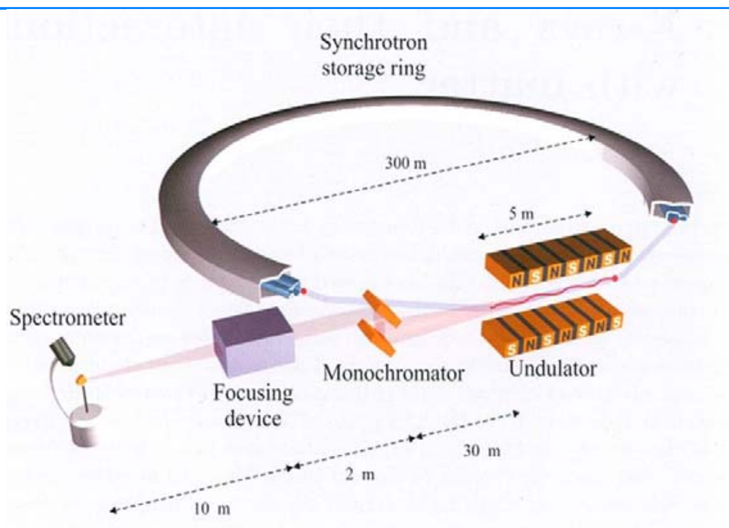
We have indicated all retarded quantities by an asterisk.



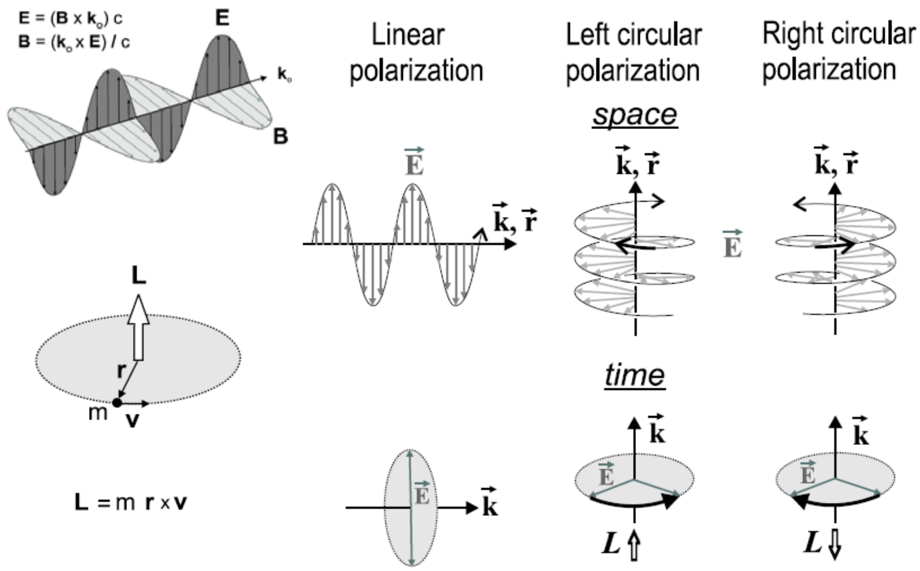


Swiss Light Source

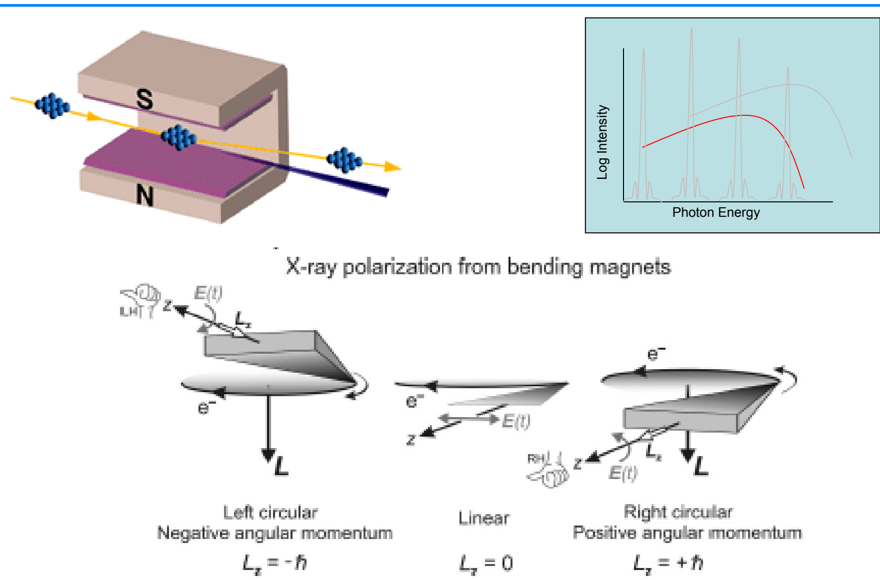
Synchrotron



Polarized Photons

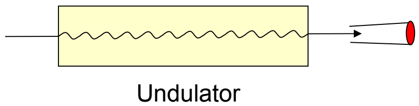


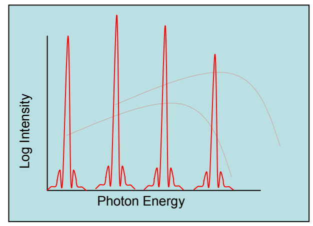
Creation of electromagnetic radiation - polarization

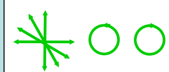


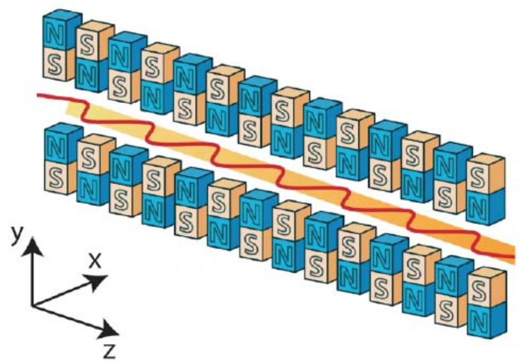
Taken from J. Stöhr

Source: undulator









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PEEM without X-rays

Spectroscopy of individual nanoclusters

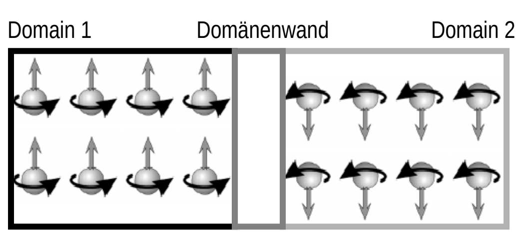
Size-dependent spin structures in iron nanoparticles

A. Fraile Rodríguez[1], A. Balan[1], A. Kleibert[1], J. Bansmann[2], A. Voitkans[2], L. J. Heyderman[1], and F. Nolting[1]

[1]Paul Scherrer Institut, Villigen PSI, CH-5232 Switzerland.
[2]Institute of Surface Chemistry and Catalysis, University of Ulm, D-89069 Ulm, Germany.
[3]Institute of Physics, University of Rostock, D-18051 Rostock, Germany.

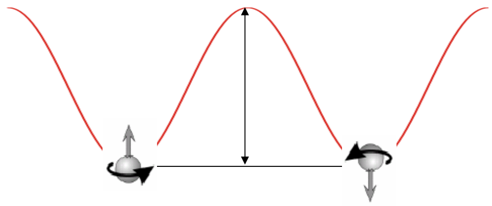
Domainwall

A domainwall needs space



How stable?

finite temperature



Energy

$\approx K_{ani} \cdot V_{particle}$

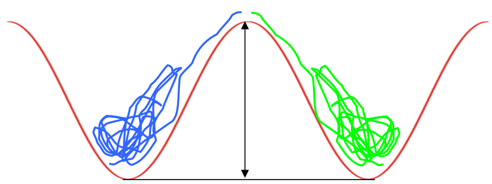
Temperature and Energy

$\overline{E_{\text{kin}}} = \frac{3}{2} k_B T$ (Ideal Gas)

Thermal energy at room temperature: 1/40 eV (0.0258472 eV)

Flipping between two states

finite temperature



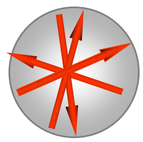
$$\frac{1}{\tau} = f \exp\left(-\frac{E_A V}{2kT}\right) \qquad f \sim 10^9 s^{-1}$$

$V^{1/3} = 14\text{ nm} \rightarrow \tau = 10^4\text{ s}$

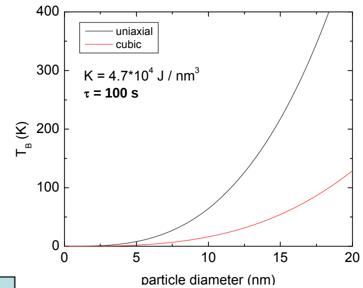
$V^{1/3} = 10\text{ nm} \rightarrow \tau = 10^{-5}\text{ s}$

How small?

Superparamagnetism



$K_{ani} \cdot V_{particle} \cong k_B \cdot T$



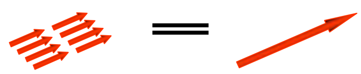
$$\frac{1}{\tau} = f \exp\left(-\frac{E_A V}{2kT}\right) \qquad f \sim 10^9 s^{-1}$$

$V^{1/3} = 14\ \mathrm{nm} \rightarrow \tau = 10^4\ \mathrm{s}$

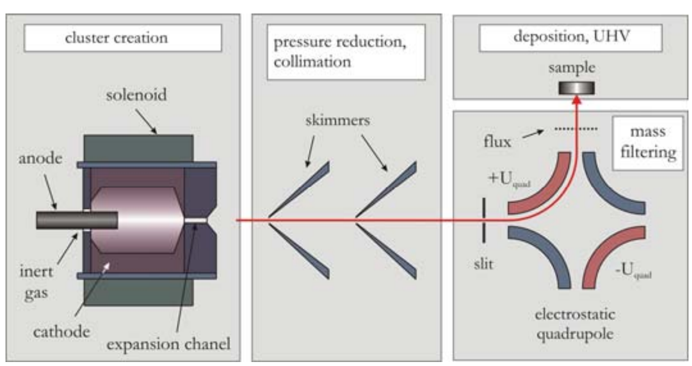
$V^{1/3} = 10\ \mathrm{nm} \rightarrow \tau = 10^{-5}\ \mathrm{s}$

Single spin model

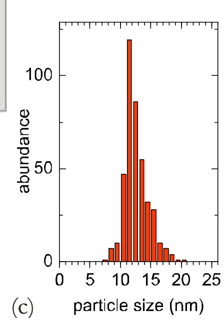
For particles smaller than 20 – 100 nm (material dependent)



Mass-filtered nanoparticles: Arc-ion source



R. P. Methling et al., EPJD **16**, 173 (2001)

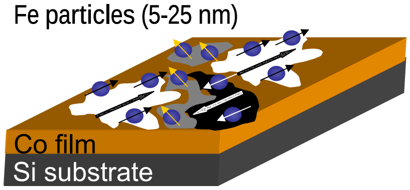


(c)

particle size tunable between 4-15nm
size distribution: ΔD/D ~10-15%
in situ deposition
transportable and UHV compatible

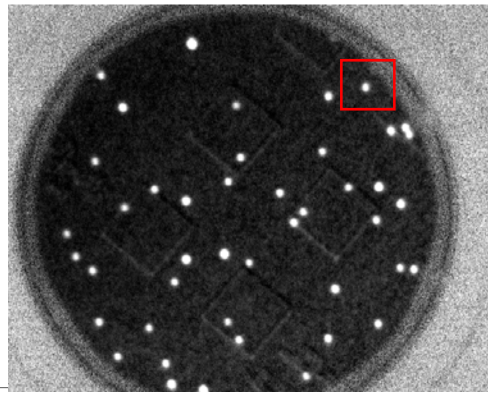
In-situ Fe particles deposited on cobalt surfaces

Fe particles (5-25 nm)

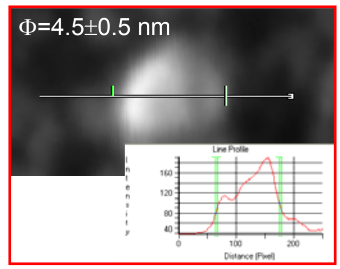


1. e-beam evaporation of cobalt thin films (2- 4 nm thickness)
2. in-situ deposition of Fe particles using arc-ion cluster source

Fe Elemental

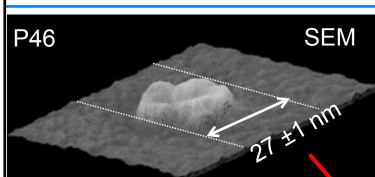


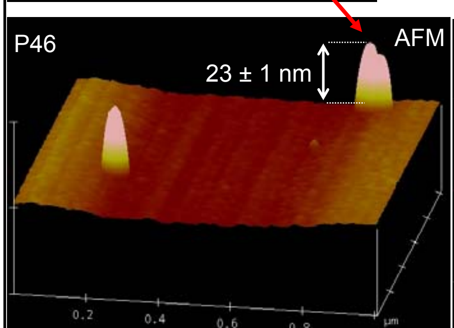
SEM

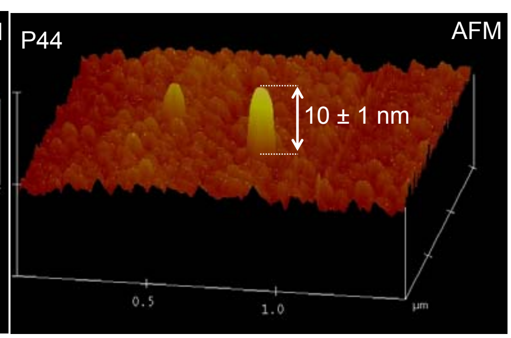


FOV = 20 micrometer

SEM, AFM







P46 (lateral size) = 27±1 nm

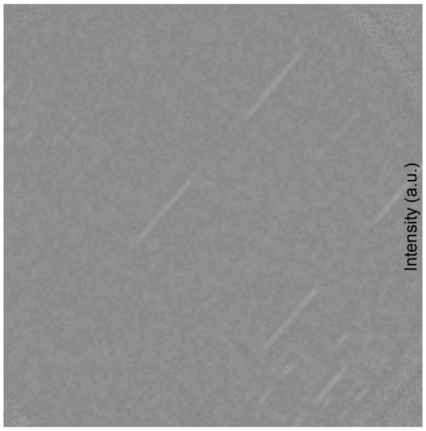
P46 (height÷width)=0.85

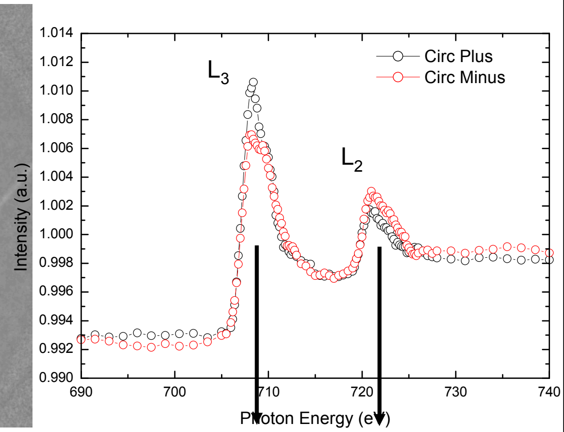
P44 (estimated lateral size) = 30±1 nm

P44 (height÷width)=0.33

Spectra of individual Fe nanoparticles

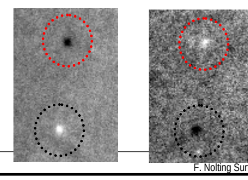
Sample: Fe nanoparticles with diameter = 9 nm





Images with increasing Photon Energy

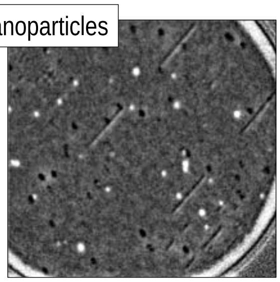
XMCD image



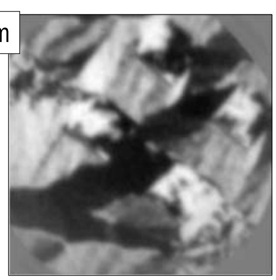
A. Fraile Rodríguez et al. PRL 104, 127201 (2010)

Coupling

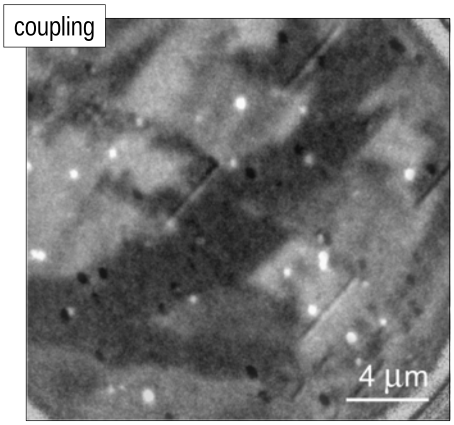
Fe Nanoparticles



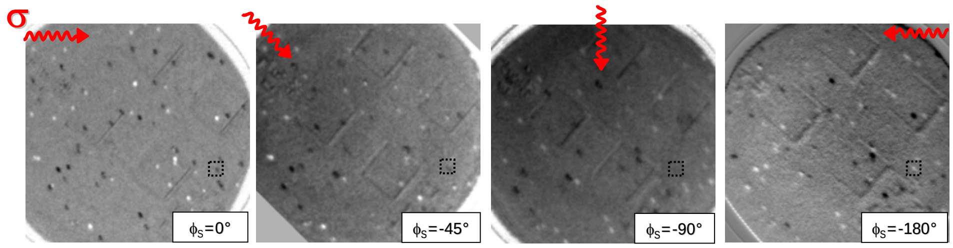
Co film

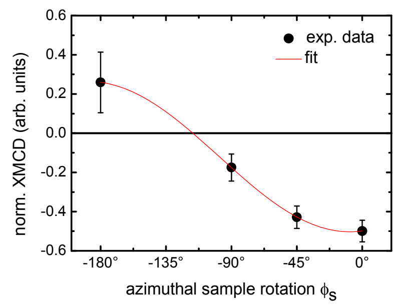


coupling

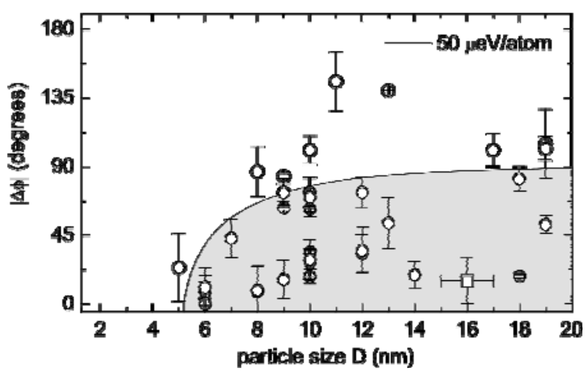


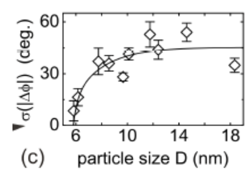
3D magnetization map

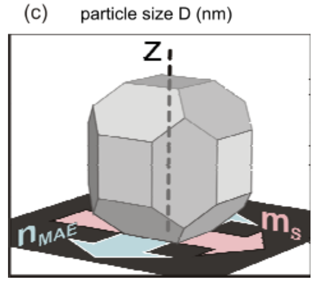




What is acting on the nanoparticle



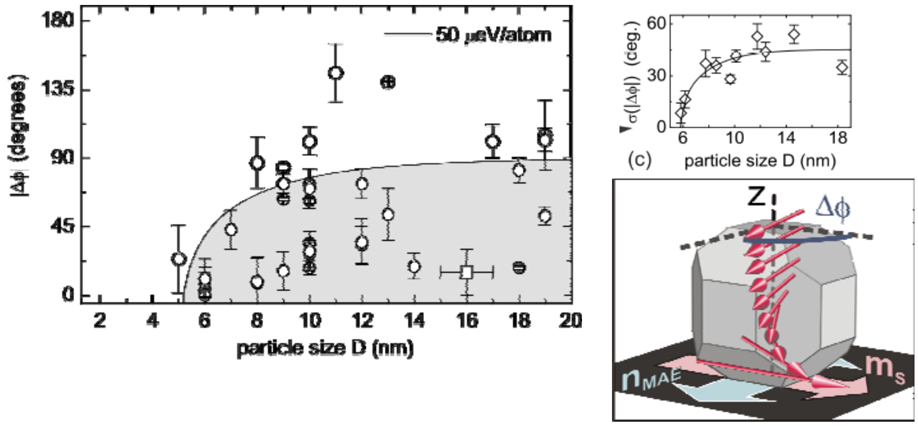




Exchange energy with ferromagnetic Co-film
Magnetocrystalline energy of the Fe-nanoparticle

A. Fraile Rodríguez, A. Kleibert, J. Bansmann, A. Voitkans, L. J. Heyderman, and F. Nolting PRL 104, 127201 (2010)

The smallest domainwall in a nanoparticle



Already for particles larger than 6 nm is the single-spin model not correct anymore

A. Fraile Rodríguez, A. Kleibert, J. Bansmann, A. Voitkans, L. J. Heyderman, and F. Nolting PRL 104, 127201 (2010)

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Research example
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PEEM without X-rays

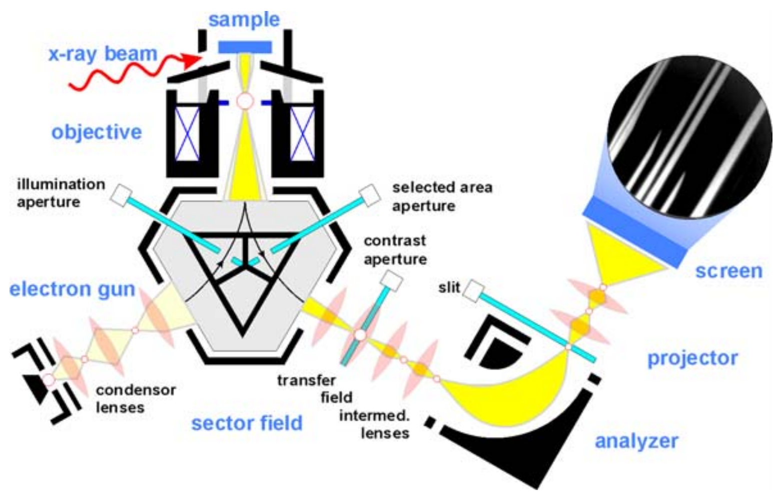
Not only with X-rays

PEEM with UV light
- 10 nm spatial resolution
- workfunction and topography contrast

PEEM with slow electrons
- 8 nm spatial resolution
- LEED, LEEM, MEM ….

PEEM with X-rays
- 50-20 nm spatial resolution
- spectromicroscopy

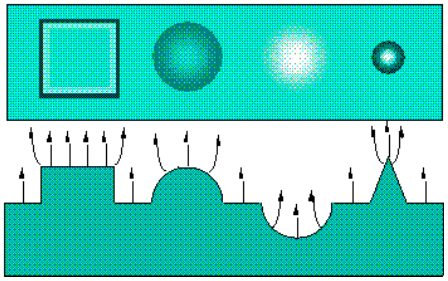
Spectromicroscope



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

Elmitec Elektronenmikroskopie GmbH
Clausthal-Zellerfeld, Germany

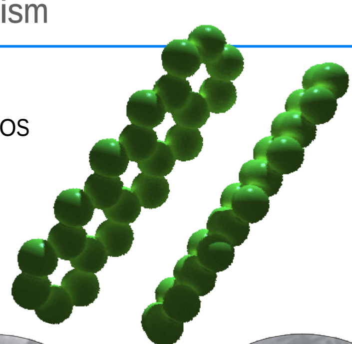
Topographical Contrast

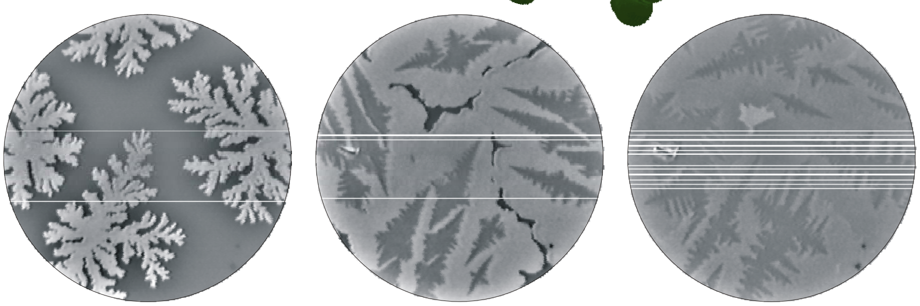


Distortion of the electric field due to topography

PEEM Contrast Mechanism

- Mercury lamp (5eV)
- Contrast either by Work Function or DOS
- Pentacene: 5 Benzene Rings
- Contrast Changes During Deposition
- FoV = 65µm



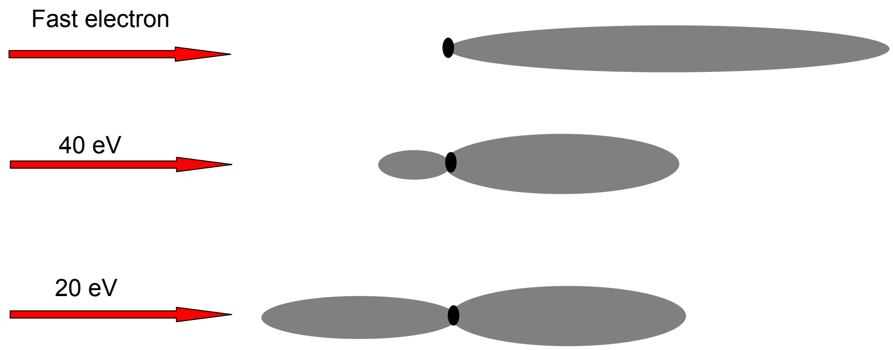


1 Layer 2 Layers 3 Layers

F.-J. Meyer zu Heringdorf et al., NATURE 412 (517), 2001

Source: slow electrons

How slow electrons interact with matter



Elastic backscattering

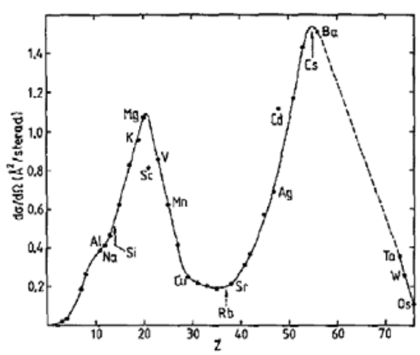


Figure 2. Elastic backscattering cross sections $d\sigma/d\Omega$ (178°) of free atoms as a fı of nuclear charge Z for 100 eV electrons. The dashed line is a simple interpolation.

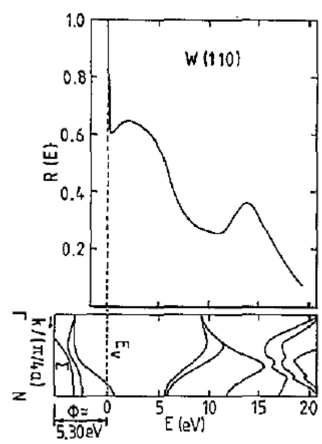
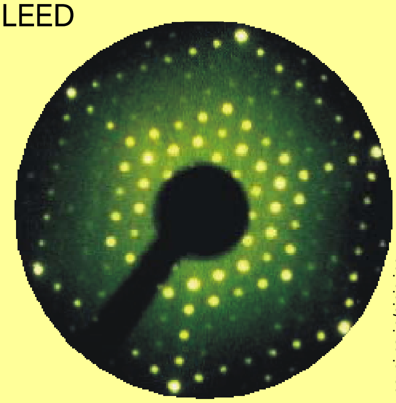


Figure 5. Reflection coefficient $R(E)$ of the W(110) surface for slow electrons (top) and bulk band structure of W along the ⟨110⟩ direction.

F. Nolting Surface Science FS 2012

Low Energy Electron Diffraction

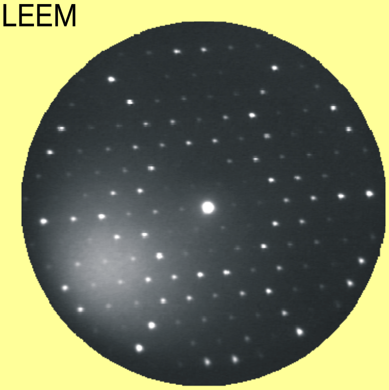
LEED



Electron Gun Visible
High Linearity
Spots move with Energy

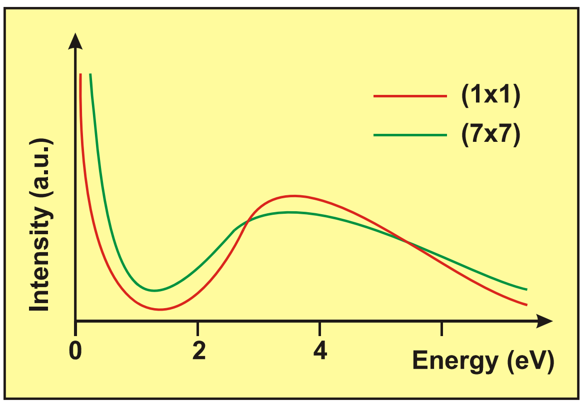
http://www.vgsysj.co.jp/sipic.jpg

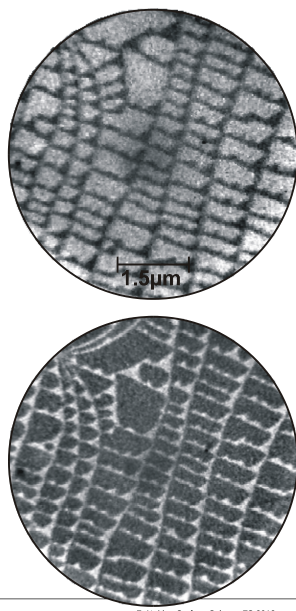
LEEM



No Apparent Electron Gun
Secondary Electrons
Spots Don't Move

Bright Field Imaging of Si (111)



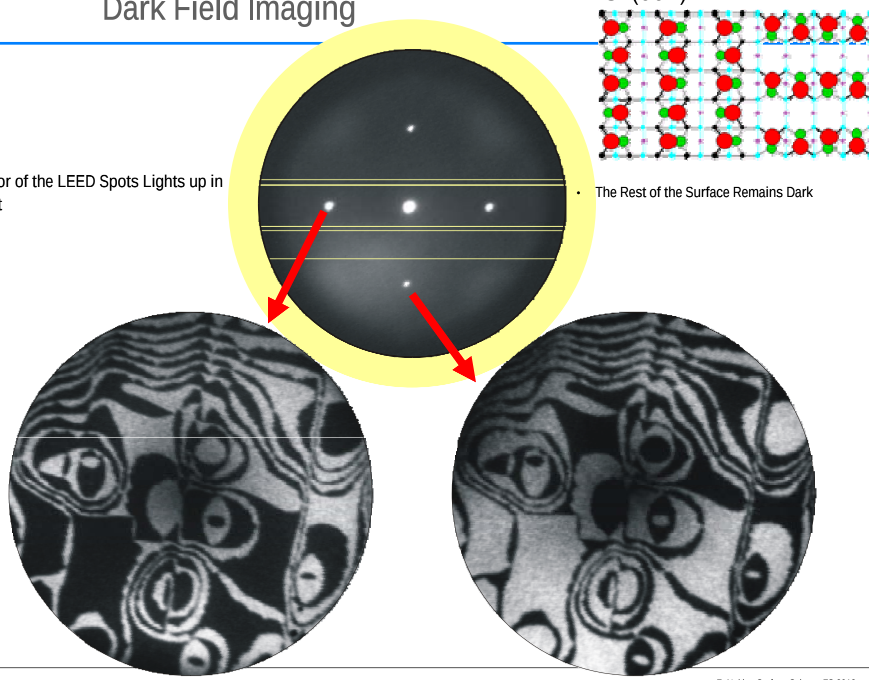


- Different Reflectivity of (1x1) and (7x7)
- Reflectivity Energy Dependent
- Contrast Reversal Dependent on Imaging Conditions

Dark Field Imaging

Si (001)

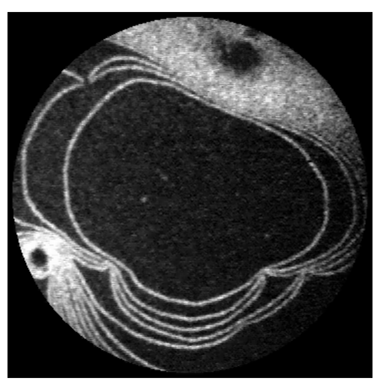
- Creator of the LEED Spots Lights up in Bright



- The Rest of the Surface Remains Dark

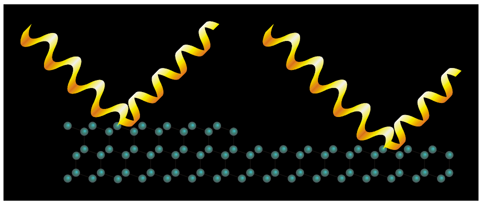
Direct Observations of Nanoscale Self-Assembly and Pattern Formation Solid Surfaces
by R. Plass, N. C. Bartelt, and G. L. Kellogg

LEEM-Movie of the growth of Pb on a Cu(111) surface.



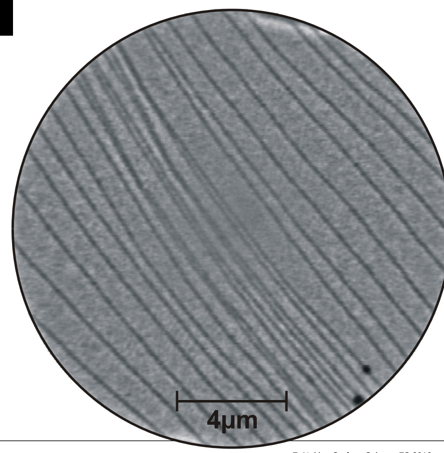
Nature, 412 (2001) 875

Phase Contrast Imaging



Analogue LEED

Electrons from Different Terraces have a Phase Shift at Out-of-Phase Conditions



Localized Phase Shift

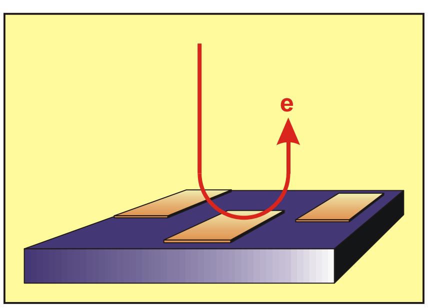
Defocusing of the Image turns Steps into Dark Lines, i.e. Centers of Destructive Interference.

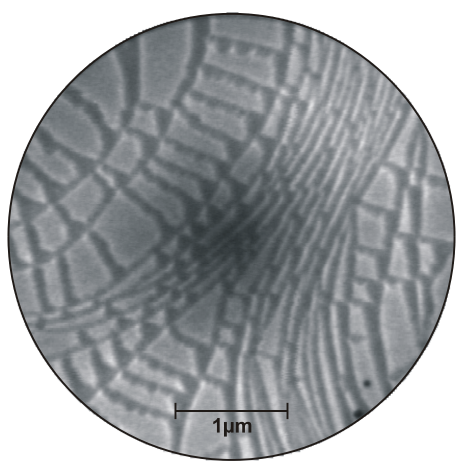
Mirror Imaging of Si(111)

Electron Energy is 0eV
- Electrons Return Before they Hit the Sample

Contrast created by outer Potential
- Workfunction

Image appears Blurred





Brief History

1930s Electron lenses/electron microscopy
Photoemission electron microscope (PEEM)
E. Brueche, Z. Phys. (1933) 448
Low Energy Electron Diffraction (LEED)
W. Ehrenberg, Philos. Mag. **18** (1934) 878

1960s improved LEED
E.J. Scheibner, L.H. Germer and C.D. Hartman,
Rev. Sci. Instrum. **31** (1960) 112
Invention of Low energy electron microscop (LEEM) by Ernst Bauer
Glass-Based Vacuum Apparatus (1962)

1985 First Operational LEEM Instrument
Telieps and Bauer, Ultramicroscopy **17** (1985) 57

1991 IBM LEEM-I
Tromp and Reuter

Brief History

Since 1990 several groups and companies:
Elmitec LEEM (Former Coworkers of E. Bauer)
Staib, Omicron
Schoenhense, Kirschner
De Stasio

Synchrotron based PEEM
pioneering G. Harp and B. Tonner, Rev. Sci. Instrum. **59** (1988) 853
Magnetism: Stöhr et al, Science **259** (1993) 658

Since 2000
world wide several beamlines for PEEM

Future
Aberration-corrected instruments (SMART / Germany, PEEMIII / USA)
spatial resolution ~ nm

Reviews (X-PEEM)
J. Stöhr et al., Surf. Rev. Lett. **6** (1998) 1297
E. Bauer, J. Phys.: Condens Matter **13** (2001) 11391
Th Schmidt et al., Surf. Rev. Lett. **9** (2002) 223

Some good books

Some good review papers

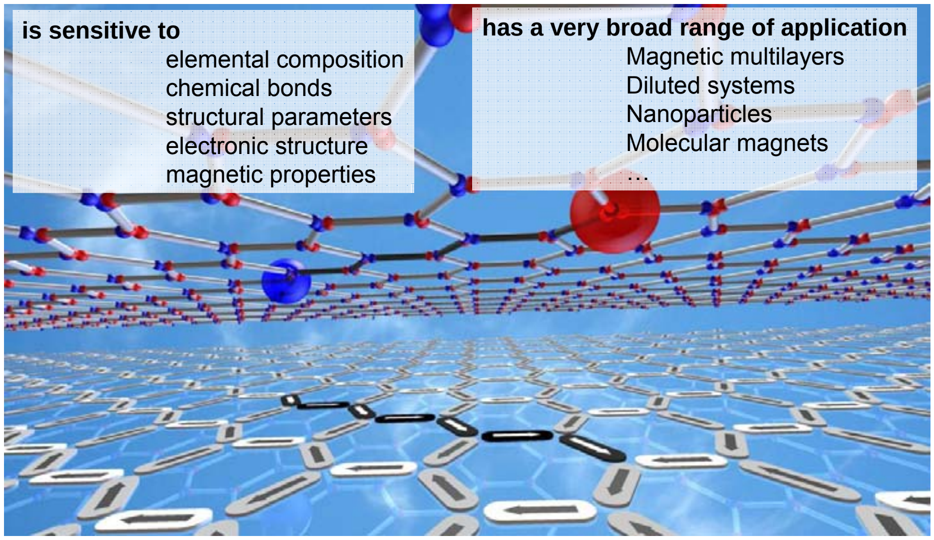
Magnetic imaging,
F U Hillebrecht, J. Phys.: Condens. Matter 13, 11163 (2001)

Photoelectron microscopy and applications in surface and materials science,
S. Günther, B. Kaulich, L. Gregoratti, M. Kiskinova, Progress in Surface Science 70, 187 (2002)

Recent advances in chemical and magnetic imaging of surfaces and interfaces by XPEEM,
A Locatelli and E Bauer J. Phys.: Condens. Matter 20, 093002 (2008)

Principles of X-ray magnetic dichroism spectromicroscopy,
J. Stöhr, S. Anders, T. Stammler, and M.R. Scheinfein, Surface Review and Letters, 5, 1297 (1998)

X-ray absorption spectroscopy (XAS)



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Repetition VIII

- Magnetic domains
- Slow electrons (surface/interface sensitivity)
- PEEM with soft X-rays as source (XMCD image)
- Polarized X-ray source
- PEEM with slow electrons as source
- Research example nanocrystals