

Chemical and magnetic imaging with x-ray photoemission electron microscopy (XPEEM)

Andrea Locatelli

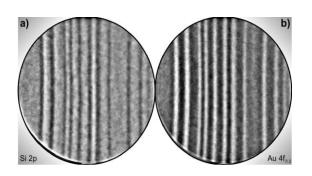
Andrea.locatelli@elettra.eu

Why do we need photoelectron microscopy?

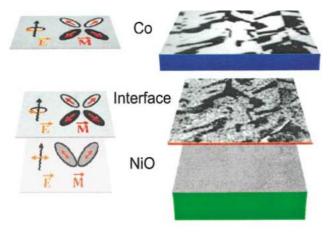


- To combine SPECTROSCOPY and MICROSCOPY to characterise the structural, chemical and magnetic properties of surfaces, interfaces and thin films
- Applications in diverse fields such as surface science, catalysis, material science, magnetism but also geology, soil sciences, biology and medicine.

Surface Science

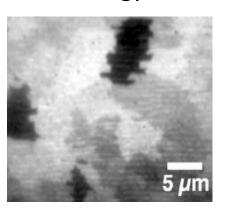


Magnetism



DOI: 10.1103/PhysRevLett.87.247201

Biology



PRL 98, 268102 (2007)

DOI: 10.1103/PhysRevLett.86.5088

Outline

- Synchrotron radiation and x-ray spectro-microscopy: basics
- Cathode lens microscopy: methods
- Applications
 - Chemical imaging of micro- structured materials
 - Graphene research.
 - Biology
 - Magnetism
 - Time-resolved XPEEM

Why does PEEM need synchrotron radiation?



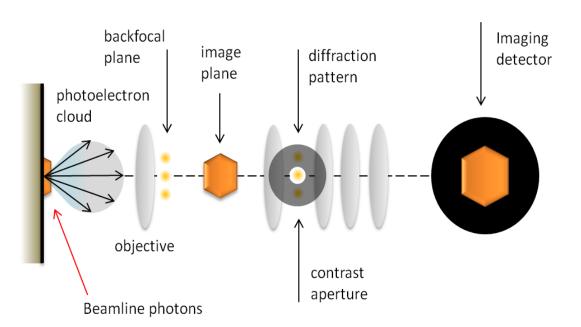
- High intensity of SR makes measurements faster
- Tuneability very broad and continuous spectral range from IR to hard X-Rays
- Narrow angular collimation
- Coherence!
- High degree of polarization
- Pulsed time structure of SR This adds time resolution to photoelectron spectroscopy!
- Quantitative control on SR parameters allows spectroscopy:
 - Absorption Spectroscopy (XAS and variants)
 - Photoemission Spectroscopies (XPS, UPS, ARPES, ARUPS)

$$J = f(h \nu, \varepsilon, \Theta, \Phi; E_{kin}^e, \sigma, \theta_e, \varphi_e)$$



PEEM basics





- Direct imaging, parallel detection
- Lateral resolution determined by electron optics: with AC, few nm possible
- Elemental sensitivity (XAS)
- Spectroscopic ability (energy filter)
- $P_{\text{max}} < 5.10^{-5} \text{ mbar}$

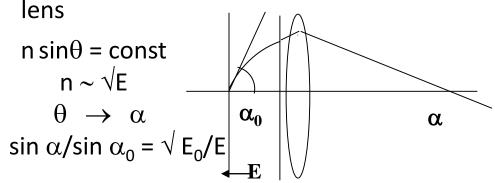
PEEM is a full-field technique. The microscope images a restricted portion of the specimen area illuminated by x-ray beam. Photoemitted electrons are collected at the same time by the optics setup, which produces a magnified image of the surface. The key element of the microscope is the objective lens, also known as cathode or immersion lens, of which the sample is part

Cathode lens operation principle



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- 1. In emission microscopy θ (emission angle) is large. Electron lenses can accept only small θ because of large chromatic and spherical aberrations
- Solution of problem: accelerate electrons to high energy before lens → Immersion objective lens or cathode



Example for E = 20000 eV:

$$E_0$$
 α for $\alpha_0 = 45^\circ$

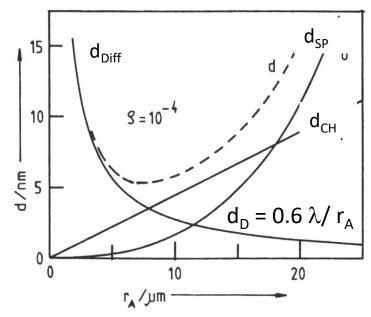
2 eV

200 eV

4.5°

3. The aberrations of the objective lens and the contrast aperture size determine the lateral resolution

$$d = \sqrt{d_{SP}^2 + d_{CH}^2 + d_D^2}$$



The different types of PEEM measurements



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PEEM

- threshold microscopy
- Laterally resolved XPS, micro-spectroscopy
- Laterally resolved UPS, microprobe ARUPS /ARPES X-rays, He lamp
- **Auger Spectroscopy**
- XAS-PEEM (XMC/LD-PEEM)

Probe

Hg lamp

X-ray

X-ray, or electrons

X rays

Measurement

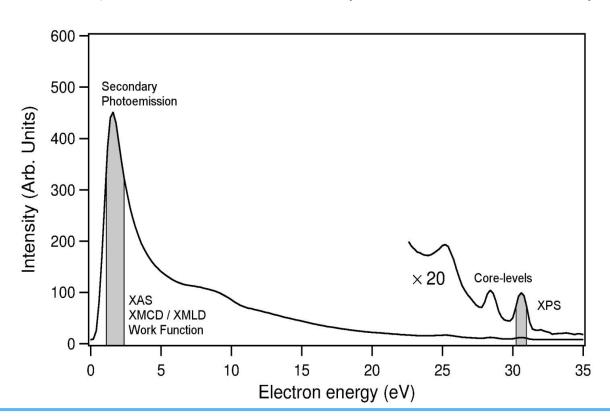
photoelectrons

core levels or VB ph.el.

VB photoelectrons

secondary electrons

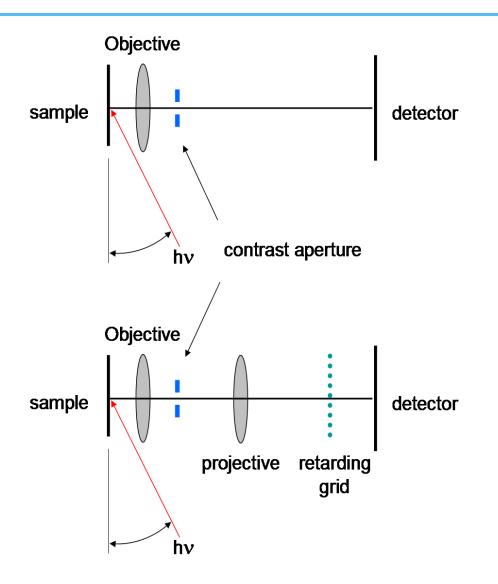
secondary electrons

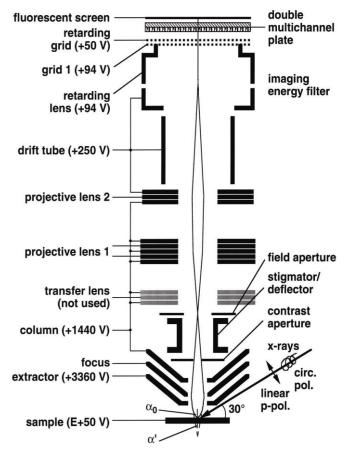


Require energy filter

Simple PEEM instruments

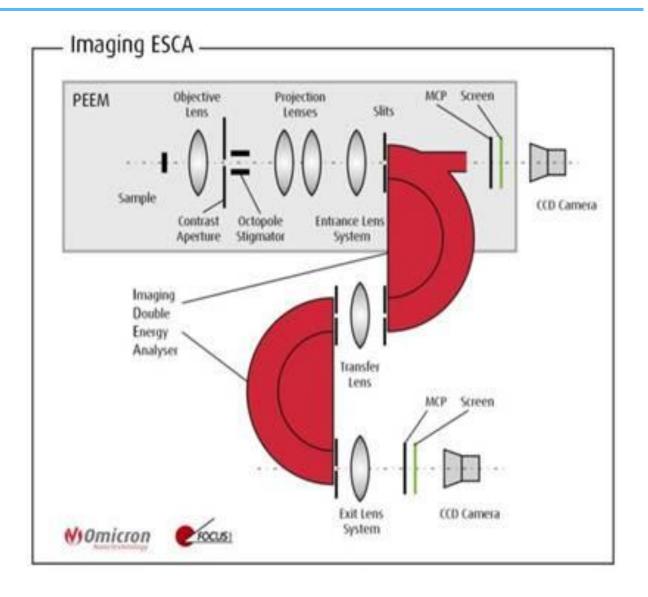






PEEM instrments with energy filter: NanoESCA

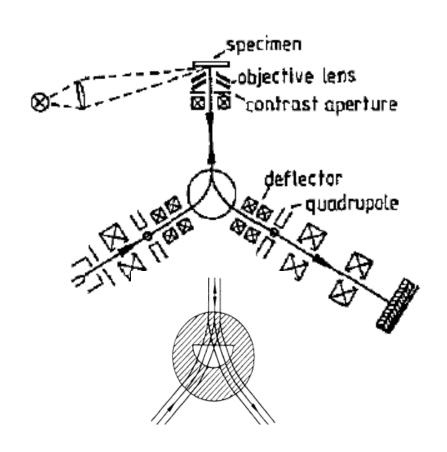




Low energy electron microscopy (LEEM)



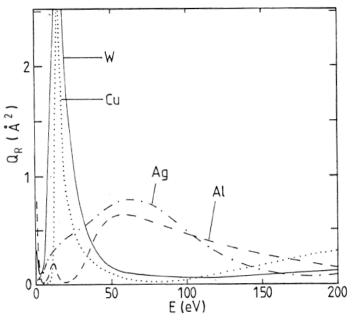
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LEEM probes surfaces with low energy electrons, using the elastically backscattered beam for imaging.

Direct imaging and diffraction imaging modes

Backscattering cross section



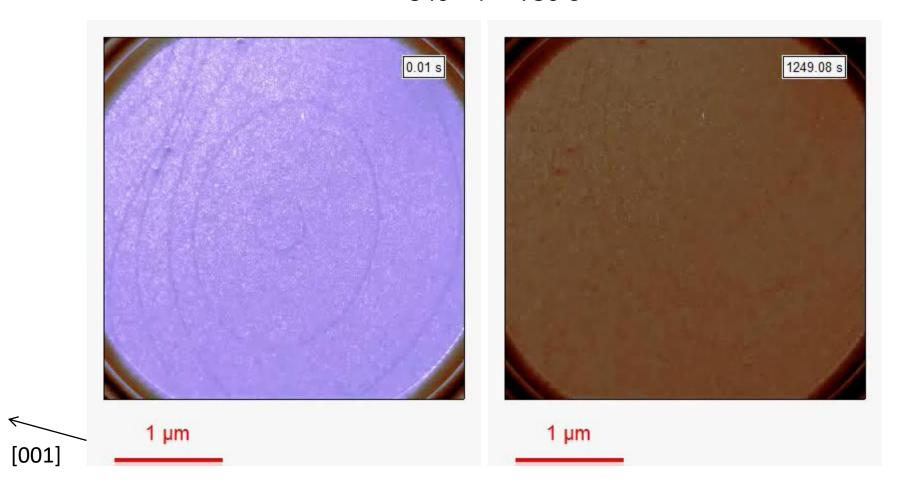
E. Bauer, Rep. Prog. Phys. 57 (1994) 895-938.

- High structure sensitivity
- High surface sensitivity
- Video rate: reconstructions, growth, step dynamics, self-organization

Imaging dynamic processes in LEEM



540 < T < 750 C



Ni growth on W(110): step flow and completion of ps ML

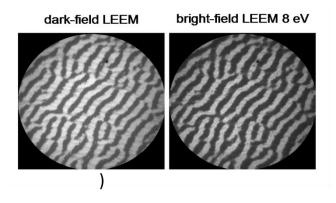
Ni growth on W(110): formation of a striped phase above 1 ps ML Ni

Image contrast in LEEM



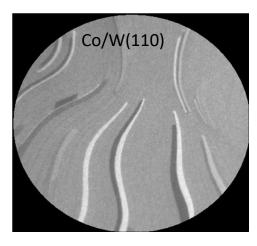
Different contrast mechanisms are available for strucutre characterization

SURFACE STRUCTURE

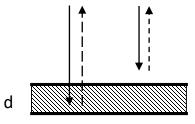


contrast
sample
objective
[h,j]
contrast
aperture

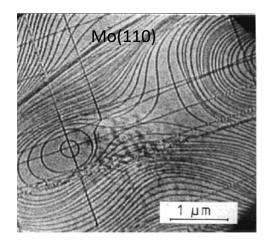
FILM THICKNESS



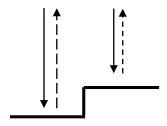
quantum size contrast



STEP MORPHOLOGY



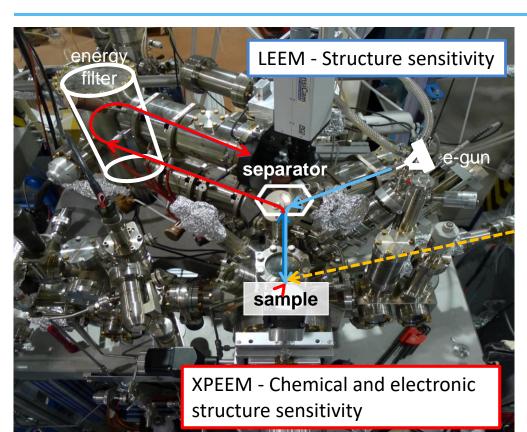
geometric phase contrast



13

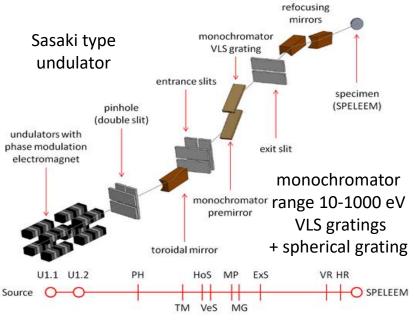
SPELEEM = LEEM + PEEM





The Nanospectroscopy beamline@Elettra

Flux on the sample: 10¹³ph/sec (microspot) intermediate energy resolution.



Applications:

characterization of materials at microscopic level, magnetic imaging of micro-structures Imaging of dynamical processes

A. Locatelli, L. Aballe, T.O. Menteş, M. Kiskinova, E. Bauer, Surf. Interface Anal. 38, 1554-1557 (2006)

T. O. Menteş, G. Zamborlini, A. Sala, A. Locatelli; Beilstein J. Nanotechnol. 5, 1873–1886 (2014)

29/09/2017

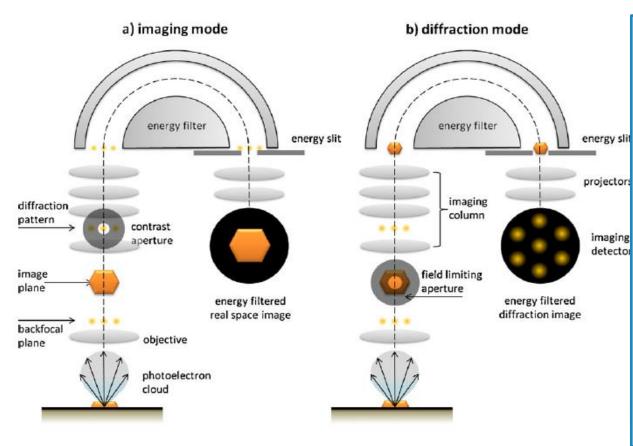
SPELEEM many methods analysis



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Spectroscopic imaging XAS-PEEM / XPEEM / LEEM microprobe-diffraction ARPES / LEED

microprobe-spectroscopy **XPS**



hv = 90 eV W 4f_{7/2} C.Ap. 20 um 100 80 60 40 20 -34 -30 Binding energy (eV) T. O. Mentes et al. Beilstein J. Nanotechnol. 5, 1873-1886 (2014). energy resolution

uXPS: 0.11 eV

spatial resolution

LEEM: 10 nm

XPEEM: 25 nm

energy resolution **XPEEM**: 0.3 eV

Limited: to 2 microns in dia.

angular resolution

transfer width: 0.01 Å-1

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SPELEEM summary



Performance: <u>lateral resolution</u> in imaging: <u>10nm</u> (LEEM)

30 nm (XPEEM)

energy resolution: 0.3 eV (0.1 eV muXPS)

Key feature: multi-method instrument to the study of surfaces and

interfaces offering imaging and diffraction techniques.

Probe: low energy e- (0-500 eV) \iff structure sensitivity

soft X-rays (50-1000 eV) ←→ chemical state, magnetic

state, electronic struct.

Applications: characterization of materials at microscopic level

magnetic imaging of microstrucutres

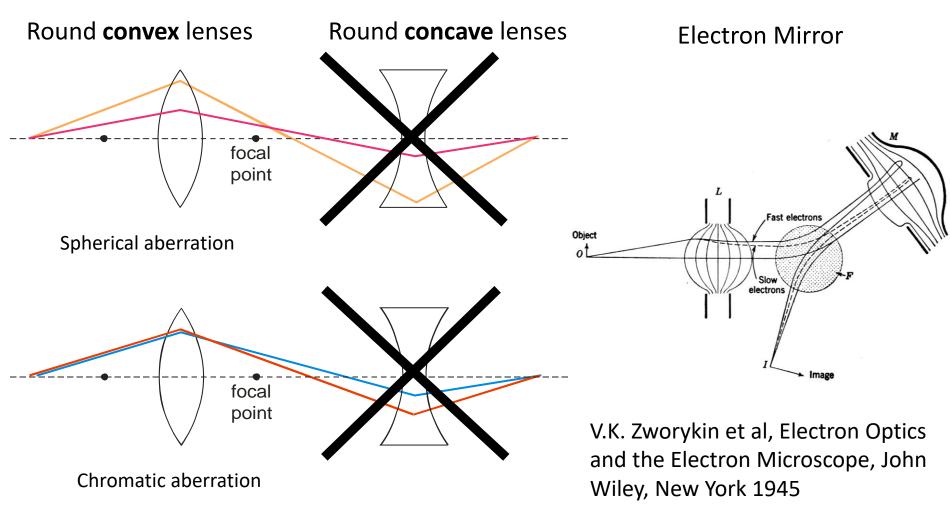
dynamical processes

Correction of spherical and chromatic aberrations



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Electron optics

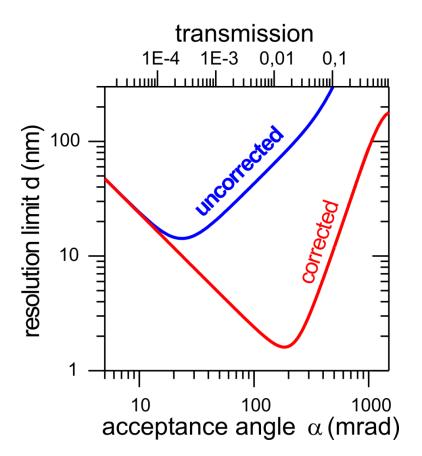


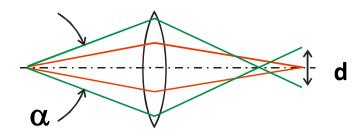
The SMART AC microscope: calculation



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Simultaneous improvement in Transmission and Resolution!!!





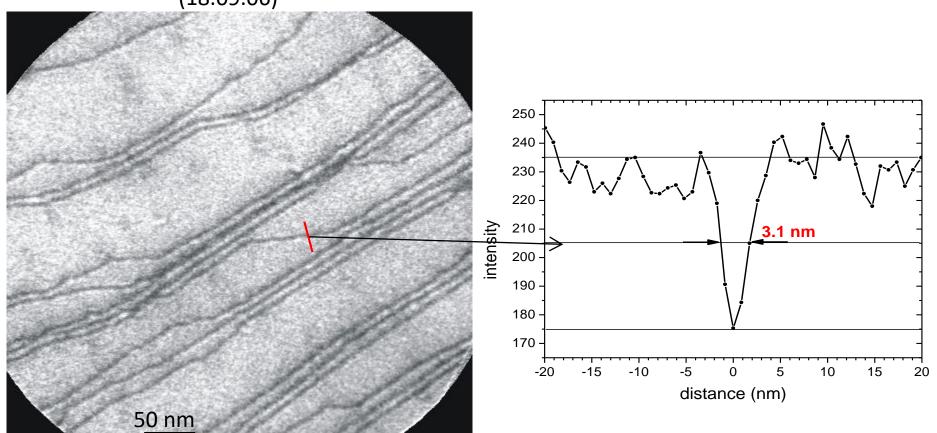
Resolution limit	without correction	with correction
Spherical	$\alpha^3 + \dots$	α^5
Chromatic	$\Delta E \alpha +$	$\Delta E \alpha^2$
		$+\Delta E^2 \alpha$
Diffraction	1/α	$1/\alpha$

D. Preikszas, H. Rose, J. Electr. Micr. 1 (1997) 1 Th. Schmidt, D. Preikszas, H. Rose et al., Surf.Rev.Lett 9 (2002) 223

First results of the SMART microscope @BESSY

Atomic steps on Au(111),

LEEM 16 eV, FoV = 444 nm x 444 nm (18.09.06)

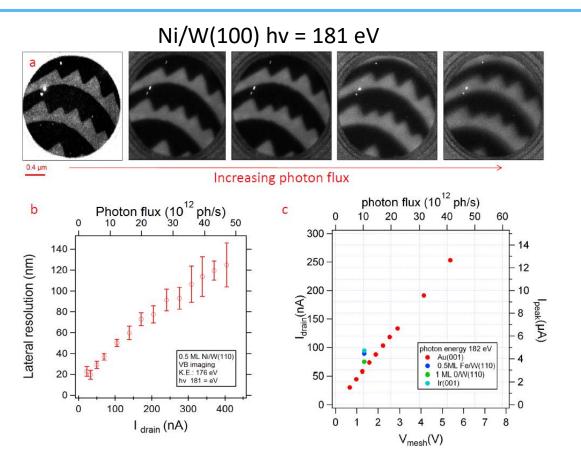


Courtesy of Th. Schmidt et al.; 5th Int. Conf. LEEM/PEEM, Himeji, 15.-19. Oct. 2006

Lateral resolution limitations: space charge



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photocurrent estimate for SPELEEM@Elettra; Au/W(110)

- 440 bunches rev. frequency: 1.157 MHz bunch length: 42 ps (2GeV)
- 1 10¹³ ph./s on sample =
 = 20000 ph./bunch
- Total photoionization yield: about 2% photons result in a photoemission event
- I peak $\approx 400 \text{ e}^{-}/42 \text{ ps}$ $\approx 1.5 \mu\text{A vs } 20 \text{ nA (LEEM)}$ 13 pA/ μ m² versus 20 nA/ μ m²
- Image blur can be observed with SR but only under very high photon fluxes.
 Must Keep into account in beamline design. No space charge in LEEM
- 2. Both the lateral and energy resolution are strongly degraded by Boersch and Loeffler effects occurring in the first part of optical path.

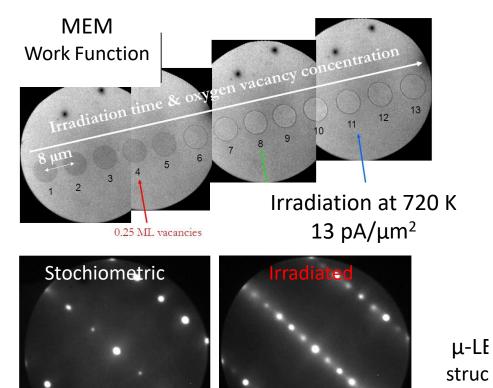


Au/TiO₂(110): controlling growth by vacancies



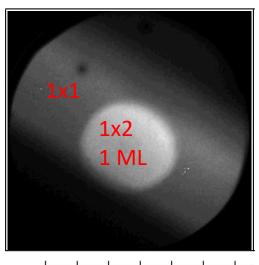
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Creation of ordered oxygen vacancies

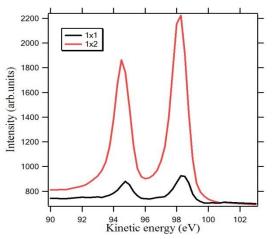


(1x1)(1x2)

Au growth on $TiO_2(110)$



XPEEM @ Au 4f



μ-XPS

Surface Oxygen on Ag: e-beam "Lithography"

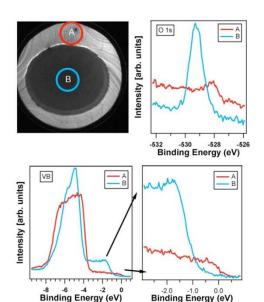


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Full oxidation of Ag using NO₂ does not occur:

$$NO_2 \rightarrow NO_{ad} + O_{ad}$$

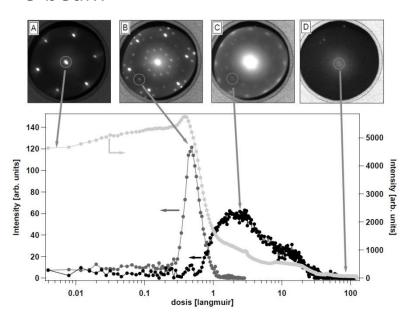
Instead: e-beam (60 eV) stimulated desorption of NO_{ad} works at RT!



A: metallic Ag B: Ag₂O Low T: NO_{ad} stays, prevents oxidation.

High T: NO_{ad} desorbs, but Ag₂O unstable.

LEED reveals path towards Ag₂O under e-beam

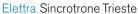


S. Günther *et al.*, *App. Phys. Lett.* 93, 233117 (2008).

S. Günther et al., Chem. Phys. Chem. 2010.

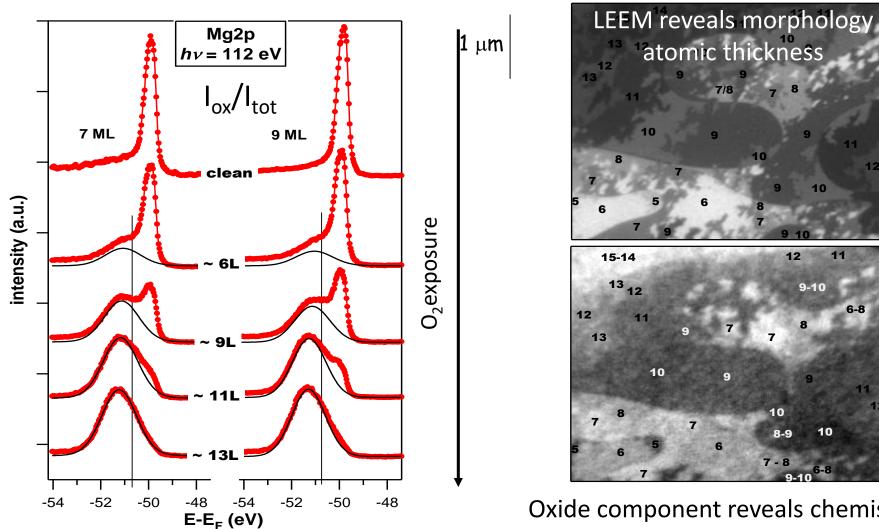
Thickness dependent reactivity in Mg





11

6-8



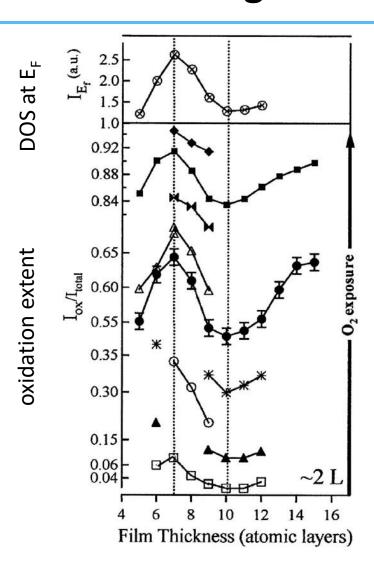
Oxide component reveals chemistry!

L. Aballe et al., Phys. Rev. Lett. 93, 196103 (2004)

Oxidation of Mg film and QWR



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FACTS

- ✓ Strong variations in the oxidation extent are correleted to thickness and to the density of states at E_F
- ✓ XPEEM is a powerful technique for correlating chemistry and electronic structure information

SIGNIFICANCE OF THE EXPERIMENTS

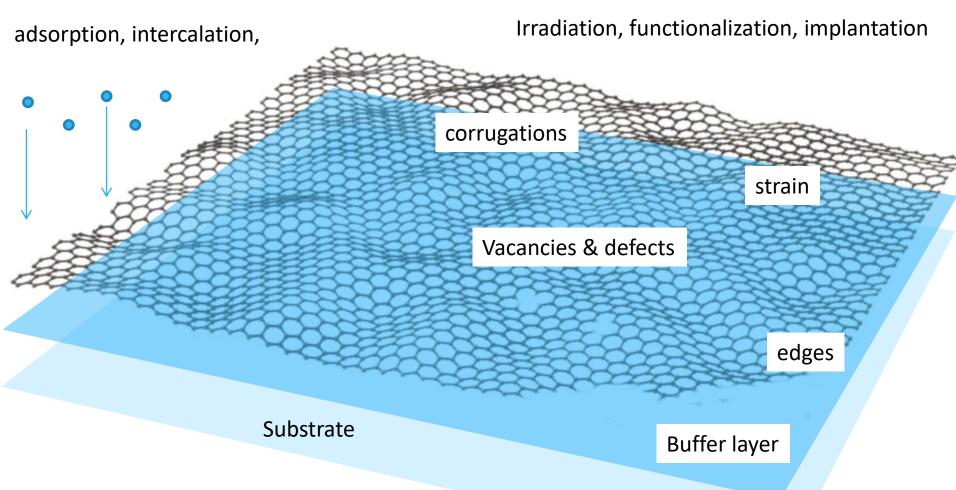
- ✓ Control on film thickness enables modifying the molecule-surface interaction
- ✓ Theoretical explanation: Decay length of QWS into vacuum is critical: it reproduces peak of reactivity in experimental data. See Binggeli and M. Altarelli, Phys.Rev.Lett. 96, 036805 (2005)

L. Aballe et al., Phys. Rev. Lett. 93, 196103 (2004)

The complexity of the metal-graphene interface



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- Understand and control the fundamental interactions occurring at the interface
- verify the properties (crystal quality, stoichiometry, electronic structure) at the mesoscale!

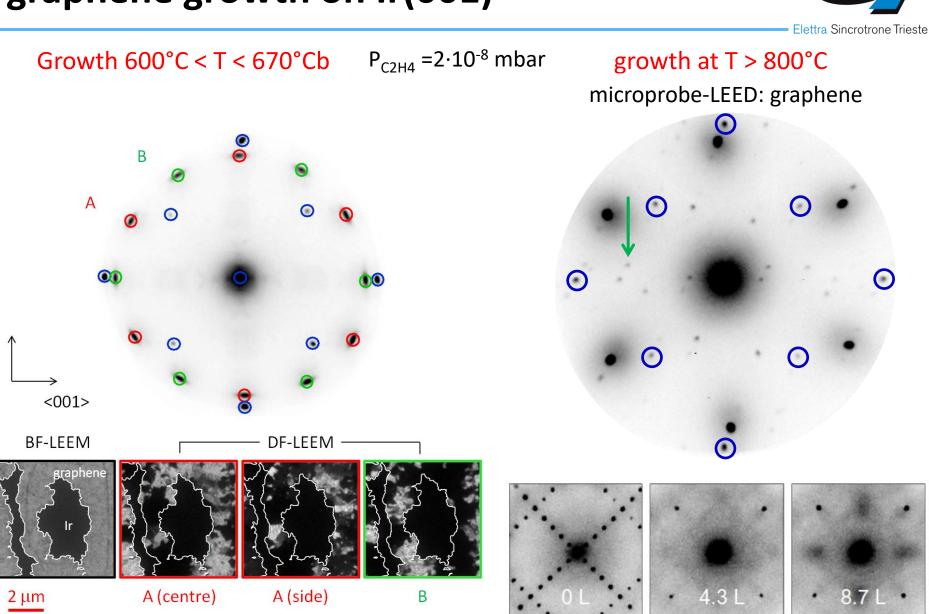
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XPEEM studies of graphene

- Effect of substrate' symmetry
 - The complex structure of g/Ir(100)
- Buffers
 - Au Intercalation
 - Carbides in graphene on Ni(111)
- Irradiation/implantation
 - [Low energy N+ ion irradiation of g/Ir(111)]
 - Irradiation with noble gases of g/Ir(100)

graphene growth on Ir(001)



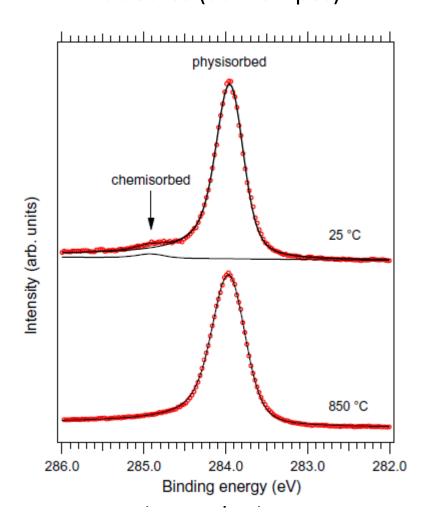


Reversible phase transformation in graphene

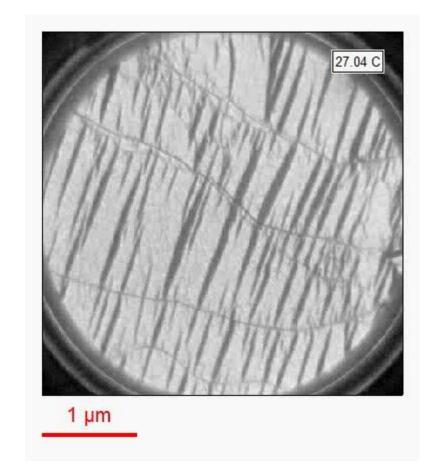


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Upon cooling a new graphene phase nucleates (dark stripes)

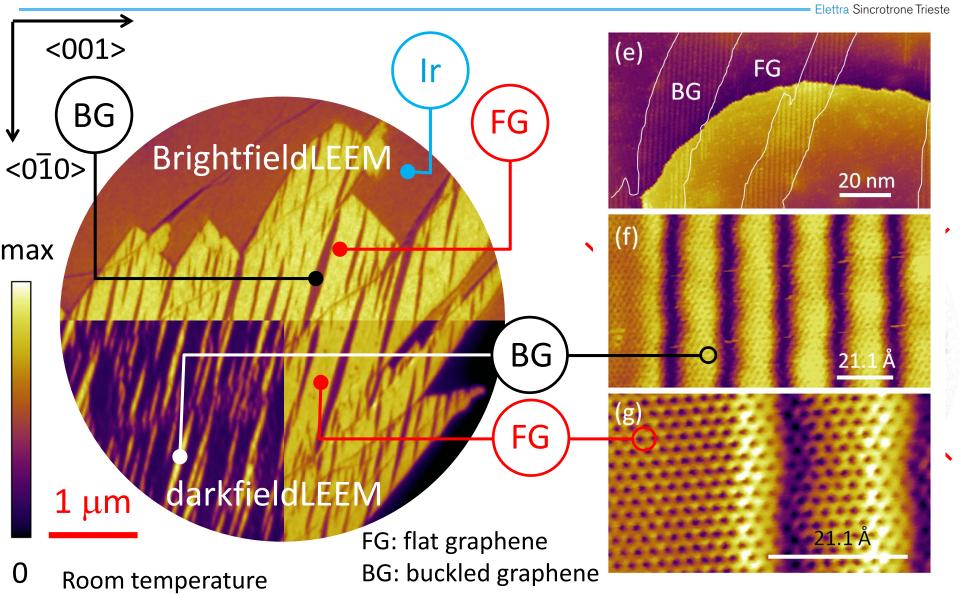


The stripes disappear upon annealing to high temperature.



Graphene/Ir(100): strucutre of FG and BG

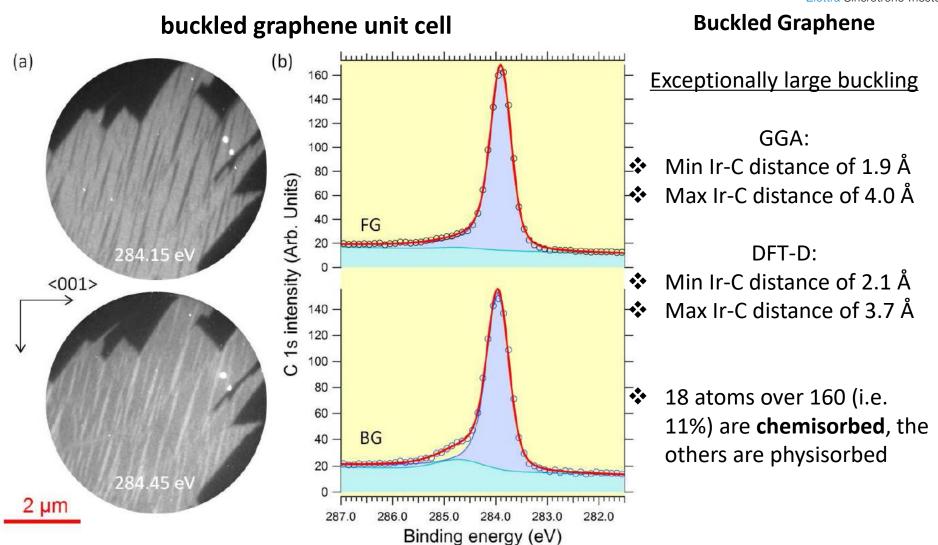




Buckled graphene unit cell by ab-initio







Buckled graphene shows regular one-dimensional ripples with periodicity of 2.1nm.

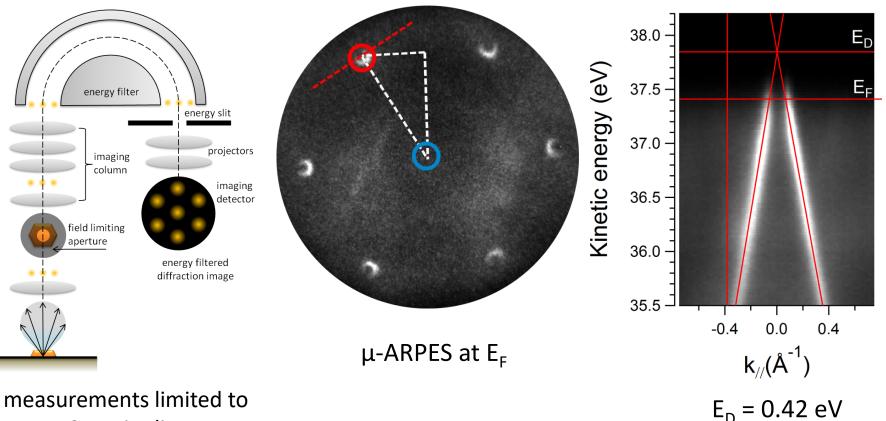
Electronic structure: graphene doping



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what is the difference in electronic structure between FG and BG? do they both show the same Dirac-like dispersion?

Diffraction Imaging

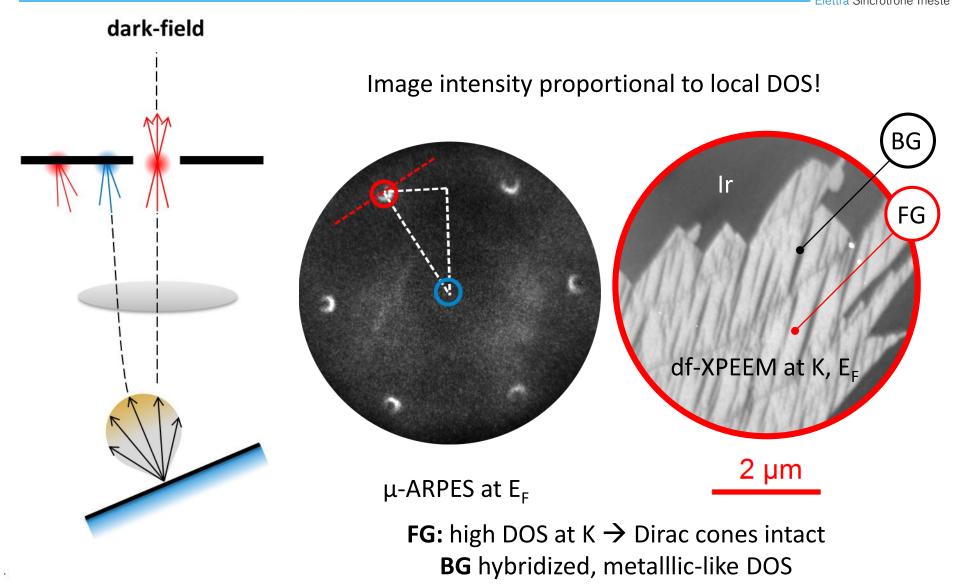


2 um in dia.

Different character of FG and BG







Decoupling graphene from substrate:

- Intercalated Au/g/Ir(100)
- Switchable formation of carbides in g/Ni(111)

Tuning the interaction by Au intercalation



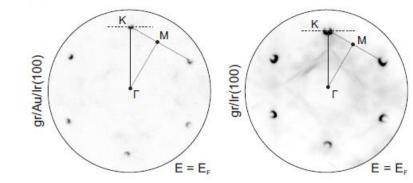
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Real time LEEM imaging during Au intercalation at 600 °C

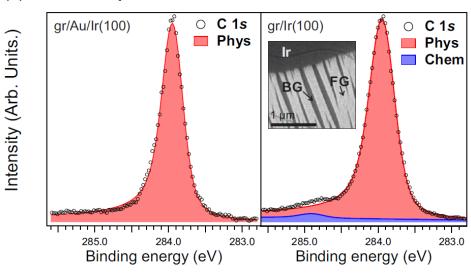
Ir Ir gr gr gr/Au gr/Au gr 1 µm

Electonic structure by microprobe ARPES



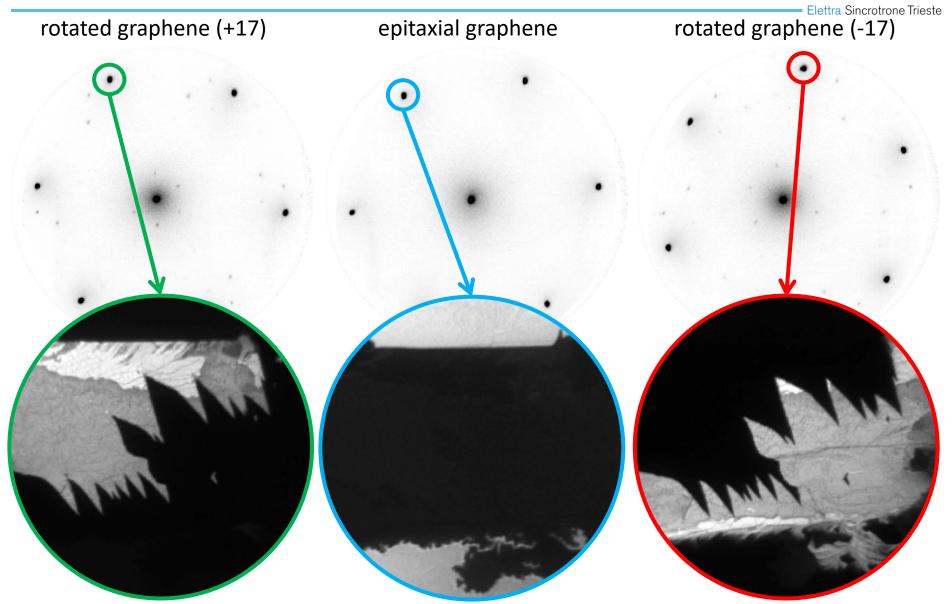


(c) Core level photoemission



Identifying crystal grains in graphene/Ni(111)



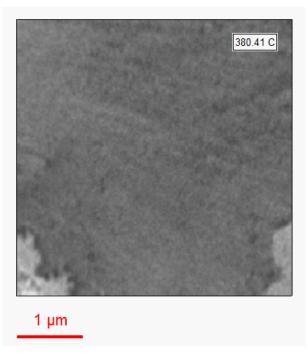


Formation/dissolution of carbides under rg/Ni(111)

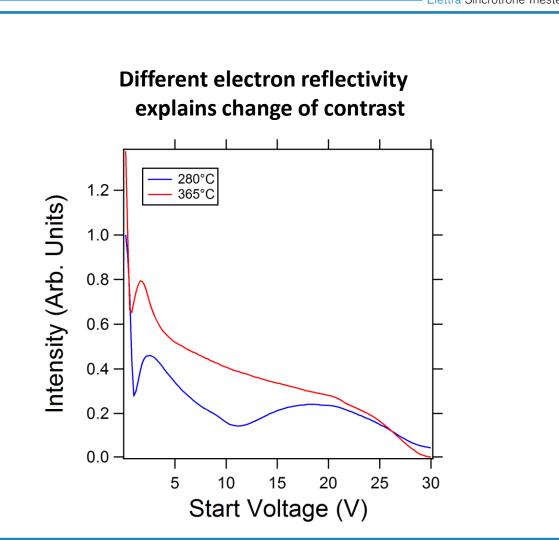




1: carbide nucleation



The Ni-carbide nucleates exclusively under rotated graphene, starting at temperatures below 340°C

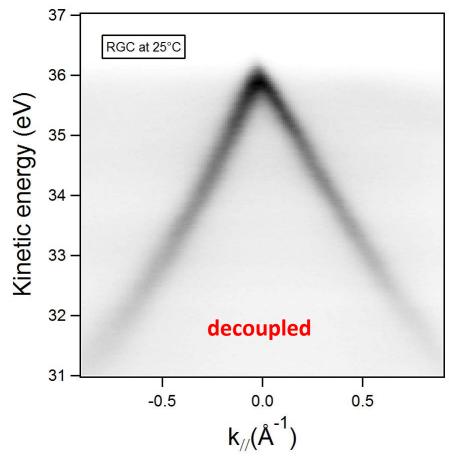


All movies: LEEM FoV 6 um, electron energy: 11 eV

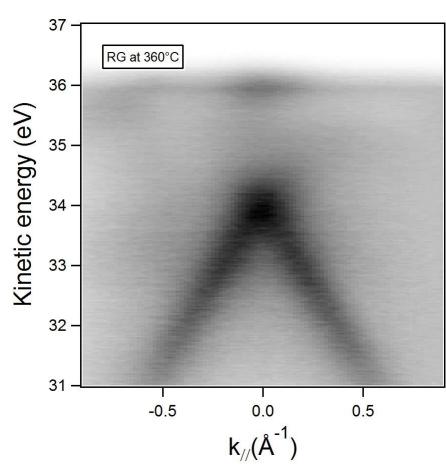
Coupling-decoupling is revealed by µ-ARPES



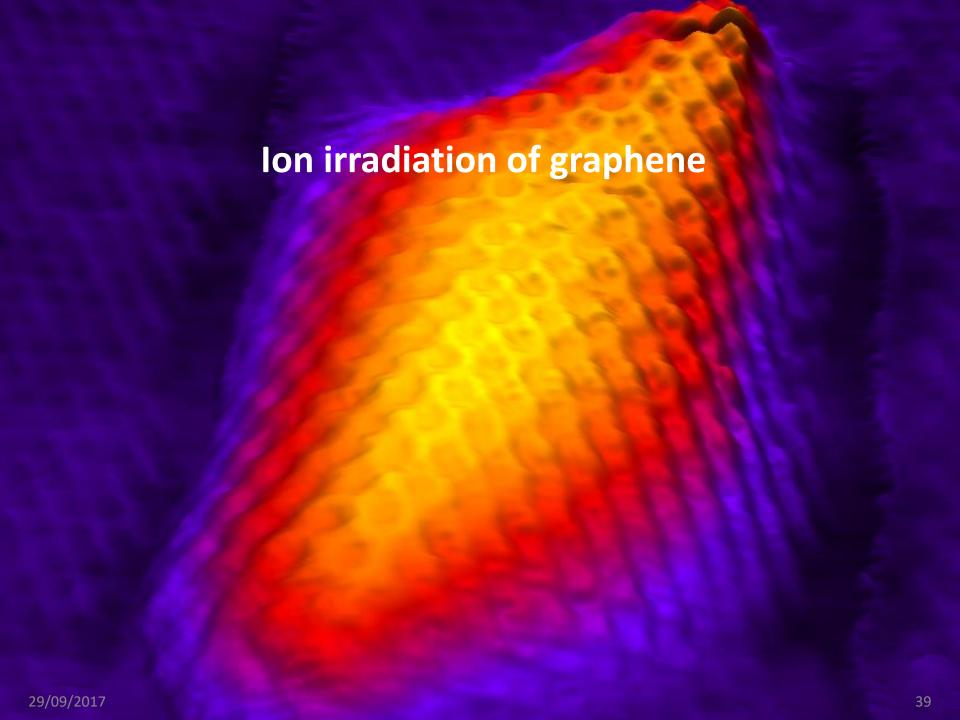




Rotated graphene with Ni-carbide underneath at room temperature; There's no double layer



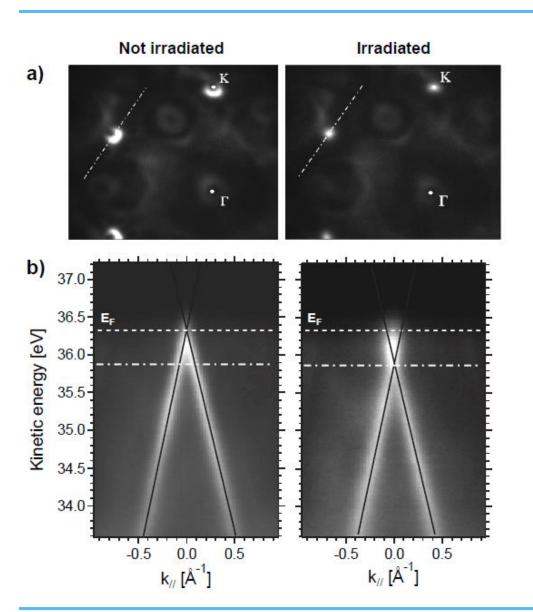
Rotated graphene without Ni-carbide underneath at 365°C



Example: Nitrogen-ion irradiated gr/Ir(111)



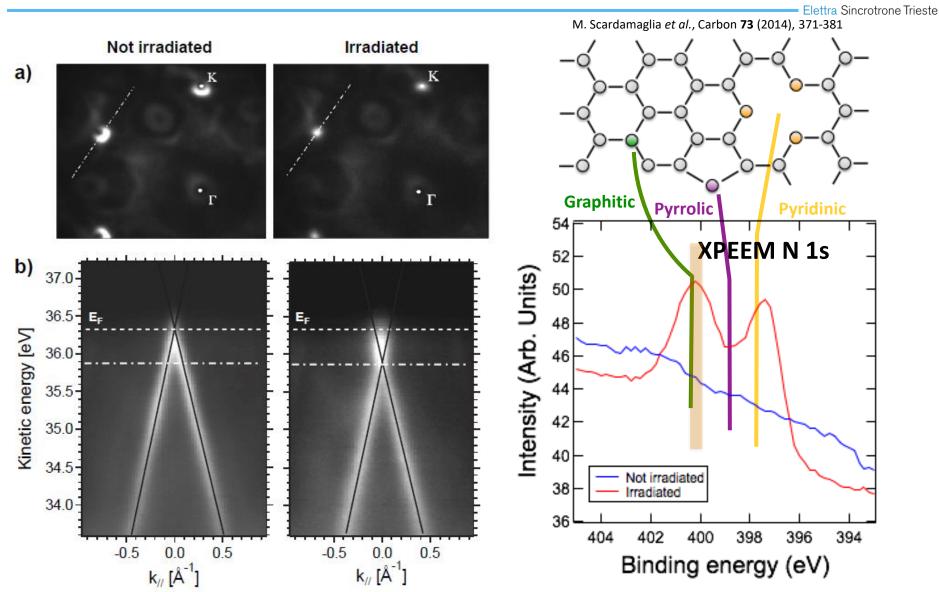
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Example: Nitrogen-ion irradiated gr/Ir(111)

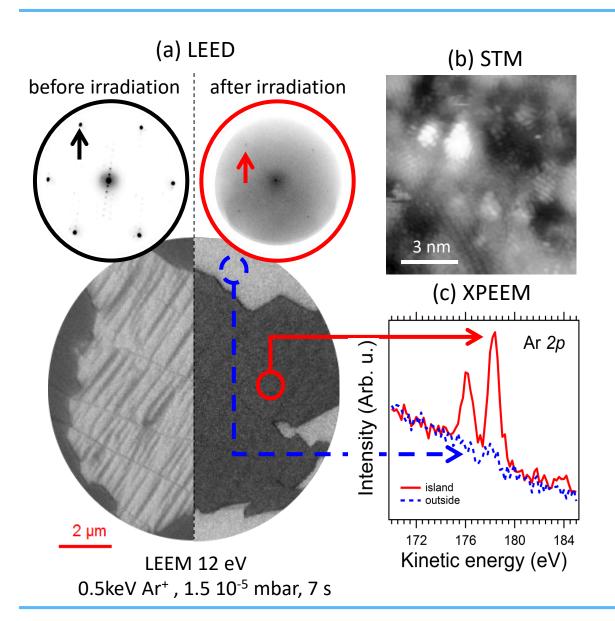




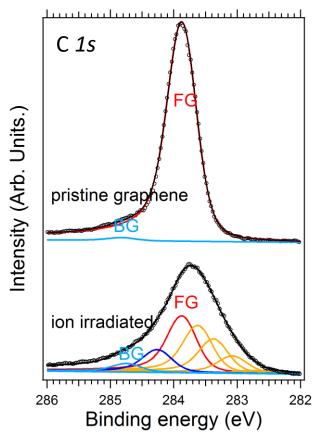
Morphology of Ar⁺ irradiated graphene/Ir(100)



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Rough morphology, but ... graphene is <u>continuous</u> average height 0.1 nm!

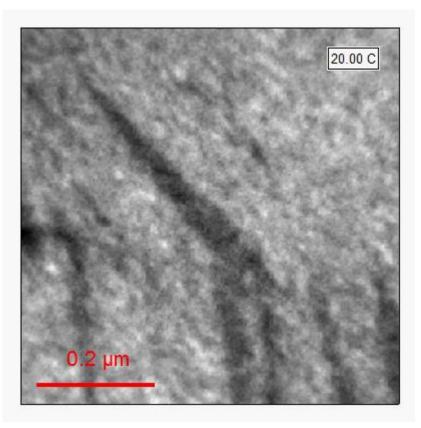


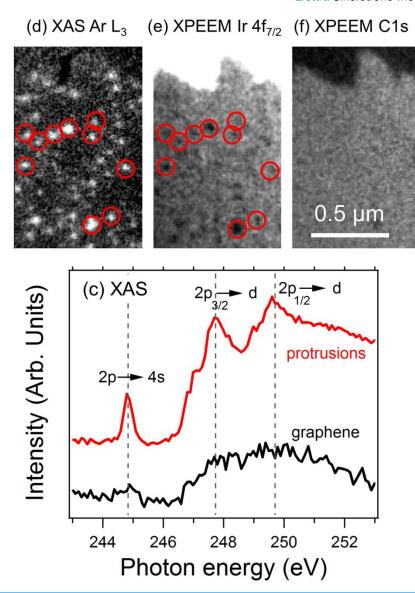
LEEM & XPEEM formation of Ar nanobubbles



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LEEM movie 12 eV

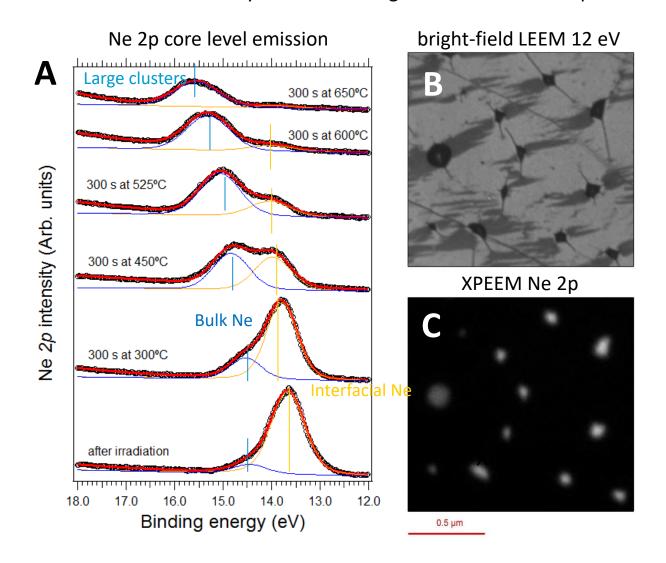




NB formation for g/Ne/Ir(100)

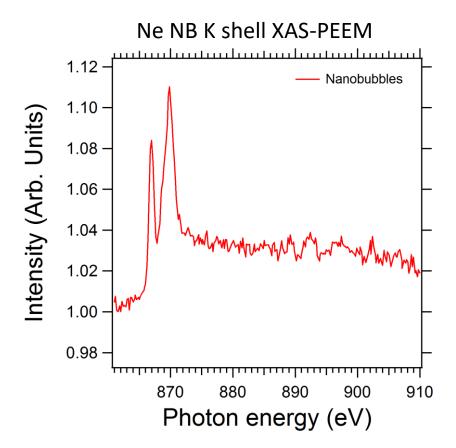


100 eV Ne+ ion irradiation was followed by 5 min annealing to 650 °C and subsequent cooling to RT



Ne state: XAS-PEEM

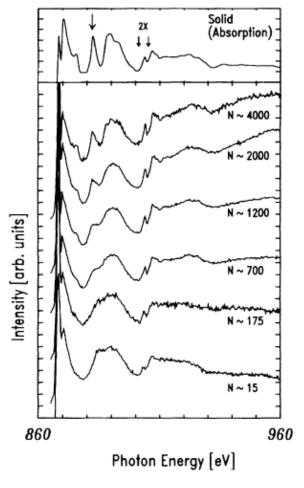




- 1s \rightarrow 3p,4p transition visible
- Spectrum is featureless at high energy

Large Ne NB are not solid!

Ne NB gas phase XPS data



F. Federmann *et al.*, Phys. Rev. Lett. **73**, 1549 (1994)

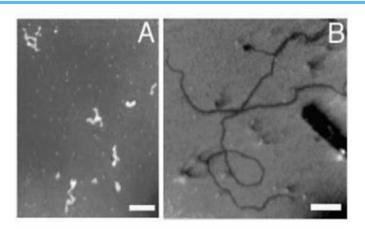
XAS-PEEM applications to biosciences

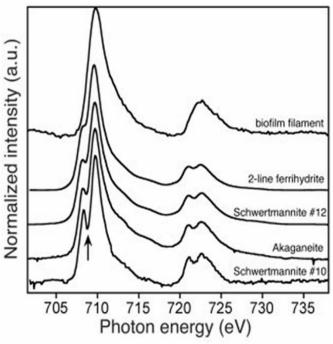


Applications of XAS in biology: biomineralization



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- Bio-mineralization resulting from microbal activity
- X-PEEM images of (A) non mineralized fibrils from the cloudy water above the biofilm (scale bar, 5 um)
- (B) mineralized filaments and a sheath from the biofilm (scale bar, 1 um); (bottom)
- X-PEEM Fe L-edge XANES spectra of the FeOOH mineralized looped filament shown in (B), compared with iron oxyhydroxide standards, arranged (bottom to top) in order of decreasing crystallinity.

P.U.P.A Gilbert *et al.* (ALS group), Science **303** 1656-1658, 2004.

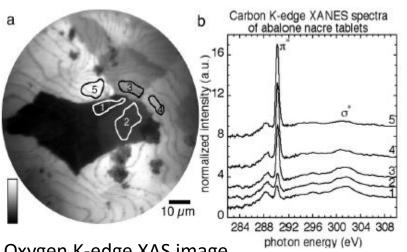
Nano-scale architecture of Nacre



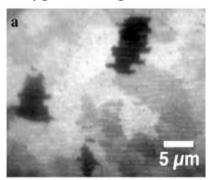
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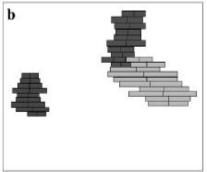
Carbon K-edge image

Carbon K-edge XANES



Oxygen K-edge XAS image





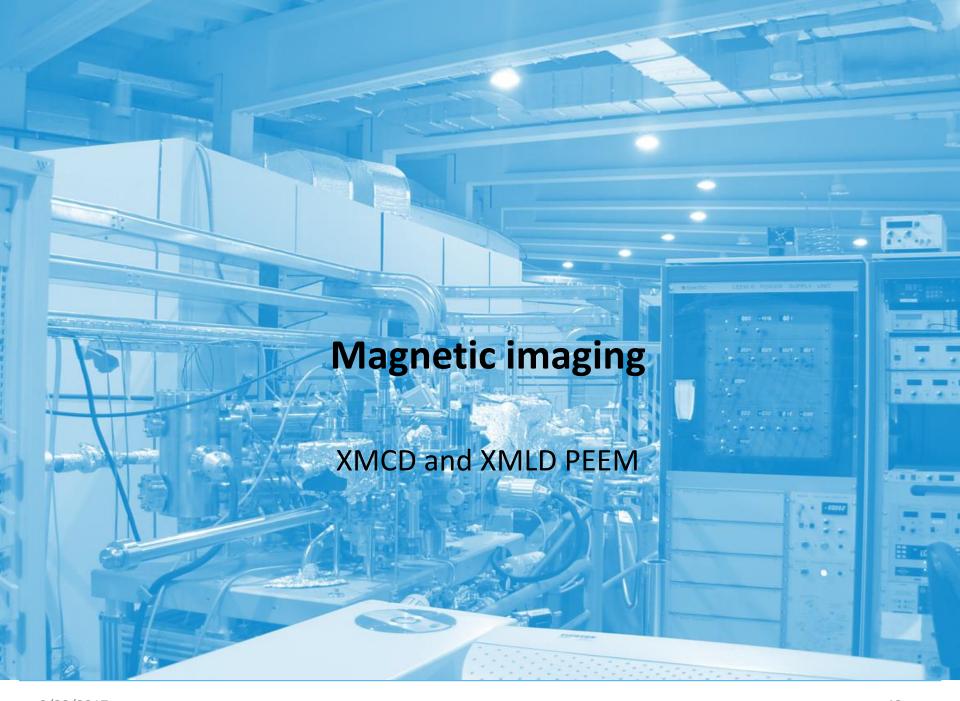
$$I(\vartheta, \theta, T) = a + b(3\cos^2\vartheta - 1)\langle Q_{zz}\rangle$$
$$+ c(3\cos^2\theta - 1)\langle M^2\rangle_T + d\sum_{i,j}\langle \hat{s}_i \cdot \hat{s}_j\rangle_T$$

Contrast is observed between adjacent individual nacre tablets, arising because different tablets have different crystal orientations with respect to the radiation's polarization vector.

The 290.3 eV peak corresponds to the C 1s \rightarrow Pi* transition of the CO bond. Synchrotron radiation is linearly polarized in the orbit plane. Under such illumination, the

intensity of the peak depends on the crystallographic orientation of each nacre tablet with respect to the polarization. This was the first observation of x-ray linear dichroism in a bio-mineral.

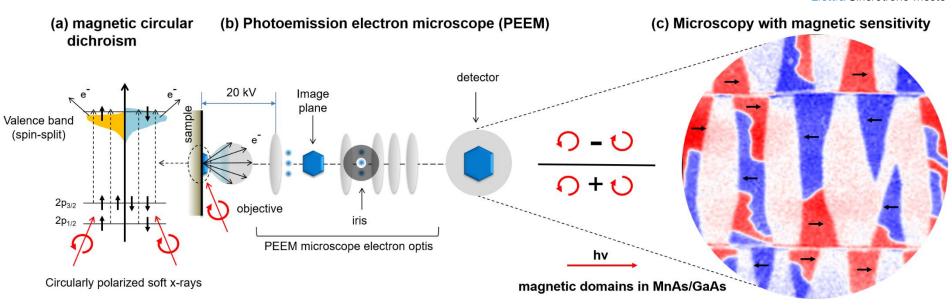
R.A. Metzler et al., Phys.Rev.Lett. 98, 268102 (2007)



Magnetic domain imaging by XMCD





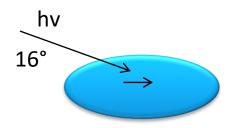


- We PROBE 3d elements by exciting 2p into unfilled 3d states
- Dominant channel: 2p → 3d
- White line intensity of the L3 and L2 resonances goes with number of empty d states (holes).
- Photoelectrons with opposite spins are created in the cases of left and right handed polarization. Spin polarization is opposite also for $p_{3/2}$ (L₃) and $p_{1/2}$ (L2) levels.
- The spin-split valence shell is a detector for the spin of the excited photoelectron. The size of the dichroism effect scales like $\cos\theta$, where θ is the angle between the photon spin and the magnetization direction.

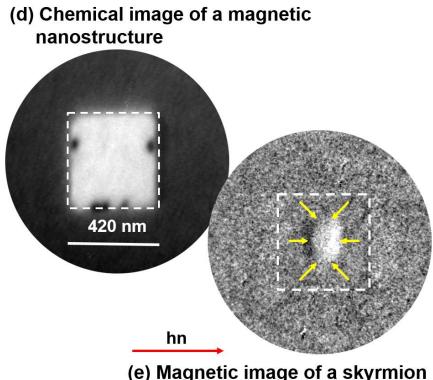
Magnetic domain imaging by XMCD







the illumination geometry, \rightarrow in plane component of M



(e) Magnetic image of a skyrmion

The spin-split valence shell is a detector for the spin of the excited photoelectron. The size of the dichroism effect scales like $\cos\theta$, where θ is the angle between the photon spin and the magnetization direction.

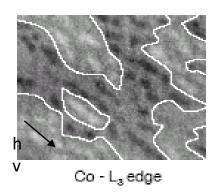
Examples of XMCD-PEEM applications



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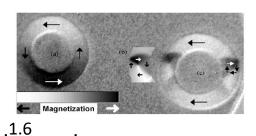
IMAGING OF MAGNETIC DOMAINS & DOMAIN WALLS

Co nanodots on Si-Ge

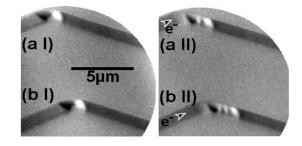


A. Mulders et al, Phys. Rev. B 71, 214422 (2005).

patterned structures

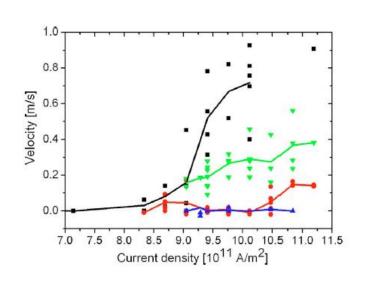


_{μm} pulse injection



M. Klaeui et al, PRL, PRB 2003 - 2010

domain wall motion induced by spin currents



Laufemberg et al, APL 88, 232507(2006).

Examples of XMCD-PEEM applications

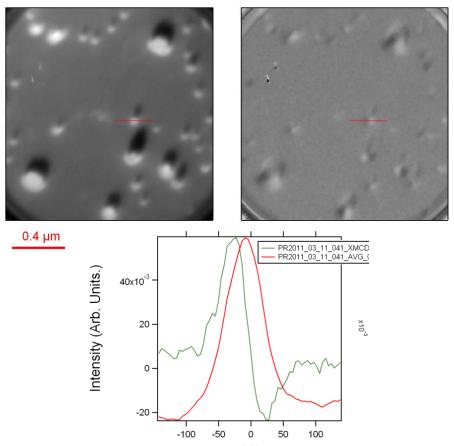


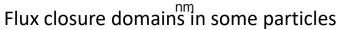
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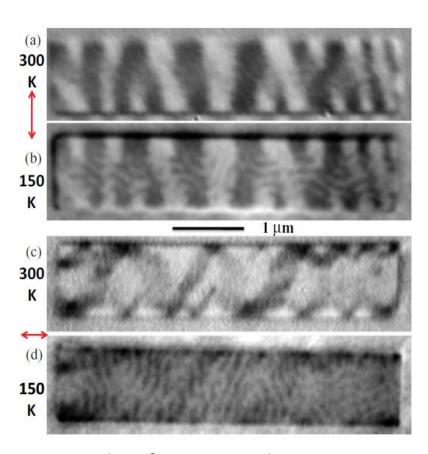
nano-magnetism of (Ga,Fe)N films

Magnetization in NiPd nanostructures

Fe L3 edge (chemical) Fe L3 edge (XMCD)



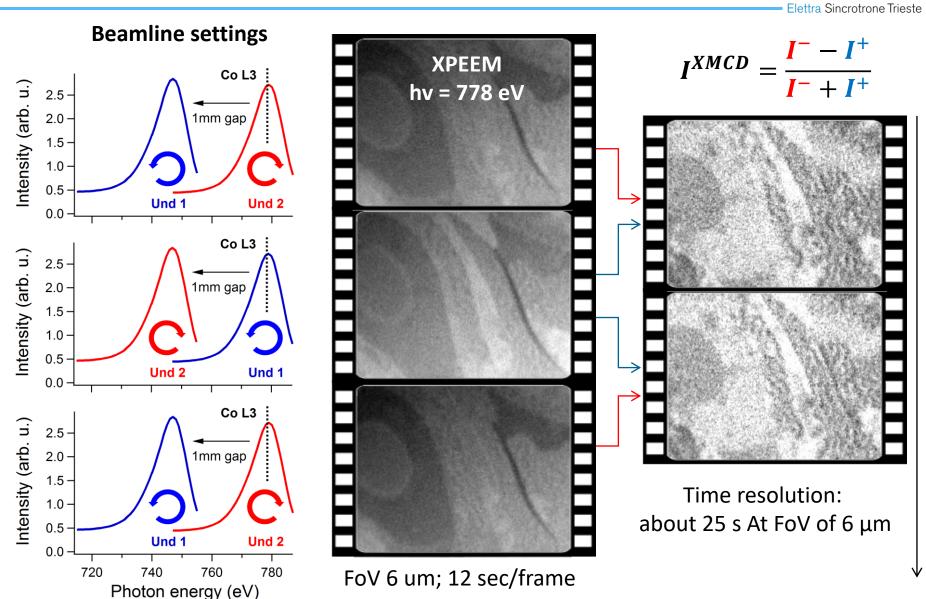




Canted configurations at low temperatures J.-Y. Chauleau, Phys. Rev. B 84, 094416 (2011)

Time evolution by XMCD-PEEM



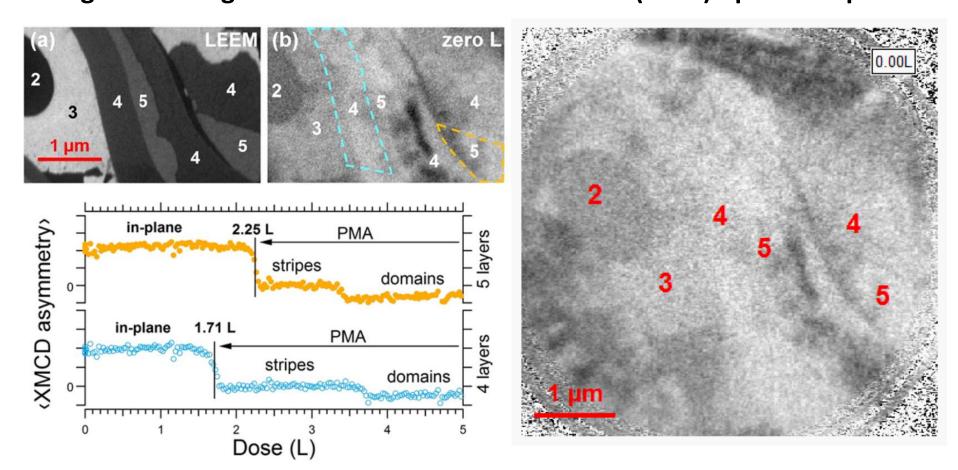


SRT upon CO uptake under photon irradiation



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Magnetic configuration of cobalt thin films on Re(0001) upon CO uptake



XMCD movie @ Co L₃ edge (780 eV); $P_{CO}=2\cdot10^{-9}$ mbar; frame acquisition 12 s, FoV 6 µm

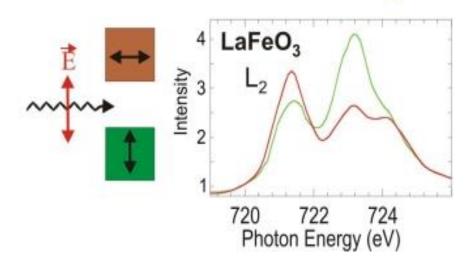
29/09/2017

Magnetic imaging basics: XMLD



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Linear Dichroism - Antiferromagnets



In the presence of spin order the spin-orbit coupling leads to preferential charge order relative to the spin direction, which is exploited to determine the spin axis in antiferromagnetic systems.

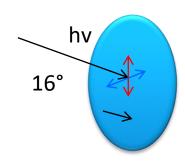
- ✓ Element sensitive technique
- ✓ Secondary imaging with PEEM determine large probing depth (10 nm), buried interfaces.
- ✓ Applied in AFM systems (oxides such as NiO)

Absorption intensity at resonance

$$I(\vartheta, \theta, T) = a + b(3\cos^2\vartheta - 1)\langle Q_{zz}\rangle$$
$$+ c(3\cos^2\theta - 1)\langle M^2\rangle_T + d\sum_{i,j}\langle \hat{s}_i \cdot \hat{s}_j\rangle_T$$

1st term: quadrupole moment, i.e.electronic charge (not magnetic!)

 2^{nd} term determines XMLD effect; Θ is the angle between E and magnetic axis A; XMLD max for E | | A;



Linear vertical and linear horizontal polarization of the photon beam

Applications of XMCD and XMLD



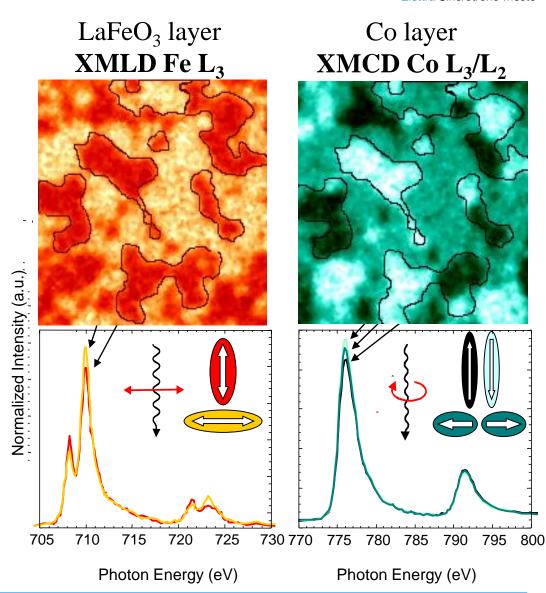
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Direct observation of the alignment of ferromagnetic spins by antiferromagnetic spins

F. Nolting*, A. Scholl*, J. Stöhr†, J. W. Seo‡§, J. Fompeyrine§, H. Siegwart§, J.-P. Locquet§, S. Anders*, J. Lüning†, E. E. Fullerton†, M. F. Toney†, M. R. Scheinfein|| & H. A. Padmore*

Nature, 405 (2000), 767.

ferromagnet/antiferromagnet
Co/LaFeO3 bilayer
interface exchange coupling
between the two materials

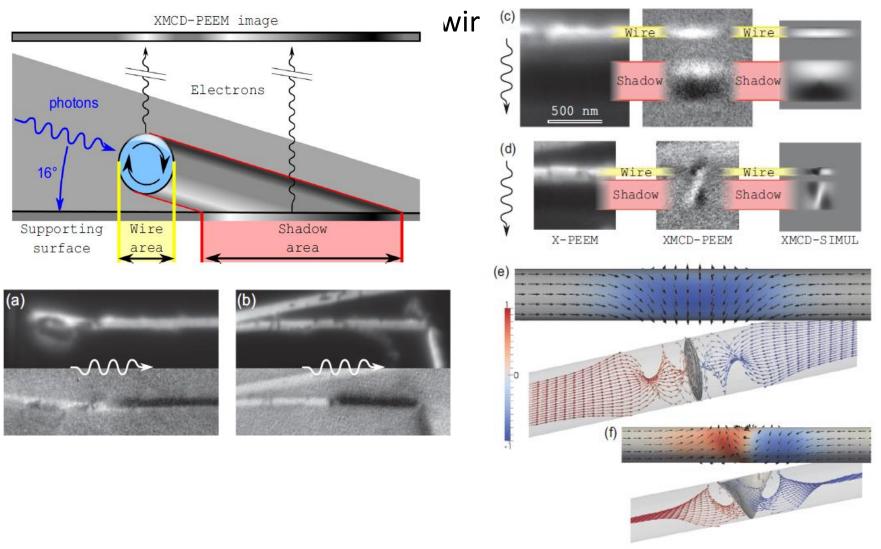


Tomographic imaging in magnetic wires



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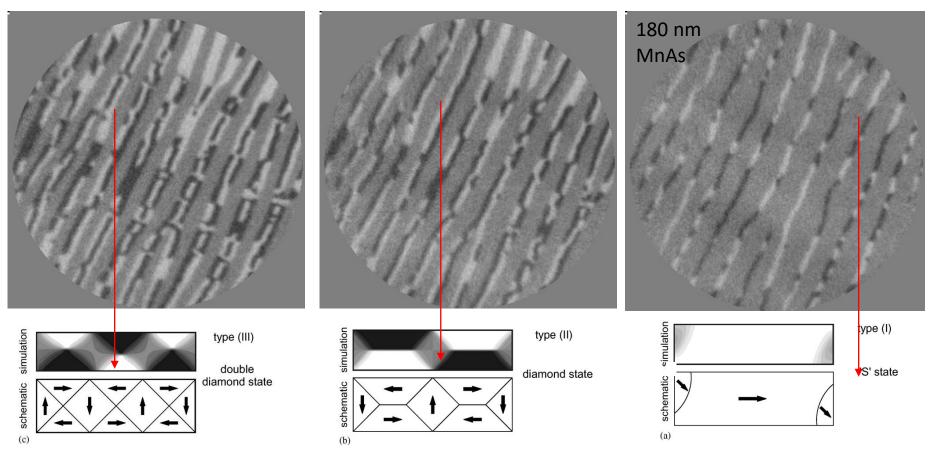
Observation of Bloch-point domain walls in cylindrical magnetic



Limited probing depth of XMCD: MnAs/GaAs

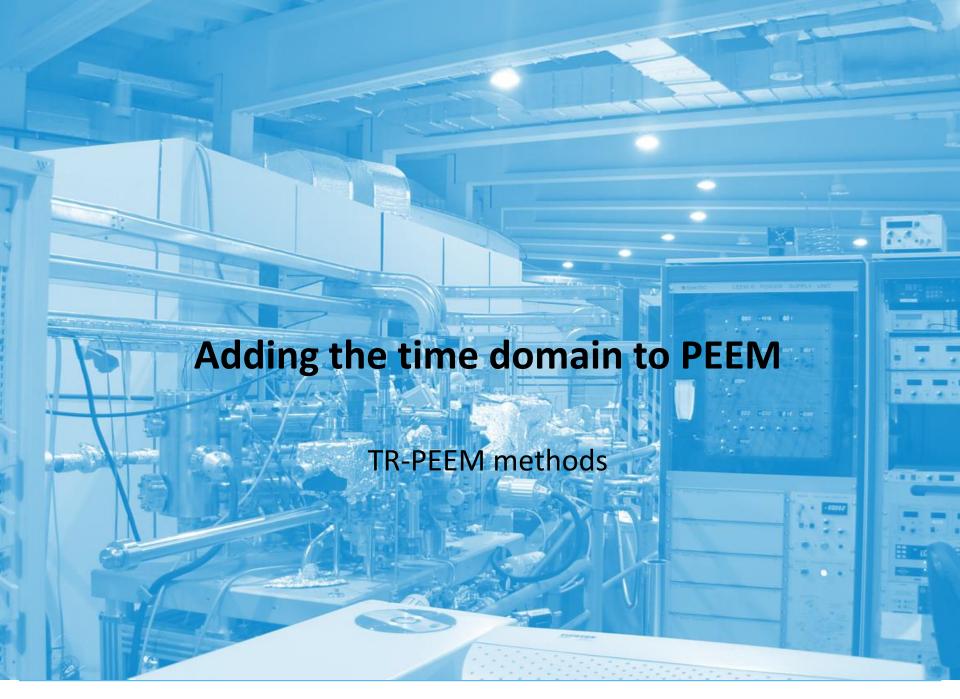


Experiment: Straight walls; Head to head domains



Simulation: Cross sectional cut: diamond state

R. Engel-Herbert et al, J. Magn. Magn. Mater. 305, (2006) 457



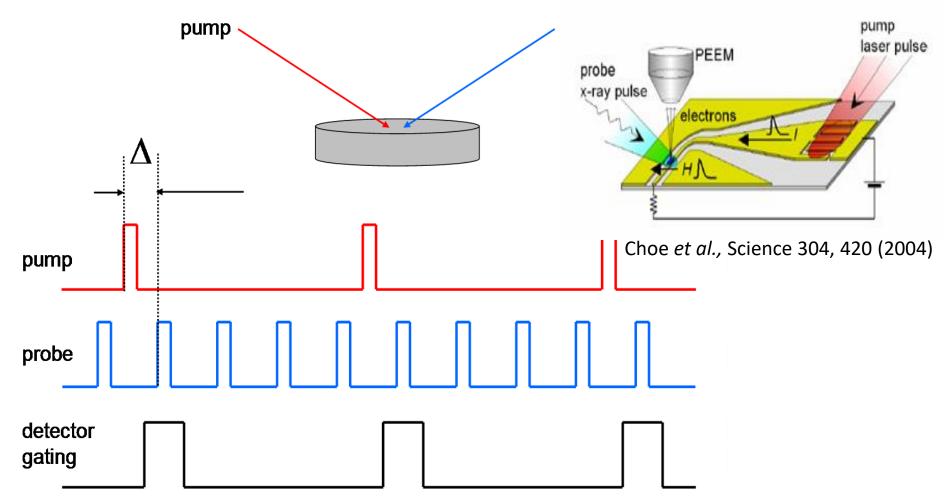
Time-resolved PEEM: the stroboscopic approach



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Stroboscopic experiments combine high lateral resolution of PEEM with high time resolution, taking advantage of pulsed nature of synchrotron radiation



Time resolved XMCD-PEEM: applications



- **Switching processes** (magnetisation reversal) in magnetic elements (in spin valves, tunnel junction)
 - Nucleation, DW propagation or both
 - Effect of surface topography, morphology crystalline structure etc.
 - Domain dynamics in Landau flux closure structures.
- response of vortices, domains, domain walls in Landau closure domains in the precessional regime
- Stroboscopic technique:
 - only reversible processes can be studied by pump probe experiments
 - Measurements are quantitative

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Magnetic excitations in LFC structures



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Quantitative Analysis of Magnetic Excitations in Landau Flux-Closure Structures Using Synchrotron-Radiation Microscopy

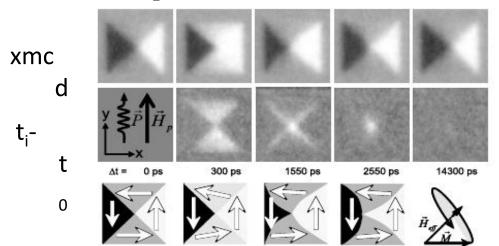
J. Raabe, 1,* C. Quitmann, 1 C. H. Back, 2 F. Nolting, 1 S. Johnson, 1 and C. Buehler 1

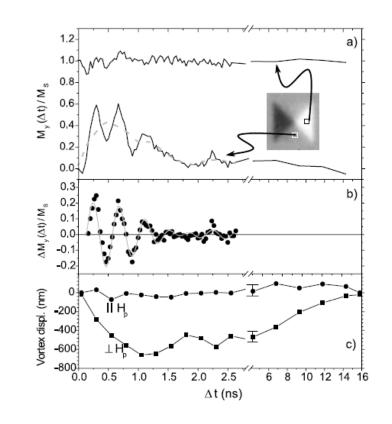
The time dependent magnetization is described by the phenomenological Landau-Lifshitz-Gilbert equation

$$\frac{d}{dt}\vec{M} = -\gamma_0 \vec{M} \times \vec{H}_{\rm eff} + \frac{\alpha}{M} \left(\vec{M} \times \frac{d}{dt} \vec{M} \right).$$

The first term describes the precession of the magnetization \vec{M} about the total effective field $\vec{H}_{\rm eff}$. The second term describes the relaxation back into the equilibrium state using the dimensionless damping parameter α .

torque
$$\vec{T} = -\gamma_0 \vec{M} \times \vec{H}_{\rm eff}$$





Summary



- XPEEM is a versatile full-field imaging technique. Combined with SR it allows
 us to implement laterally resolved versions of the most popular x-ray
 spectroscopies taking advantage of high flux of 3rd generation SR light sources.
- In particular, XAS-PEEM combines element sensitivity with chemical sensitivity (e.g. valence), and, more importantly, magnetic sensitivity. Magnetic imaging has been the most successful application of PEEM (next tutorial lecture!).
- XPEEM or energy-filtered PEEM adds true chemical sensitivity to PEEM.
 Modern instruments allow to combine chemistry with electronic structure using ARUPS.
- XPEEM can be complemented by LEEM, which adds structure sensitivity and capability to monitor dynamic processes.
- Lateral resolution will approach the nm range as AC instruments become available. Limitations due to space charge are not yet clear
- Novel application field are being approached, such as biology, geology and earth sciences. HAXPES will increase our capabilities to probe buried structures (bulk).

Review work



Reviews and topical papers on x-ray spectromicroscopy and XPEEM

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