

# The Nanospectroscopy Beamline at ELETTRA

elettra

S. Heun

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# Coworkers

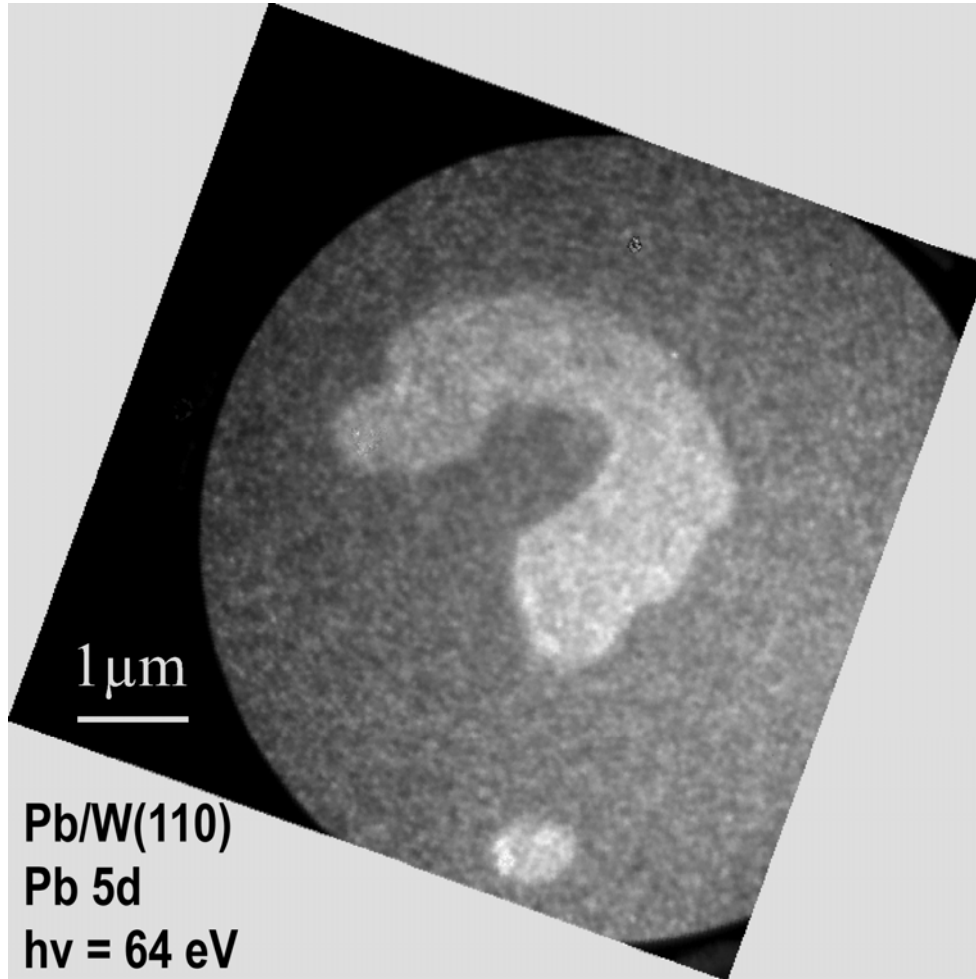
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# Motivation



## Why spectro-microscopy ?

- (semiconductor) nanostructures
  - lithography
  - self-organization
- devices
- laterally inhomogeneous surfaces
- segregation at defects
- alloying (silicide formation)
- 2-compound growth on surfaces
- **XMCD with lateral resolution**

# Outline

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1. Spectromicroscopy at Elettra
2. The SPELEEM microscope
3. The Nanospectroscopy Beamline
4. First Results: MnAs on GaAs

# Outline

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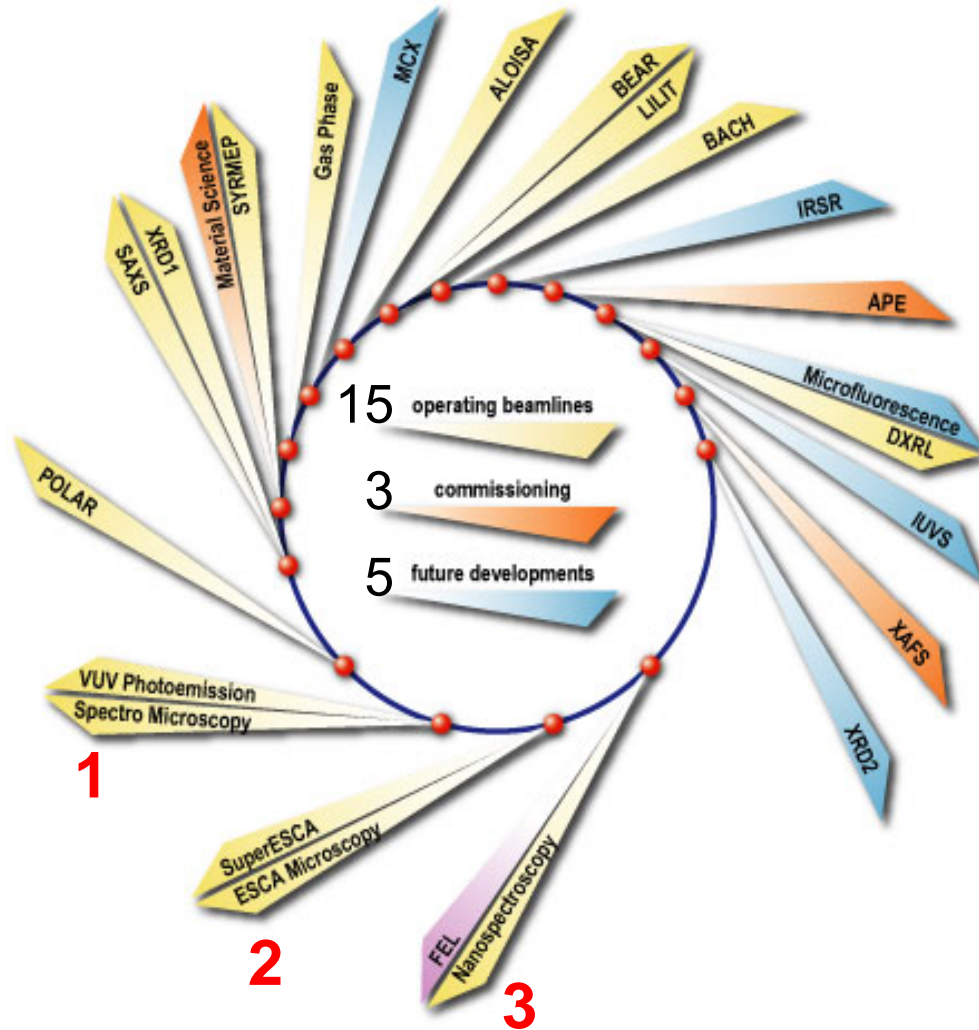
1. Spectromicroscopy at Elettra
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# Location of Elettra

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# Elettra Beamlines

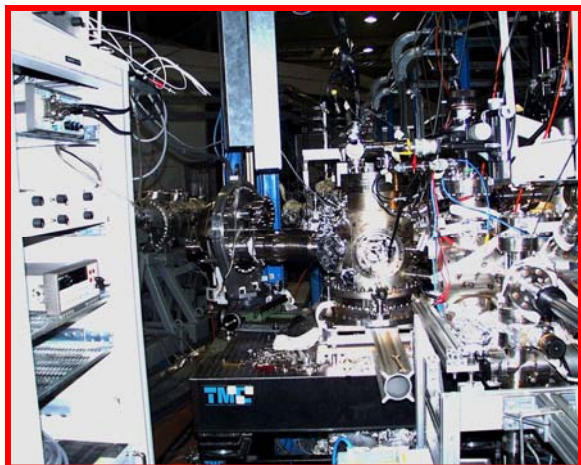


2.0 GeV / 320 mA  
2.4 GeV / 140 mA

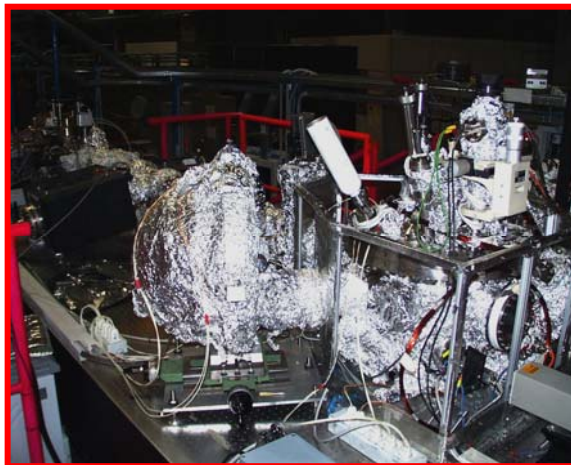
exit	beamline	source
1.2L	Nanospectroscopy *	id
1.2R	FEL (Free-Electron Laser)	-
2.2L	ESCA Microscopy	id
2.2R	SuperESCA	id
3.2L	Spectro Microscopy	id
3.2R	VUV Photoemission	id
4.2	Circularly Polarised Light	id
5.2L	SAXS (Small Angle X-Ray Scattering)	id
5.2R	XRD1 (X-ray Diffraction)	id
6.1L	Material science	bm
6.1R	SYRMEP (SYnchrotron Radiation for MEDical Physics)	bm
6.2R	Gas Phase	id
7.1	MCX (Powder Diffraction Beamline)	bm
7.2	ALOISA (Advanced Line for Overlayer, Interface and Surface Analysis)	id
8.1L	BEAR (Bending magnet for Emission Absorption and Reflectivity) *	bm
8.1R	LILIT (Lab of Interdisciplinary LIThography)	bm
8.2	BACH (Beamline for Advanced DiChroism) *	id
9.1	IRSR (Infrared Synchrotron Radiaton Microscopy)	bm
9.2	APE (Advanced Photoelectric-effect Experiments) **	id
10.1L	X-ray microfluorescence	bm
10.1R	DXRL (Deep-etch Lithography)	bm
10.2	IUVS (Inelastic Ultra Violet Scattering)	id
11.1	XAFS (X-ray Absorption Fine Structure)	bm
11.2	XRD2 (X-ray Diffraction)	id

# Operating Beamlines

U2.2 EscaMicroscopy



U3.2 SpectroMicroscopy



U1.1 Nanospectroscopy

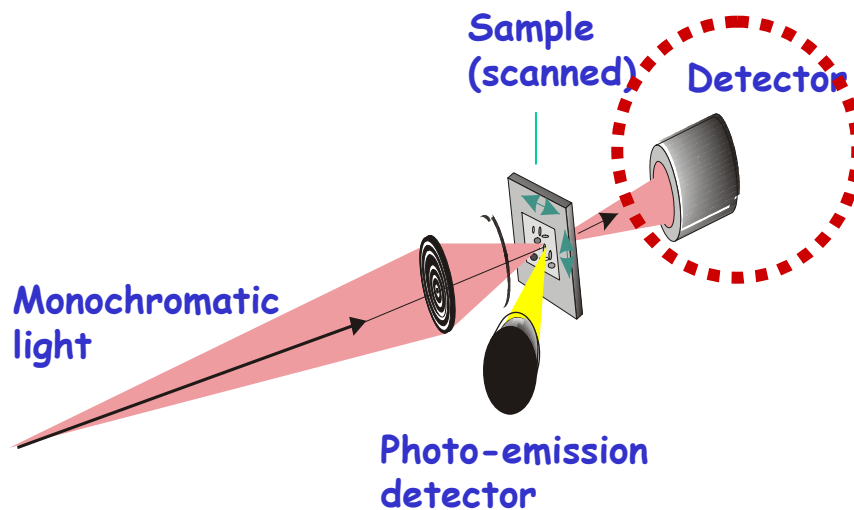


	<u>EM</u>	<u>SM</u>	<u>NS</u>
E (eV):	400-750	20-110	40-1000
SR (nm):	90	500	40 (20)
SR (eV):	0.25	0.07	0.4 (0.25)
Flux (ph/s):		$10^9-10^{10}$	$10^{11}-10^{13}$
Methods:	XPS(XAS)	XPS	XPS-XAS
Polarization:		Linear	Linear&circular



# Photoemission Microscopy

## Scanning (PE&T)

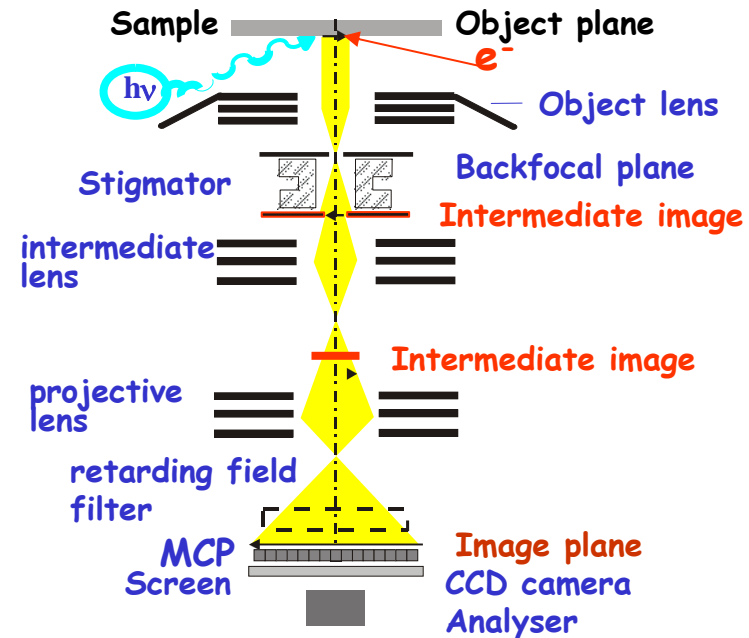


Photon optics is demagnifying the beam:

### Scanning Instrument

1. Whole power of XPS in a small spot mode.
2. Flexibility for adding different detectors.
3. Rough surfaces can be measured.
4. Limited use for fast dynamic processes.
5. Lower resolution than imaging instruments.

## XPEEM



Electron optics to magnify irradiated area:

### Imaging Instrument

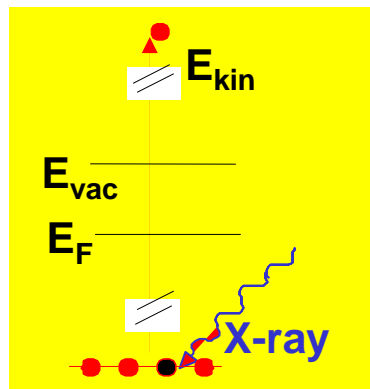
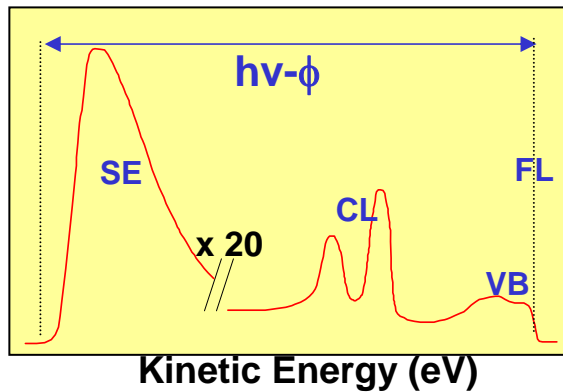
1. High lateral resolution (20 nm).
2. Multi-method instrument (XPEEM/PED).
3. Excellent for monitoring dynamic processes.
4. Poorer spectroscopic ability.
5. Sensitive to rough surfaces.

# Concepts of Spectromicroscopy

**XPS – mode: hv=const**

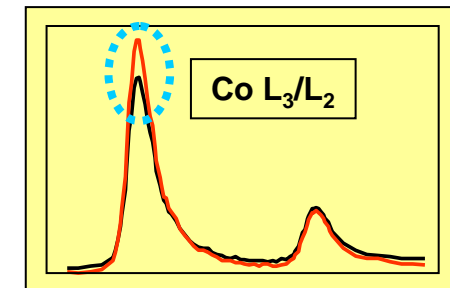
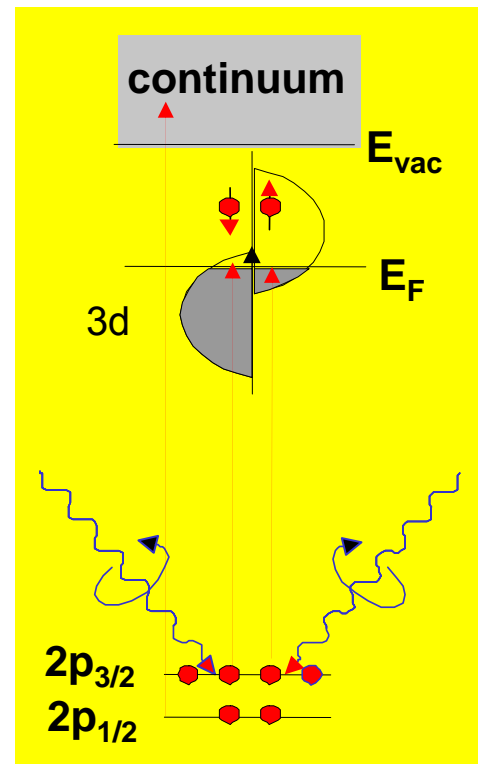
hv in / e<sup>-</sup> out

+ energy filtering of electrons



**XAS – mode: hv scanned**

hv in / e<sup>-</sup> out (TEY)

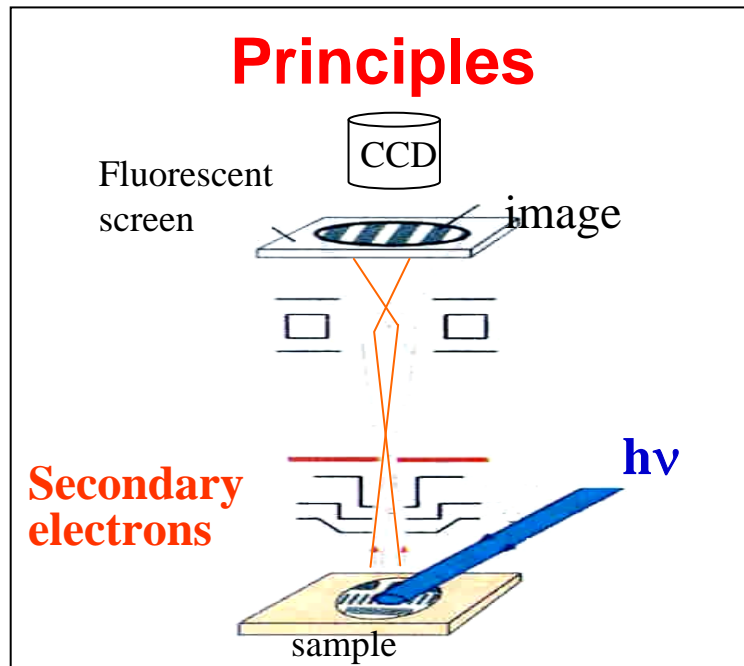


**XANES:**  
tuning on molecular  
orbitals

**XMLD:** imaging  
antiferromagnets

**XMCD:** imaging  
ferromagnets

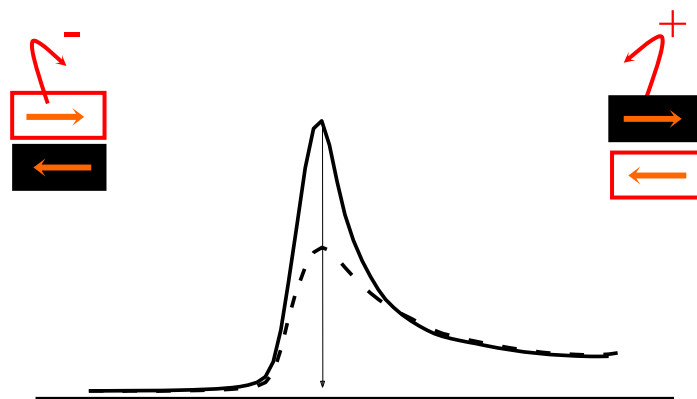
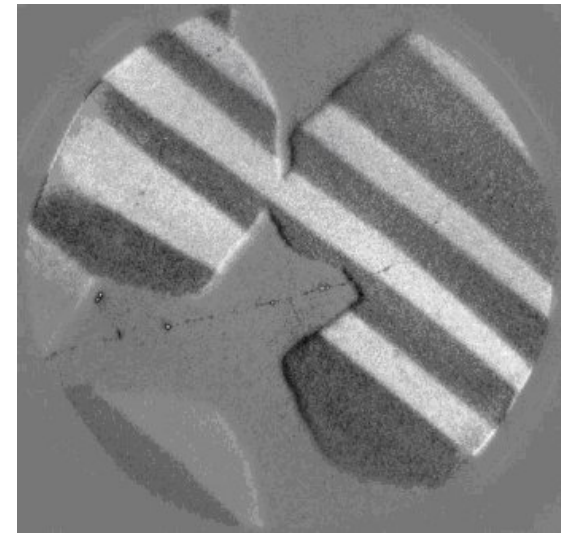
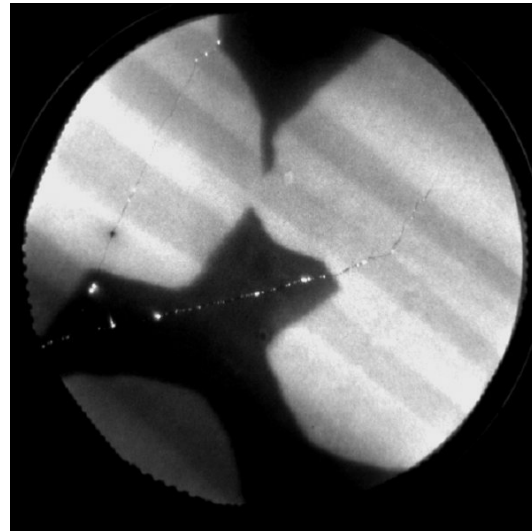
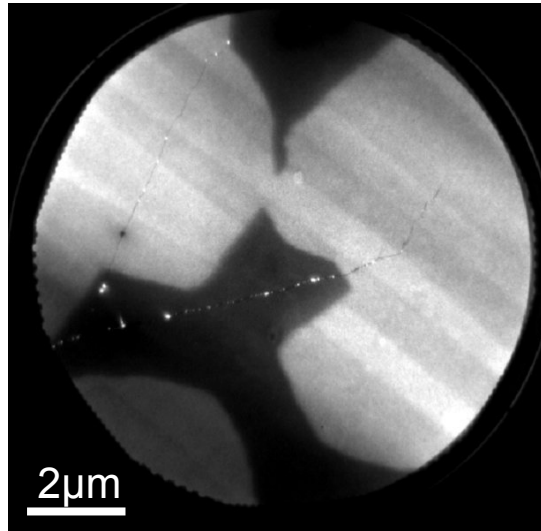
**Sum rules: Magnetic moment values**



## Characteristics

- Elemental resolution
- Lateral resolution
- Magnetic domain imaging
- Magnetic moment values

# XMCD-PEEM



$I^- \text{ max} - I^+ \text{ max}$   
Magnetic contrast  
XMCD- PEEM

# Outline

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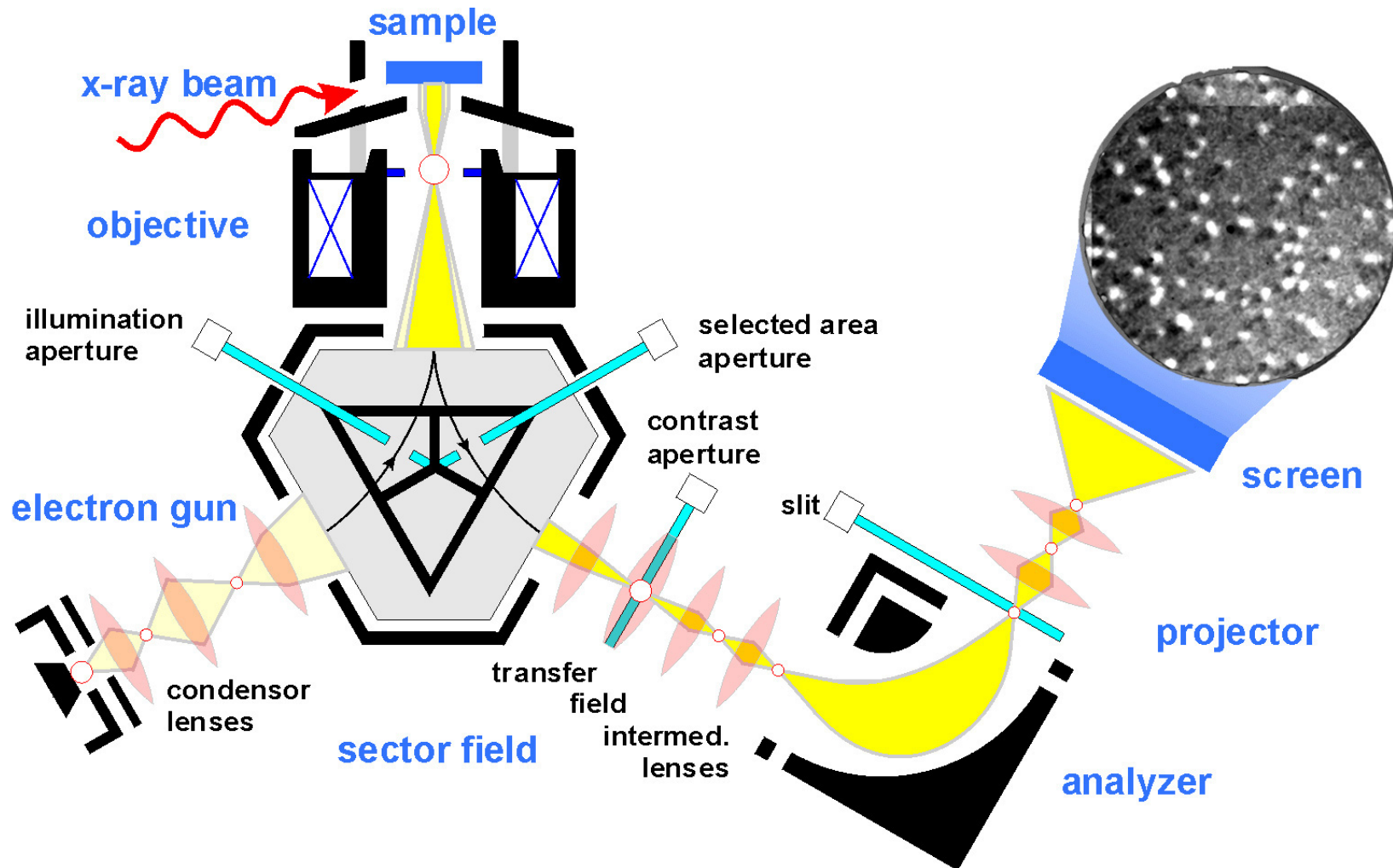
1. Spectromicroscopy at Elettra
2. The SPELEEM microscope
3. The Nanospectroscopy Beamline
4. First Results: MnAs on GaAs

# The SPELEEM at ELETTRA

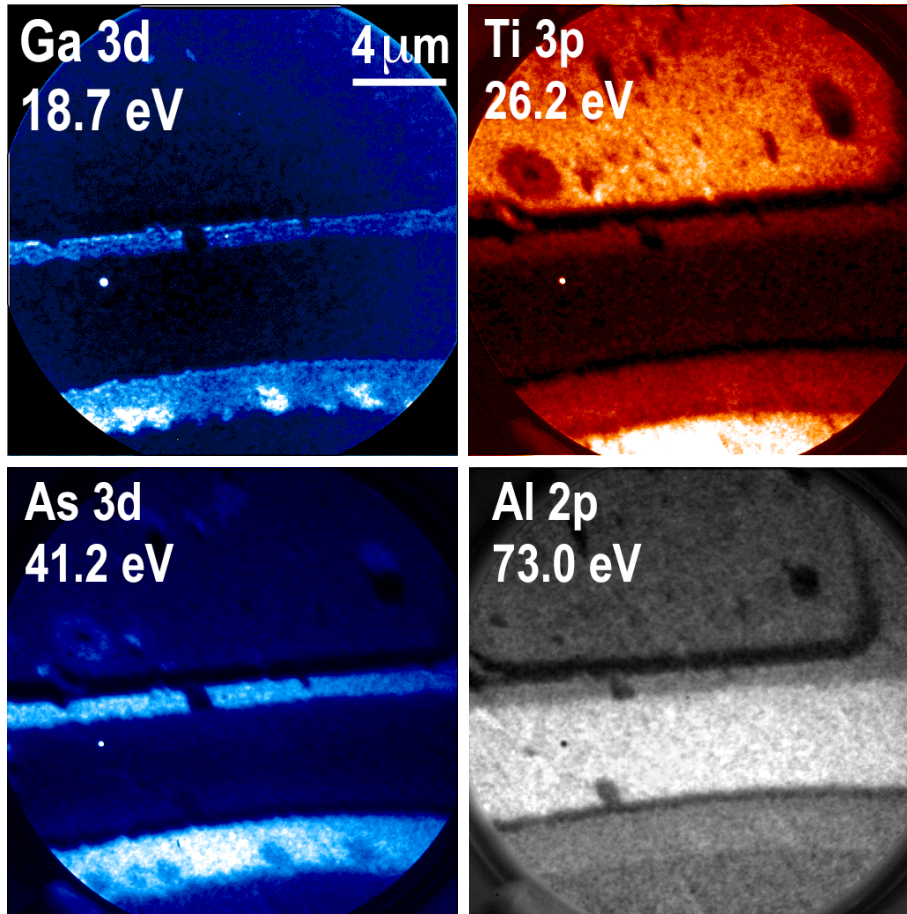


# The SPELEEM

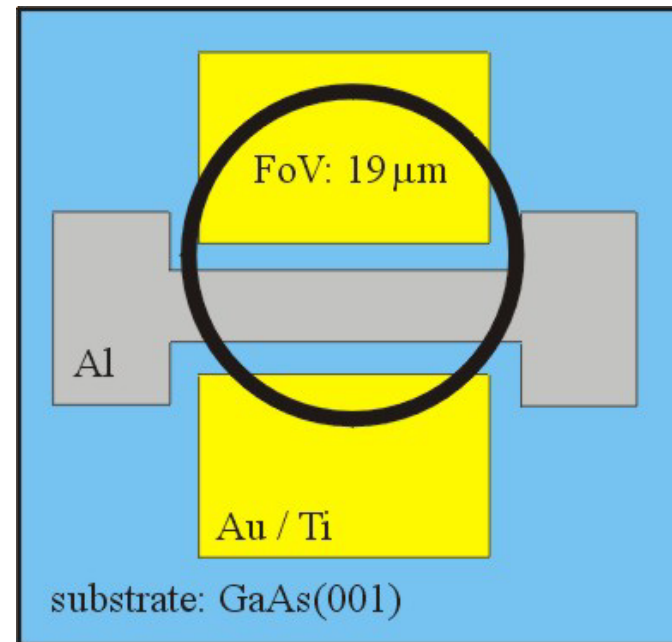
Spectroscopic photoemission and low energy electron microscope



# Spectroscopic Microscopy



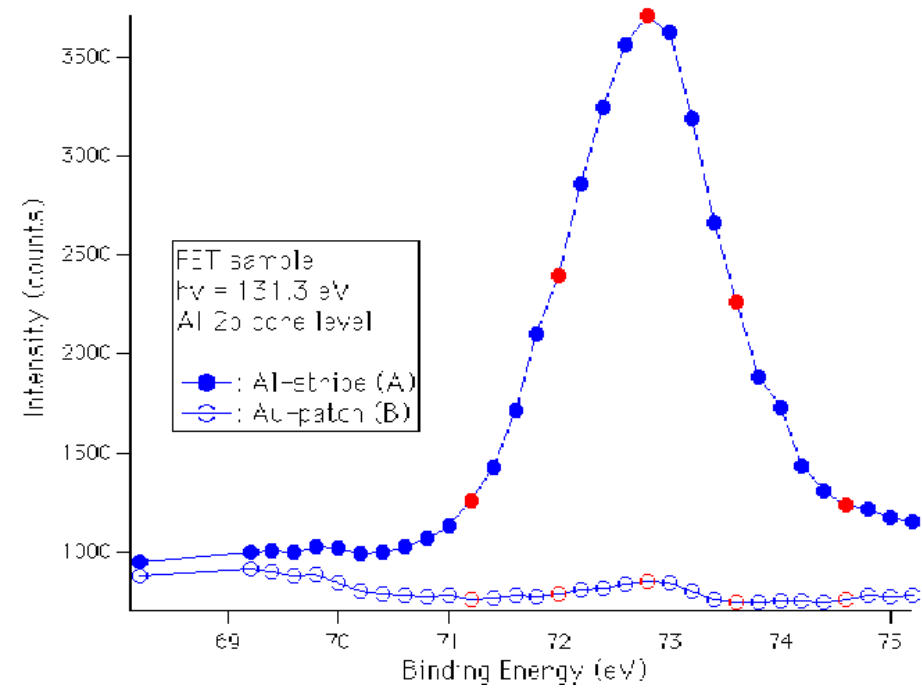
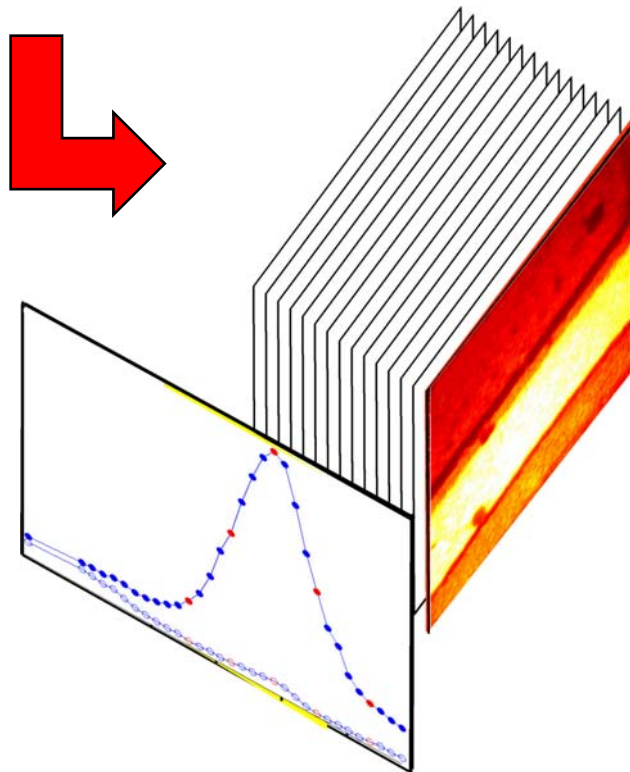
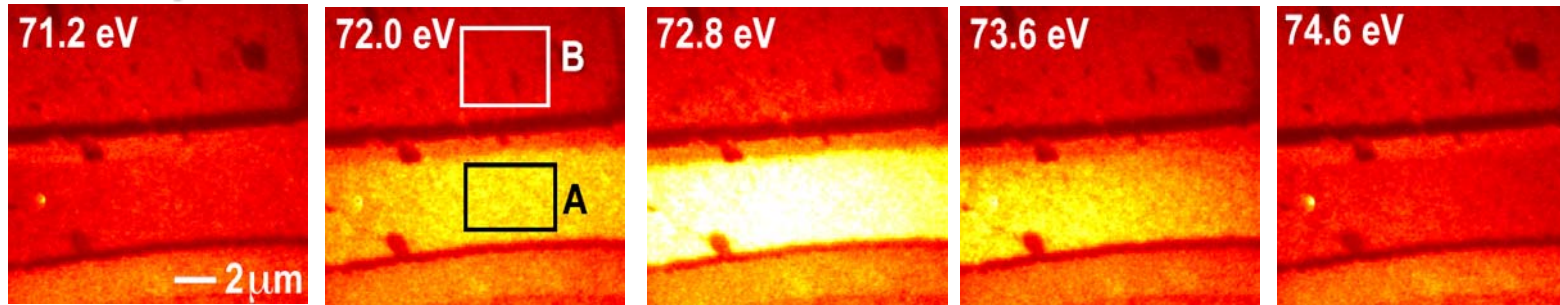
Images from a Field Effect Transistor (FET) at different binding energies. Photon energy  $h\nu = 131.3$  eV.



Sample from M. Lazzarino, L. Sorba, and F. Beltram, Laboratorio TASC-INFM, Trieste, Italy



# XPEEM

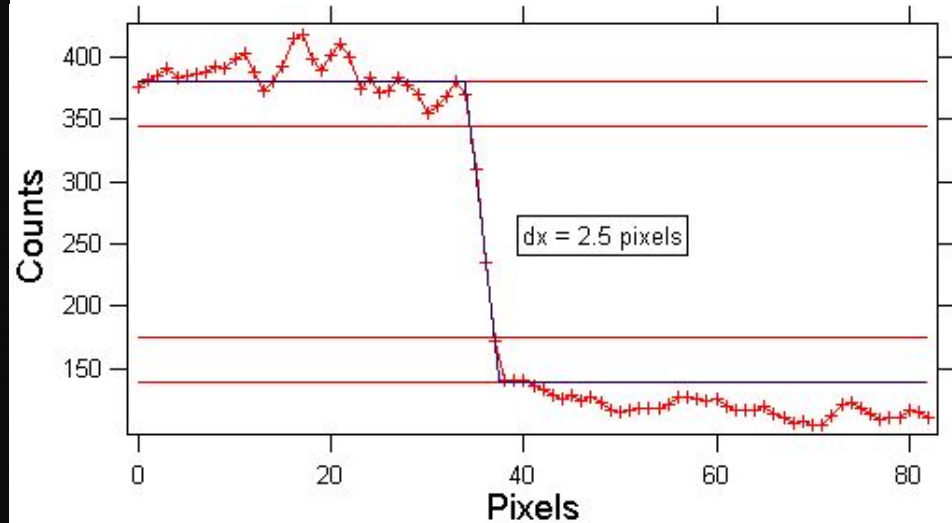
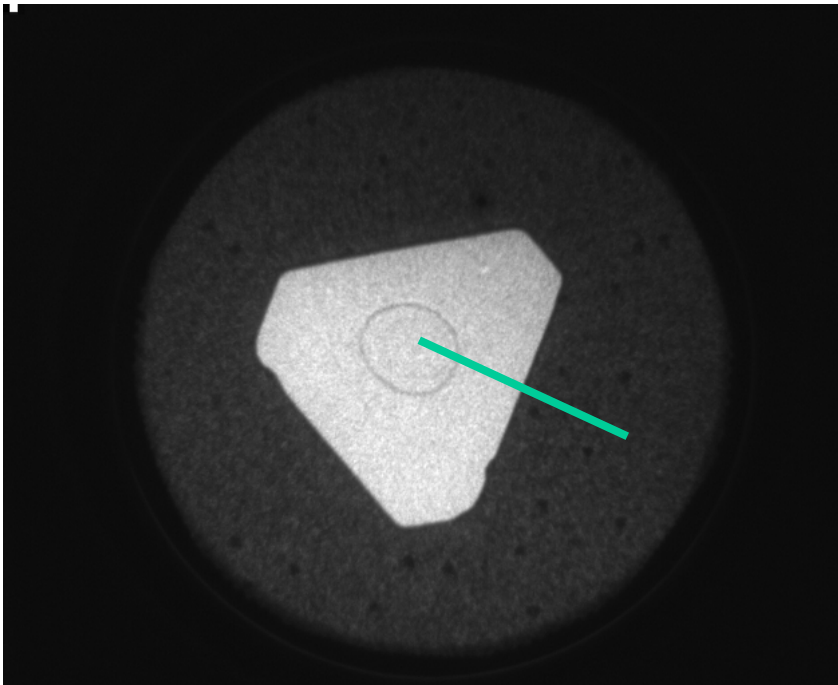


# Lateral Resolution of LEEM

FoV = 2.65  $\mu\text{m}$   
STV = 7.5 eV  
12.5  $\mu\text{m}$  energy slit  
30  $\mu\text{m}$  contrast aperture  
100 ms int. time, 2x2 binning

Pb on Si (111)  
LEEM – lateral resolution  
13/11/2002 image\_003

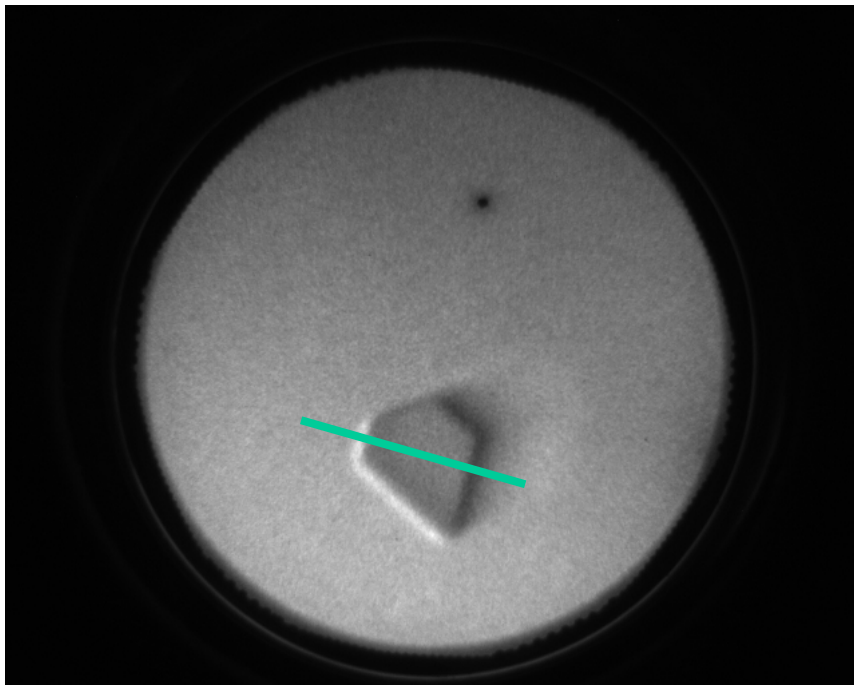
Profile line width = 3 pixels



Spatial resolution is **15 nm**.

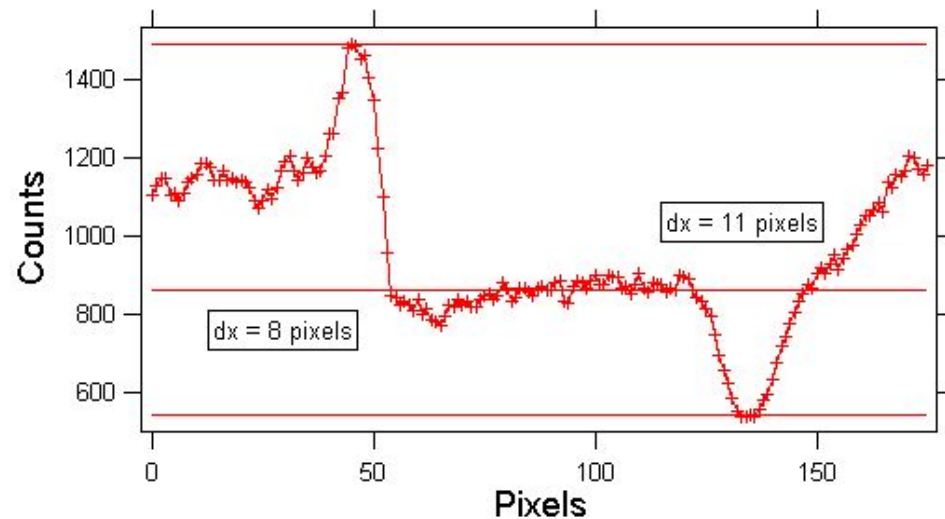
# Lateral Resolution of XPEEM

FoV = 2.65  $\mu\text{m}$   
STV = 1.2 eV,  $h\nu = 54.5$  eV  
12.5  $\mu\text{m}$  energy slit  
20  $\mu\text{m}$  contrast aperture  
15 s int. time, 2x2 binning



Pb on Si (111)  
XPEEM – lateral resolution  
imaging secondaries  
12/11/2002 image\_025

Profile line width = 7 pixels

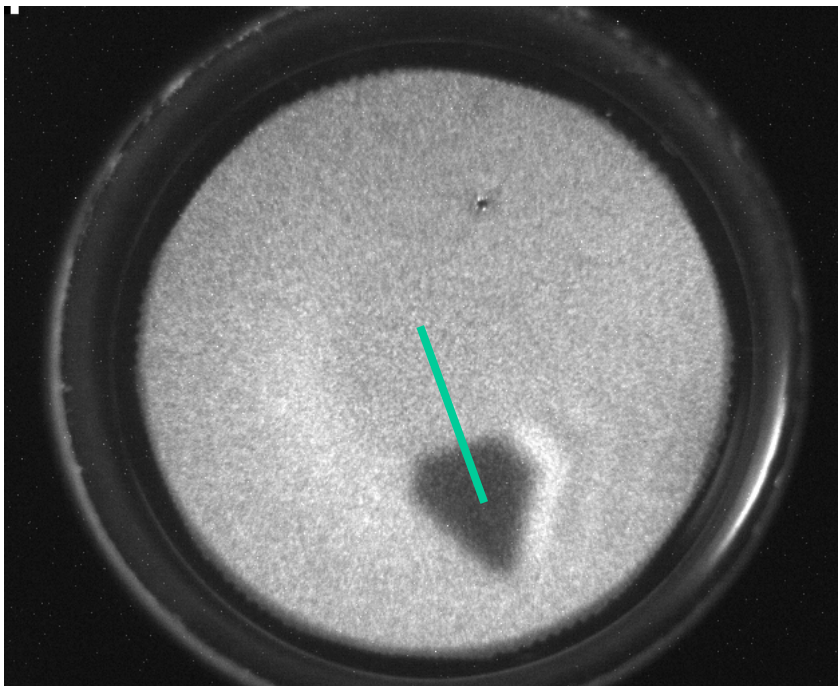


Spatial resolution is **40 nm**.

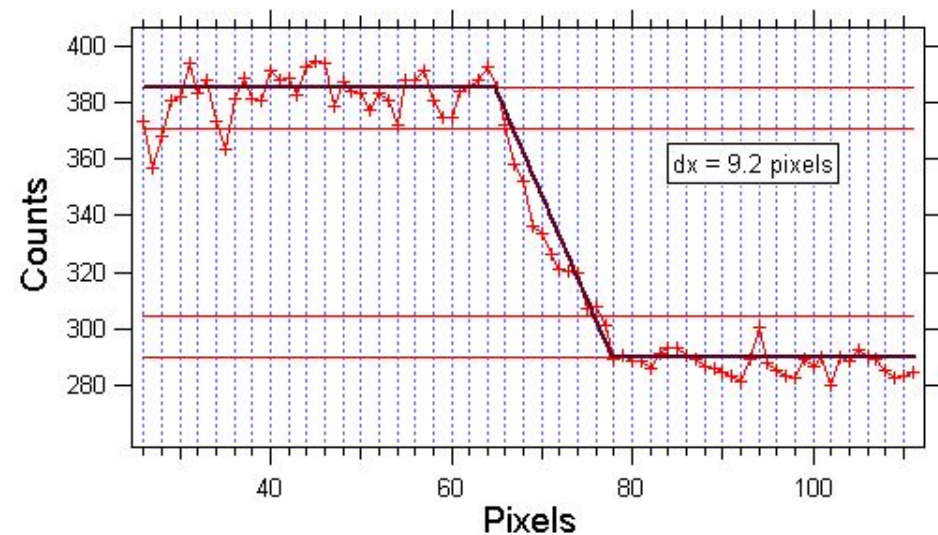
# Lateral Resolution of XPEEM

FoV = 2.65  $\mu\text{m}$   
STV = 43.2 eV,  $h\nu = 144.0$  eV  
12.5  $\mu\text{m}$  energy slit  
30  $\mu\text{m}$  contrast aperture  
240 s int. time, 2x2 binning

Pb on Si (111)  
XPEEM – lateral resolution  
core level imaging – Si 2p  
12/11/2002 image\_033



Profile line width = 7 pixels



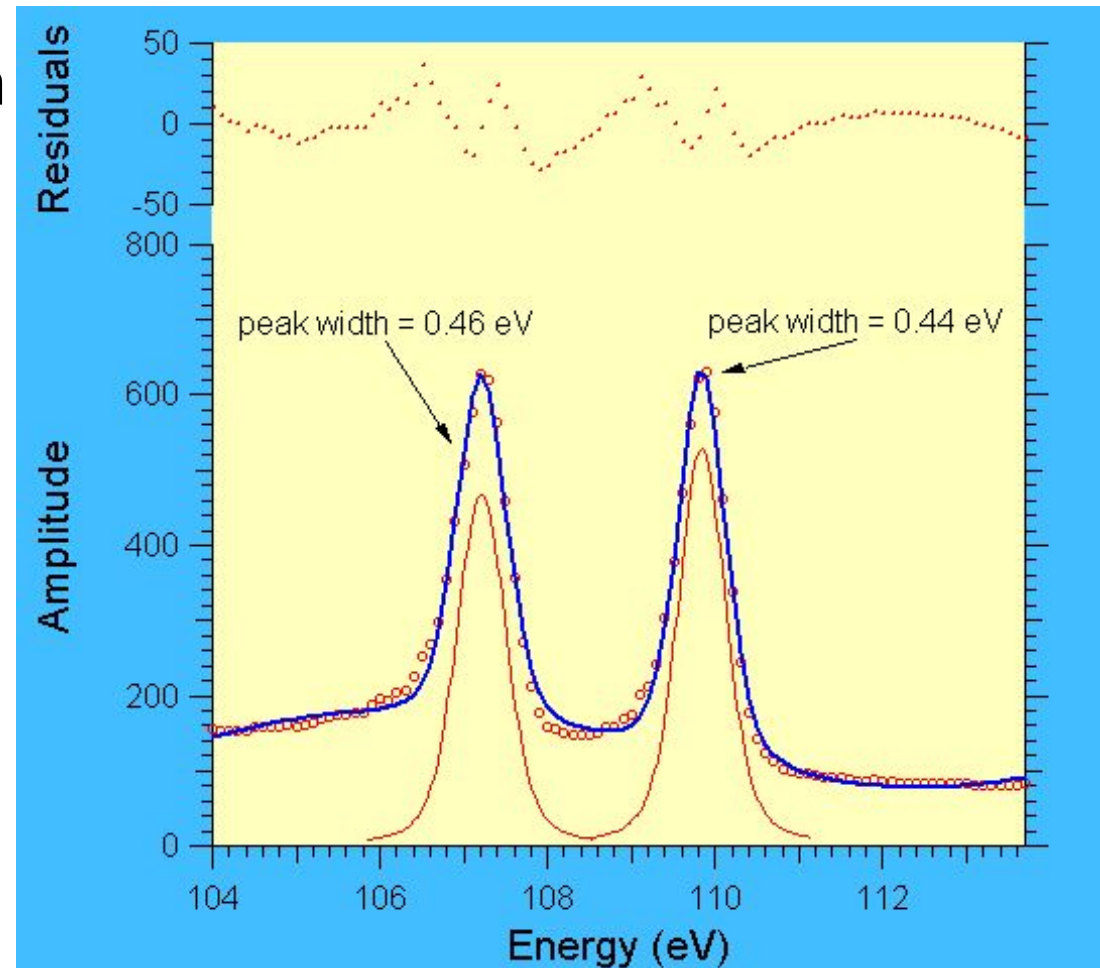
Spatial resolution is **55 nm**.

# Energy Resolution of XPEEM

Pb on Si (111)  
XPEEM – energy resolution  
Pb 5d – Voigt fit  
13/11/2002 scan\_002

FoV = 2.65  $\mu\text{m}$   
 $h\nu = 130.0 \text{ eV}$   
12.5  $\mu\text{m}$  energy slit  
30  $\mu\text{m}$  contrast aperture  
30 s int. time, 4x4 binning

Energy resolution  
better than **0.45 eV**.



# Outline

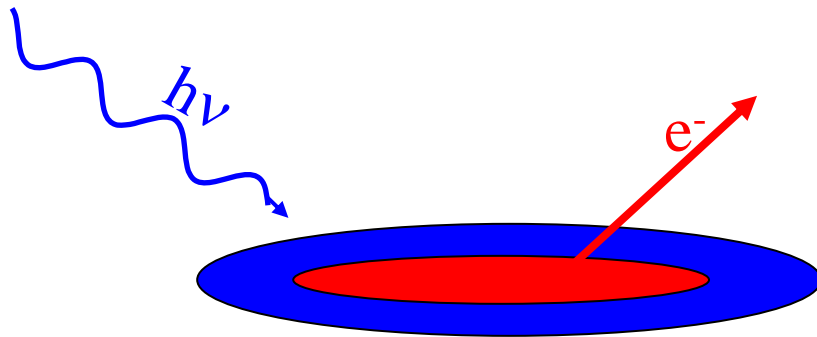
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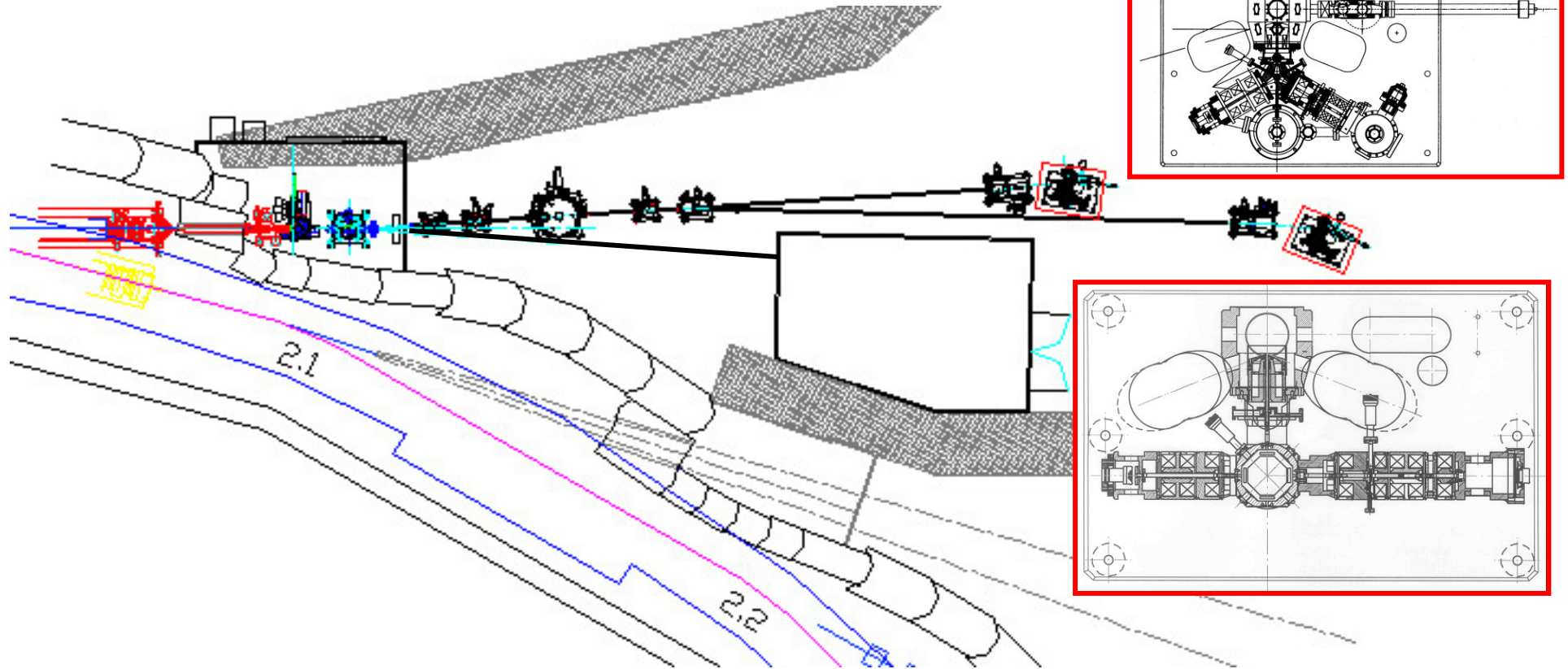
# Requirements

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- Source:** Variable Polarization
- Monochromator:** Wide spectral range  
Medium spectral resolution
- Spot:** High photon flux density on sample  
Small variable spot size ( $\sim\mu\text{m}$ )  
Homogeneous illumination



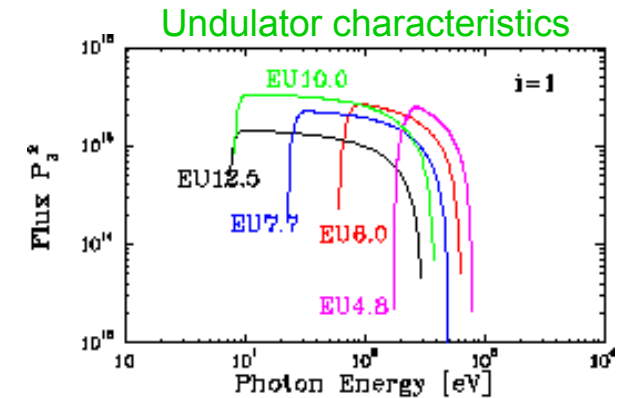
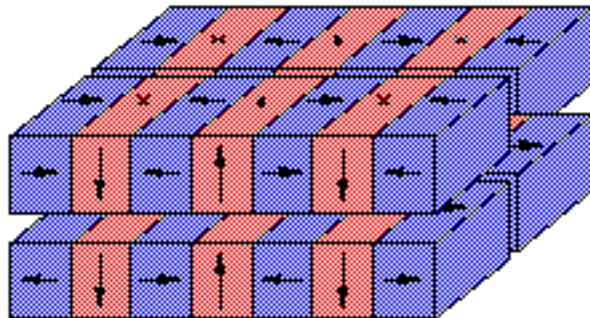
# Beamline Layout

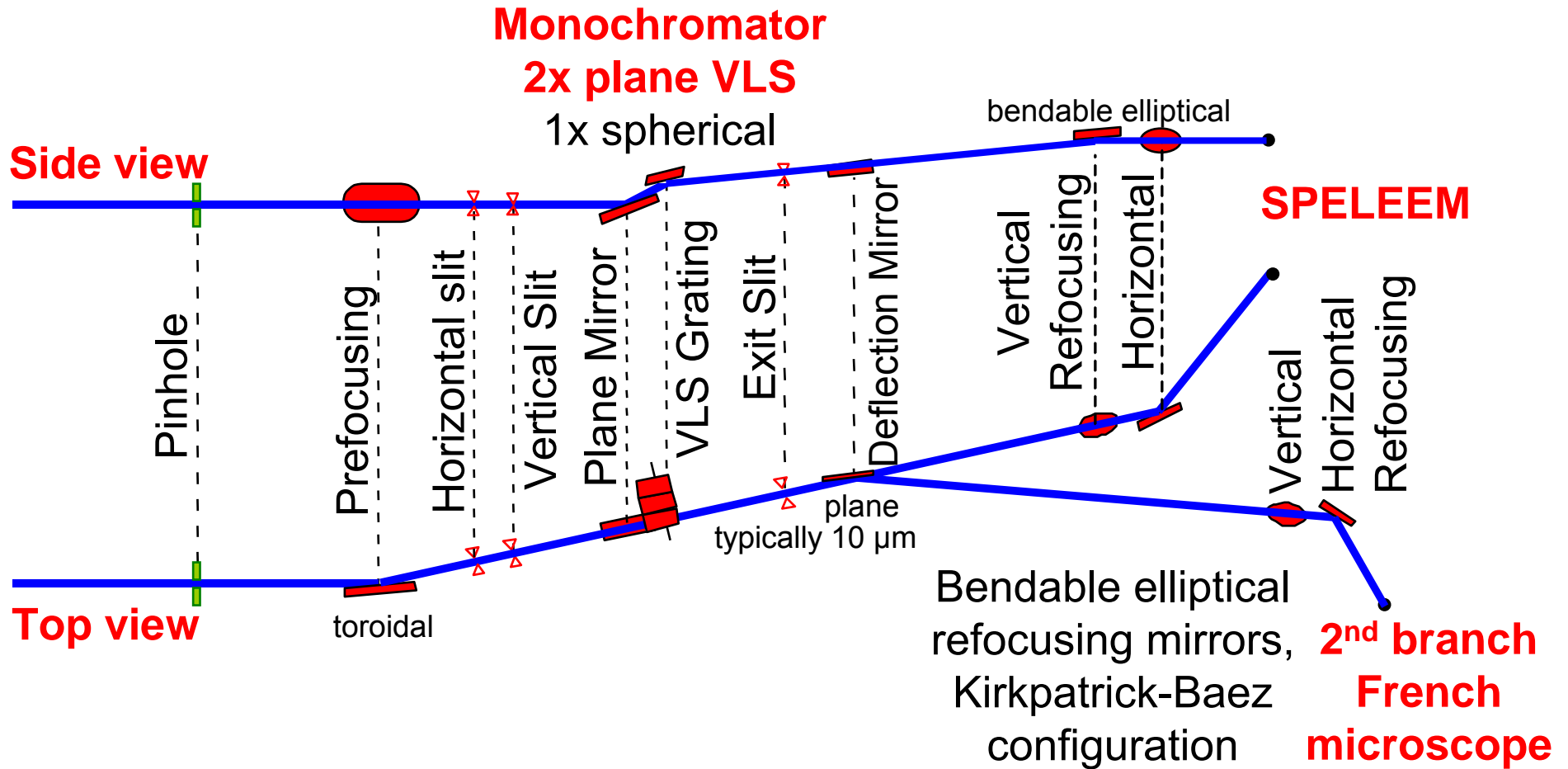




# Source Characteristics

- FEL/Nanospectroscopy undulator
- Sasaki Apple II type undulator
- 2 sections with phase modulation electromagnet
- 2 x 20 periods of length 10 cm
- Polarization: elliptical (horizontal, circular, and vertical)
- Source dim.: 560  $\mu\text{m}$  x 50  $\mu\text{m}$





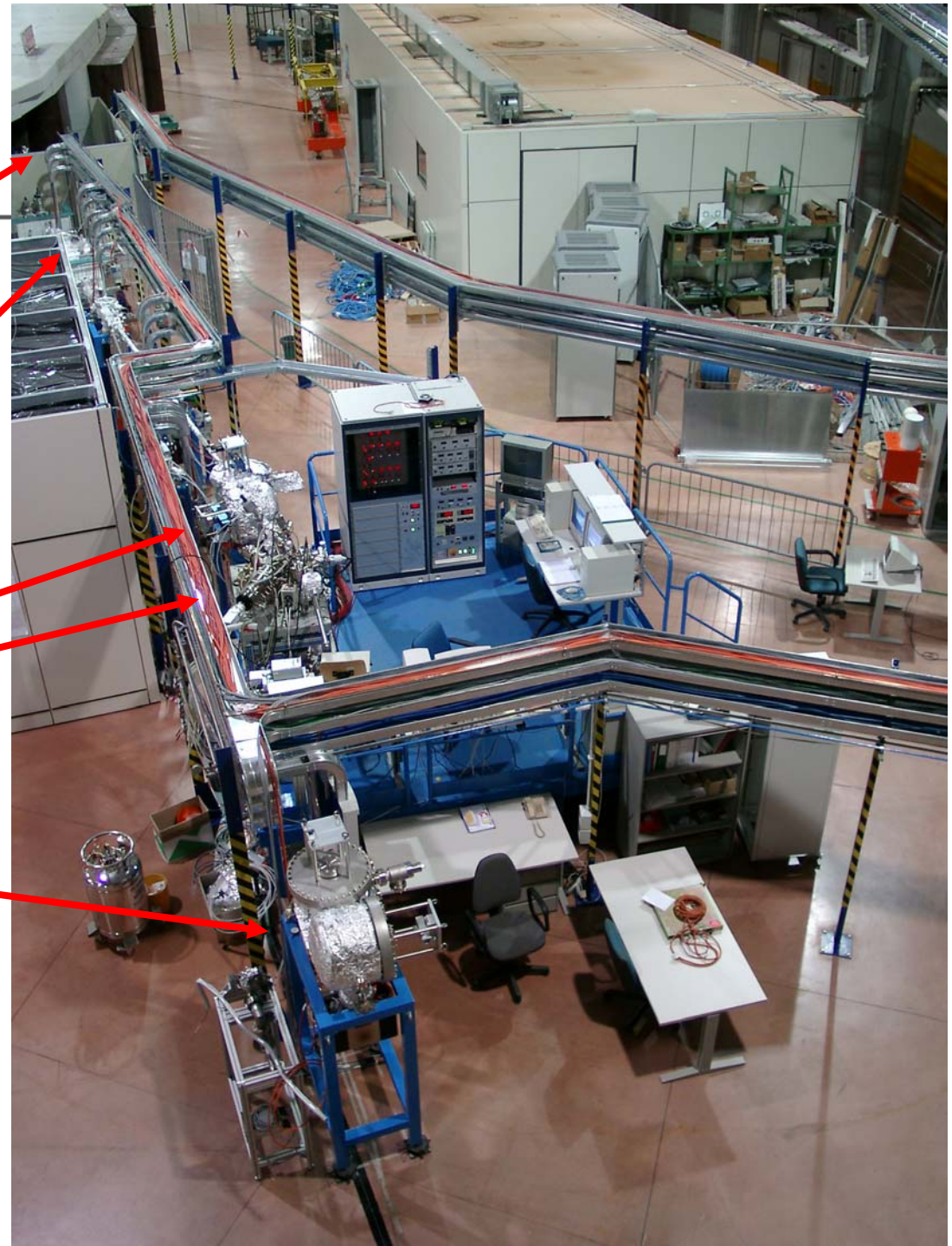
Front End

Monochromator

Refocusing Mirrors

SPELEEM

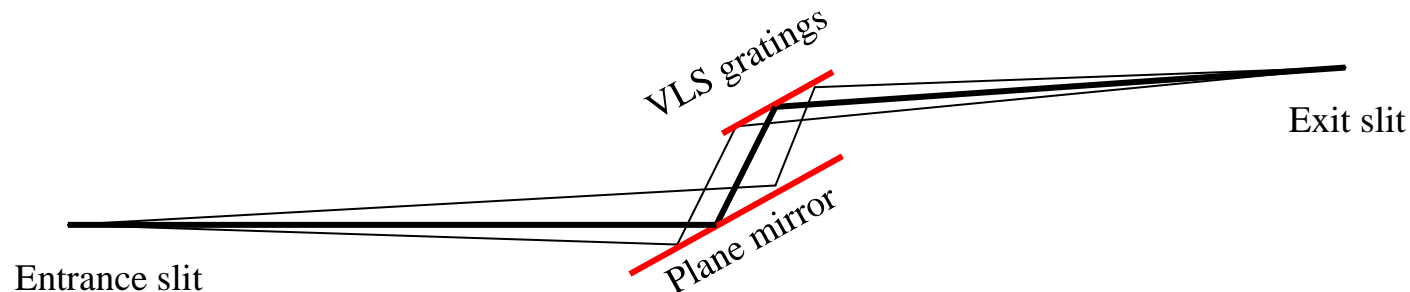
2nd Branch



# Monochromator

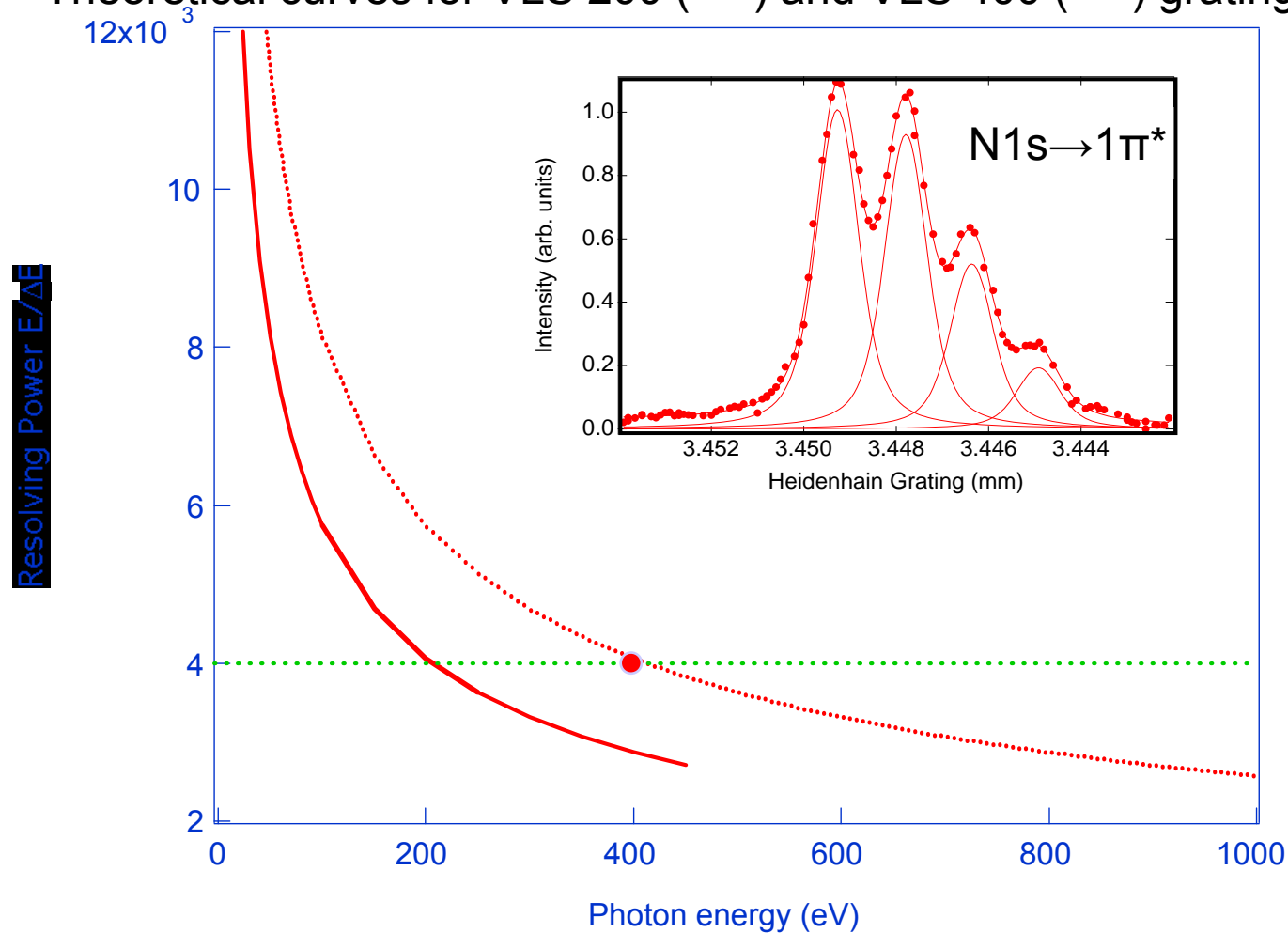
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- 2 VLS (variable line spacing) gratings of low groove density
  - 200/mm for 20 - 250 eV
  - 400/mm for 200 - 1000 eV
- 1 spherical grating (5 - 40 eV)

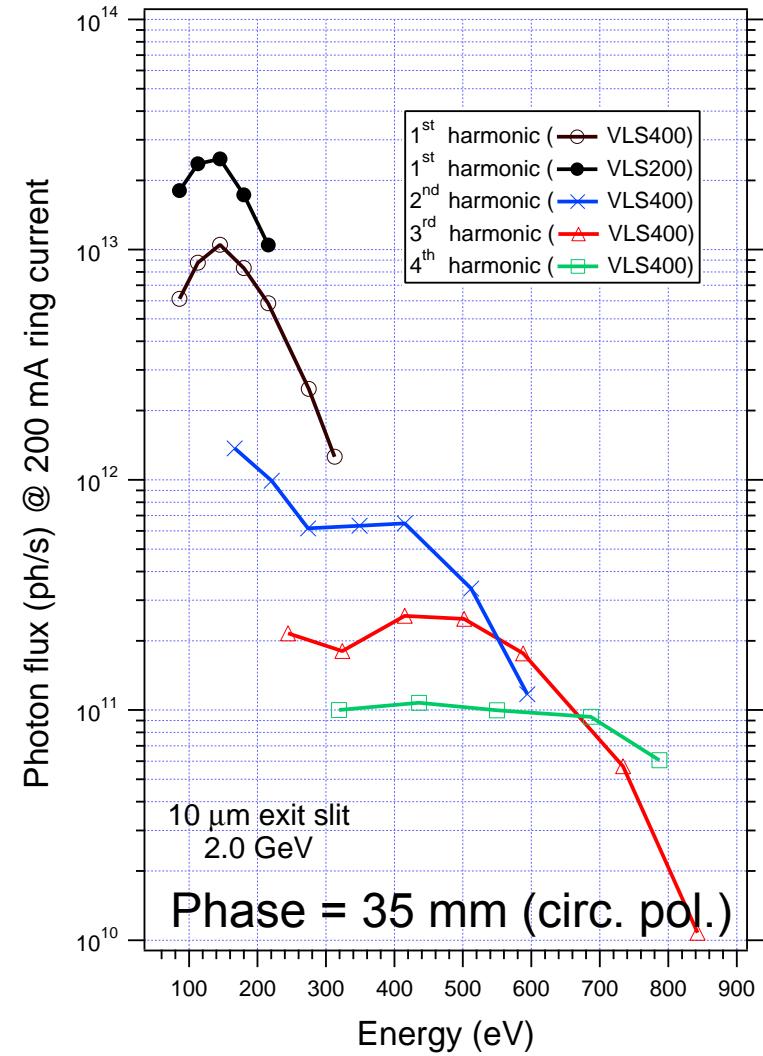
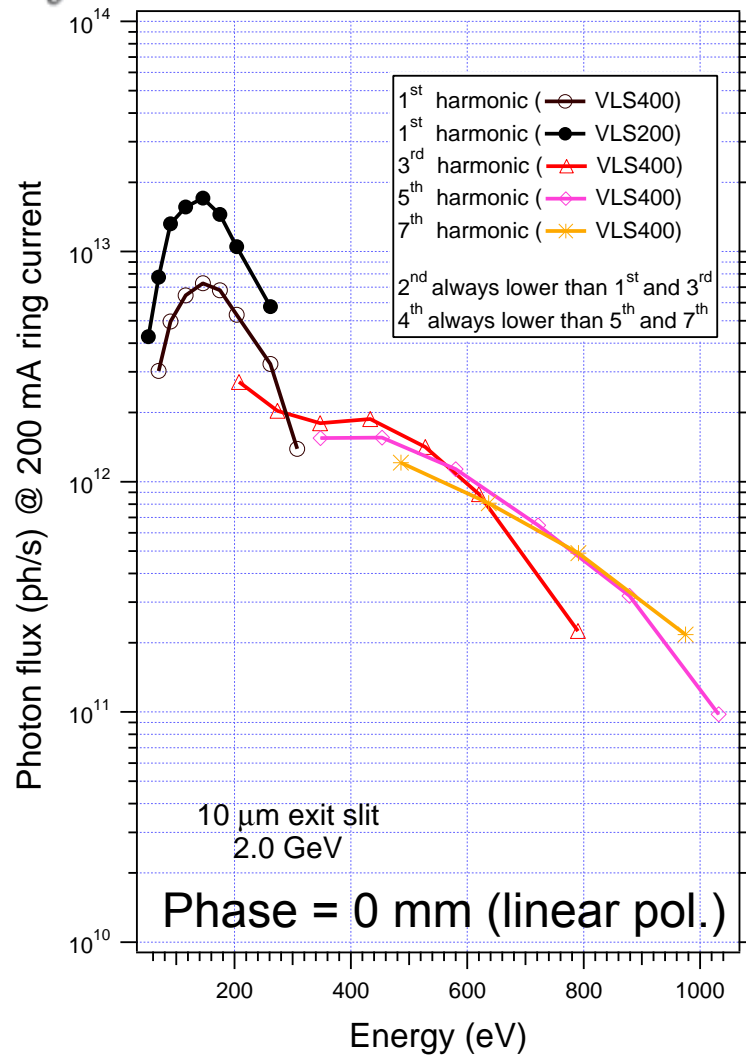


# Energy Resolution

Theoretical curves for VLS 200 (—) and VLS 400 (⋯) gratings



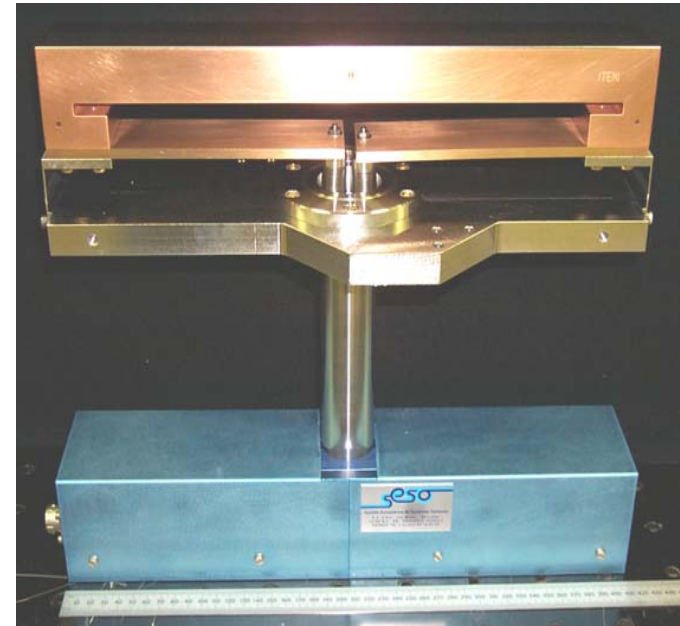
# Photon Flux



# Photon Beam Refocusing

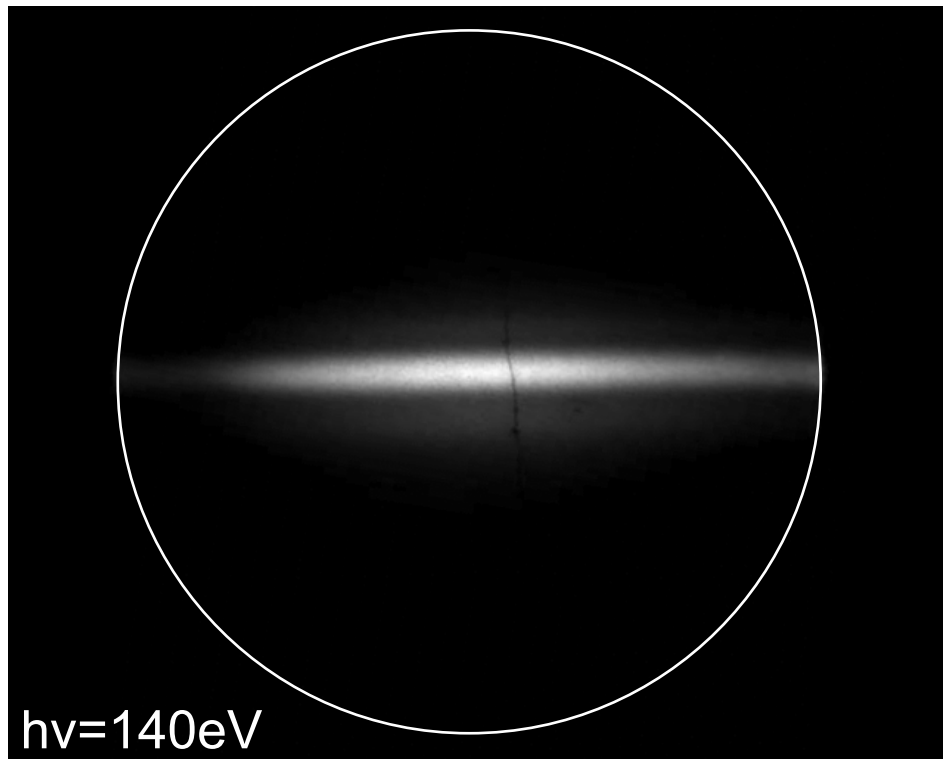
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- Need:
  - Homogeneous micro-spot
  - Highest photon flux in the field of view of the microscope
- Two adaptive plane elliptical mirrors («bendable mirrors»)
- Bend by applying unequal moments to their ends
- Kirkpatrick-Baez configuration
- Theoretical spot size:  
1.6  $\mu\text{m}$  (vert) x 6.1  $\mu\text{m}$  (hor)



# Best Focus: Spot Size on Sample

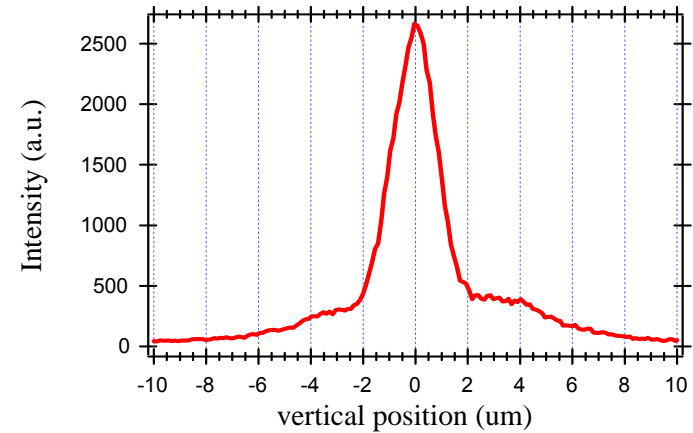
Best Focus



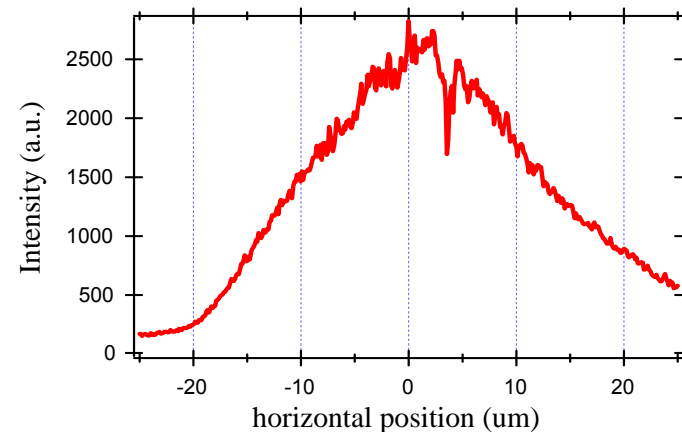
hv=140eV

Field of view: 55  $\mu\text{m}$

vertical line profile (FWHM 2  $\mu\text{m}$ )



horizontal line profile (FWHM 25  $\mu\text{m}$ )  
corrected for grazing incidence: 7  $\mu\text{m}$

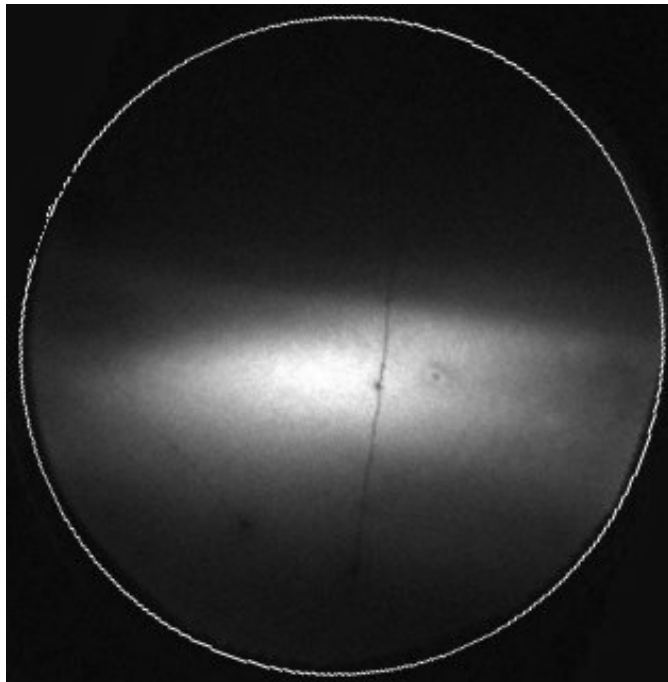




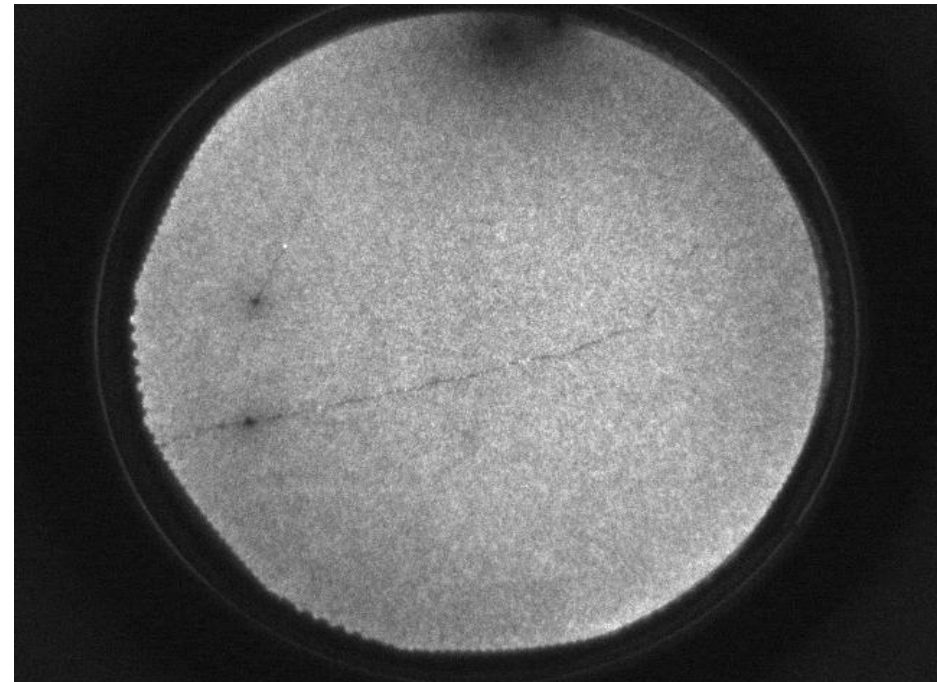
# Increased Spot Size

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Field of view  $\sim 50 \mu\text{m}$   
HRM roll misalignment (-700 steps)



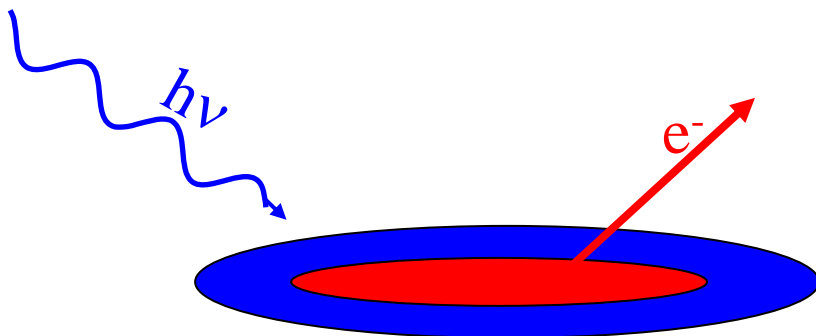
XPEEM image at  $5 \mu\text{m}$  FOV  
Homogeneous illumination



# Summary

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- Source:** Sasaki Apple II type undulator  
Polarization: circular, elliptical, and linear
- Monochromator:** Spectral range: 20 - 1000 eV  
Spectral resolution:  $E/\Delta E \sim 4000$  @ 400 eV
- Spot:** Flux on sample:  $10^{11} - 10^{13}$  ph/s/200mA  
Focused spot size:  $2 \mu\text{m} \times 7 \mu\text{m}$   
Vertical spot size from  $2 \mu\text{m}$  to  $10 \mu\text{m}$



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1. Spectromicroscopy at Elettra
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4. First Results: MnAs on GaAs

# Structural and Magnetic Phase Transition in MnAs/GaAs epitaxial Films Probed by LEEM and XMCD-PEEM

*eleonora*

S. Cherifi, S. Heun, A. Locatelli  
*Sincrotrone Trieste, Italy*

L. Däweritz, M. Kästner  
*Paul-Drude-Institut, Berlin, Germany*

E. Bauer  
*Arizona State University, Tempe, USA*



# Epitaxial MnAs on GaAs : Candidate for Spin Injection ?

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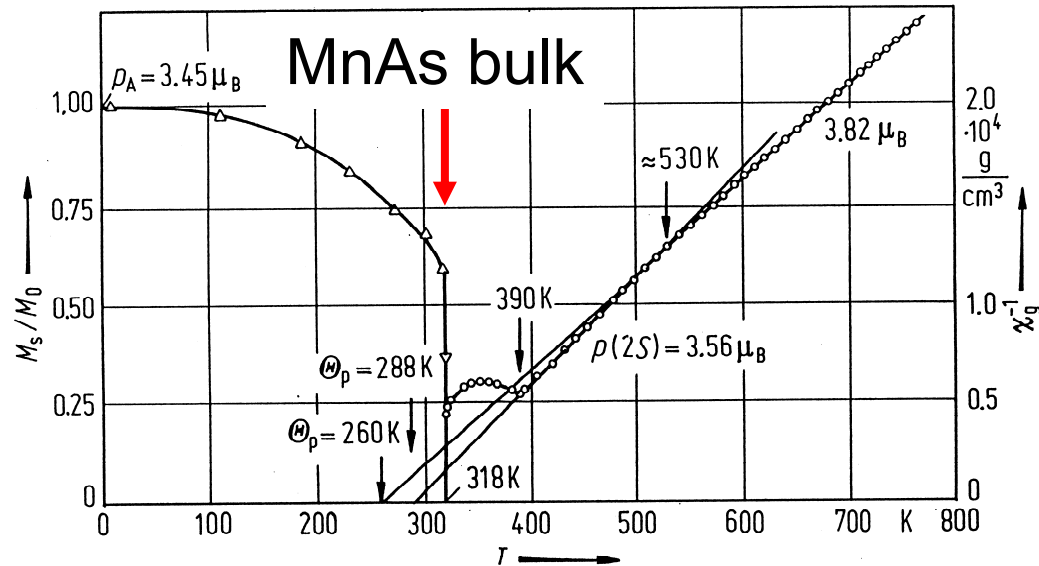
Ferromagnetic epitaxial MnAs films on GaAs:

- combination of ferromagnetic and conventional semiconductor
- spin injection

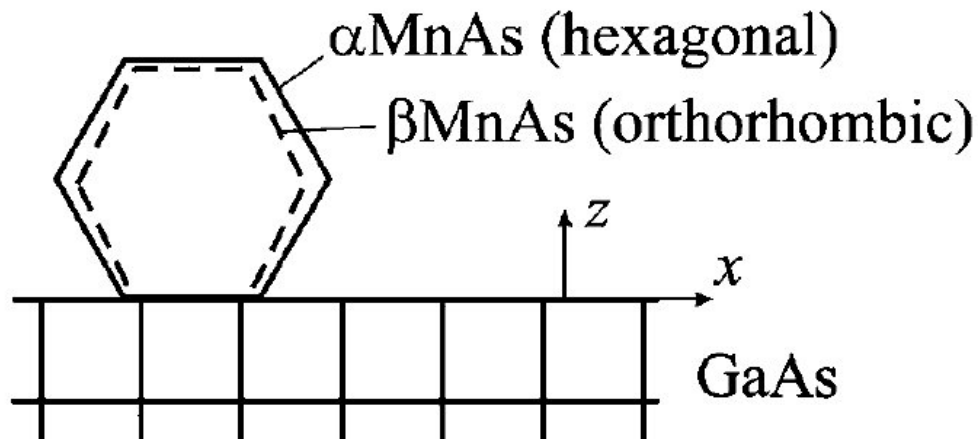
Here: XMCD-PEEM and LEEM study

Magnetic properties vs. structure

# Background



First order phase transition from the ferromagnetic hexagonal  $\alpha$  phase to the paramagnetic orthorhombic  $\beta$  phase



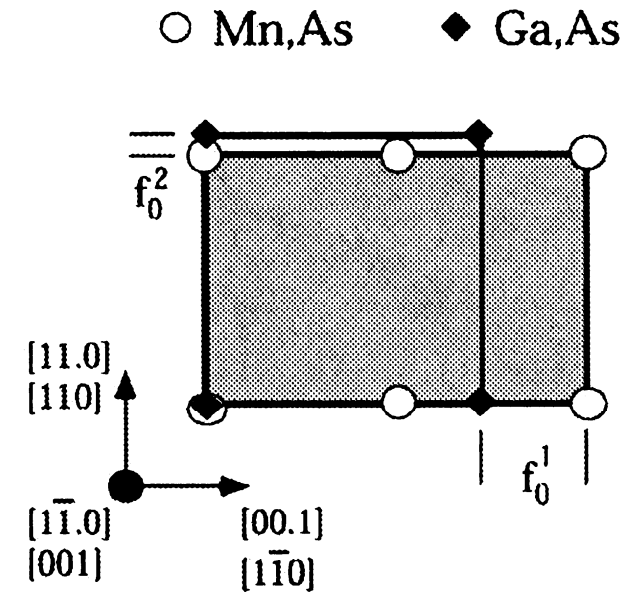
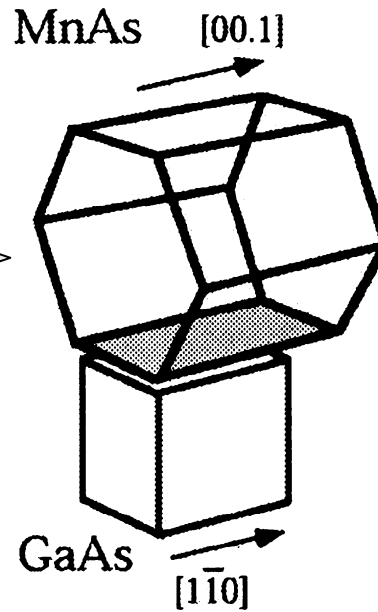
Large exchange splitting



High magnetic moment  
( $3.45 \mu_B$ )

# Background

**Hexagonal MnAs  
on cubic GaAs  
epitaxial films**

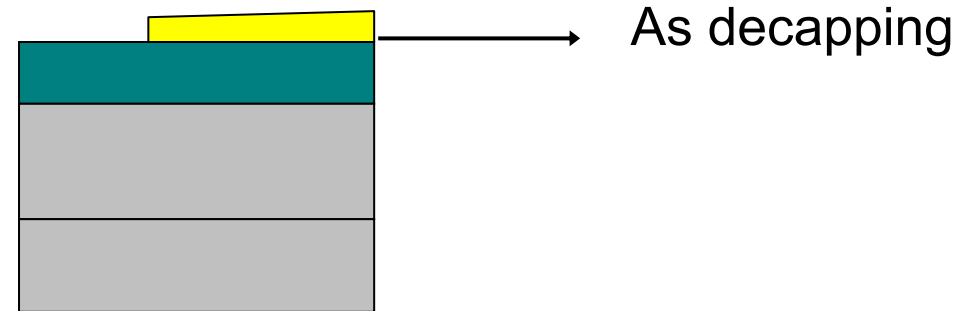
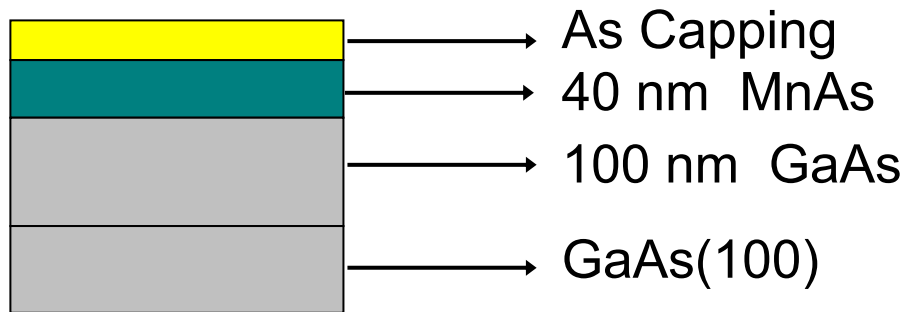


$\alpha$  MnAs(1-100) // GaAs(001)  
 $\alpha$  MnAs[0001] // GaAs[1-10]

**Large lattice misfit**  
 $\Downarrow$   
**Large strain**

# Samples

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MnAs films:

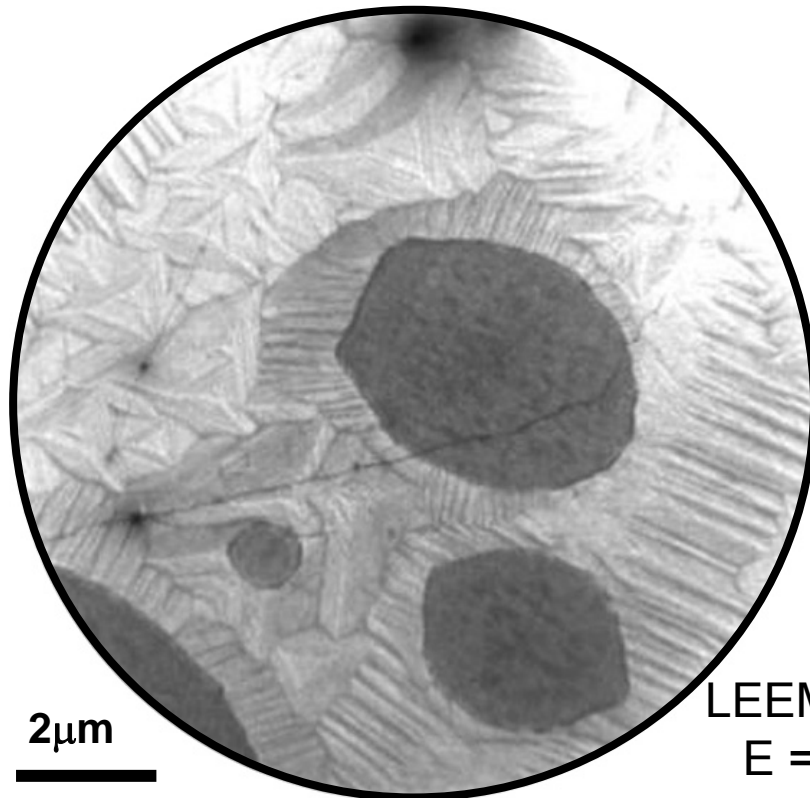
- grown at 250°C by MBE,
- annealed at 400°C, and
- capped with As before removal from the MBE chamber.

As-decapping at 320°C in the SPELEEM sample preparation chamber



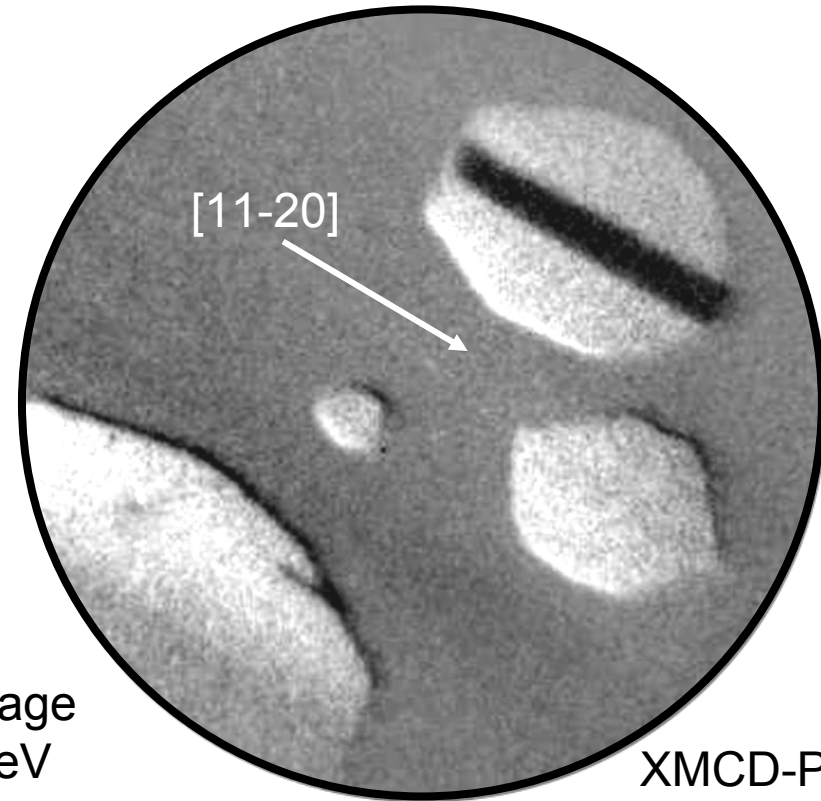
# Incompletely decapped MnAs Layer

$T < 0^\circ\text{C}$ : Ferromagnetic state



LEEM image  
 $E = 10\text{ eV}$

MnAs islands (dark) are surrounded by crystallized As



XMCD-PEEM

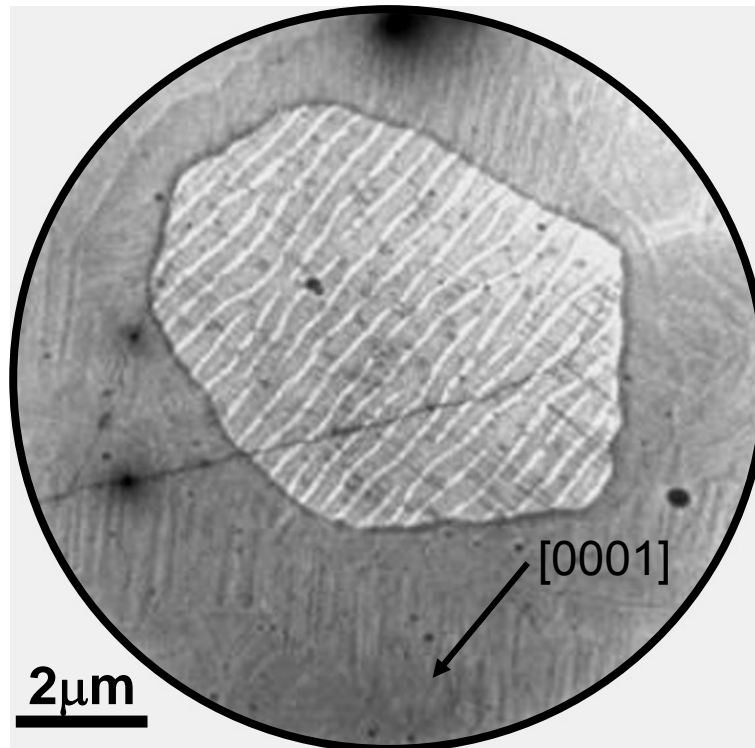
MnAs islands in a completely ferromagnetic state

# Incompletely decapped MnAs Layer

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T = Room temperature

Intermediate ferromagnetic-paramagnetic state



The misfit strain causes coexistence of two phases over a temperature range of about 30°C around room temperature.

LEEM image  
*electron energy = 4.5 eV*

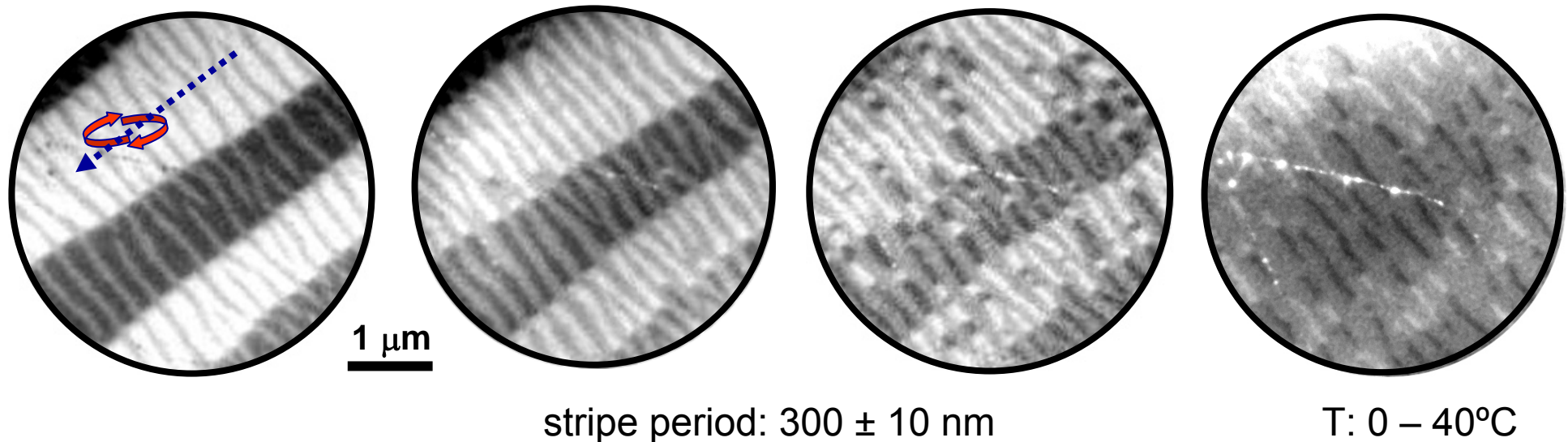
MnAs crystal showing a striped structure with alternating regions of the hexagonal  $\alpha$  phase and the orthorhombic  $\beta$  phase.

# Completely decapped MnAs Layer

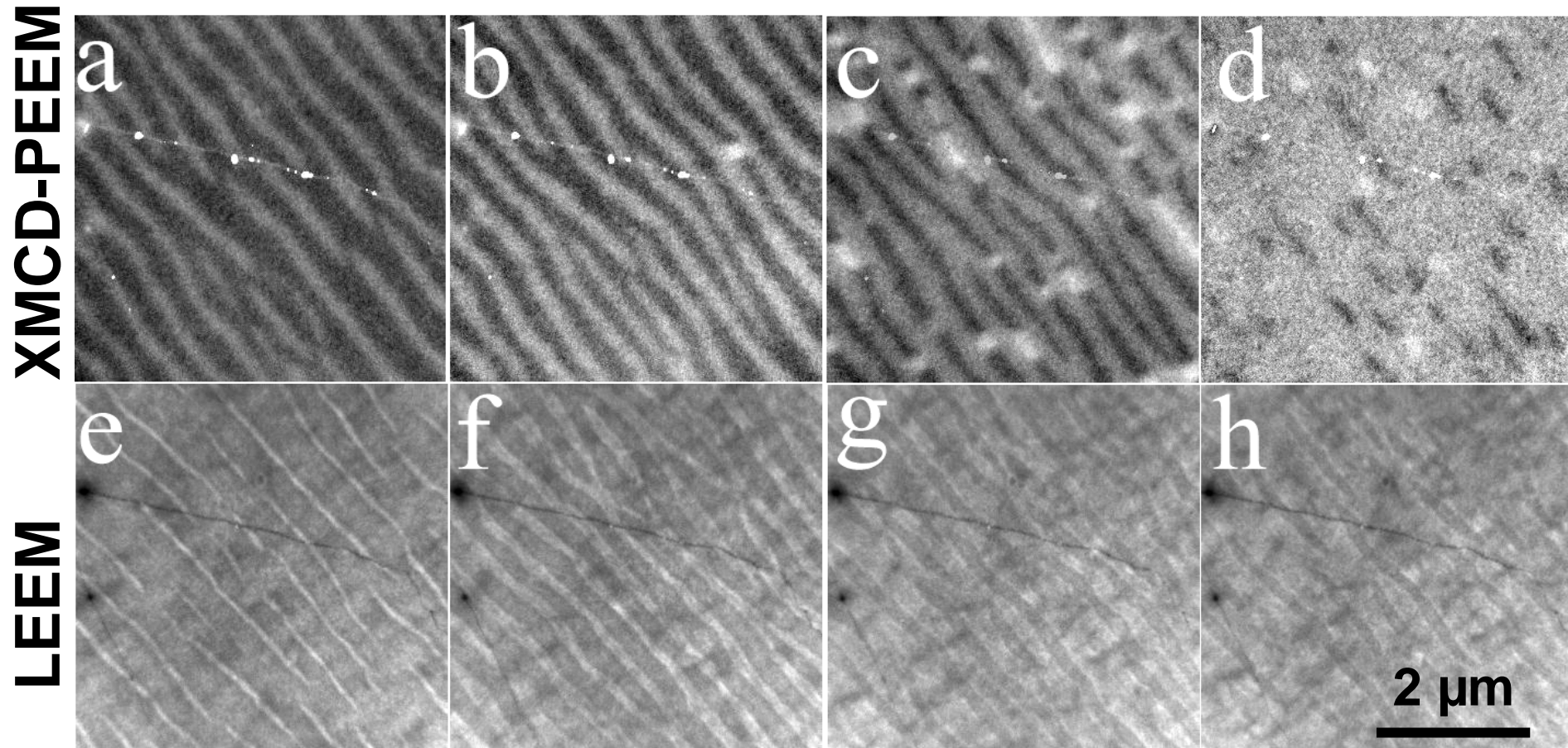
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Magnetic phase transition stage (multi-domain state)  
*XMCD-PEEM images at the Mn  $L_3$  edge*

MnAs[11-20] easy magnetization axis is in the plane of incidence of the photon beam for optimum contrast



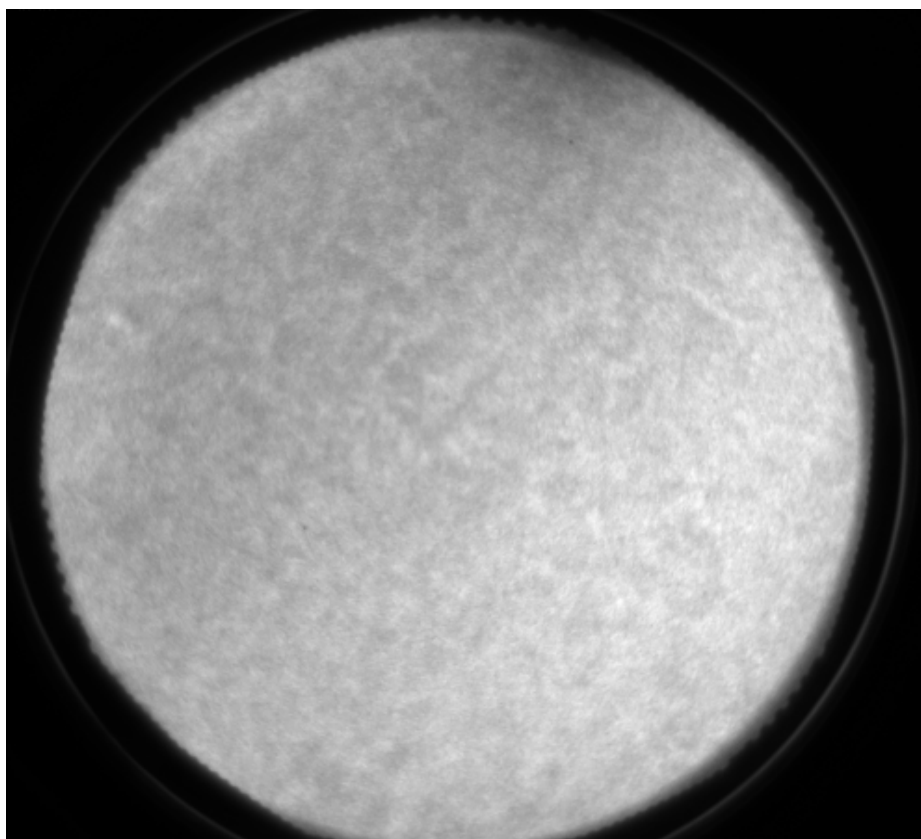
# Structural and Magnetic Properties



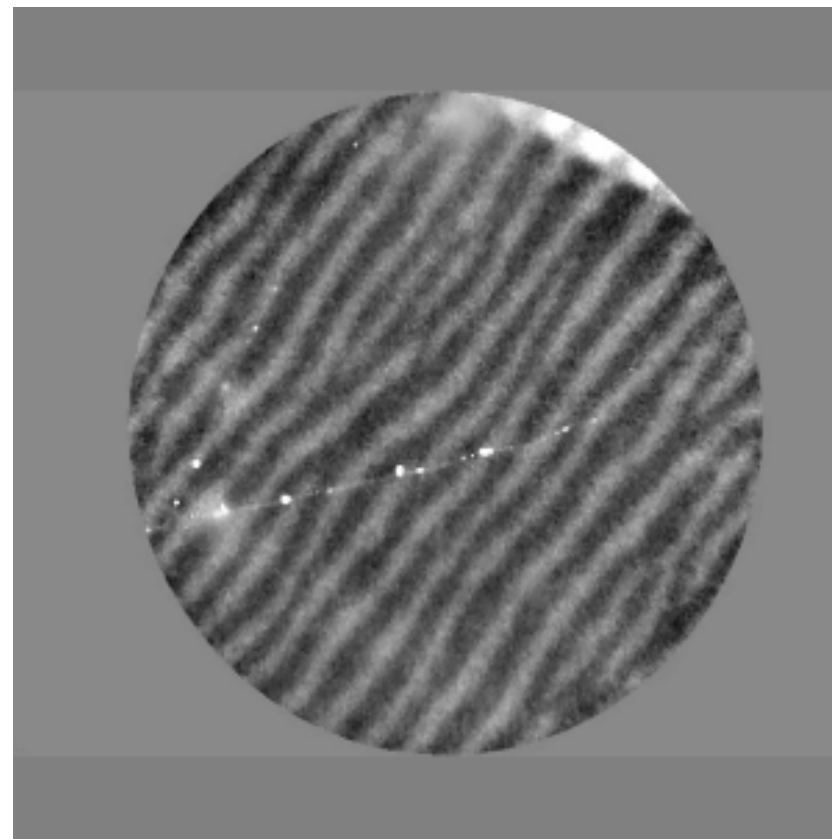
Magnetic and structural phase transition stages during heating

# Movies

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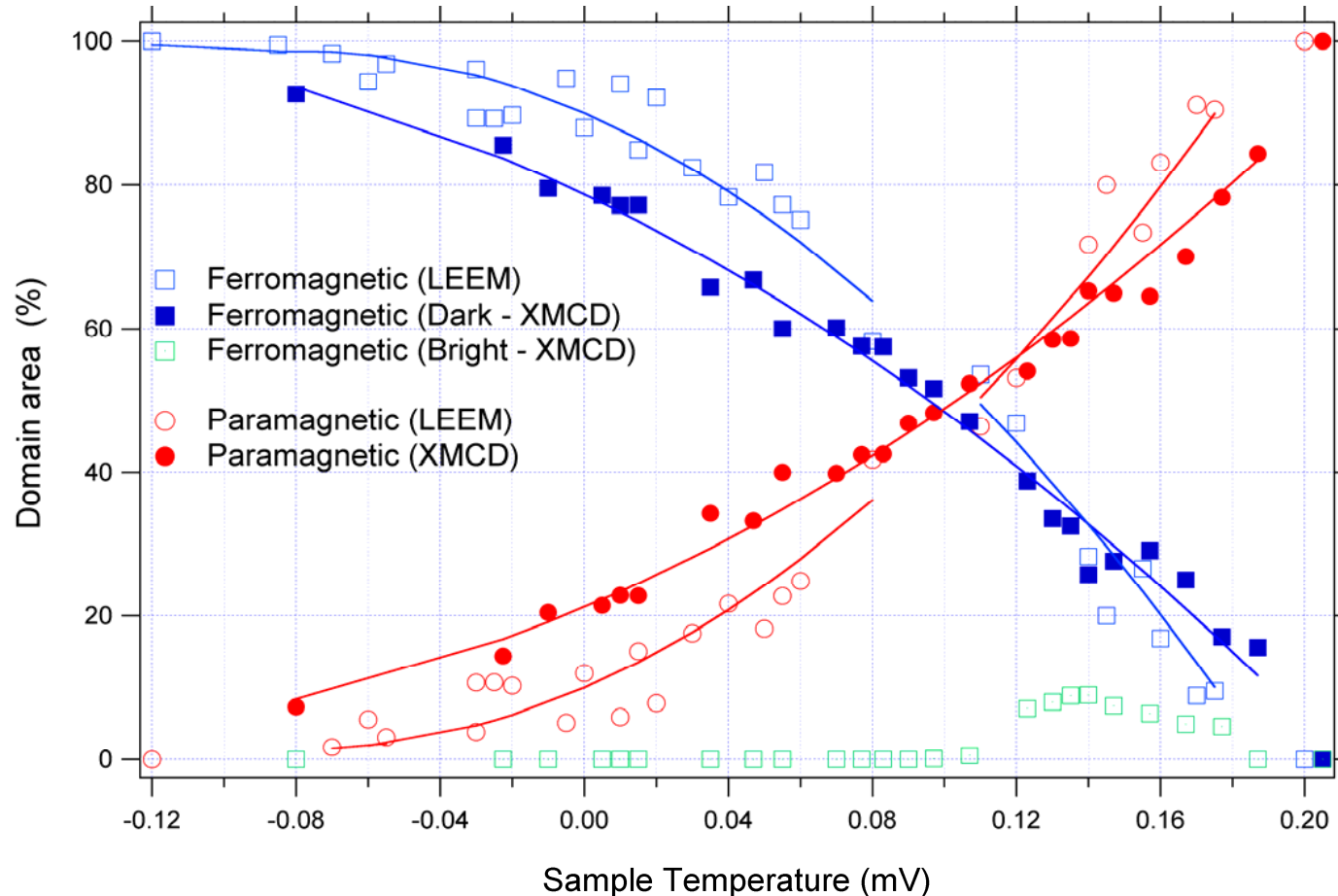


LEEM



XMCD-PEEM

# Area Fractions



Evolution of the area fractions of the magnetic phases (ferromagnetic and paramagnetic) and structural phases ( $\alpha$  and  $\beta$ ) derived from rsp. XMCD-PEEM and LEEM images taken during heating.

# Summary

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- The phase transition of epitaxial MnAs films on GaAs(100) from the ferromagnetic  $\alpha$  phase to the paramagnetic  $\beta$  phase occurs via phase separation into a ferromagnetic and paramagnetic striped phase without noticeable decrease of the magnetic moment.
- The stripe period (300 nm) is constant during this transition, causing a continuous decrease of the ferromagnetic stripe width.
- The stripes remain correlated until their distance exceeds a critical value at which they break up into small domains with opposite magnetization.