The Nanospectroscopy Beamline at ELETTRA

S. Heun
Sincrotrone Trieste
Coworkers

A. Locatelli, S. Cherifi, M. Marsi, and M. Pasqualetto
Nanospectroscopy Group, Sincrotrone Trieste

A. Bianco, G. Sostero, D. Cocco
X-ray Optics Group, Sincrotrone Trieste

E. Bauer
Arizona State University
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
1. Spectromicroscopy at Elettra

2. The SPELEEM microscope

3. The Nanospectroscopy Beamline

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

2. The SPELEEM microscope

3. The Nanospectroscopy Beamline

4. First Results: MnAs on GaAs
Location of Elettra
Elettra Beamlines

<table>
<thead>
<tr>
<th>exit</th>
<th>beaml ine</th>
<th>source</th>
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<tbody>
<tr>
<td>1.2L</td>
<td>Nanospectroscopy *</td>
<td>id</td>
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<tr>
<td>1.2R</td>
<td>FEL (Free-Electron Laser)</td>
<td>-</td>
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<td>2.2L</td>
<td>ESCA Microscopy</td>
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<tr>
<td>2.2R</td>
<td>SuperESCA</td>
<td>id</td>
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<td>Spectro Microscopy</td>
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<td>3.2R</td>
<td>VUV Photoemission</td>
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<td>4.2</td>
<td>Circularly Polarised Light</td>
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<td>SAXS (Small Angle X-Ray Scattering)</td>
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<td>5.2R</td>
<td>XRD1 (X-ray Diffraction)</td>
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<td>6.1L</td>
<td>Material science</td>
<td>bm</td>
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<td>6.1R</td>
<td>SYRMEP (SYnchrotron Radiation for MEdical Physics)</td>
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<td>7.1</td>
<td>MCX (Powder Diffraction Beamline)</td>
<td>bm</td>
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<td>7.2</td>
<td>ALOISA (Advanced Line for Overlayer, Interface and Surface Analysis)</td>
<td>id</td>
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<td>8.1L</td>
<td>BEAR (Bending magnet for Emission Absorption and Reflectivity) *</td>
<td>bm</td>
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<td>8.1R</td>
<td>LILIT (Lab of Interdisciplinary LITHography)</td>
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<td>8.2</td>
<td>BACH (Beamline for Advanced DiCHroism) *</td>
<td>id</td>
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<tr>
<td>9.1</td>
<td>IRSR (Infrared Synchrotron RadiatIon Microscopy)</td>
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<td>APE (Advanced Photoelectric-effect Experiments) **</td>
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<td>X-ray microfluorescence</td>
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<td>10.1R</td>
<td>DXRL (Deep-etch Lithography)</td>
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<td>10.2</td>
<td>IUVS (Inelastic Ultra Violet Scattering)</td>
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<td>11.1</td>
<td>XAFS (X-ray Absorption Fine Structure)</td>
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<td>11.2</td>
<td>XRD2 (X-ray Diffraction)</td>
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2.0 GeV / 320 mA
2.4 GeV / 140 mA
### Operating Beamlines

<table>
<thead>
<tr>
<th>Beamline</th>
<th>U2.2 EscaMicroscopy</th>
<th>U3.2 SpectroMicroscopy</th>
<th>U1.1 Nanospectroscopy</th>
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<tbody>
<tr>
<td><strong>E (eV):</strong></td>
<td>400-750</td>
<td>20-110</td>
<td>40-1000</td>
</tr>
<tr>
<td><strong>SR (nm):</strong></td>
<td>90</td>
<td>500</td>
<td>40 (20)</td>
</tr>
<tr>
<td><strong>SR (eV):</strong></td>
<td>0.25</td>
<td>0.07</td>
<td>0.4 (0.25)</td>
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<tr>
<td><strong>Flux (ph/s):</strong></td>
<td>$10^9-10^{10}$</td>
<td>$10^{11}-10^{13}$</td>
<td></td>
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<tr>
<td><strong>Methods:</strong></td>
<td>XPS(XAS)</td>
<td>XPS</td>
<td>XPS-XAS</td>
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<tr>
<td><strong>Polarization:</strong></td>
<td>Linear</td>
<td></td>
<td>Linear&amp;circular</td>
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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.

Electron optics to magnify irradiated area: 
**Imaging Instrument**
1. High lateral resolution (20 nm).
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⁻ out
- + energy filtering of electrons

**XAS – mode: hv scanned**
- hv in / e⁻ 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

Magnetic contrast

I^- max - I^+ max

2µm
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The SPELEEM at ELETTRA
The SPELEEM

Spectroscopic photoemission and low energy electron microscope
Images from a Field Effect Transistor (FET) at different binding energies. Photon energy $hv = 131.3$ eV.

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

Lateral Resolution of LEEM

FoV = 2.65 µm
STV = 7.5 eV
12.5 µm energy slit
30 µm 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

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

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.

FoV = 2.65 µm
STV = 43.2 eV, hv = 144.0 eV
12.5 µm energy slit
30 µm contrast aperture
240 s int. time, 2x2 binning
Pb on Si (111)
XPEEM – energy resolution
Pb 5d – Voigt fit
13/11/2002 scan_002

FoV = 2.65 µm
hv = 130.0 eV
12.5 µm energy slit
30 µm contrast aperture
30 s int. time, 4x4 binning

Energy resolution better than 0.45 eV.
Outline

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Requirements

Source: Variable Polarization

Monochromator: Wide spectral range
Medium spectral resolution

Spot: High photon flux density on sample
Small variable spot size (~µm)
Homogeneous illumination
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 μm x 50 μm

Undulator characteristics
Optics

Front End
Monochromator
Refocusing Mirrors
SPELEEM
2nd Branch
Monochromator

- 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

Resolving Power $E/\Delta E \sim 4000$

@ 400.8 eV

Photon Flux

Photon Beam Refocusing

- 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:
  - $1.6 \, \mu m$ (vert) x $6.1 \, \mu m$ (hor)

Best Focus: Spot Size on Sample

Best Focus

Field of view: 55 µm

hv=140eV

vertical line profile (FWHM 2 µm)

horizontal line profile (FWHM 25 µm) corrected for grazing incidence: 7 µm

Increased Spot Size

Field of view $\sim$50 µm
HRM roll misalignment (-700 steps)

XPEEM image at 5µm FOV
Homogeneous illumination
**Summary**

**Source:** Sasaki Apple II type undulator  
Polarization: circular, elliptical, and linear

**Monochromator:** Spectral range: 20 - 1000 eV  
Spectral resolution: $E/\Delta E \sim 4000 \, @ \, 400 \, \text{eV}$

**Spot:** Flux on sample: $10^{11} - 10^{13}$ ph/s/200mA  
Focused spot size: 2 µm x 7 µm  
Vertical spot size from 2 µm to 10 µm
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Structural and Magnetic Phase Transition in MnAs/GaAs epitaxial Films Probed by LEEM and XMCD-PEEM

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?

Ferromagnetic epitaxial MnAs films on GaAs:
• combination of ferromagnetic and conventional semiconductor
• spin injection

Here: XMCD-PEEM and LEEM study
Magnetic properties vs. structure
First order phase transition from the ferromagnetic hexagonal $\alpha$ phase to the paramagnetic orthorhombic $\beta$ phase.

Large exchange splitting

High magnetic moment

(3.45 $\mu_B$)

Hexagonal MnAs on cubic GaAs epitaxial films

\[ \alpha \text{ MnAs}(1-100) \parallel \text{ GaAs}(001) \]
\[ \alpha \text{ MnAs}[0001] \parallel \text{ GaAs}[1-10] \]

Large lattice misfit
\[ \Downarrow \]
Large strain

Samples

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°C: Ferromagnetic state

MnAs islands (dark) are surrounded by crystallized As

MnAs islands in a completely ferromagnetic state

Incompletely decapped MnAs Layer

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

Magnetic phase transition stage (multi-domain state)

**XMCD-PEEM images at the Mn L₃ edge**

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

stripe period: 300 ± 10 nm

T: 0 – 40°C

Structural and Magnetic Properties

Magnetic and structural phase transition stages during heating

Evolution of the area fractions of the magnetic phases (ferromagnetic and paramagnetic) and structural phases (α and β) derived from esp. XMCD-PEEM and LEEM images taken during heating.
Summary

• 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.