Desorption dynamics of **L**ocal **A**nodic **O**xidation-oxide nanostructures

S. HEUN,[1,2] G. MORI,[1,3] D. ERCOLANI,[1,4] M. LAZZARINO,[1] A. LOCATELLI,[2] and L. SORBA[1,4]

[1] Laboratorio Nazionale TASC-INFM, I-34012 Trieste, Italy

[2] Sincrotrone Trieste S.c.p.a., I-34012 Trieste, Italy

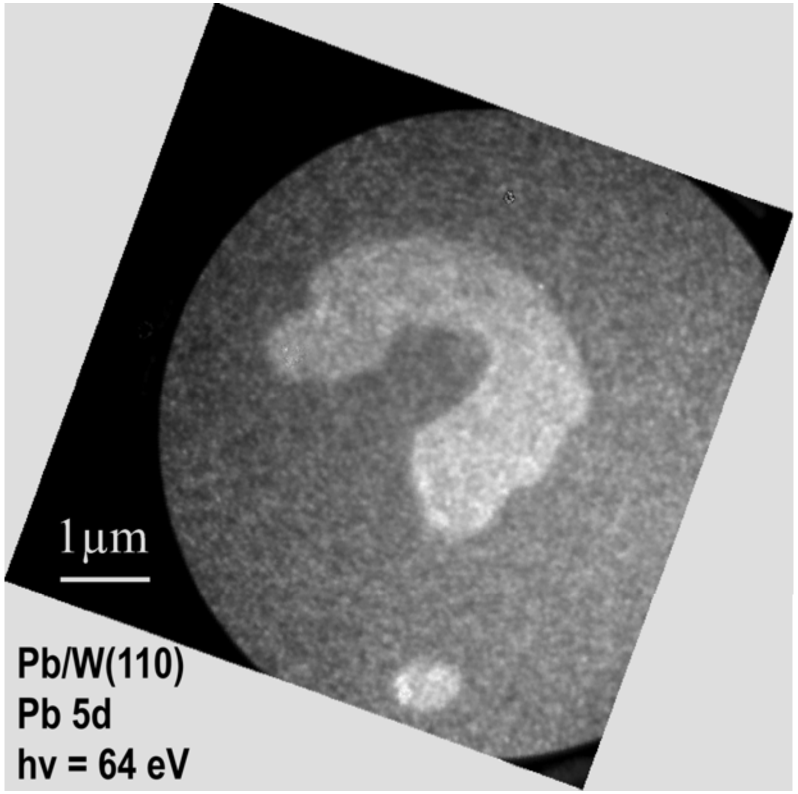
[3] Università degli Studi di Trieste, I-34100 Trieste, Italy

[4] Università degli Studi di Modena e Reggio Emilia, I-41100 Modena, Italy

Outline

- A brief introduction to spectromicroscopy
- The SPELEEM at Elettra
  - SPELEEM = spectroscopic photoemission and low energy electron microscope
- Application example:
  - Local Anodic Oxidation (LAO) by AFM: GaAs oxides

Motivation



Why XPS?

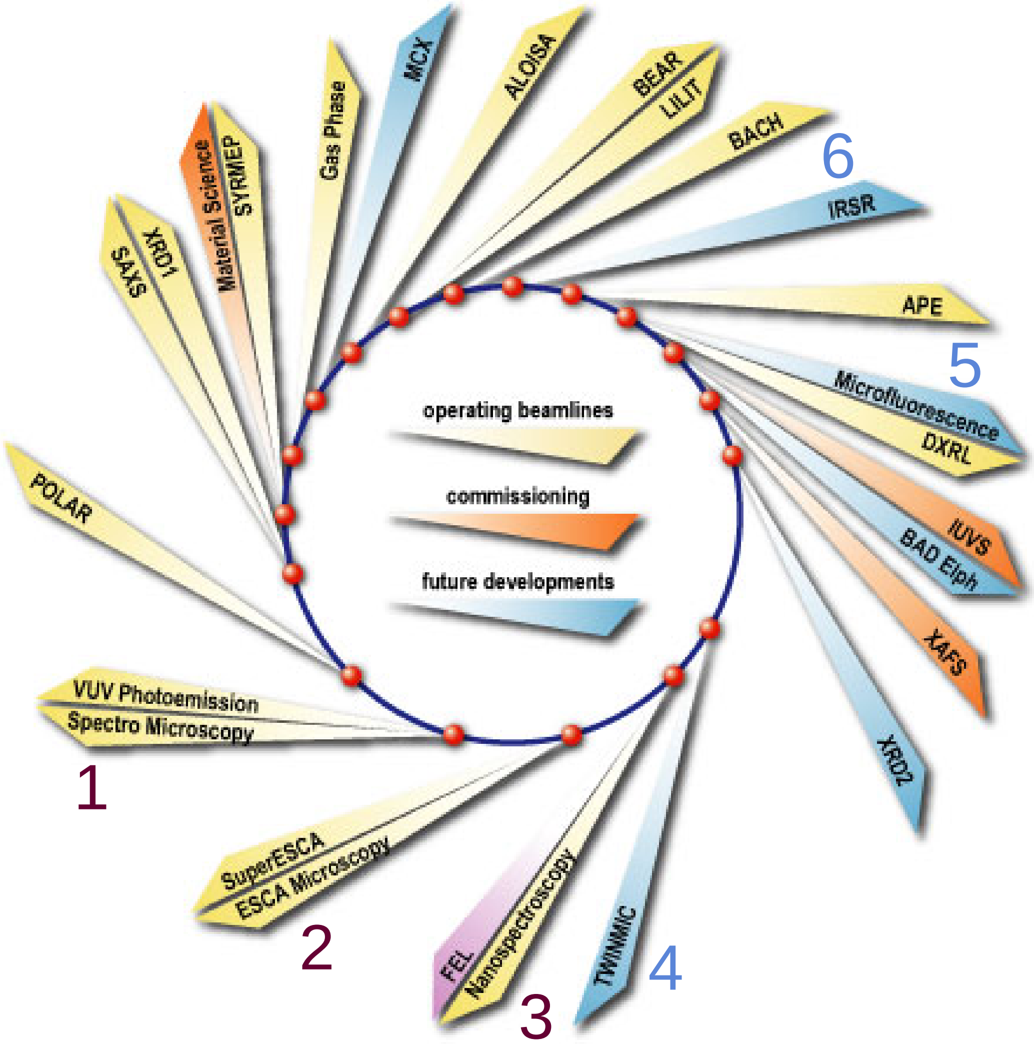
- chemical state information
- surface sensitive
- ease of quantification
- (in general) nondestructive

Why spectromicroscopy ?

- (semicond.) nanostructures: self-organization, lithography
- devices
- diffusion, segregation
- alloying (silicide formation)
- catalysis, chemical waves
- surface magnetism (XMCD)

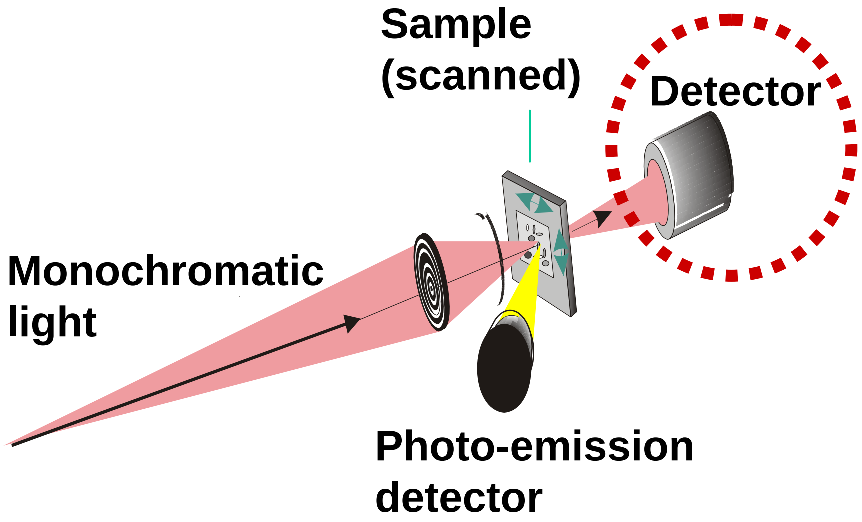
Location of TASC and Elettra

Elettra Beamlines



exit	beamline	source
1.1L	TWINMIC	short id
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
10.2L	IUVS (Inelastic Ultra Violet Scattering)	id
10.2R	BAD Elph	id
11.1	XAFS (X-ray Absorption Fine Structure)	bm
11.2	XRD2 (X-ray Diffraction)	id

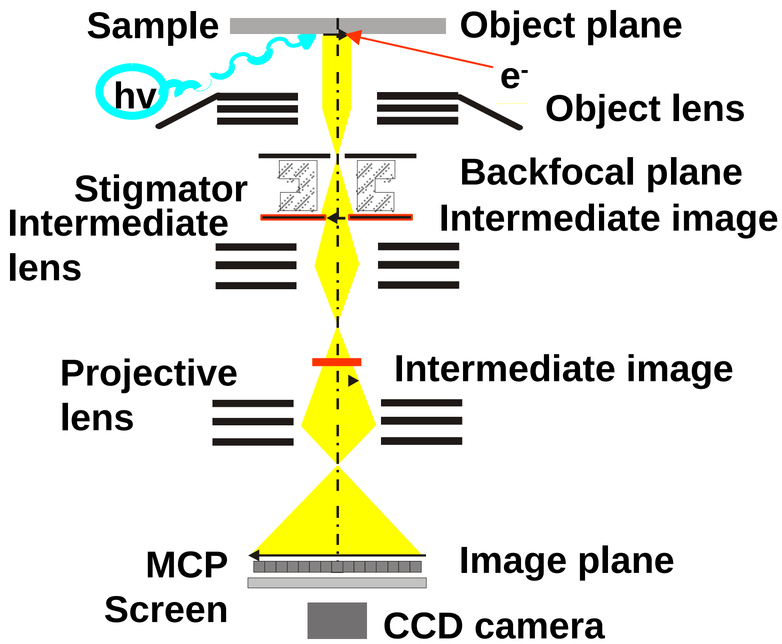
Scanning vs. direct imaging type



Photon optics is demagnifying the beam:

Scanning Instrument

- Whole power of XPS in a small spot mode
- Flexibility for adding different detectors
- Rough surfaces can be measured
- Limited use for fast dynamic processes
- Lower lateral resolution than imaging instruments



Electron optics to magnify irradiated area:

Imaging Instrument

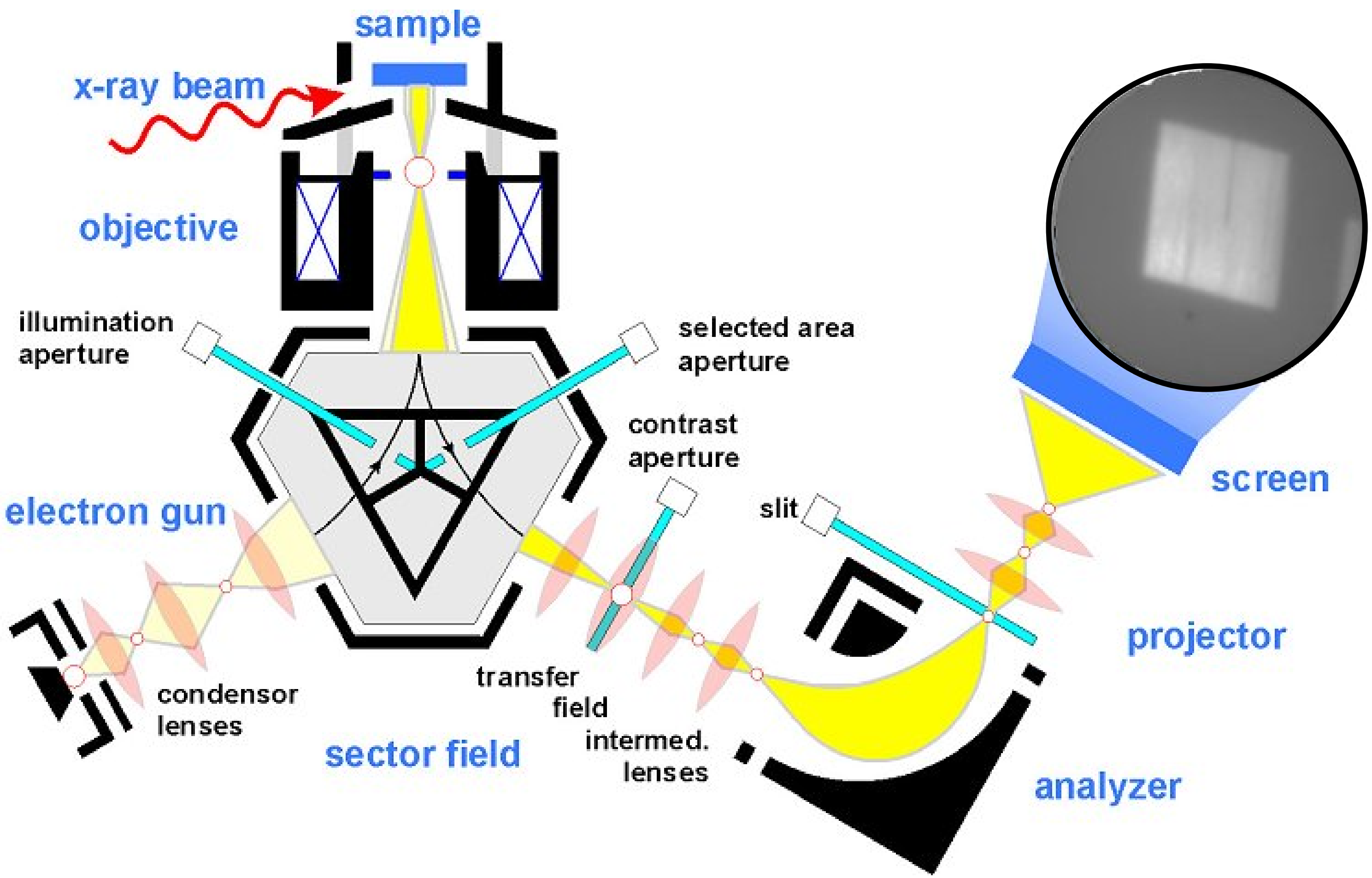
- High lateral resolution (20 nm)
- Multi-method instrument (XPEEM/PED)
- Excellent for monitoring dynamic processes
- Poorer spectroscopic ability
- Sensitive to rough surfaces

Outline

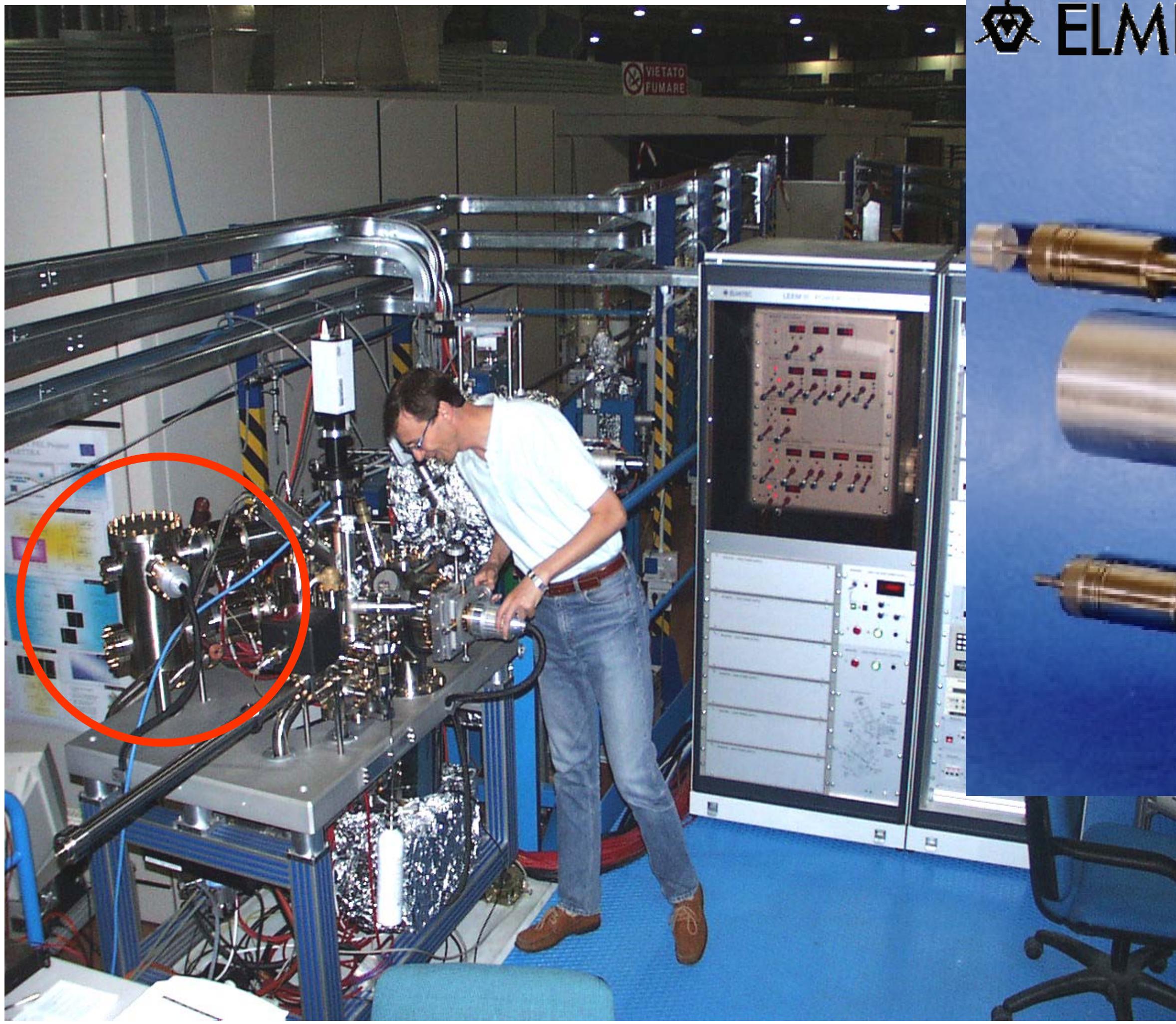
- A brief introduction to spectromicroscopy
- The SPELEEM at Elettra
  - SPELEEM = spectroscopic photoemission and low energy electron microscope
- Application example:
  - Local Anodic Oxidation (LAO) by AFM: GaAs oxides

The SPELEEM at Elettra

Spectroscopic photoemission and low energy electron microscope

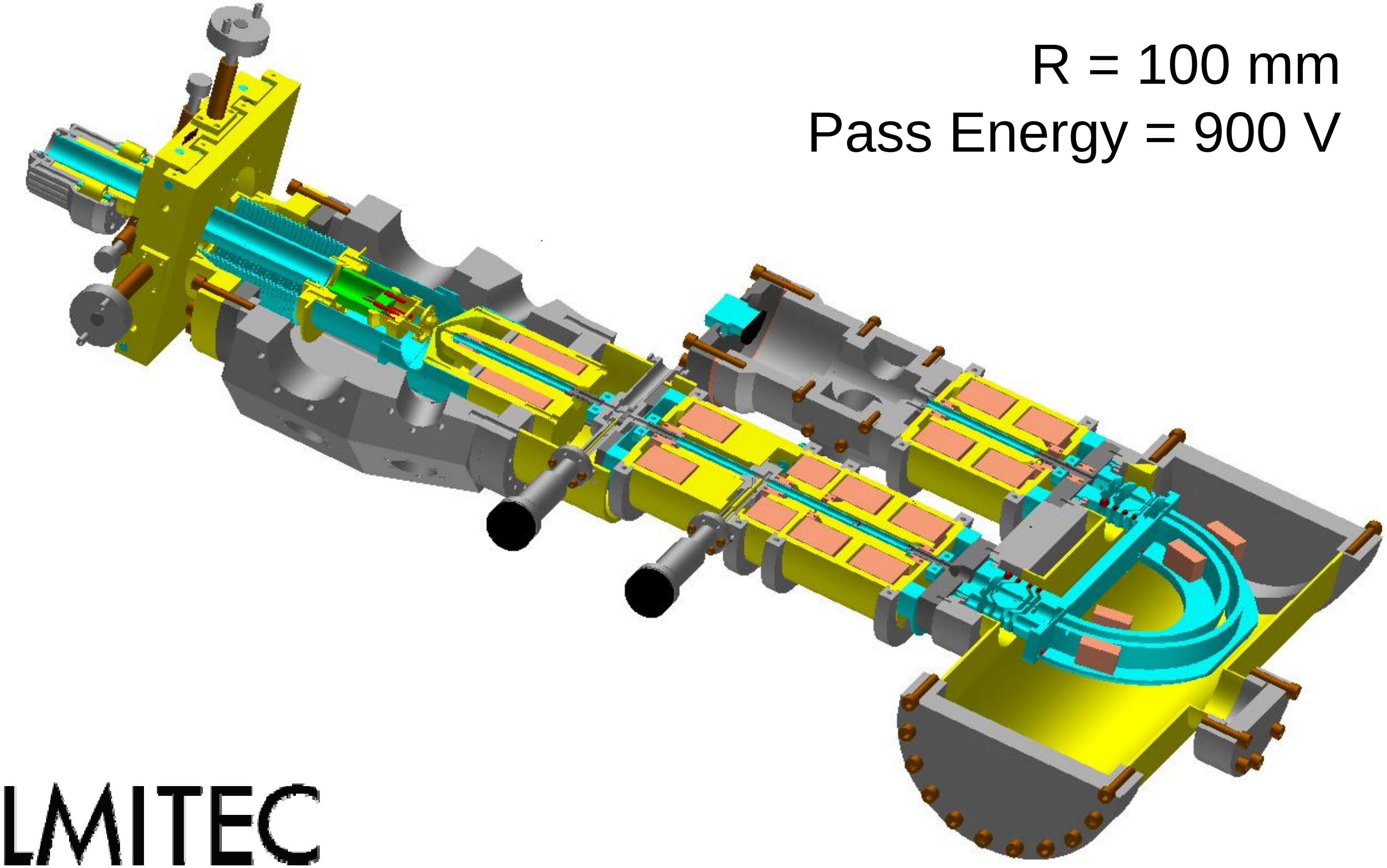


The SPELEEM at ELETTRA



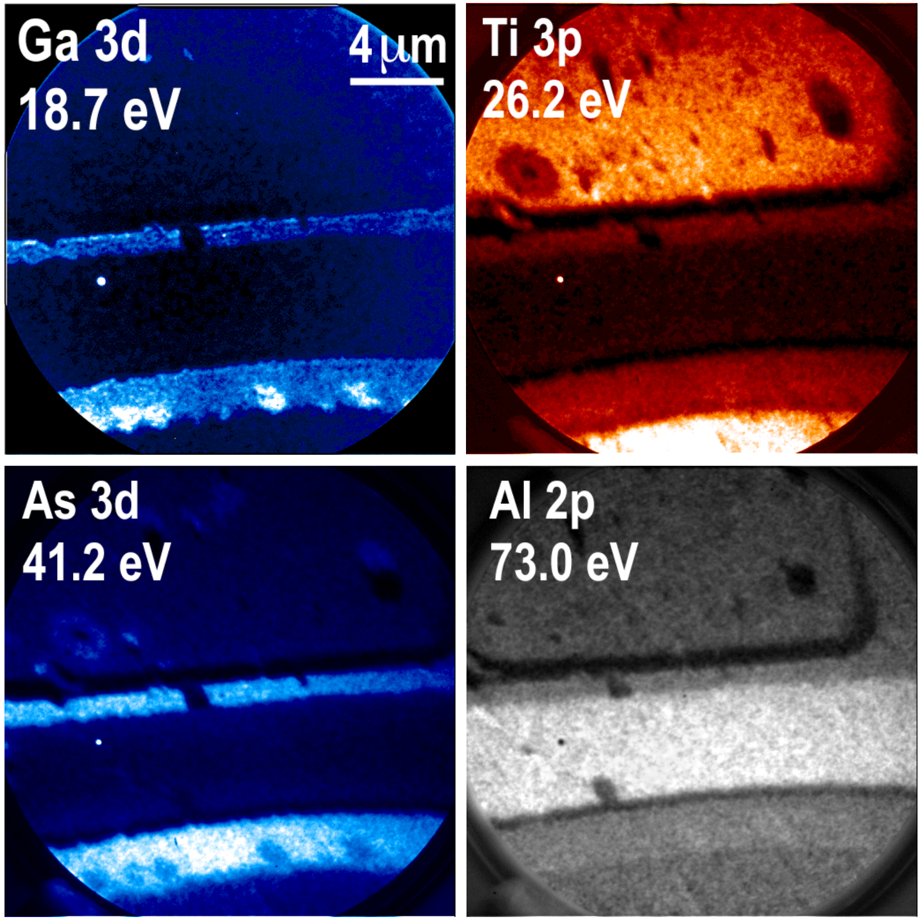
The energy filter

R = 100 mm
Pass Energy = 900 V

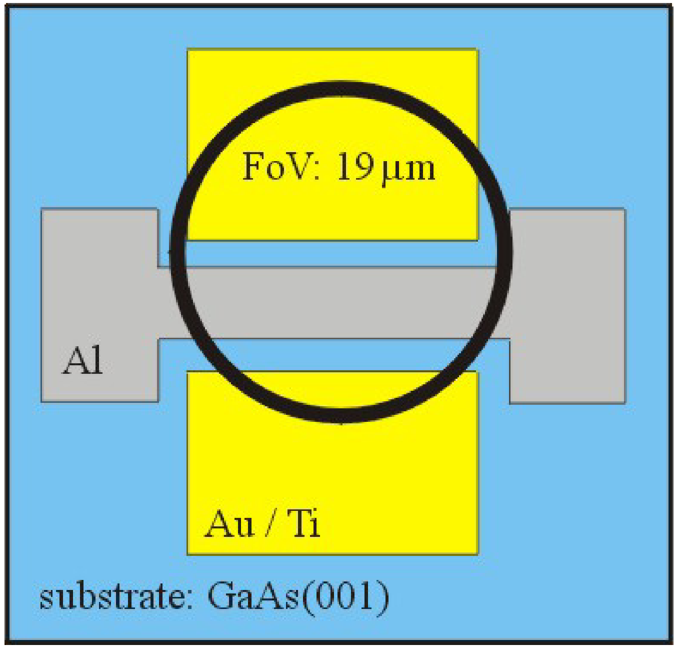


ELMITEC

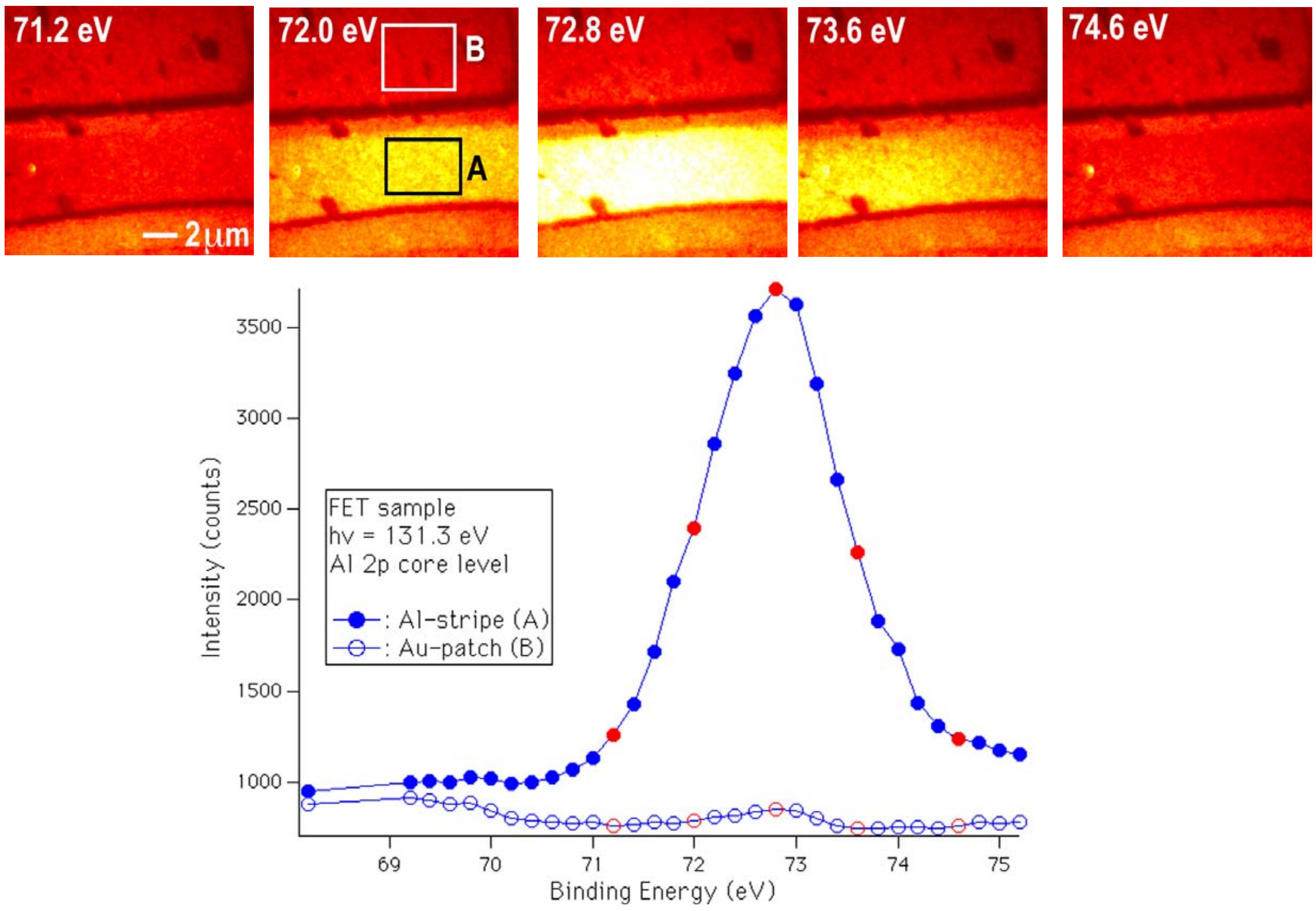
XPEEM: Spectroscopic Microscopy



- Images from a Field Effect Transistor (FET) at different binding energies.
- Photon energy 131.3 eV.

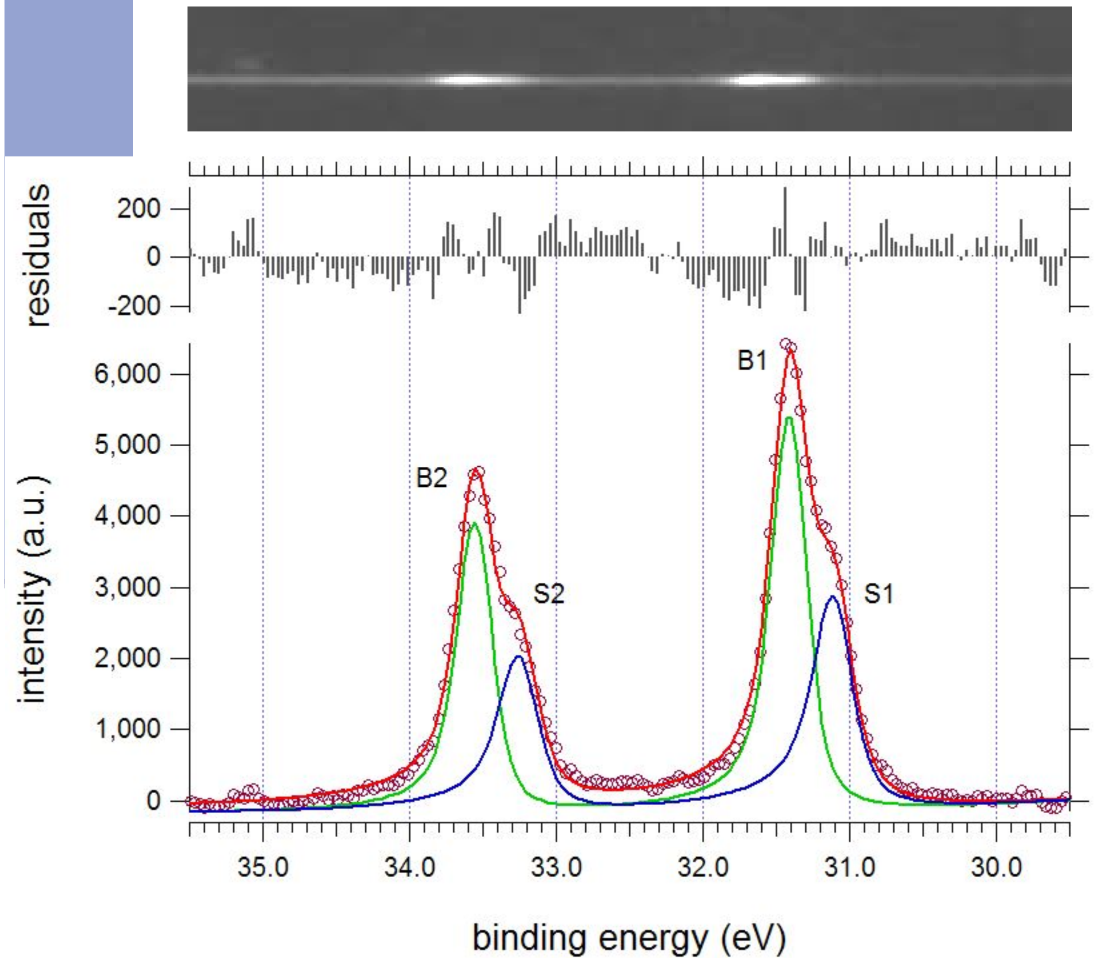


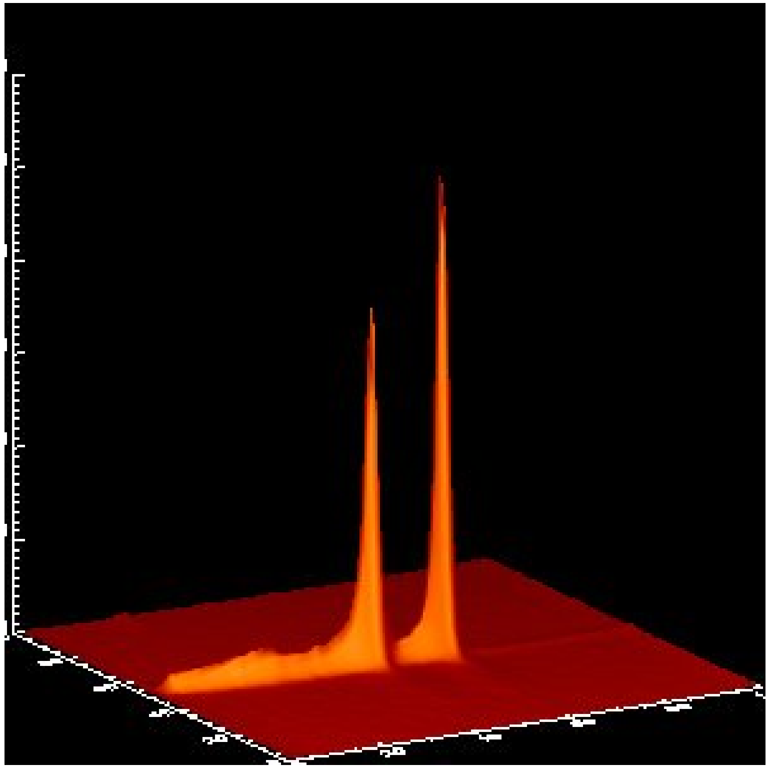
XPEEM: Core Level Spectroscopy



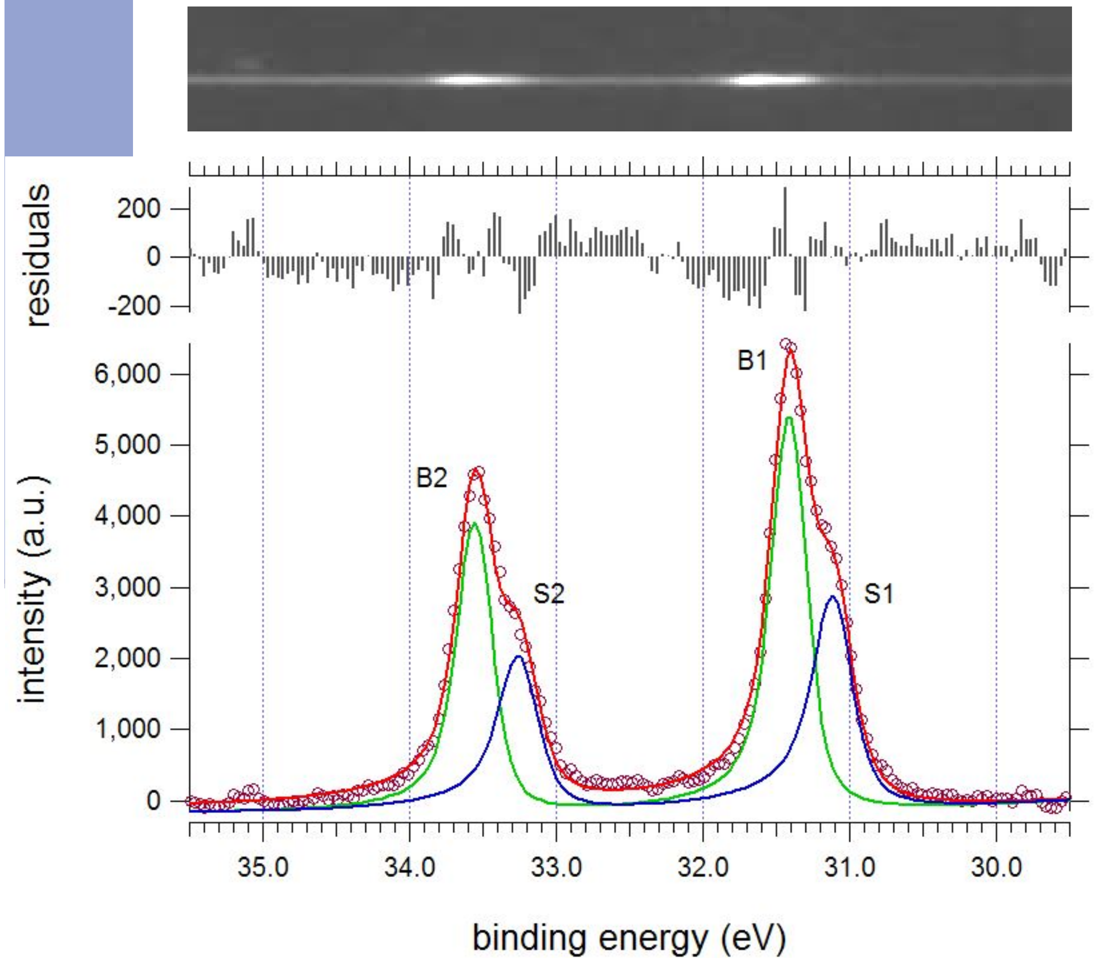
Imaging of Dispersive Plane

- W{110} clean surface
- W 4f core level





Imaging of Dispersive Plane



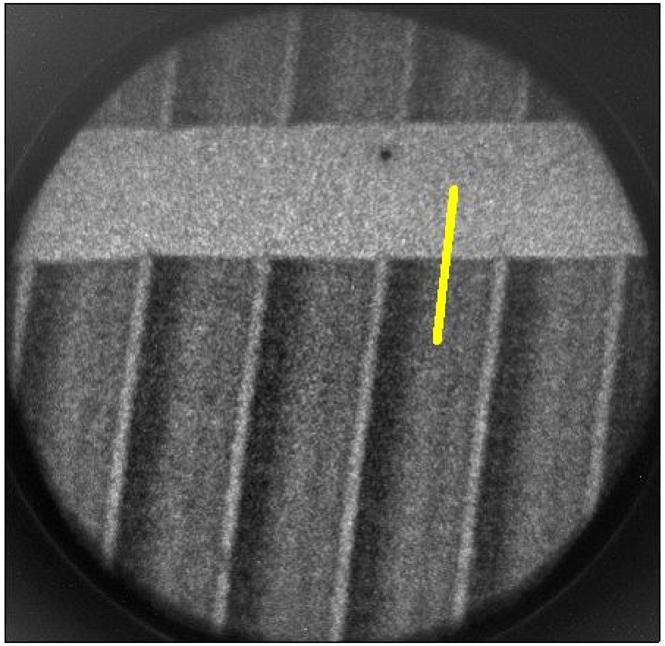
- W{110} clean surface
- W 4f core level
- hv = 98 eV
- Resolution 210 meV

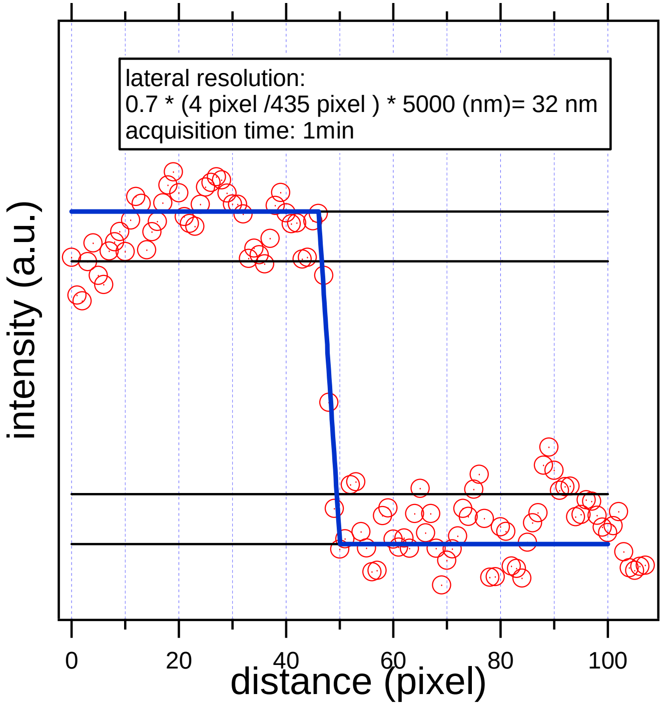
parameter	Ref. (1)	Our fit
Gamma B (eV)	0.06	Fixed
Alpha B	0.035	Fixed
Gamma S (eV)	0.084	Fixed
Alpha S	0.063	Fixed
Gauss. Broad. (eV)	0.04	0.21
$W_{7/2}$-$W_{5/2}$ BE diff. (eV)	2.2 (2)	2.14
SCLS (eV)	0.321	0.304

(1) Riffe et al., PRL **63** (1989) 1976.
(2) Webelements

Lateral resolution

- C 1s image (hv= 350 eV, KE = 62 eV)





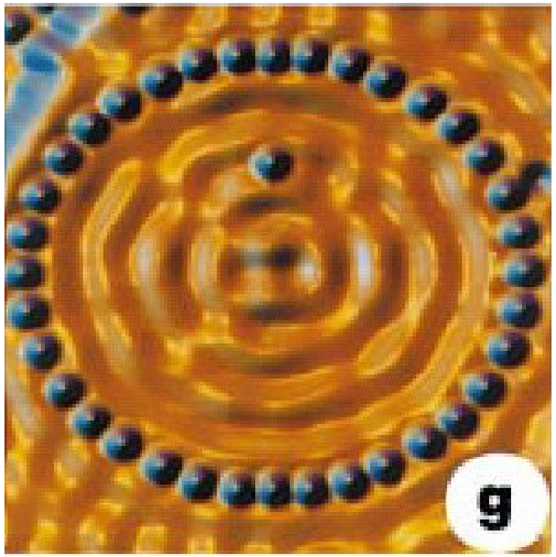
- Lateral resolution: 32 nm

Outline

- A brief introduction to spectromicroscopy
- The SPELEEM at Elettra
  - SPELEEM = spectroscopic photoemission and low energy electron microscope
- Application example:
  - Local Anodic Oxidation (LAO) by AFM: GaAs oxides

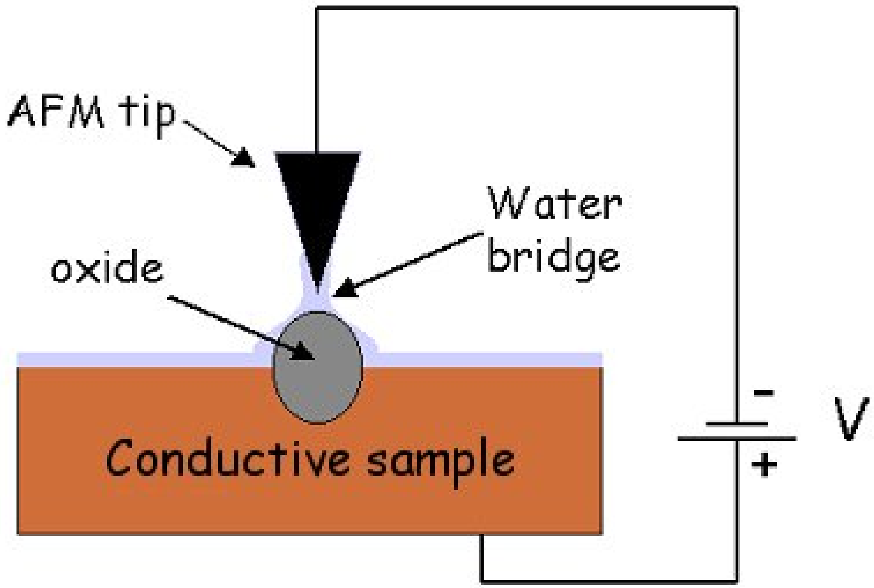
Motivation

- Lithography for fabrication of state-of-the-art semiconductor nanostructures
- Basic research and quantum device applications
- Approaches:
  - Traditional lithography
  - Proximal probes (STM or AFM)



H.C. Manoharan, C.P. Lutz,
D.M. Eigler: Nature **403** (2000) 512

Local Anodic Oxidation (LAO)



- Water electrolysis $H_2O \rightarrow H^+ + OH^-$.
- OH^- groups migrate towards the sample.
- Oxide penetration induced by the intense local electric field.

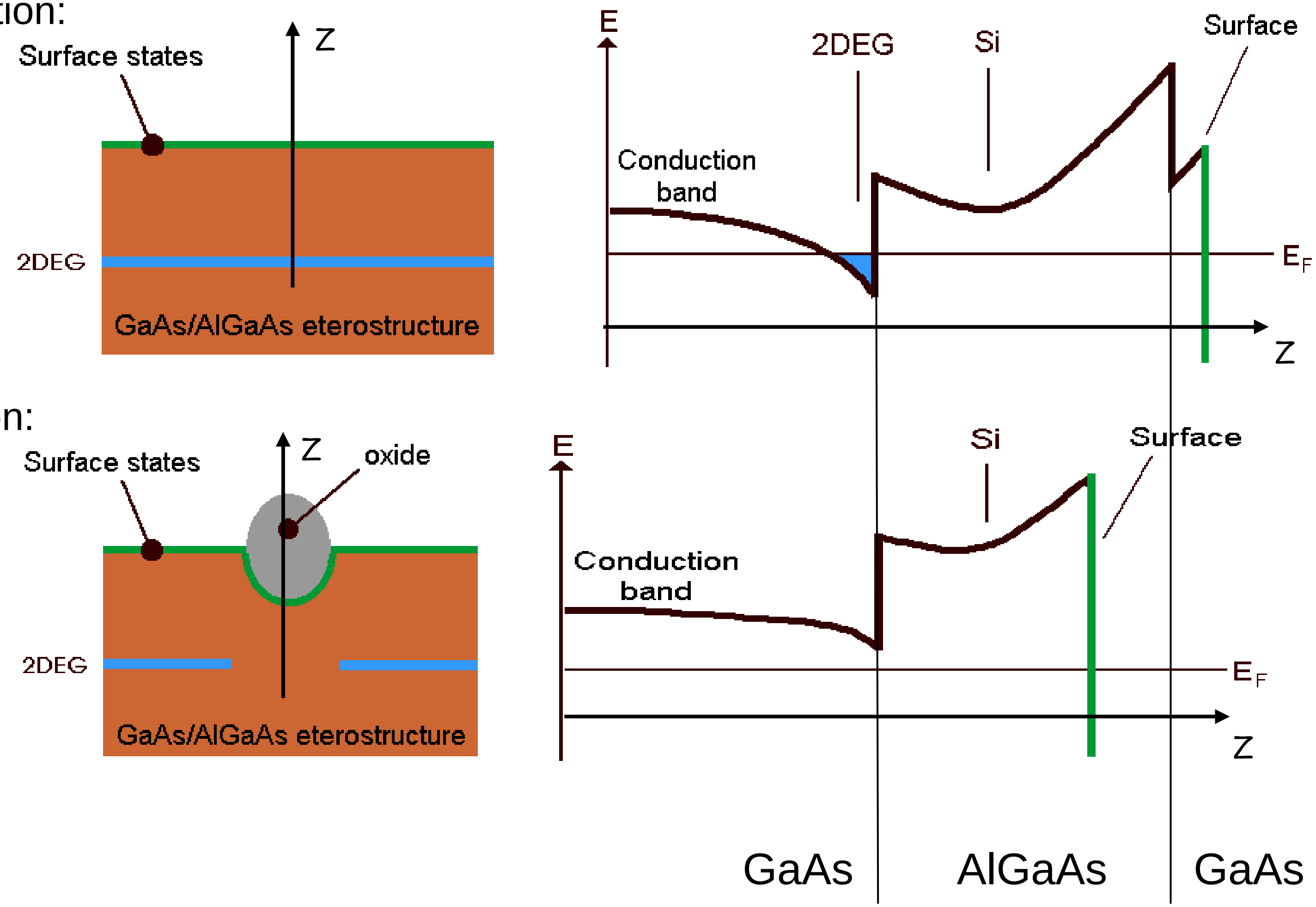
Versatile tool at relatively low cost

High lateral resolution but small area

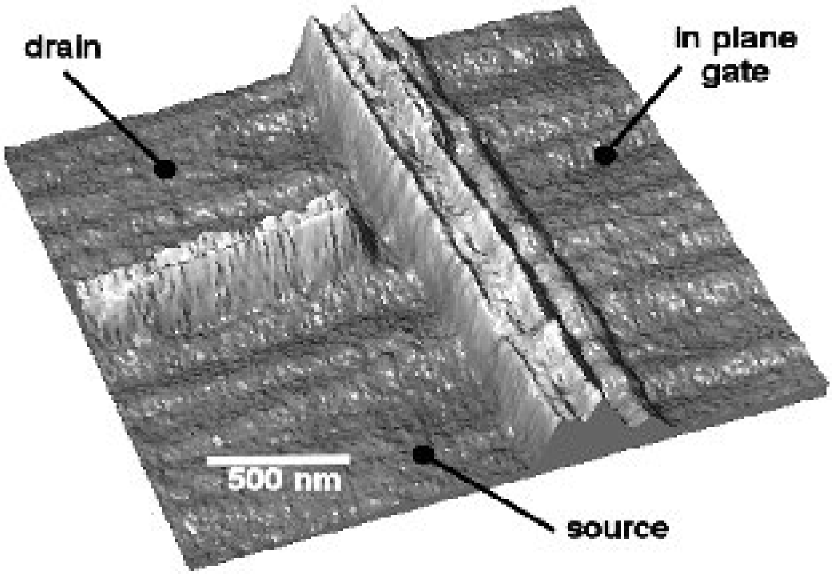
LAO on GaAs/AlGaAs

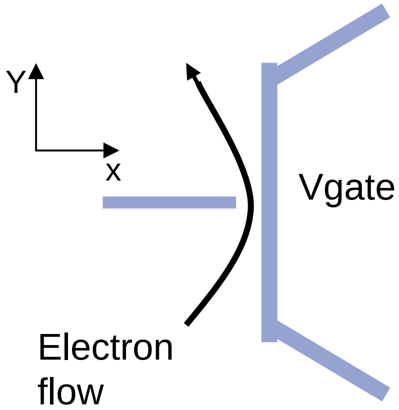
Before oxidation:

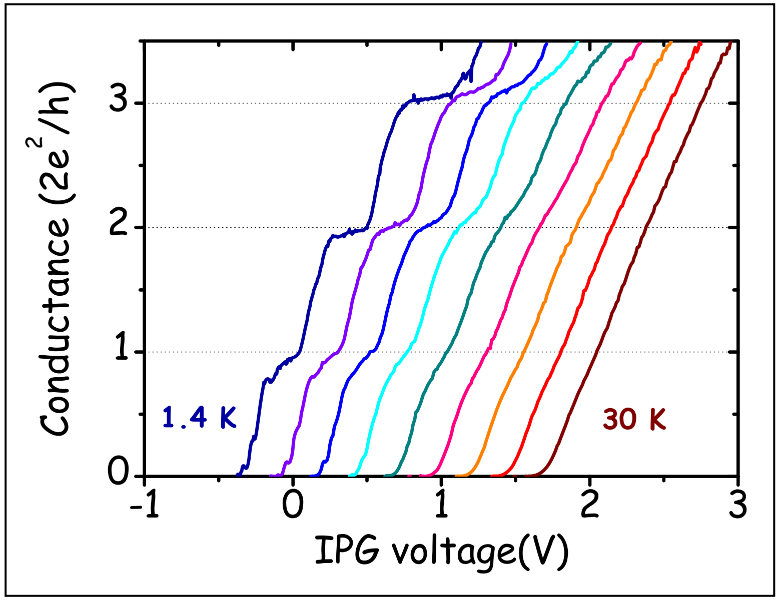
After oxidation:



Quantum Point Contact







G. Mori et al, JVST B **22** (2004) 570.

Setup for Lithography on GaAs

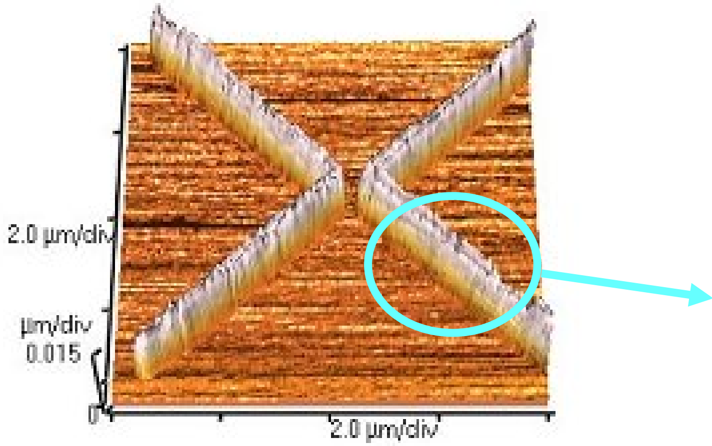
Thermomicroscope Microcope CP-Resource

Why Spectroscopic Microscopy?

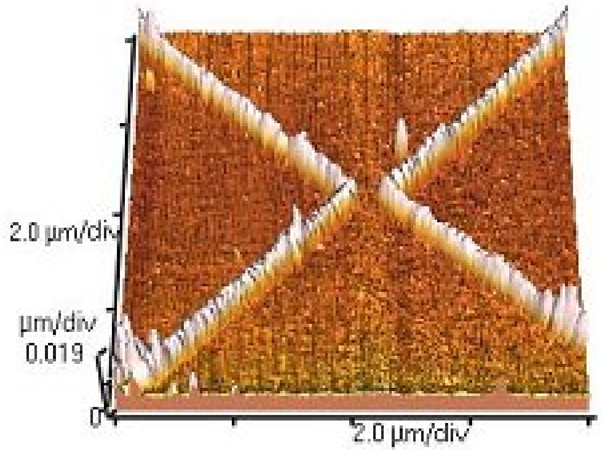
- Lack of information on the oxidation process and on the chemical nature of the oxides.
- Lack of reliable microscopic techniques able to perform chemical analysis on such small structures.

- Understand the composition of the AFM-grown oxides (electrical and chemical properties, effect of oxidation parameters).
- Improve the fabrication of devices with LAO.

GaAs Oxide: Photon Exposure



AFM before: height 18nm



AFM after: height 13nm

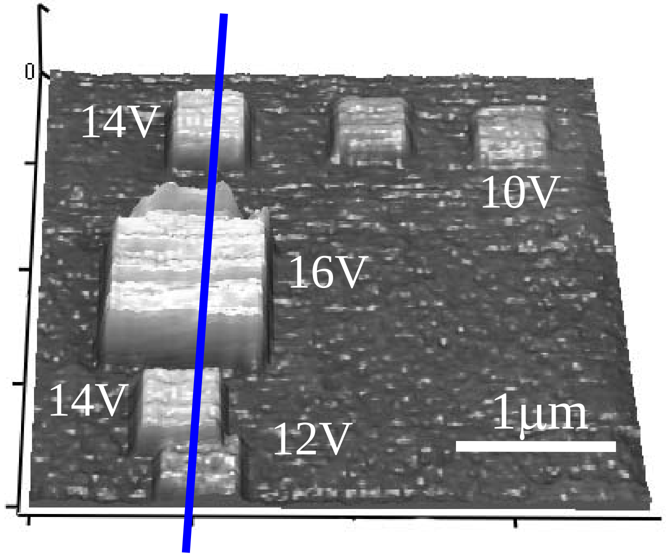
Images taken with secondary electrons

- Photon energy: 125 eV
- Kinetic energy: 4 eV
- Field of view: 10 µm
- One image every 2 sec

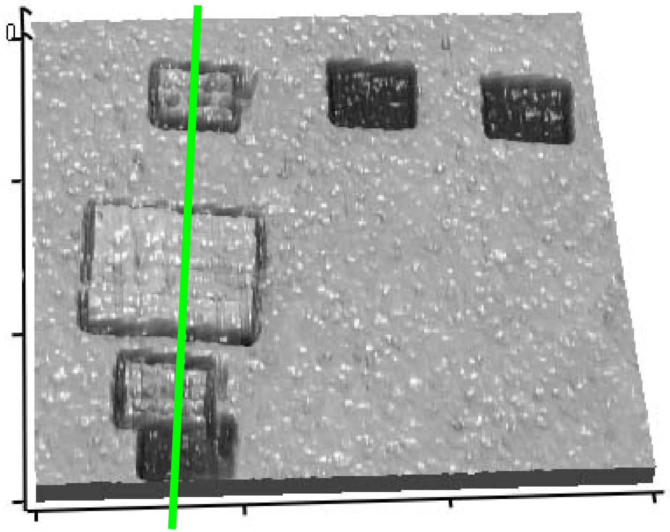
GaAs Oxide: Desorption

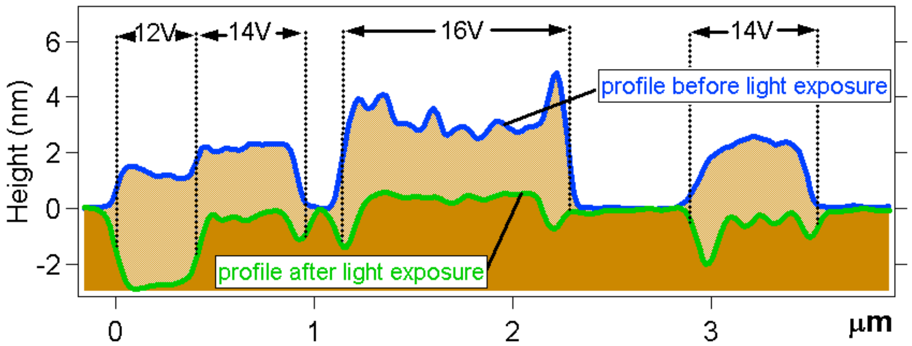
Before photon exposure

After hours of exposure



hv

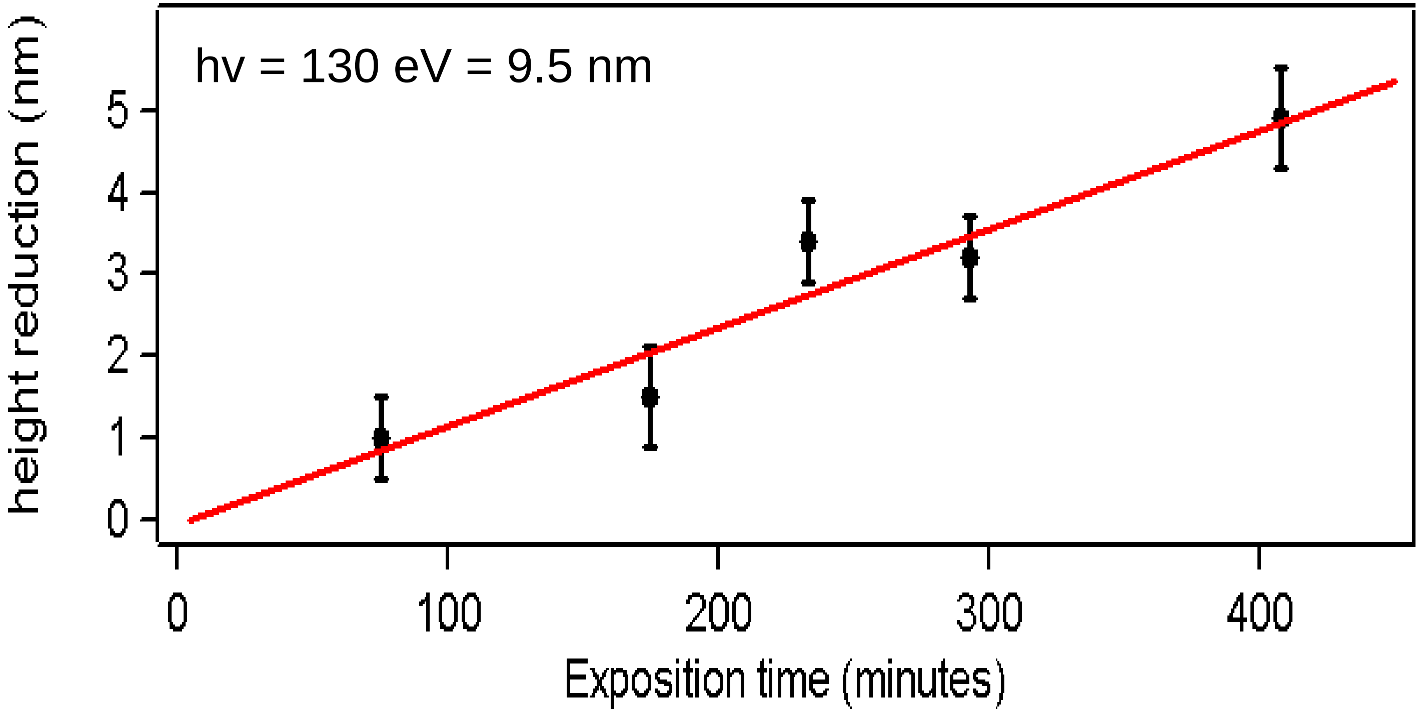




Writing speed 0.5 µm/s
Humidity 50 %

Photon energy 130 eV
Photon flux 10^{17} ph $cm^{-2}sec^{-1}$

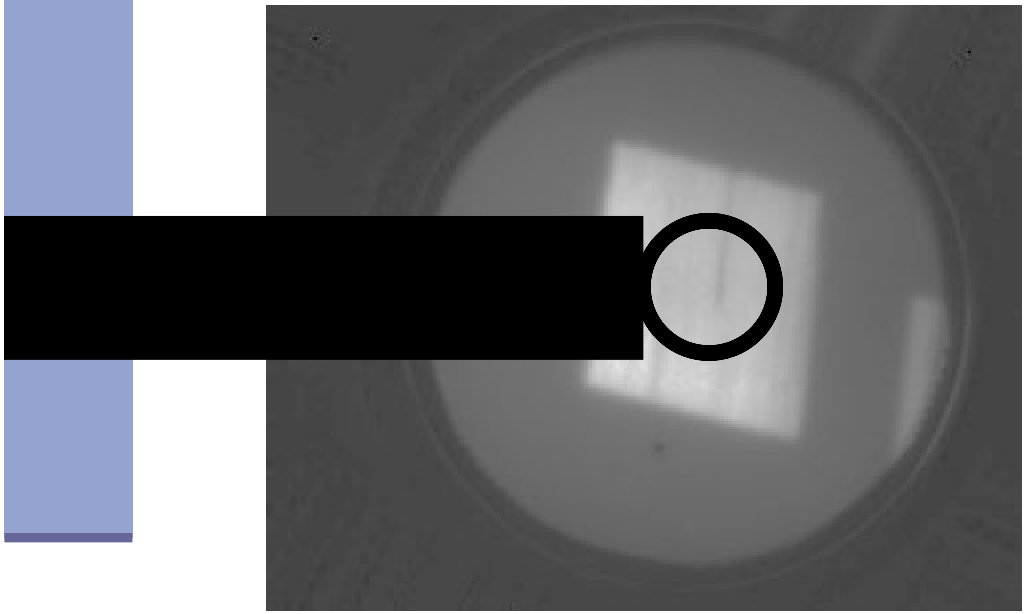
Height reduction vs. exposure time



- We observe a linear relation between exposure time and height reduction.
- A dependence on other oxidation parameters (bias, writing speed) could not be detected.

Spectra From AFM GaAs Oxide

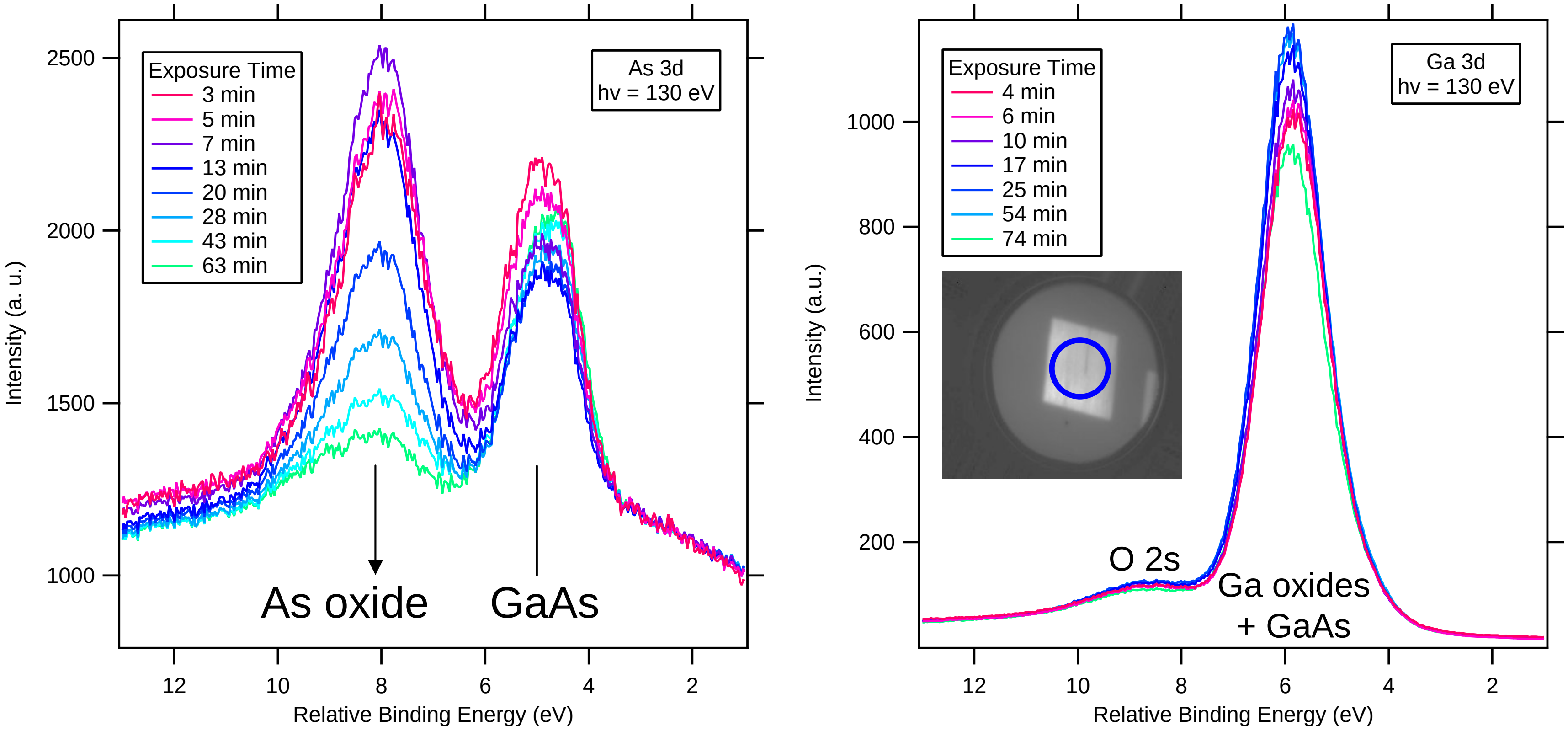
Time resolved spectroscopy with SPELEEM using Dispersive Plane (hv = 130 eV)



- Sample S03B
- Hole (3,2)
- Writing voltage 15 V
- Structure height 3 nm
- Image taken with secondary electrons:
  - Photon energy: 130 eV
  - Kinetic energy: 0.3 eV
  - Field of view: 10 µm

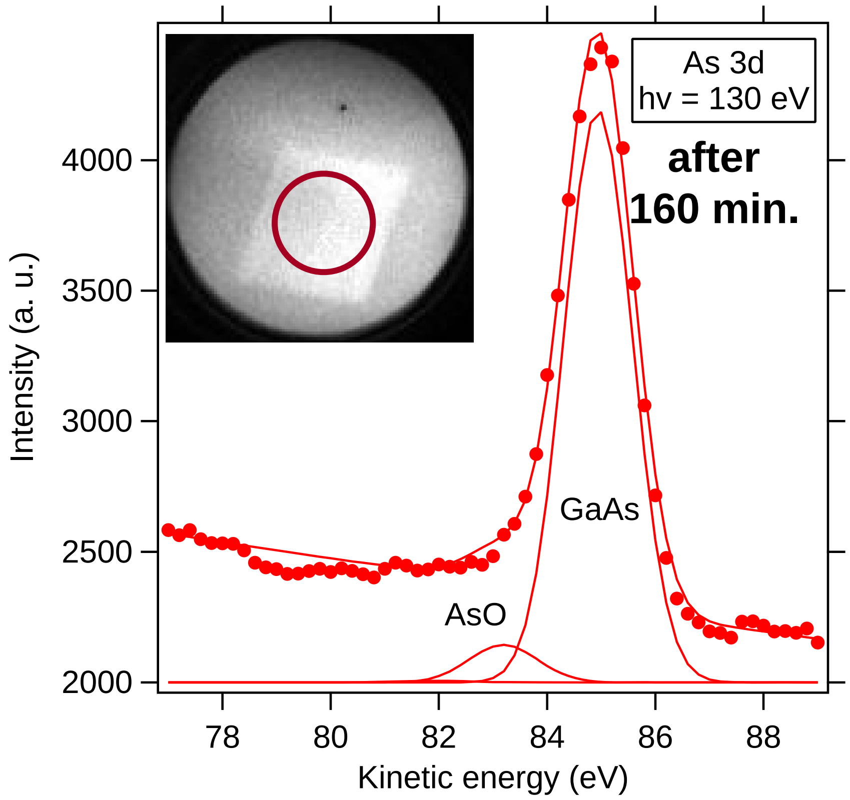
Spectra From AFM GaAs Oxide

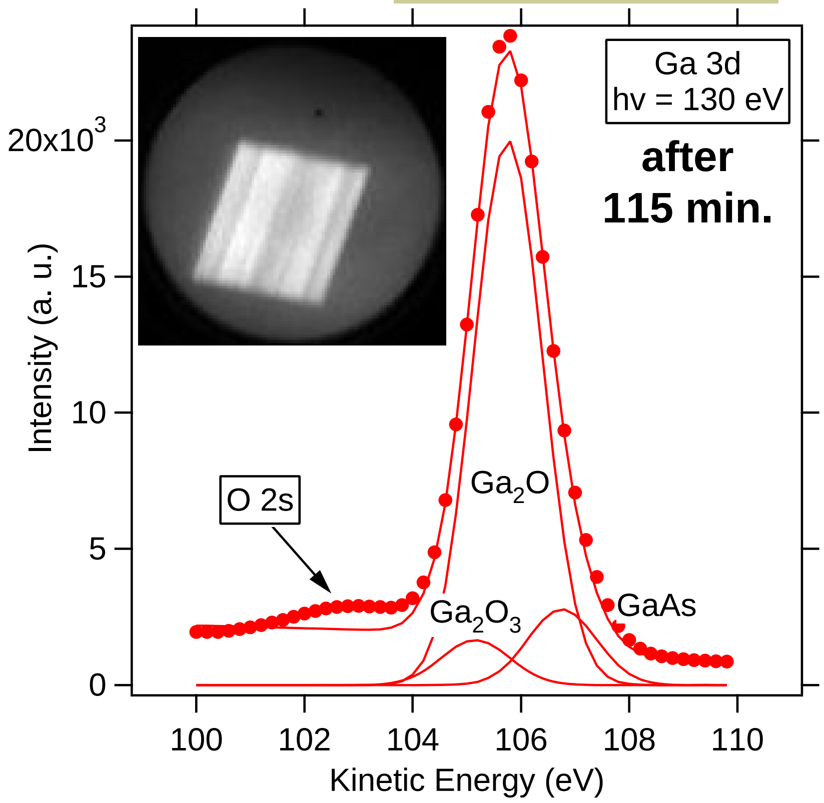
Time resolved spectroscopy with SPELEEM using Dispersive Plane (hv = 130 eV)



- The As-oxide signal disappeares with time.
- The Ga-oxide signal remains unchanged (early stage of exposure).

Spectra From AFM GaAs Oxide



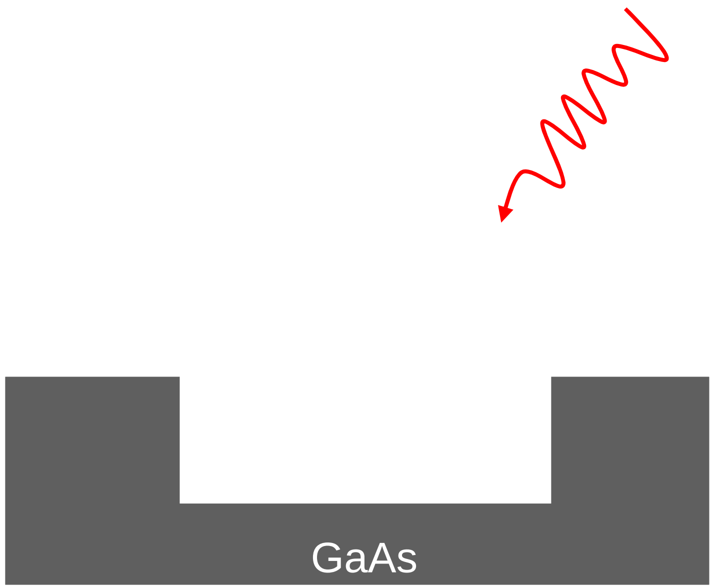


- The AFM-oxide is mainly composed of Ga_2O.
- After 3 hours exposure, only traces of As-oxides observed in As 3d.
- Absolute ratio As : Ga = 1 : 5

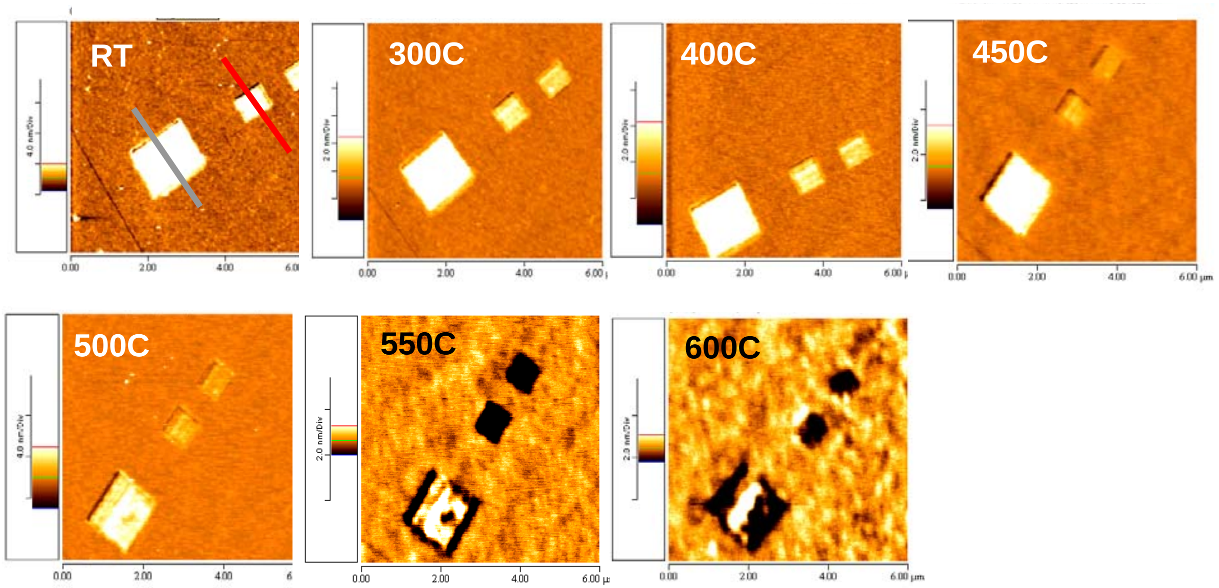
A possible model for the desorption

Our Observations:

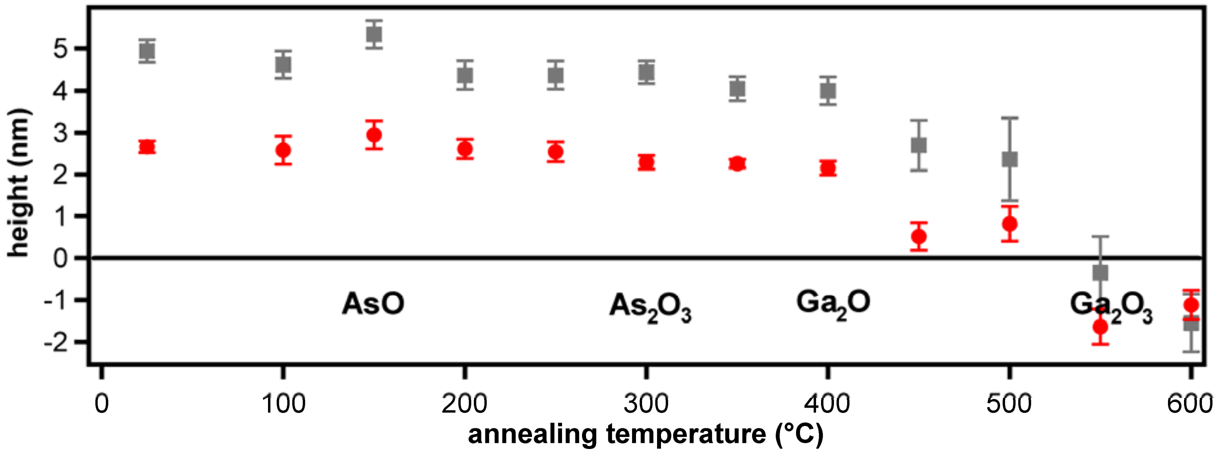
- The AFM-grown oxide exposed to 130 eV photons desorbs linearly with exposure time.
- The AFM-oxide is mainly composed of Ga_2O with traces of As-oxide.
- The shape of the Ga peak does not change with exposure time (early stage of desorption).
- The As-oxides desorb completely after 3 hours of exposure.
- Not even traces of As-oxides detected in scanning Auger microscopy.



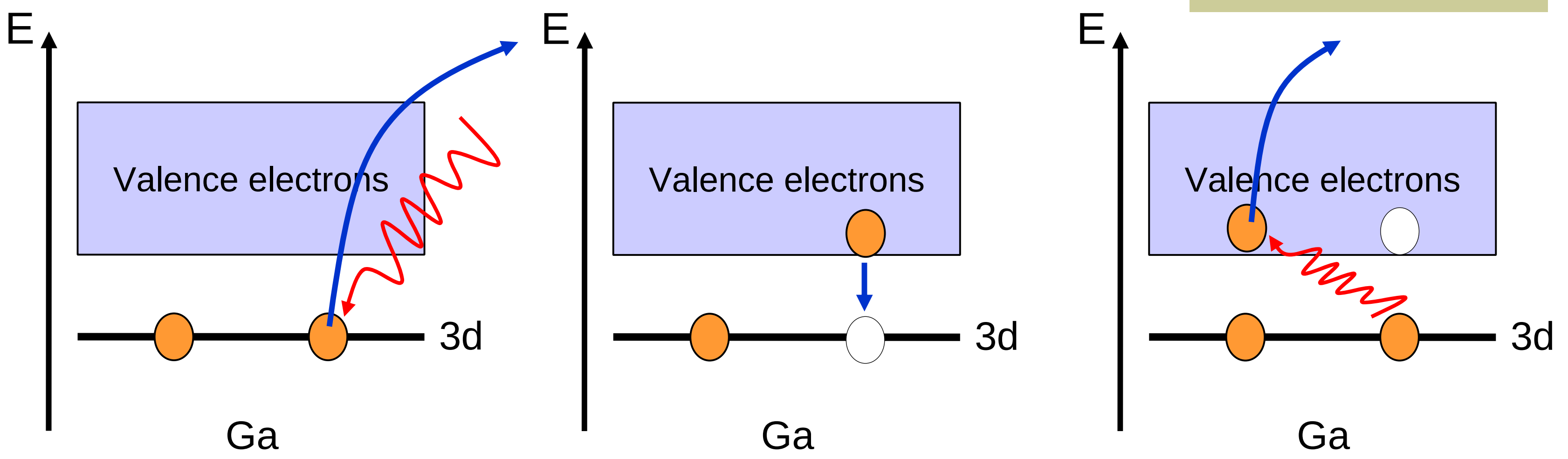
Thermal stability



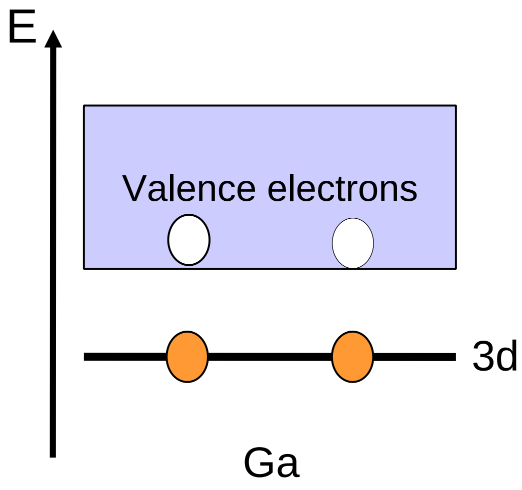
Each annealing step:
10 minutes
in N_2 atmosphere



The Knotek-Feibelman mechanism



Initial state



Final state

- The valence electrons are mainly localized at O atoms.
- This Auger decay leads to a final state with two vacancies in valence band weakening the bond between Ga and O.

Summary

- The AFM-oxide is mainly composed of Ga_2O with traces of As-oxide at the surface.
- Photon assisted partial desorption of the AFM-grown oxide was observed.
- All As oxides and the oxygen-rich Ga oxides are desorbed.
- We proposed a simple model for the dynamics of the desorption.