Local Anodic Oxidation of GaAs: A Nanometer-Scale Spectroscopic Study with PEEM

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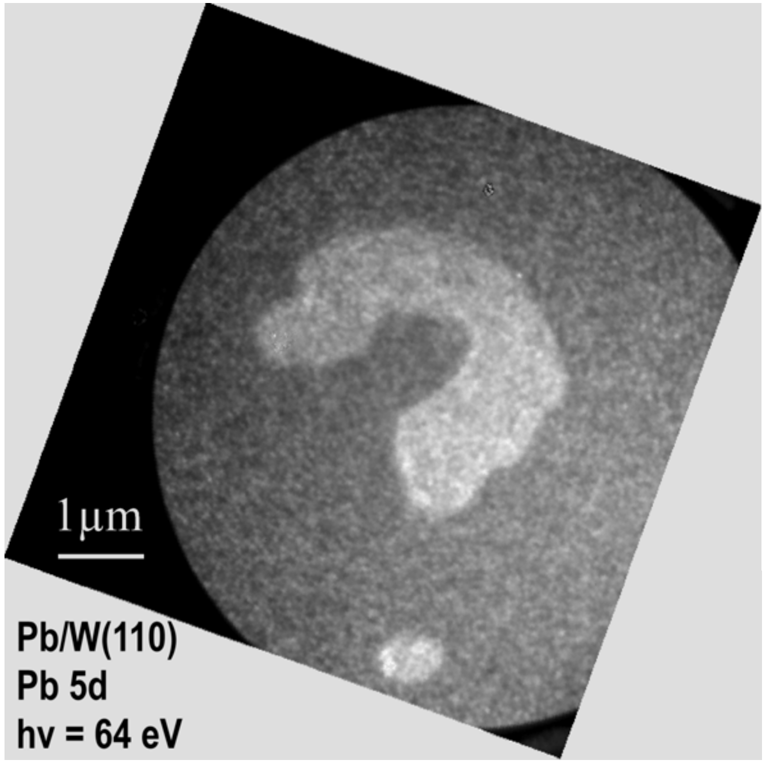
Outline

- A brief introduction to spectromicroscopy
  - The spectroscopic photoemission and low energy electron microscope (SPELEEM)
- Local Anodic Oxidation (LAO) of GaAs

Motivation



Why XPS?

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

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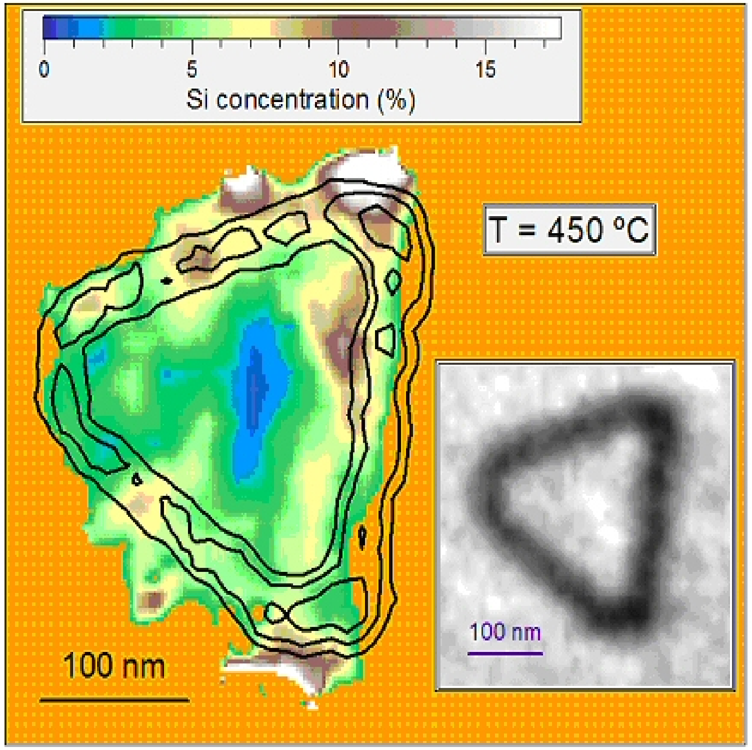

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Why spectromicroscopy ?

- semicond. nanostructures: self-organized islands (dots)

F. Ratto et al.: Appl. Phys. Lett. **84** (2004) 4526.

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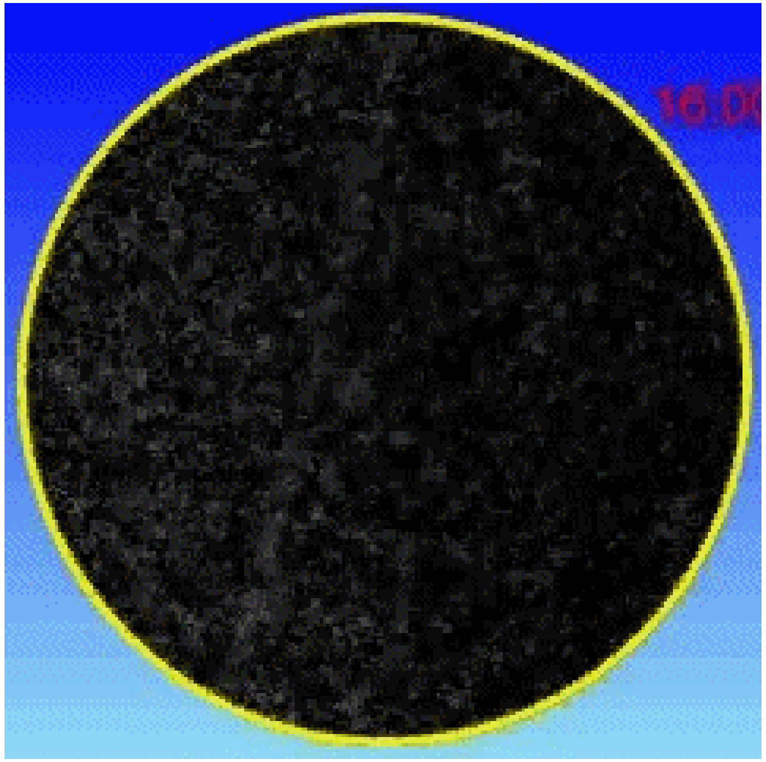

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F.-J. Meyer zu Heringdorf et al.: Phys. Rev. Lett. **86** (2001) 5088.

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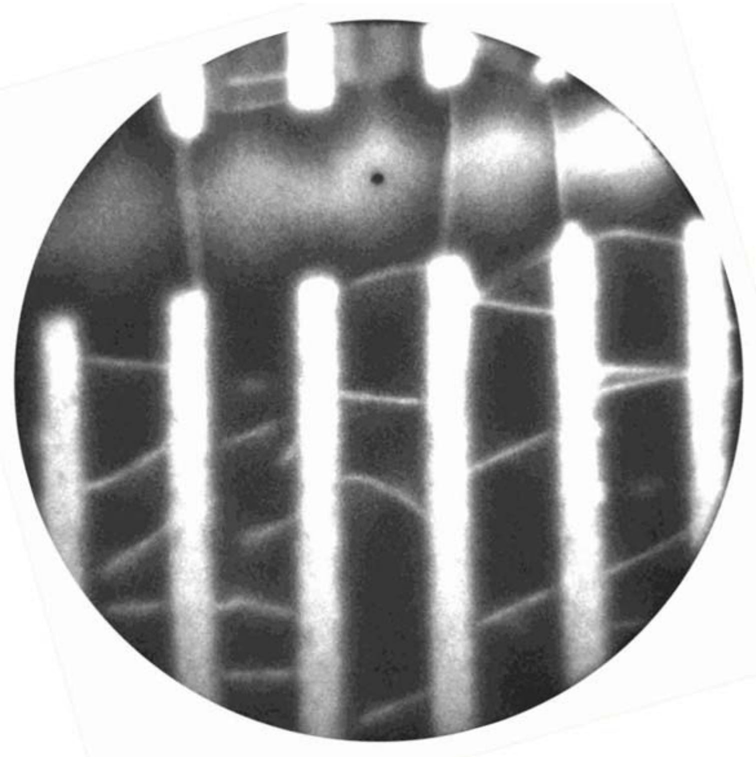

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S. Suzuki et al.: Appl. Phys. Lett. **85** (2004) 127.

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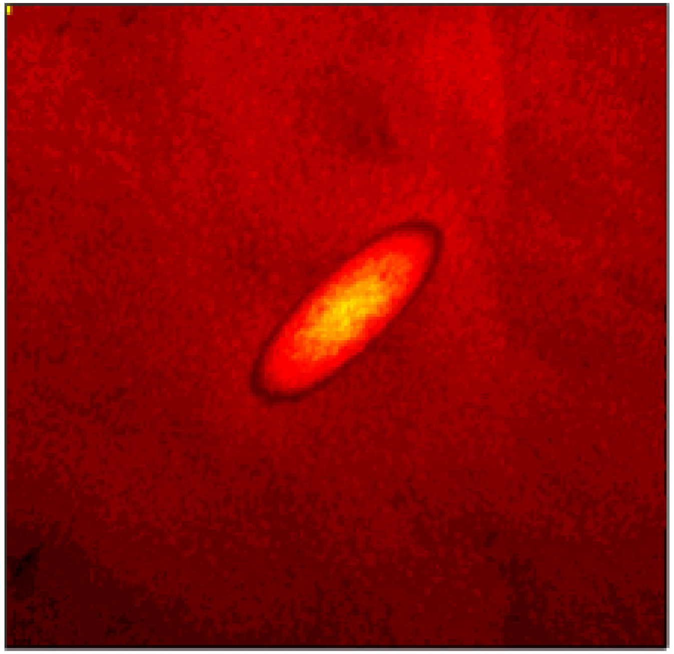

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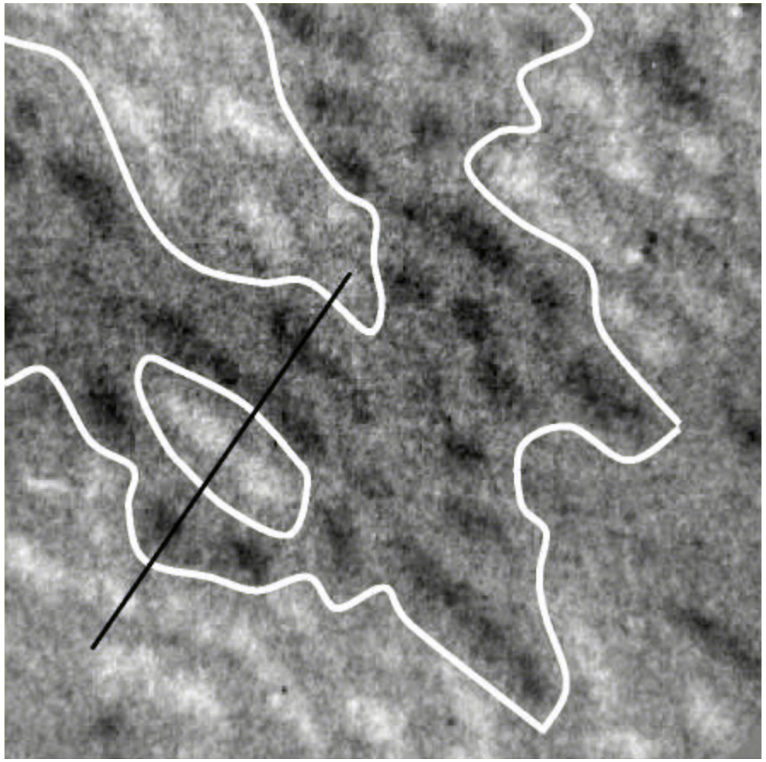
- catalysis, chemical waves

A. Locatelli et al.: J. Am. Chem. Soc., in press.

Motivation



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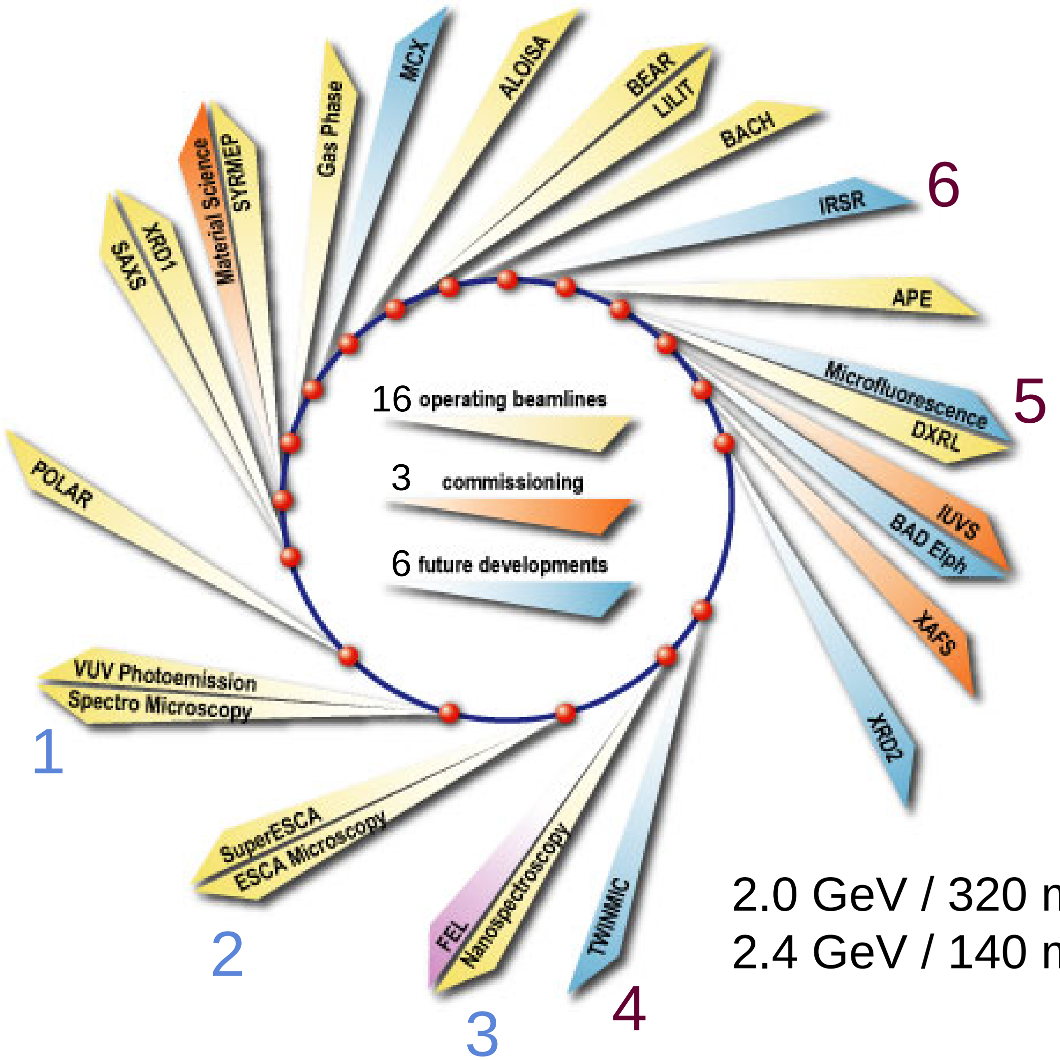
- surface faceting (wires)
- carbon nanotubes
- catalysis, chemical waves
- surface magnetism (XMCD)

A. M. Mulders et al.: Phys. Rev. B, submitted.

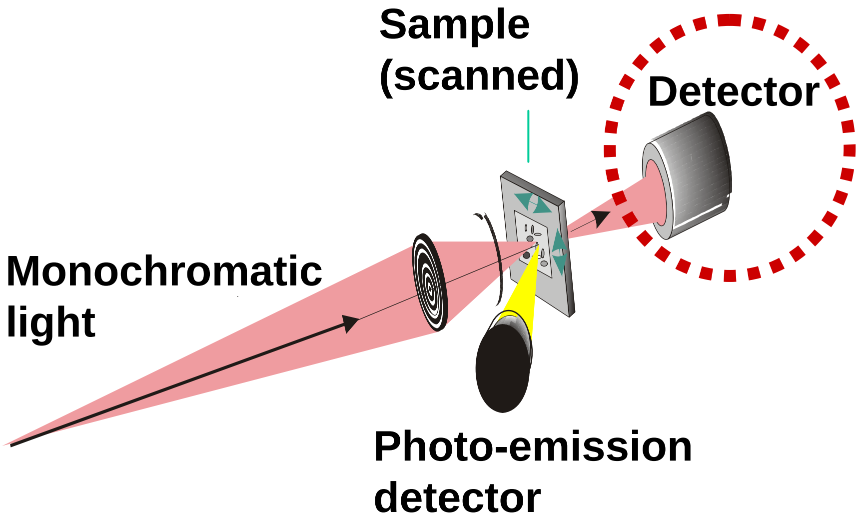
Spectromicroscopy at Elettra



2.0 GeV / 320 mA
2.4 GeV / 140 mA

1: Spectromicroscopy
2: ESCA Microscopy
3: Nanospectroscopy
4: TwinMic
5: Microfluorescence
6: IR Microscopy

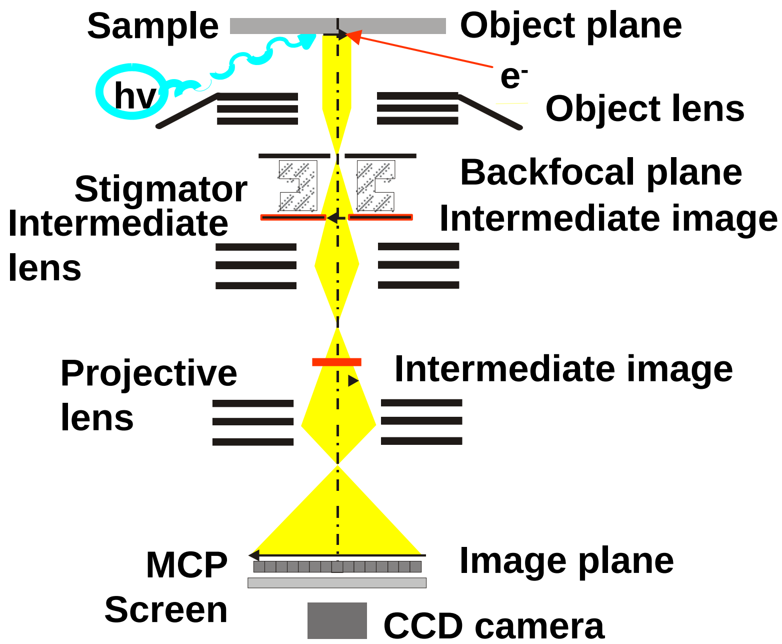
Scanning vs. direct imaging type



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



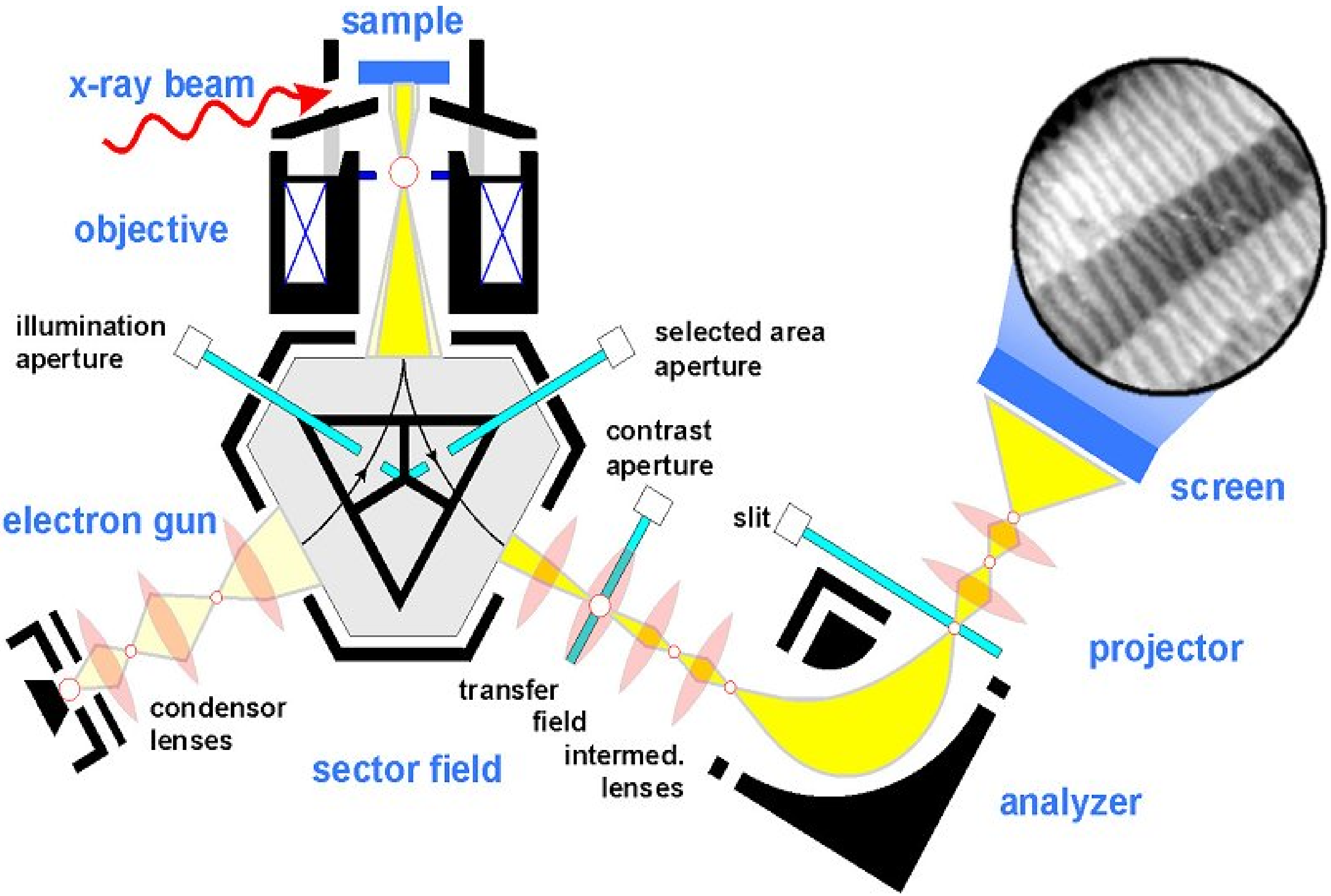
Electron optics to magnify irradiated area:

Imaging Instrument

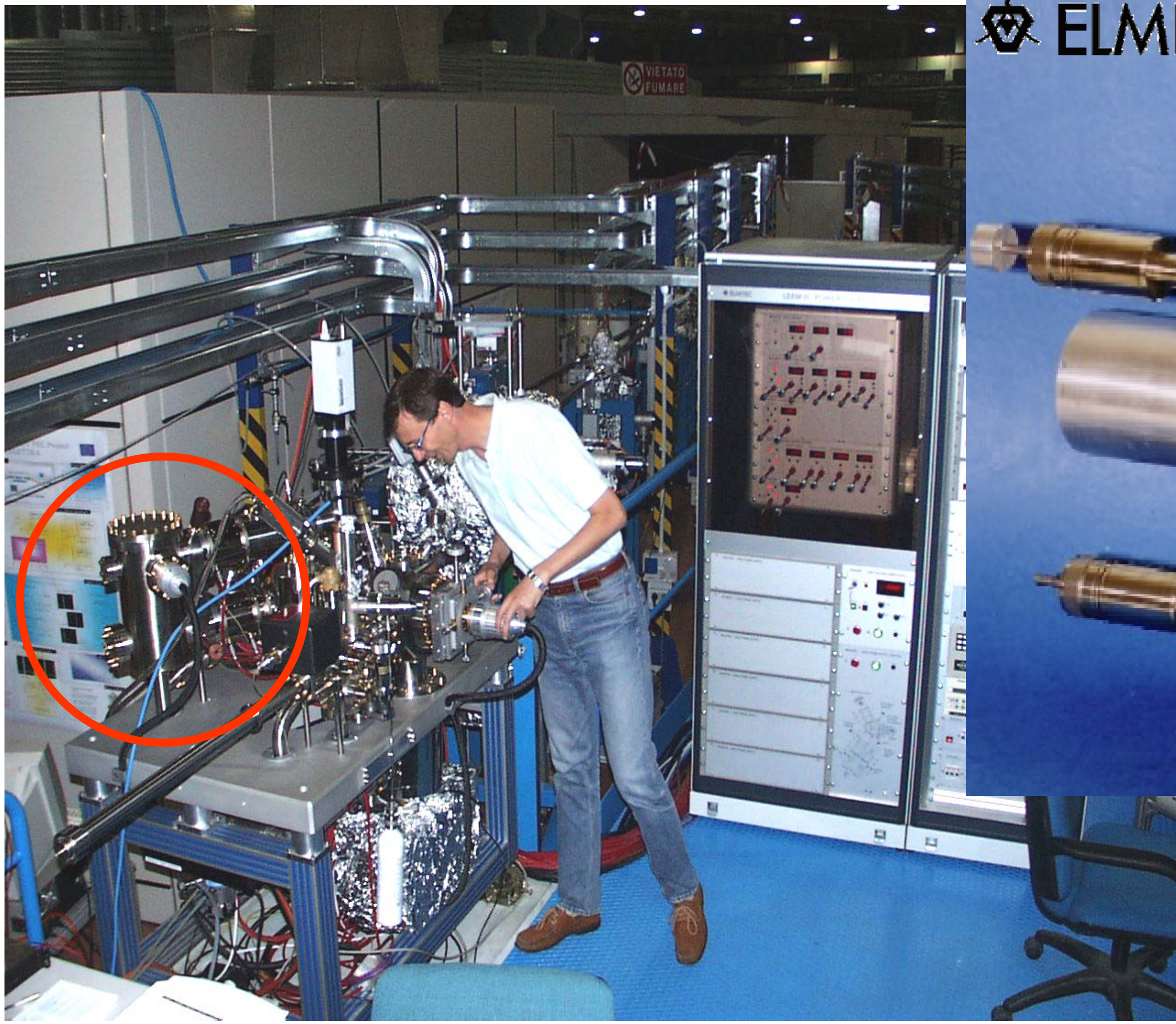
- High lateral resolution (20 nm)
- Multi-method instrument (XPEEM/PED)
- Excellent for monitoring dynamic processes
- Poorer spectroscopic ability
- Sensitive to rough surfaces

The SPELEEM

Spectroscopic photoemission and low energy electron microscope

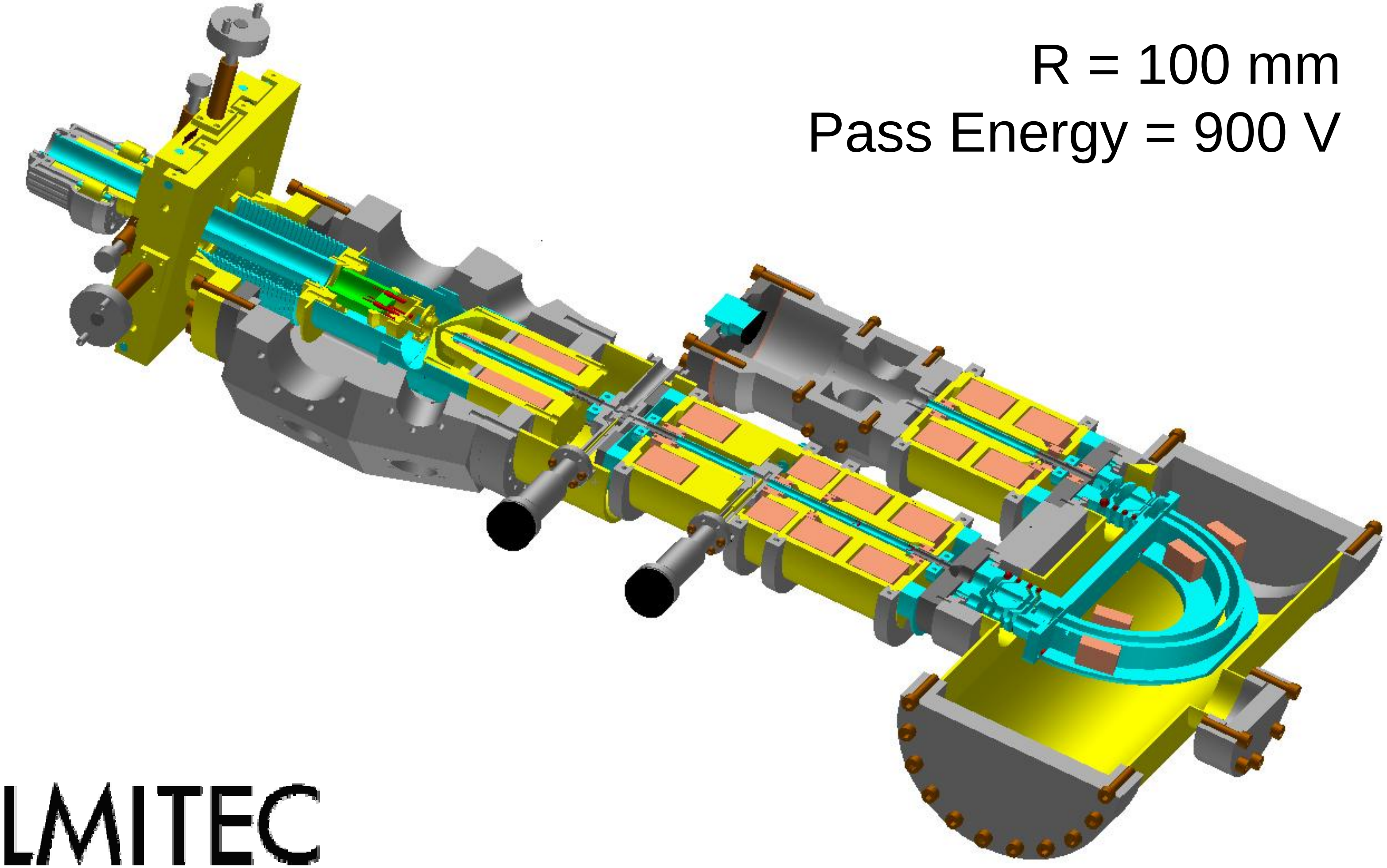


The SPELEEM at ELETTRA



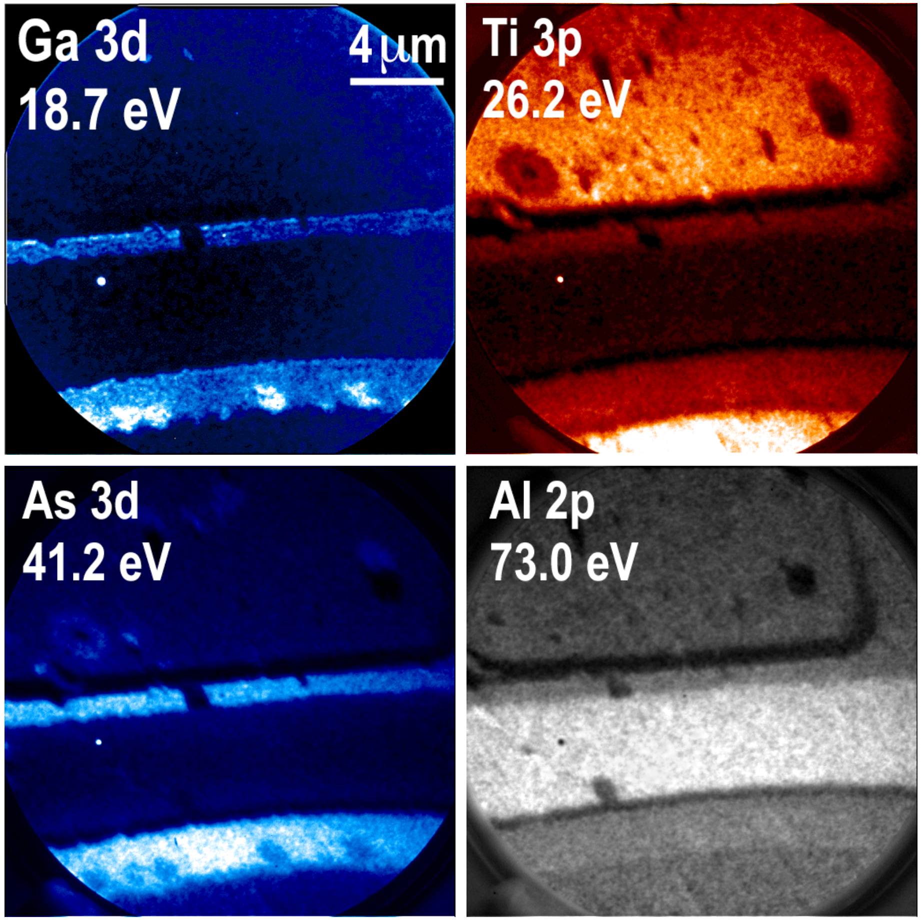
The Energy Filter

R = 100 mm
Pass Energy = 900 V

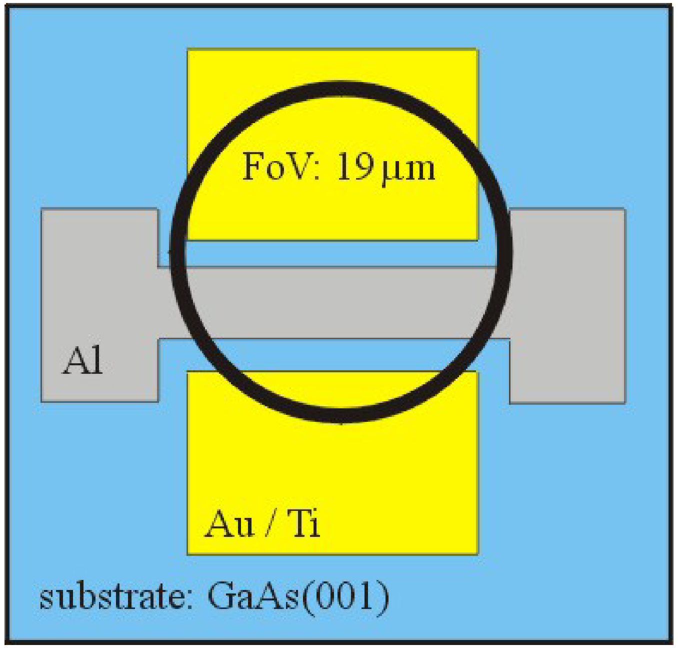


ELMITEC

XPEEM: Spectroscopic Microscopy



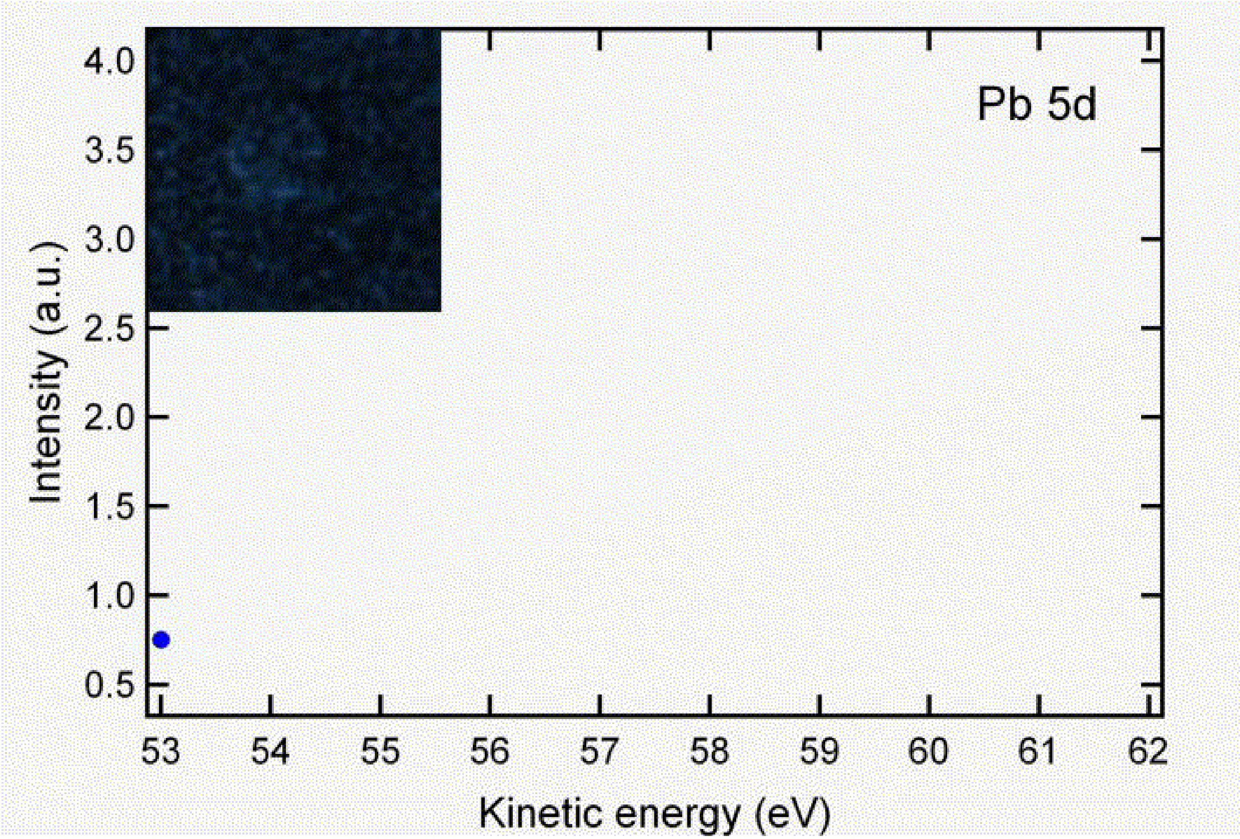
- Images from a Field Effect Transistor (FET) at different binding energies.
- Photon energy 131.3 eV.



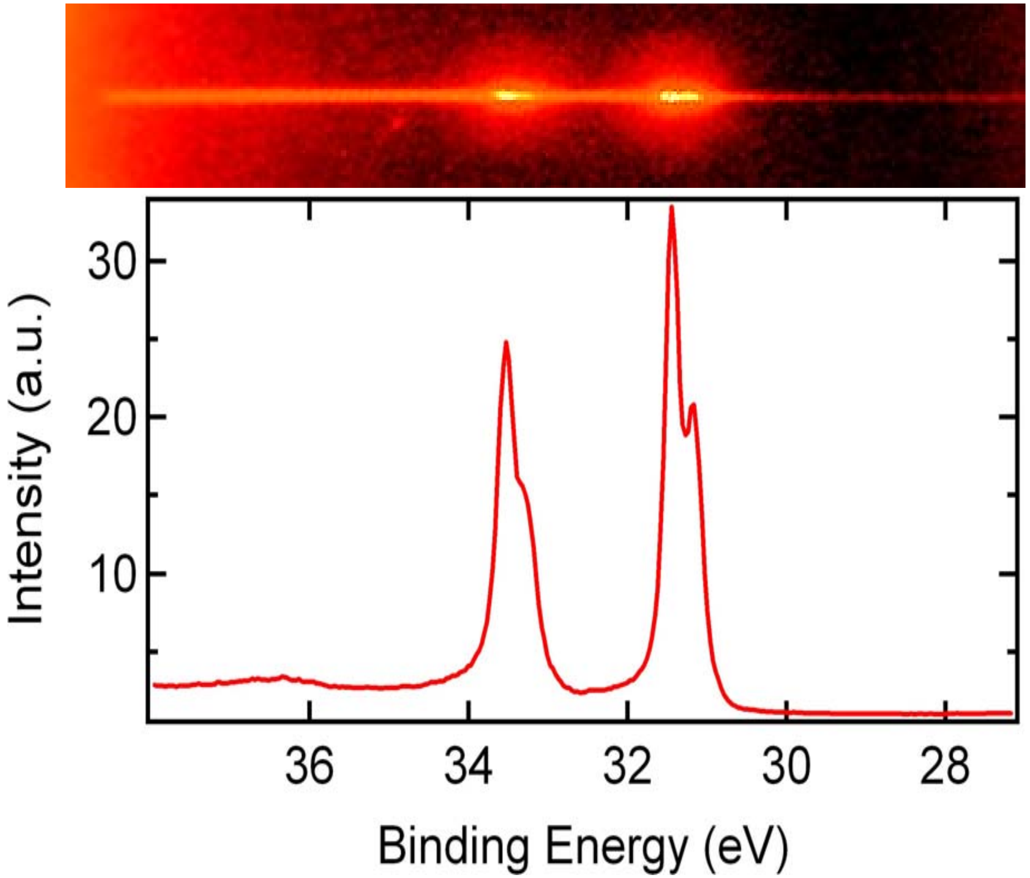
Sample from M. Lazzarino, L. Sorba, and F. Beltram

XPEEM: Core Level Spectroscopy

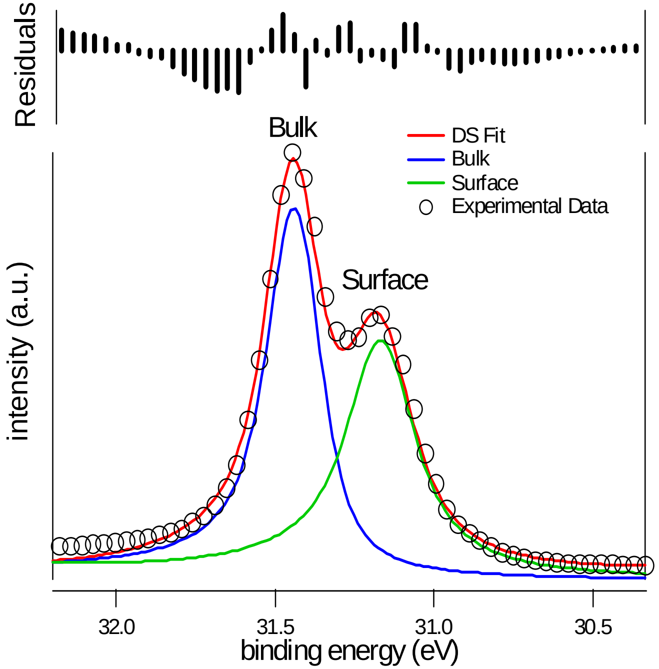
- Pb/W(110), Pb 5d core level, hν = 80 eV
- Best energy resolution: 250 meV



Imaging of Dispersive Plane

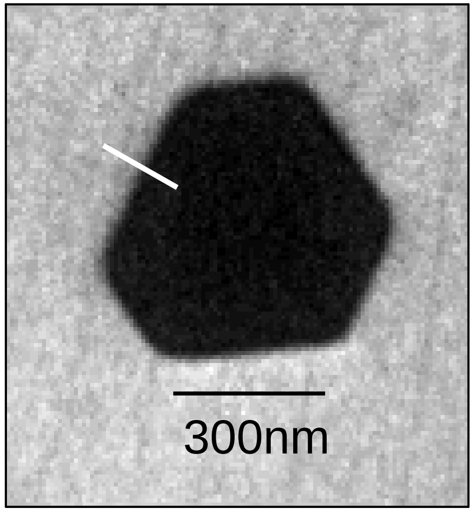


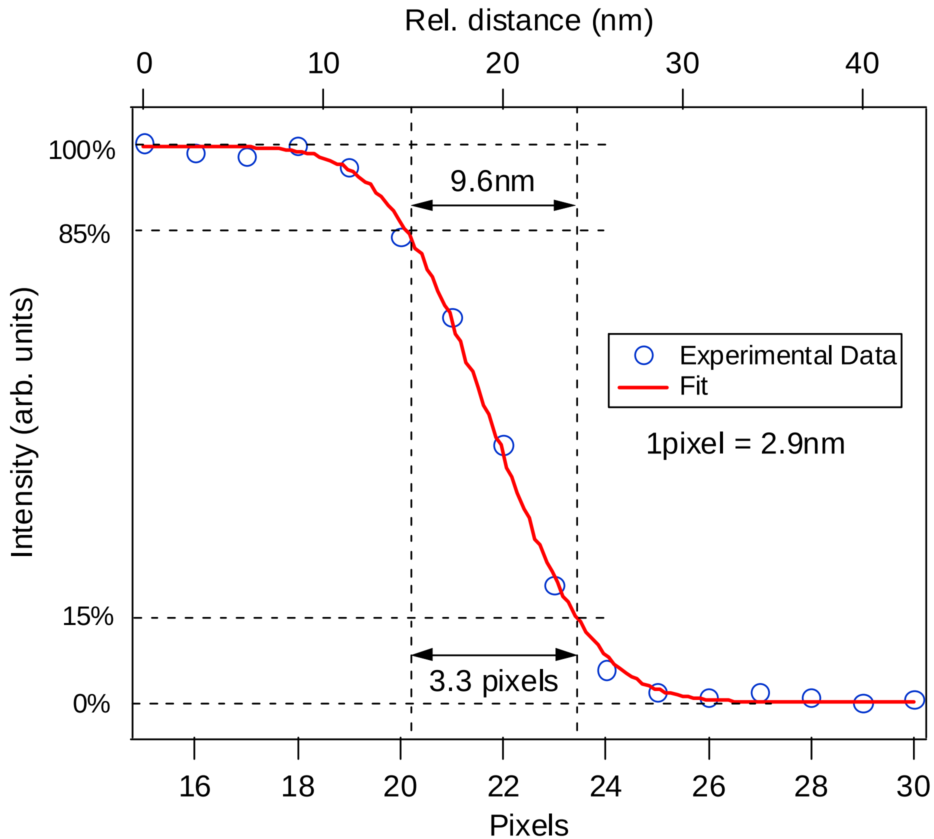
- W{110} clean surface
- W 4f core level
- hν = 131 eV
- Resolution 130 meV



Lateral resolution in LEEM mode

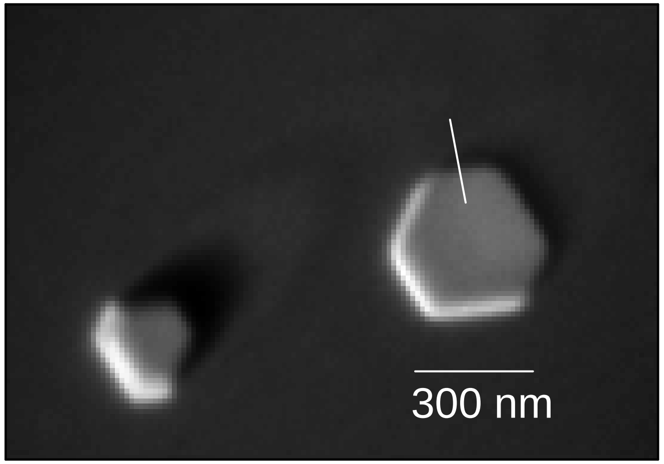
- Pb/W(110), E = 11 eV
- Lateral resolution: 10 nm

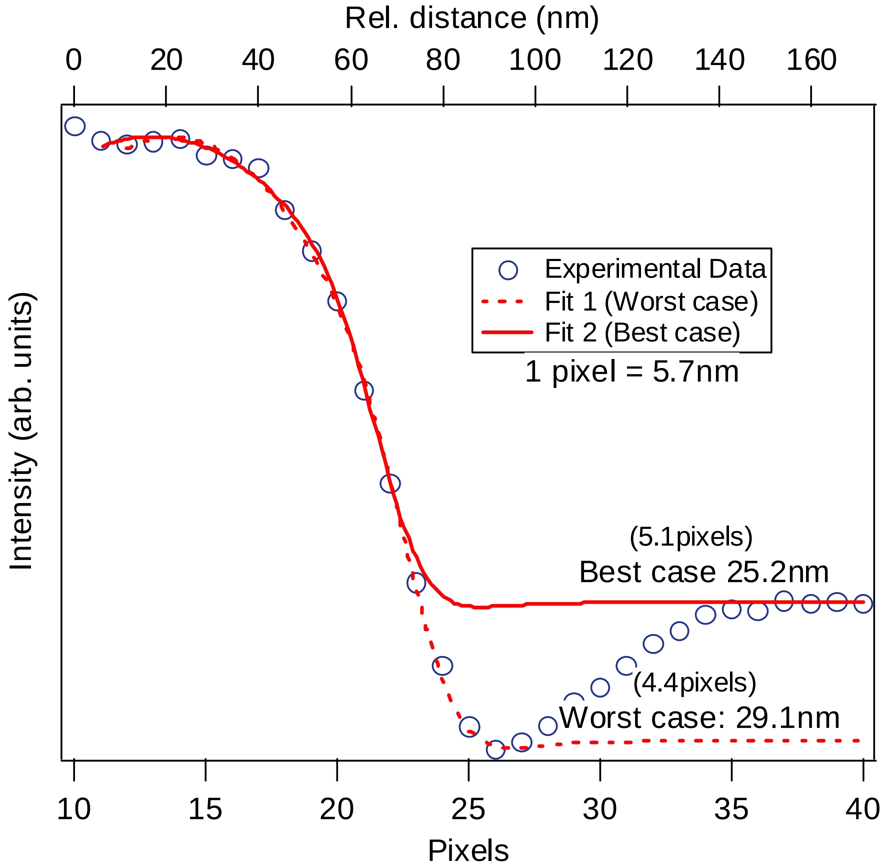




Lateral resolution in XPEEM mode

- Pb/W(110), Pb $5d_{5/2}$, hv = 80 eV, KE = 58.2 eV
- Lateral resolution: 27 nm





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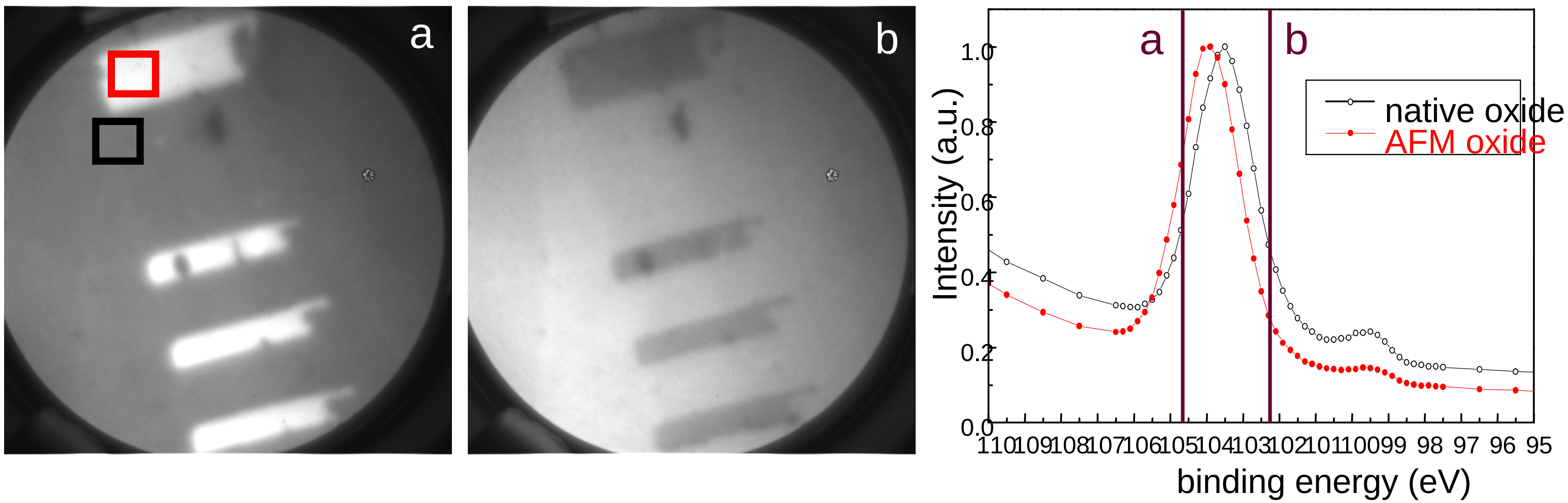
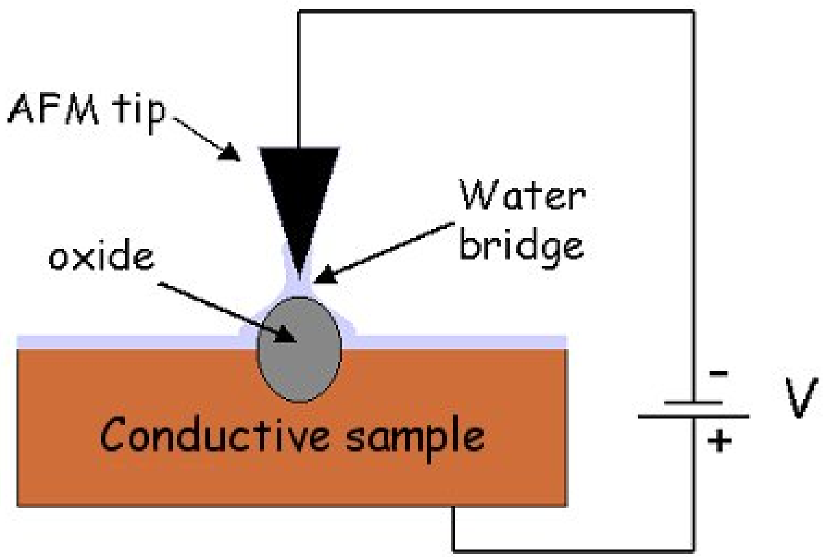


M. Lazzarino, S. Heun, B. Ressel, K. C. Prince, P. Pingue, and C. Ascoli: Appl. Phys. Lett. **81** (2002) 2842.

Local Anodic Oxidation (LAO)



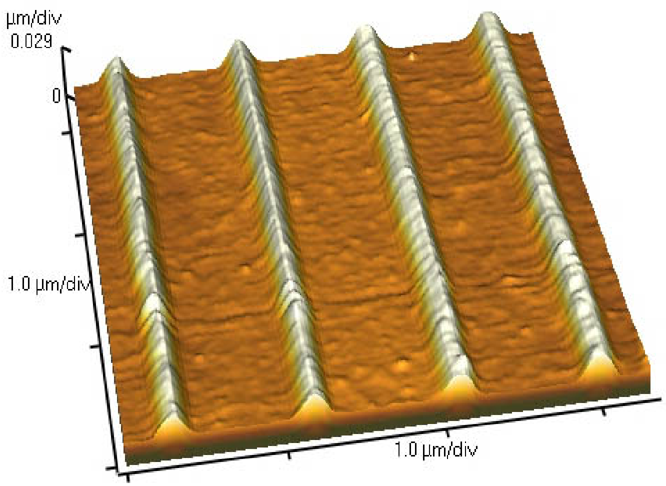
Commonly accepted model:

- Water electrolysis
  $H_2O \rightarrow H^+ + OH^-$.
- OH^- groups (or O^-) migrate towards the substrate-oxide interface.
- Oxide penetration induced by the intense local electric field ($>10^7$ V/cm).

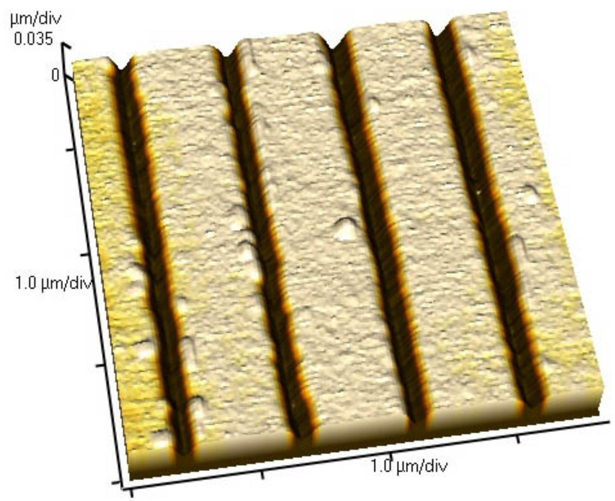
Versatile tool at relatively low cost

High lateral resolution but small area

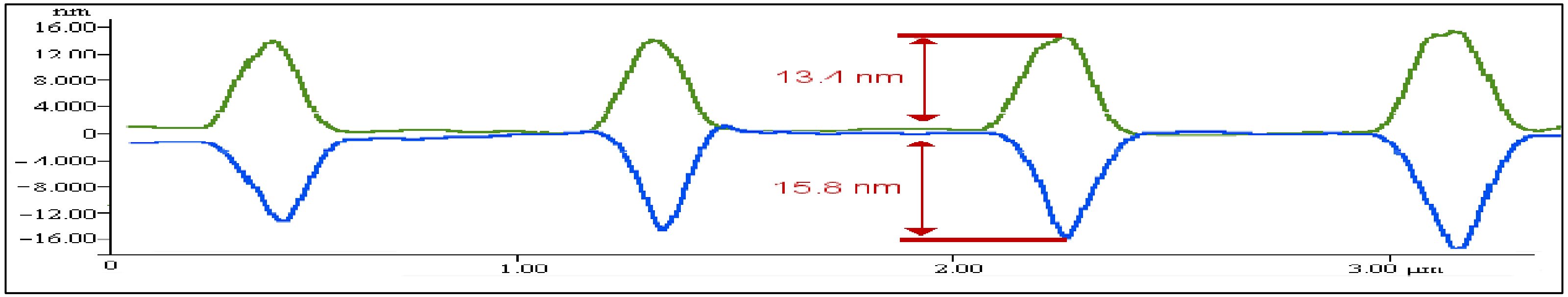
Local Anodic Oxidation (LAO)



After oxidation.



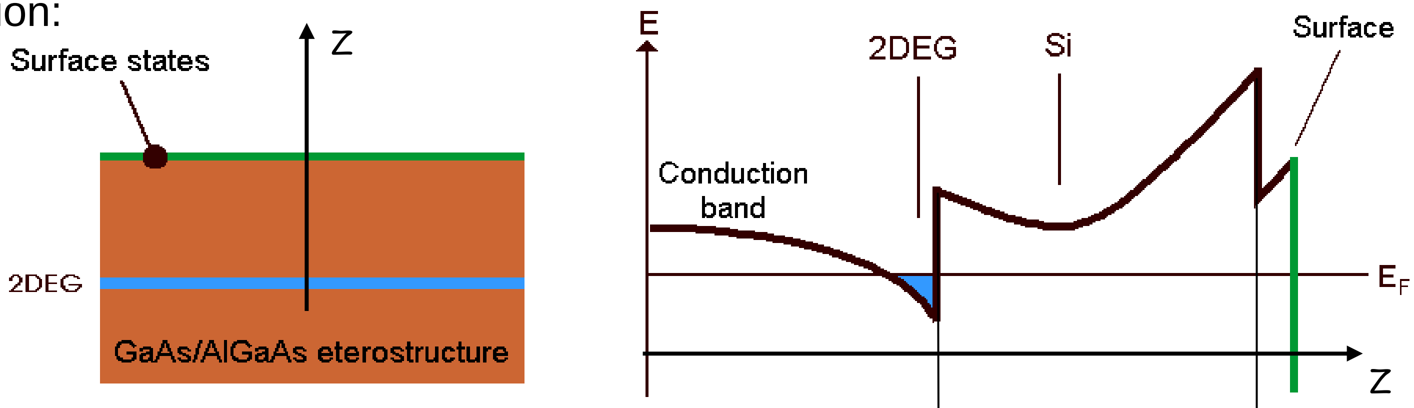
Oxide removal with HF 10%, 30 s.



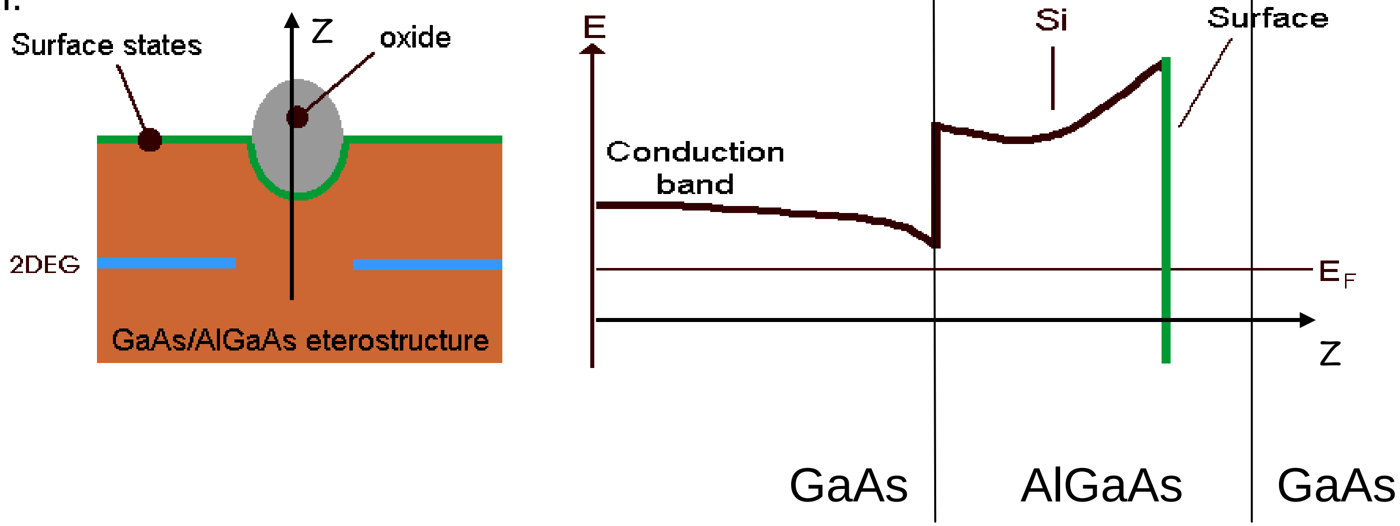
The penetration depth is 1.0-1.5 times the oxide height.

LAO on GaAs/AlGaAs

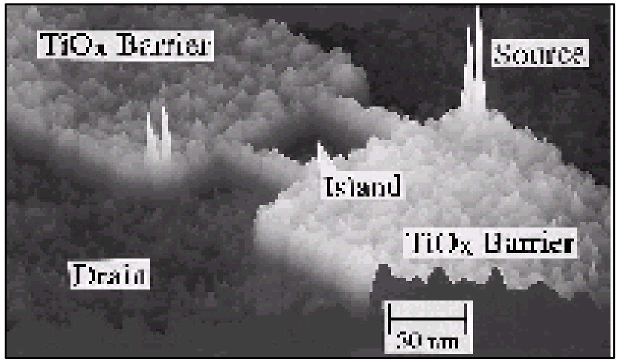
Before oxidation:



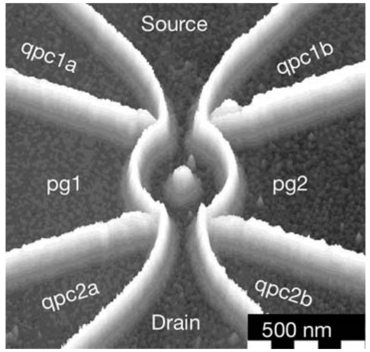
After oxidation:



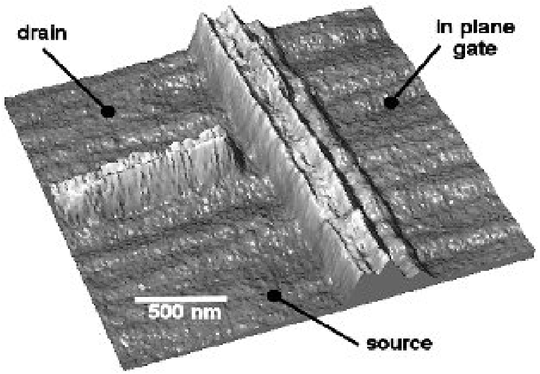
Devices made with LAO



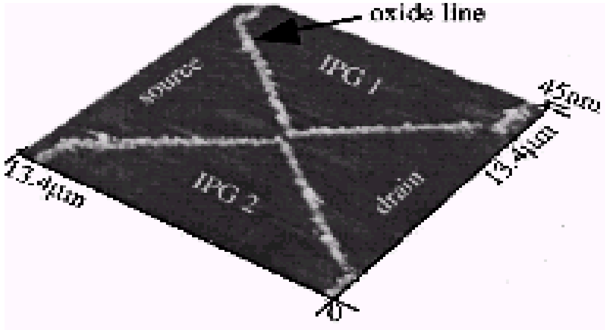
K. Matsumoto *et al*.,
APL **68** (1996) 34.



A. Fuhrer *et al*.,
Nature **413** (2001) 822.



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



R. Held *et al*.,
APL **73** (1998) 262.

Setup for Lithography on GaAs

voltage source

Hygrometer

AFM
camera

Plexiglass hood

AFM

$N_2 + H_2O$

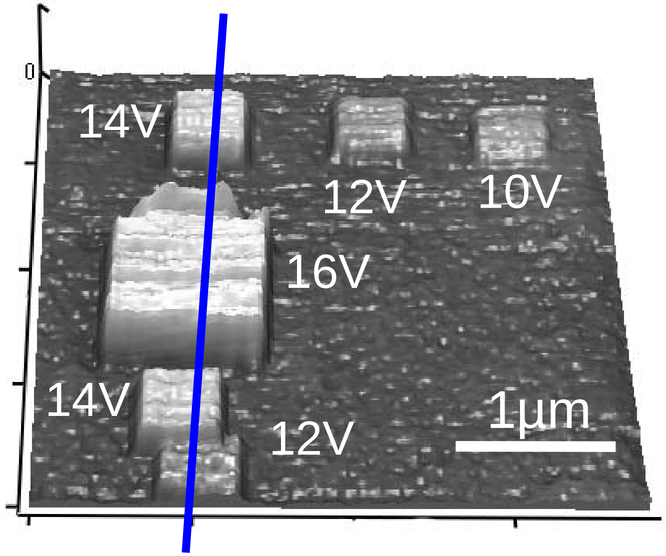
water bottle

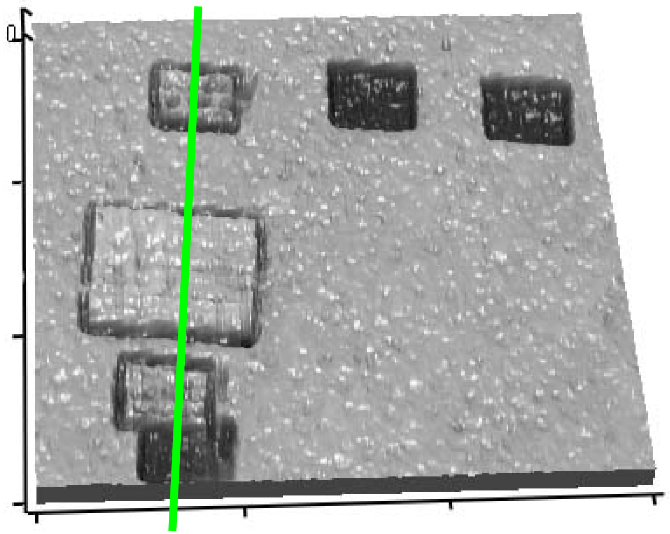
Thermomicroscope Microcope CP-Resource

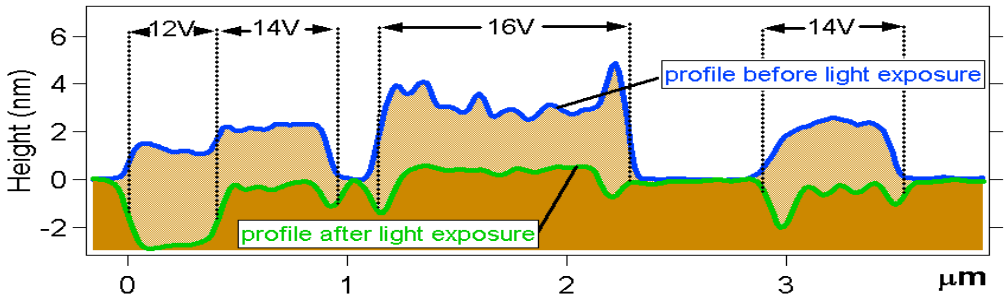
GaAs LAO-Oxide: Desorption

Before photon exposure

After hours of exposure

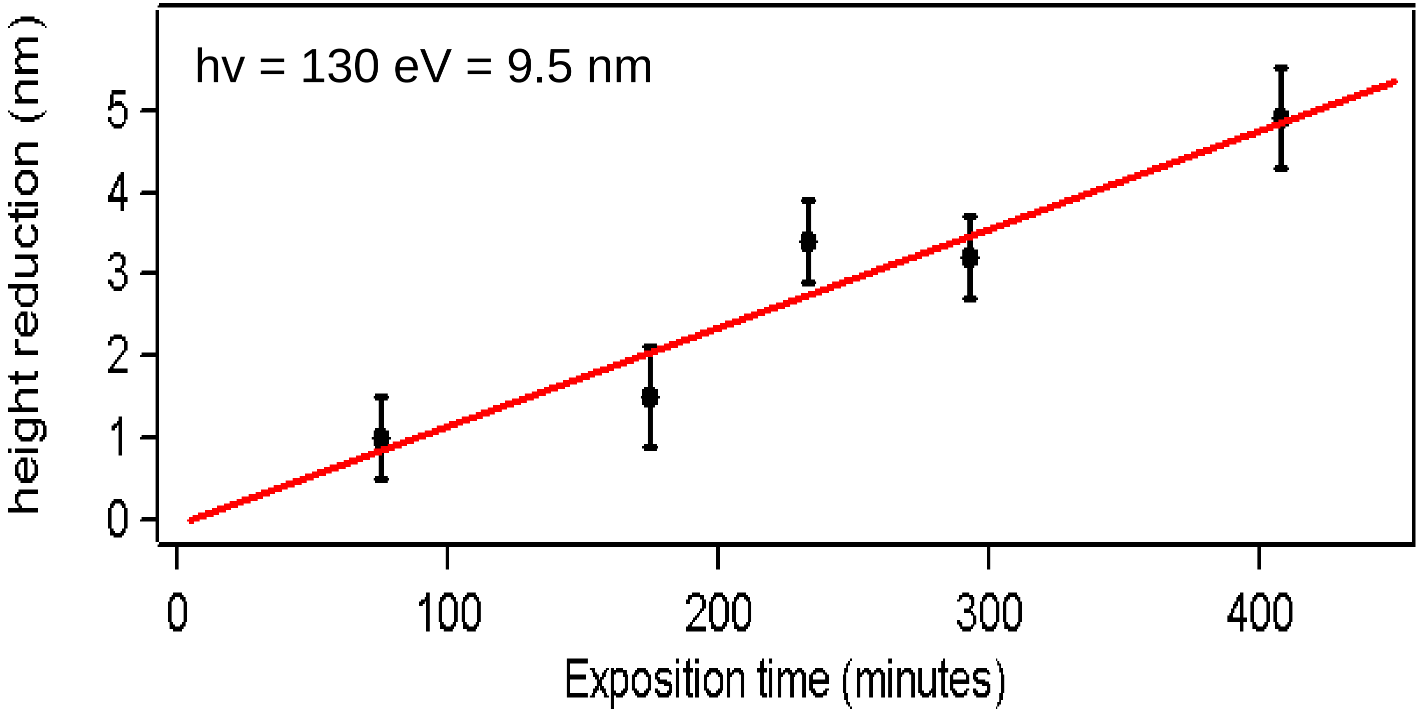






D. Ercolani et al.: Adv. Funct. Mater., in press.

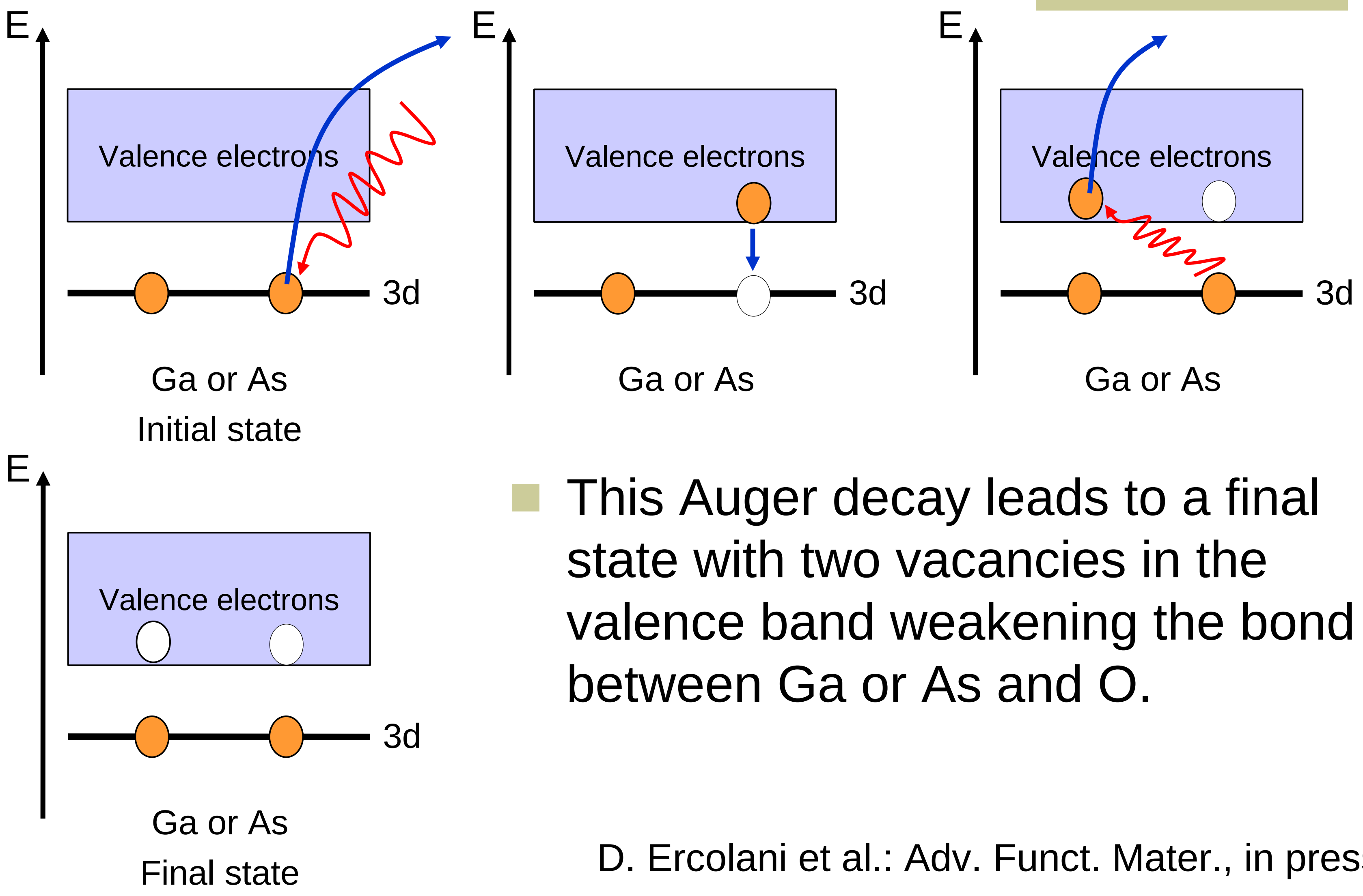
Height reduction vs. exposure time



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

D. Ercolani et al.: Adv. Funct. Mater., in press.

The Knotek-Feibelman mechanism

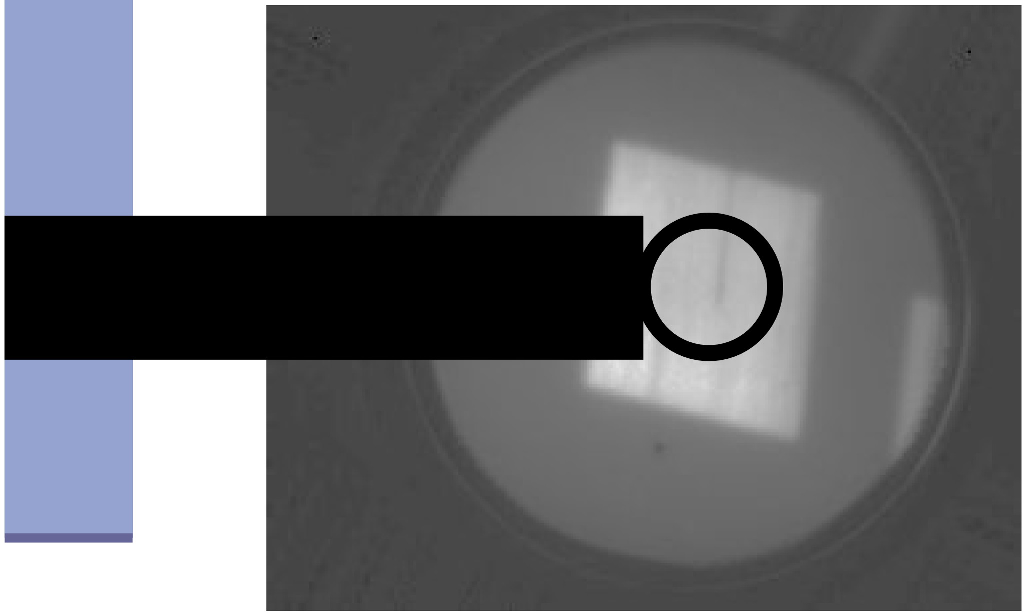


- This Auger decay leads to a final state with two vacancies in the valence band weakening the bond between Ga or As and O.

D. Ercolani et al.: Adv. Funct. Mater., in press.

Spectra From GaAs LAO-Oxide

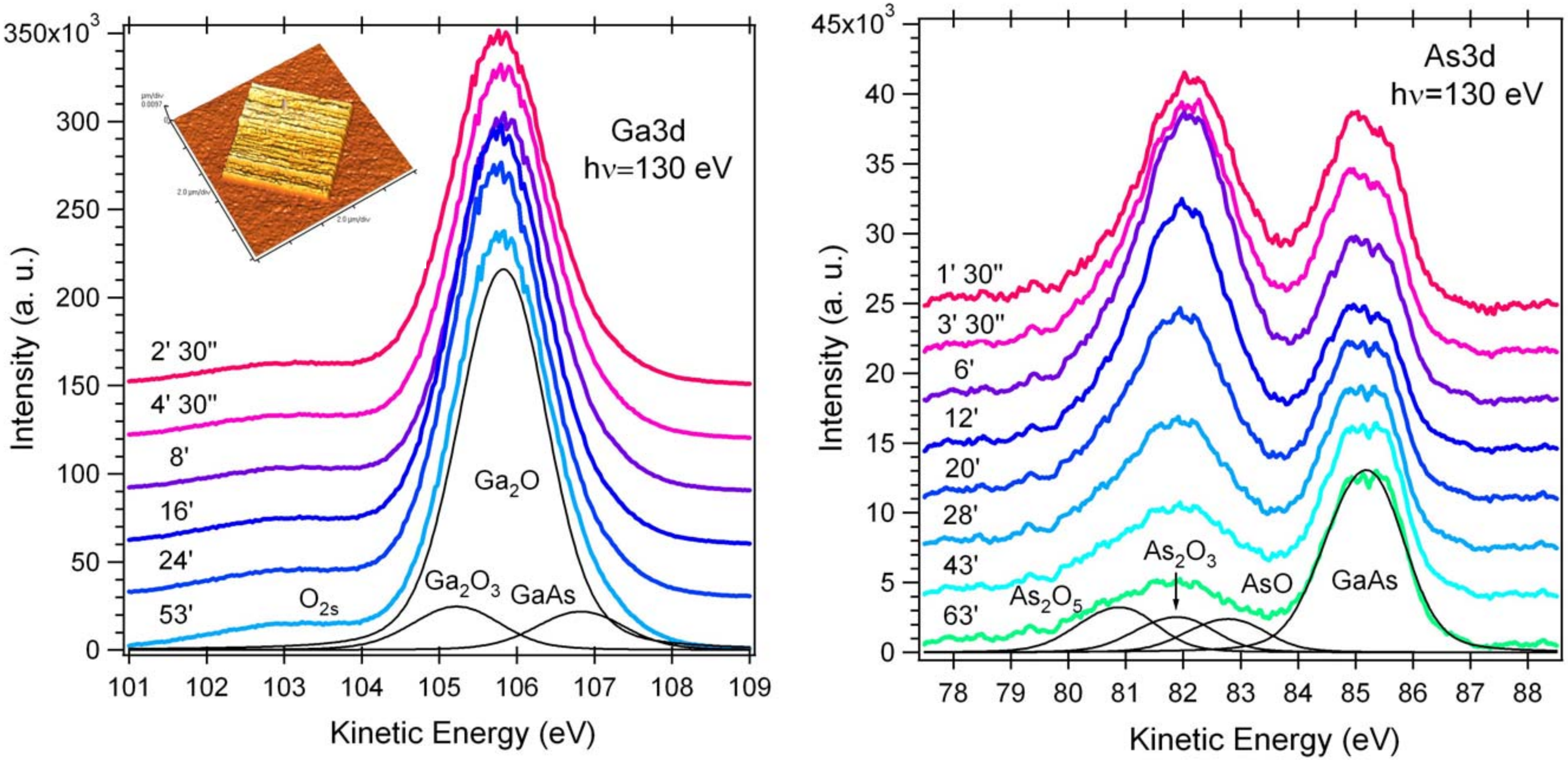
Time resolved spectroscopy with SPELEEM using Dispersive Plane (hv = 130 eV)



- 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

G. Mori et al.: J. Appl. Phys., submitted.

DP-Spectra From GaAs LAO-Oxide

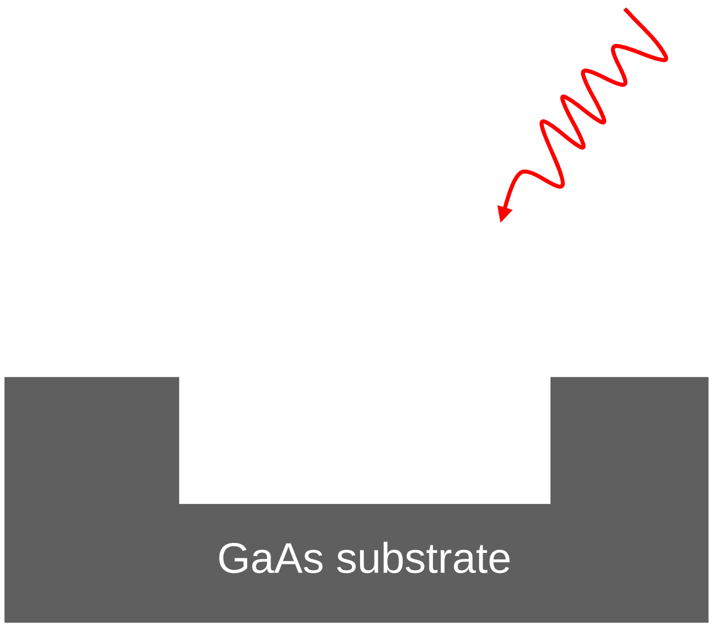


- Ga 3d remains unchanged, As-oxides signal disappeares with time.
- Ga-oxides mainly Ga_2O with small contribution of Ga_2O_3.

G. Mori et al.: J. Appl. Phys., submitted.

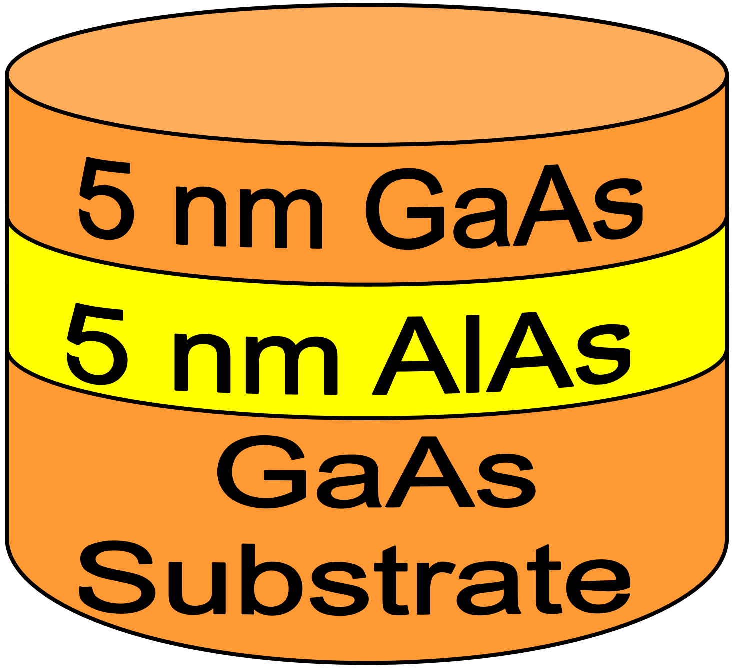
Chemistry of the GaAs LAO-Oxide

- Photon assisted partial desorption of the AFM-grown oxide was observed.
- The AFM-oxide is mainly composed of Ga_2O, with a small fraction of Ga_2O_3 and As-oxides.
- The shape of the Ga peak does not change with exposure time (early stage of desorption), however, Ga-oxides desorb.
- All As-oxides desorb completely. No As-oxides detected after some hours of exposure.
- The As-oxides are located only at the surface.
- Evidence for the presence of unoxidized GaAs in the LAO-oxide.
- Chemical composition does not depend on writing bias.

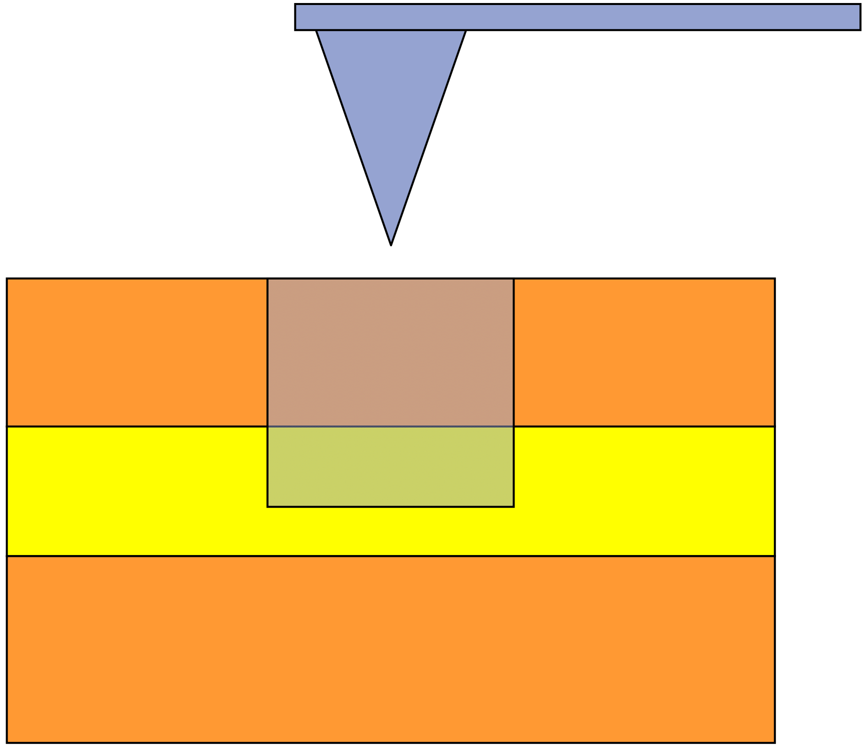


G. Mori et al.: J. Appl. Phys., submitted.

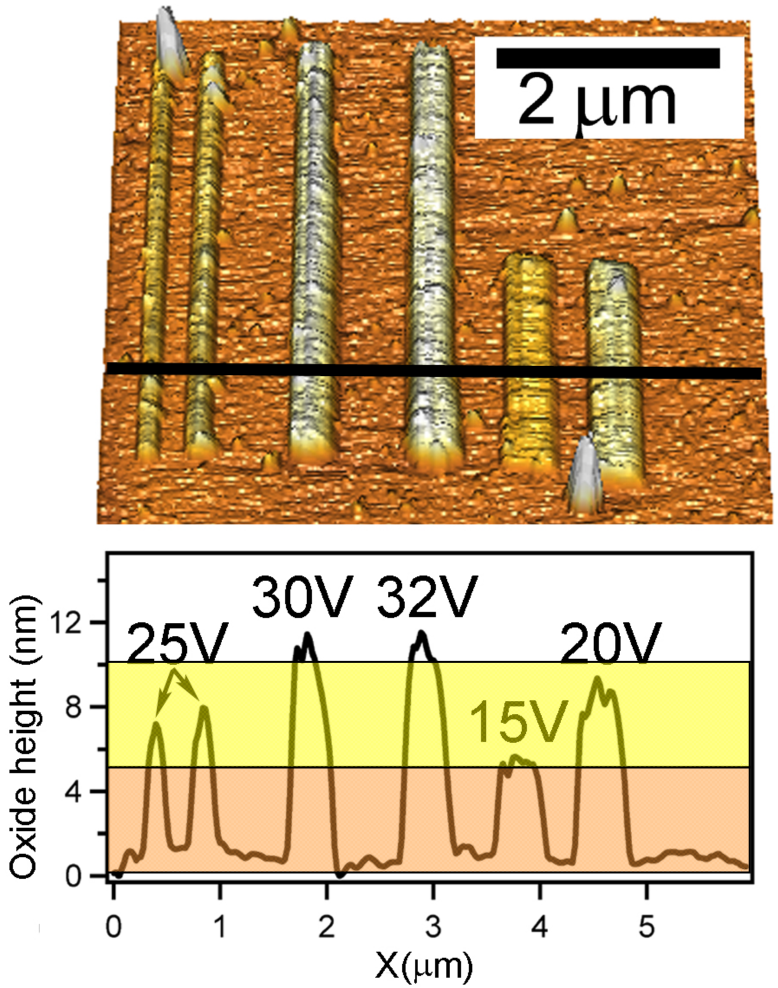
LAO on III-V Heterostructures



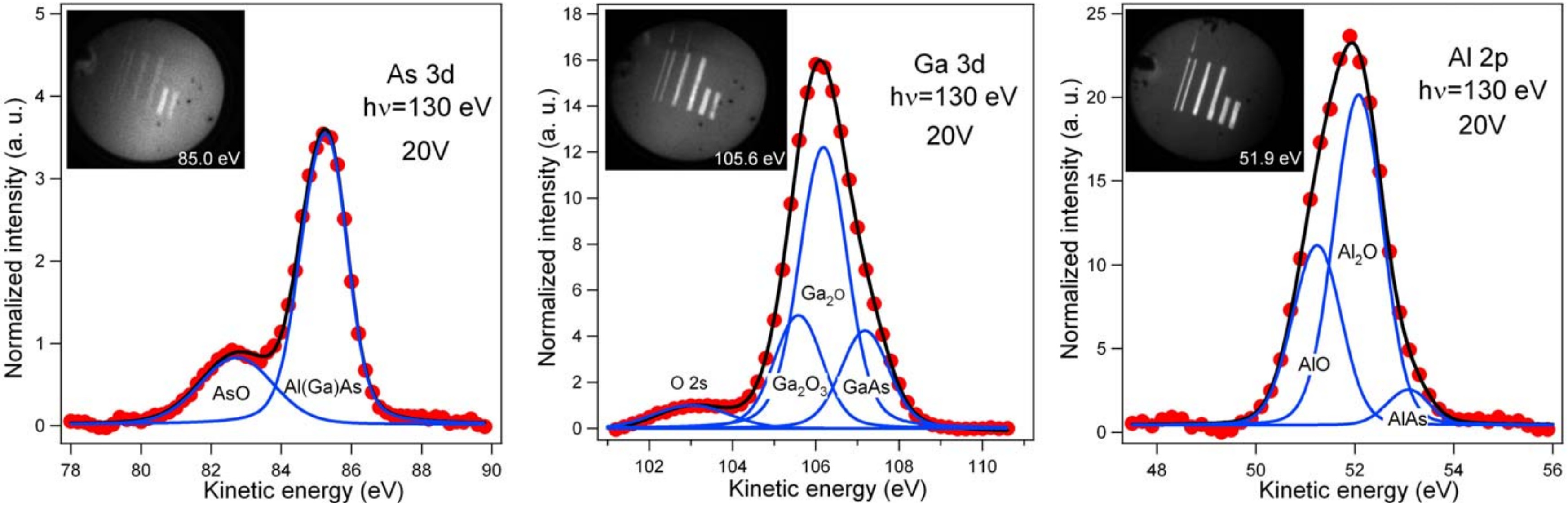
Sample I



AFM-LAO on Sample I

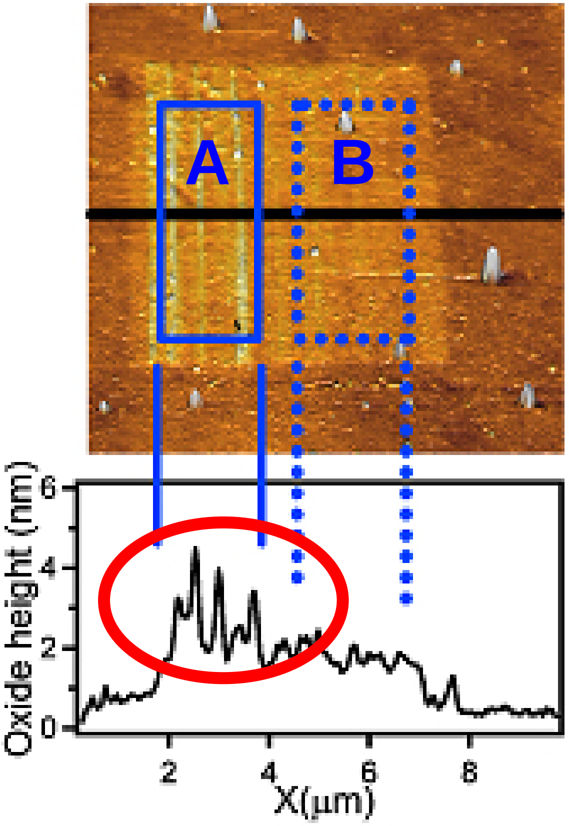


XPEEM Core Level Spectra

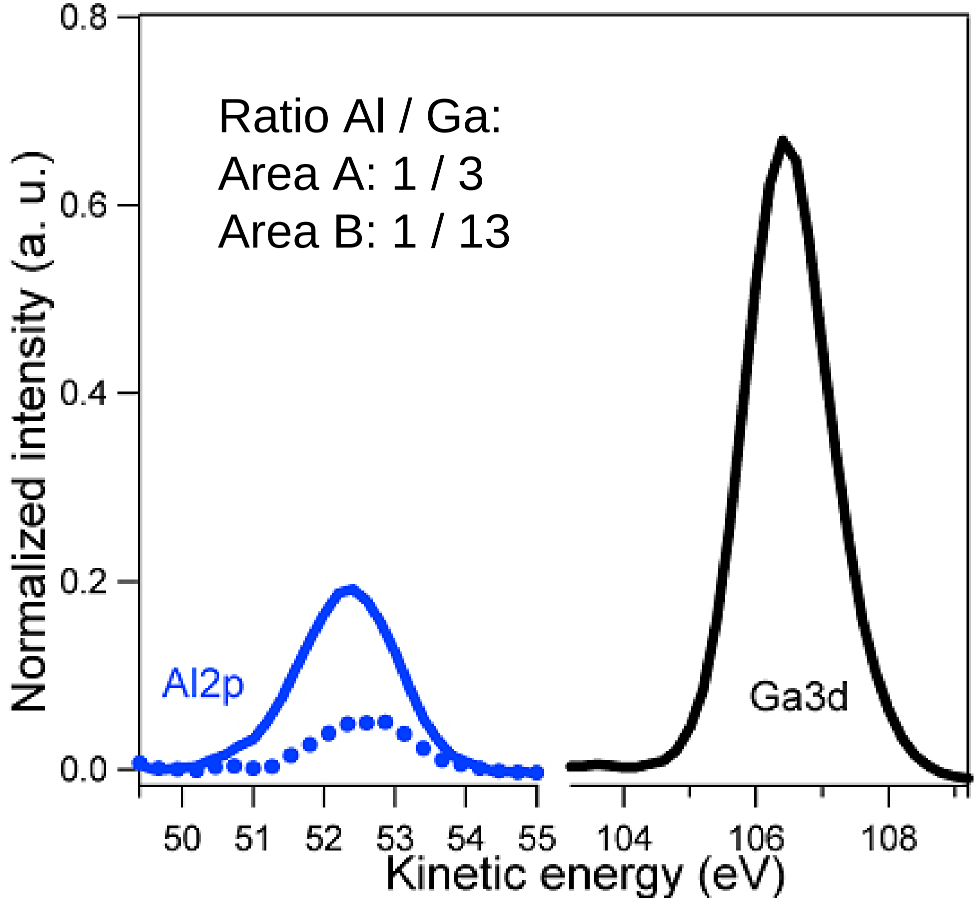


- Aluminium observed at the surface of the LAO oxide
- Unoxidized regions: No Al 2p signal
- Main component: Al_2O
- No chemical difference between different bias

Shallow Oxidations



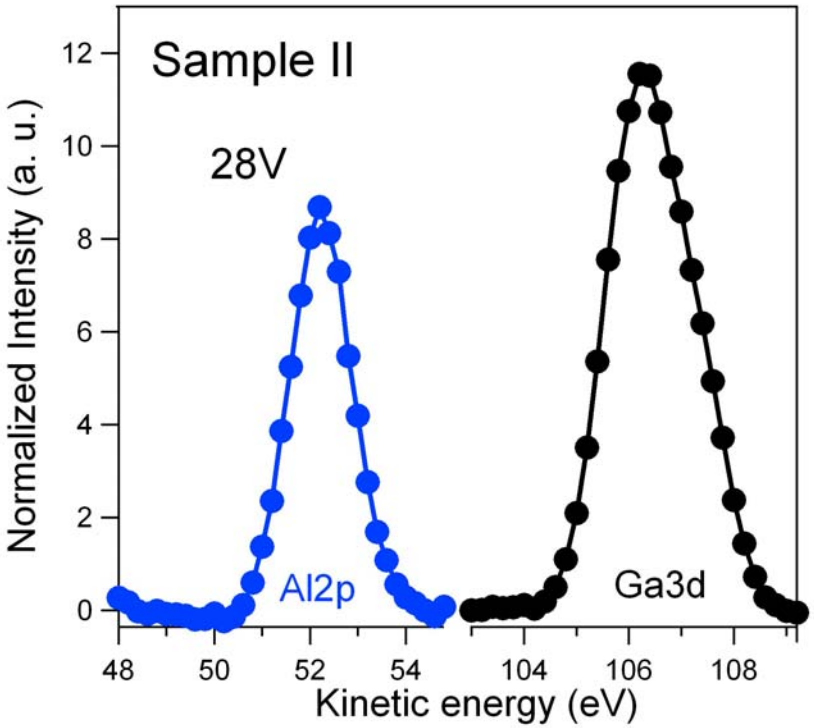
Average oxide height:
Area A: 1.5 ± 0.6 nm
Area B: 1.0 ± 0.2 nm



Surface Roughness might be important !

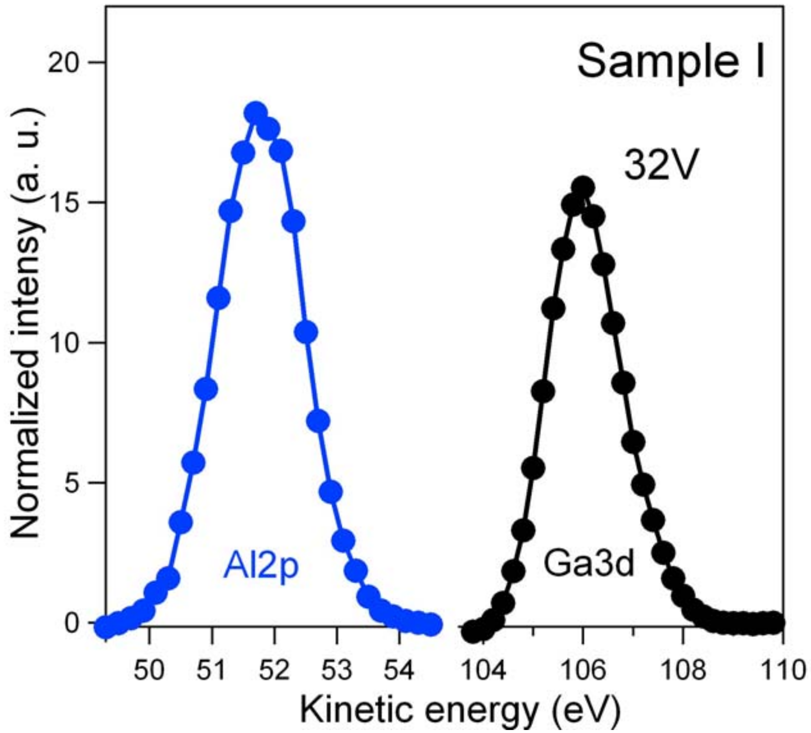
Variation in Marker Thickness

2 nm AlAs
(h = 9.1 ± 1 nm)



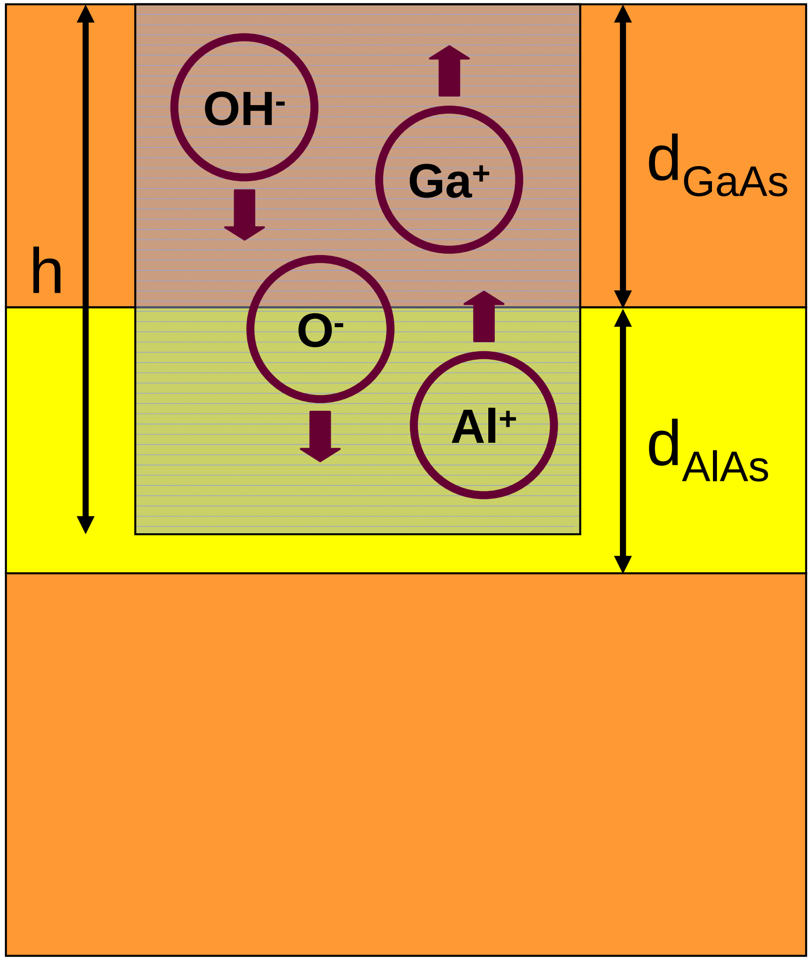
Ratio Al / Ga: 0.57

5 nm AlAs
(h = 8.9 ± 0.9 nm)



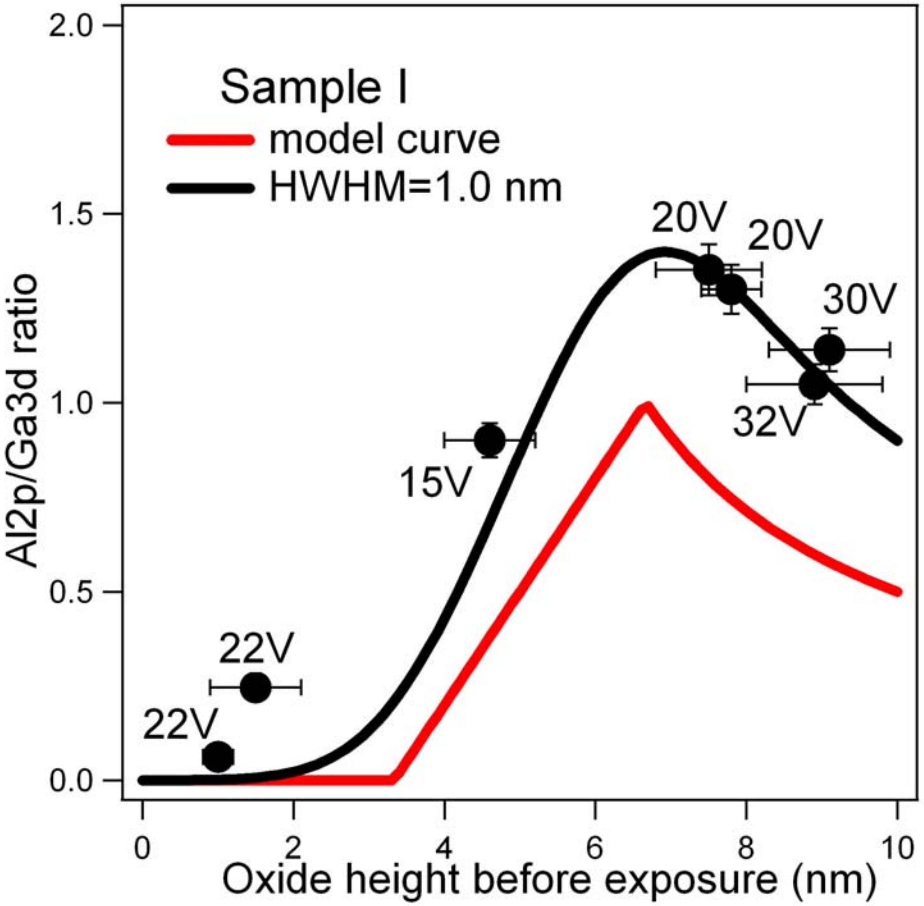
Ratio Al / Ga: 1.12

Refined Model



- Diffusion of oxygen-rich ions **plus** substrate ions
- Homogeneous mixing of components
- Ratio Al / Ga:
  - 0 for $h < d_{GaAs}$
  - $(h - d_{GaAs}) / d_{GaAs}$ for $d_{GaAs} < h < d_{GaAs} + d_{AlAs}$
  - $d_{AlAs} / (h - d_{AlAs})$ for $h > d_{GaAs} + d_{AlAs}$
- Surface and interface roughness have to be considered!

Comparison to Experiment



Al intensity too high.

⬇

Normalization Factor.

Average RMS roughness of the LAO-oxide: 1 nm.

⬇

Convolution with Gaussian curve.

Summary

- Presence of Al-oxides in the topmost layers of GaAs/AlAs/GaAs heterostructures detected.
- Classical model of LAO process has to be revised.
- Alternative model includes diffusion of ionized substrate atoms to the surface.
- Good agreement between model and experiment.
- Al diffusion might be faster than Ga diffusion.