

# AFM Nanolithography Studied by Spectromicroscopy

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# Outline

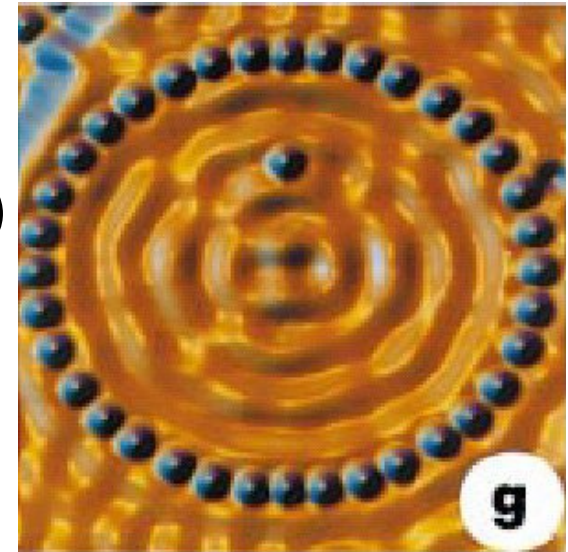
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- AFM local anodic oxidation
  - How to fabricate nanodevices with AFM
- Spectromicroscopy
  - How to measure chemical properties with lateral resolution on a nanometer scale
- Si oxides
- GaAs oxides

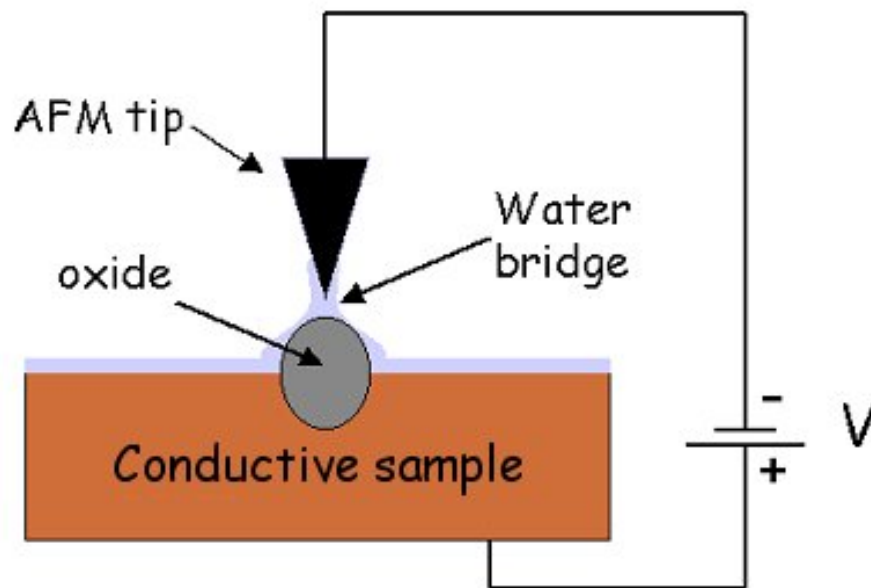
# Motivation

- Nanolithography 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)

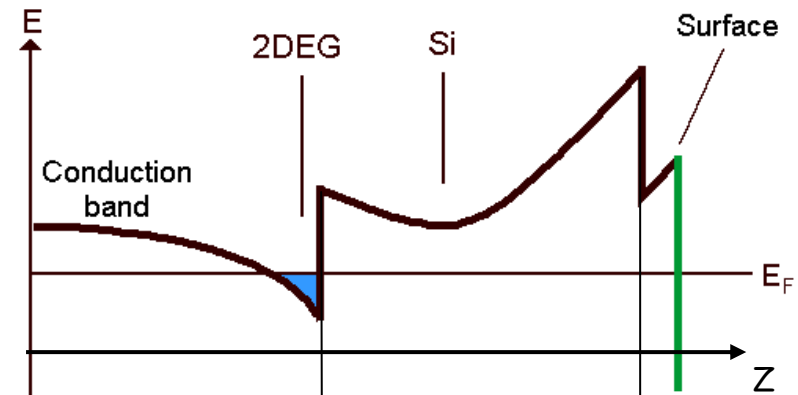
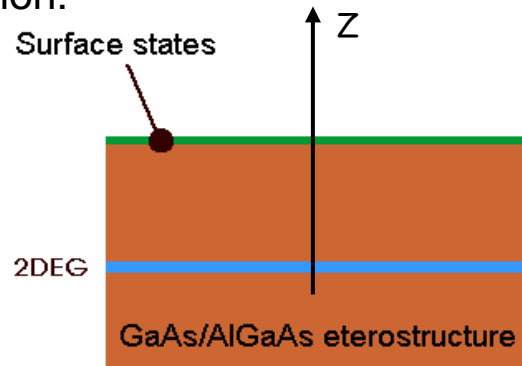


- Water electrolysis  
 $\text{H}_2\text{O} \rightarrow \text{H}^+ + \text{OH}^-$ .
- $\text{OH}^-$  groups migrate towards the sample.
- Oxide penetration induced by the intense local electric field.

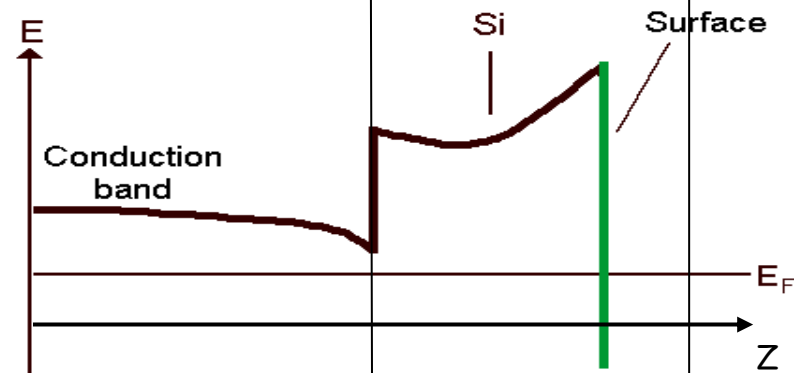
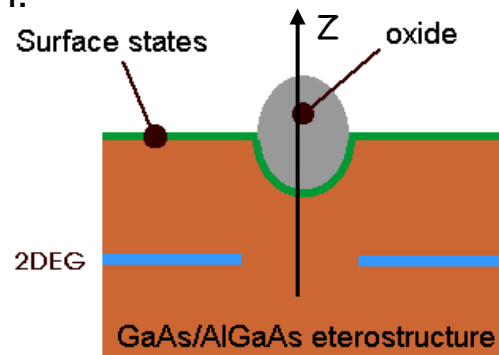
Versatile tool at relatively low cost  
High lateral resolution but small area

# LAO on GaAs/AlGaAs

Before oxidation:



After oxidation:

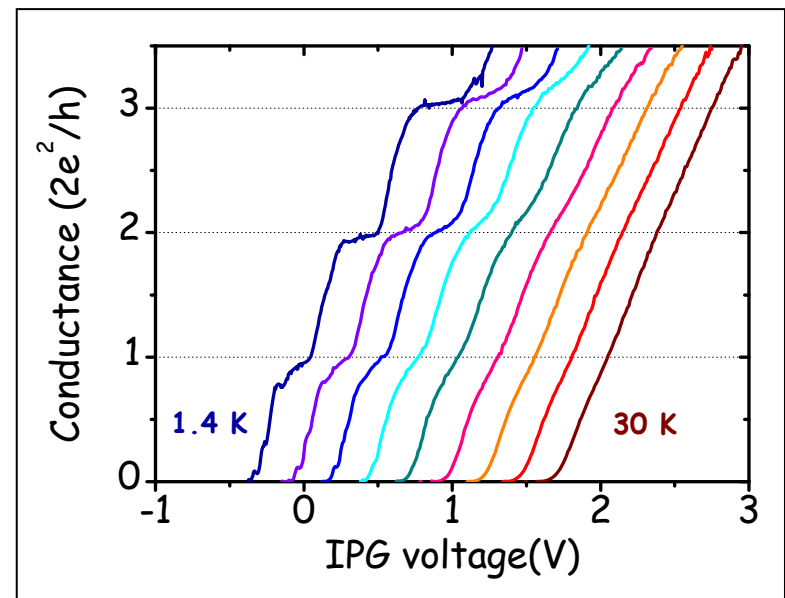
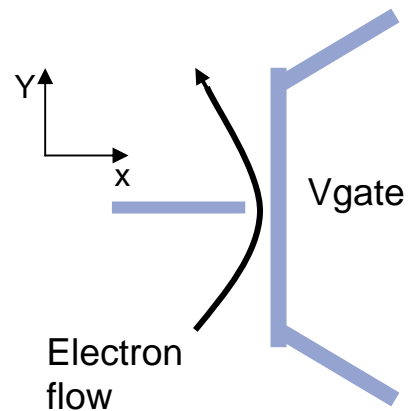
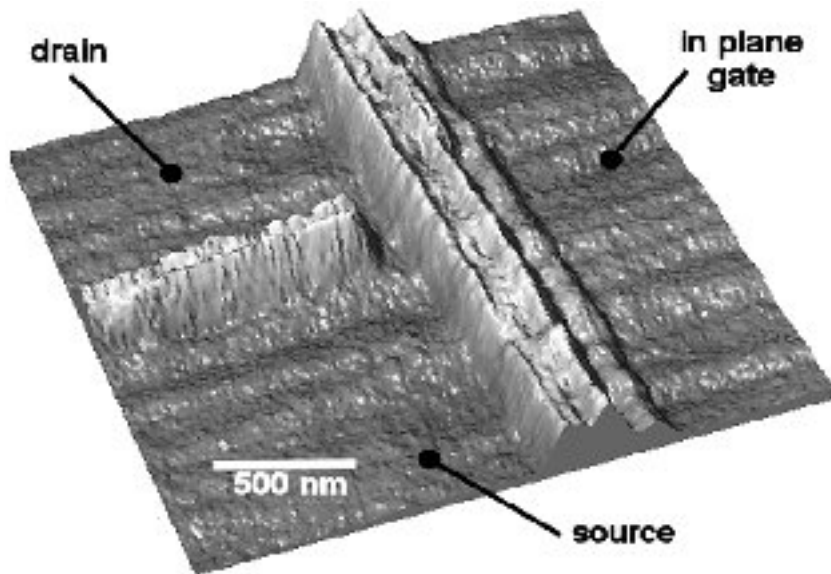


GaAs

AlGaAs

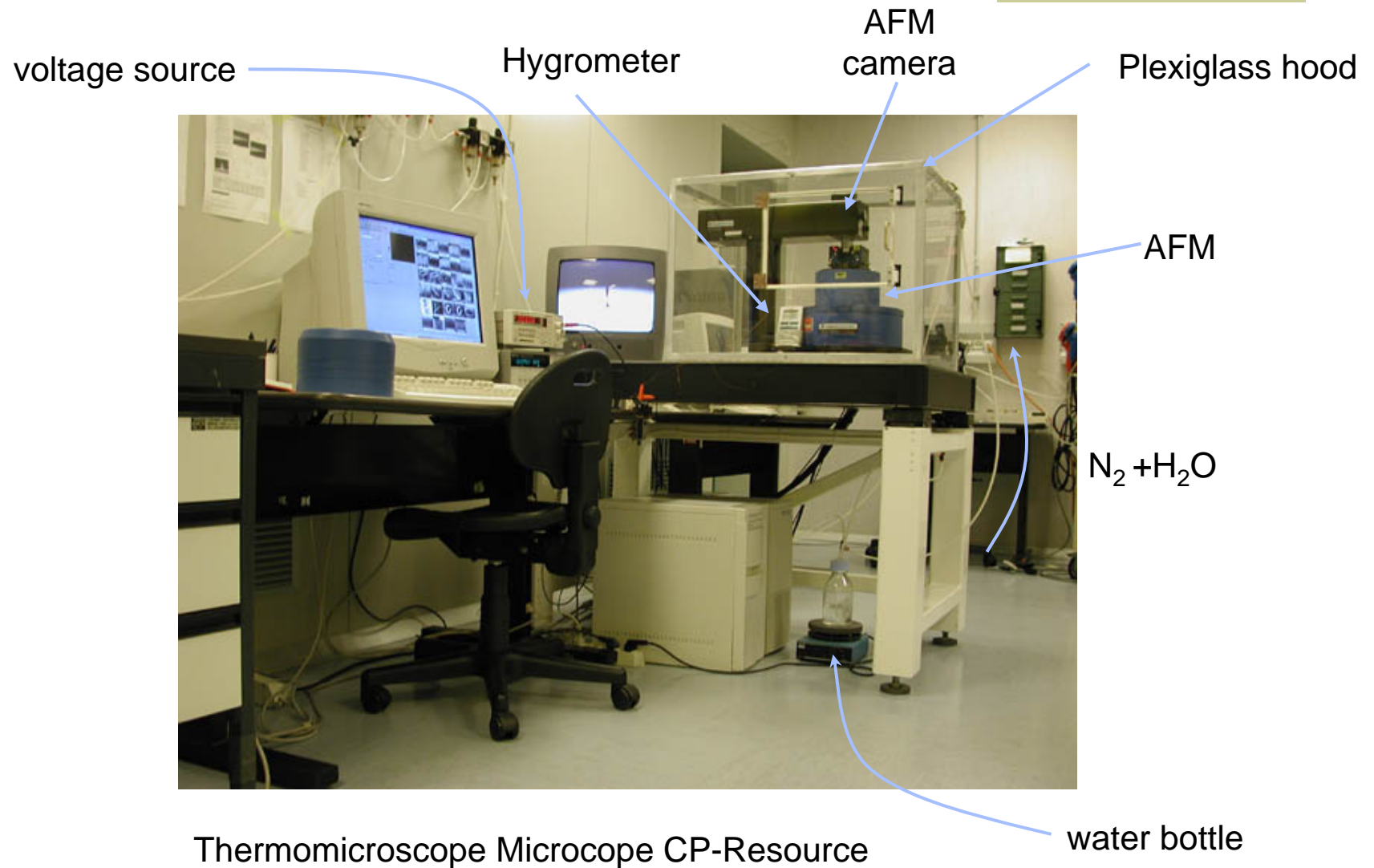
GaAs

# Quantum Point Contact



G. Mori et al, JVST B, submitted.

# Setup for Lithography on GaAs



# Sample Preparation Parameters

## ■ Si samples:

- Si(100) n-type,  $\rho = 4 \Omega\text{cm}$ .
- AFM in air.
- Contact mode.
- Si tip.
- Humidity 65%.
- Scan speed  $0.5 \mu\text{m/s}$ .
- Substrate grounded, tip voltage from  $-6 \text{ V}$  to  $-14 \text{ V}$ .

## ■ GaAs samples:

- GaAs(100) n-type,  $\rho = 2 \times 10^{-3} \Omega\text{cm}$ .
- AFM in controlled atm.
- Tapping mode.
- Si tip (B doped).
- Humidity controlled.
- Scan speed  $0.5 \mu\text{m/s}$ .
- Tip grounded, substrate voltage from  $+12 \text{ V}$  to  $+18 \text{ V}$ .



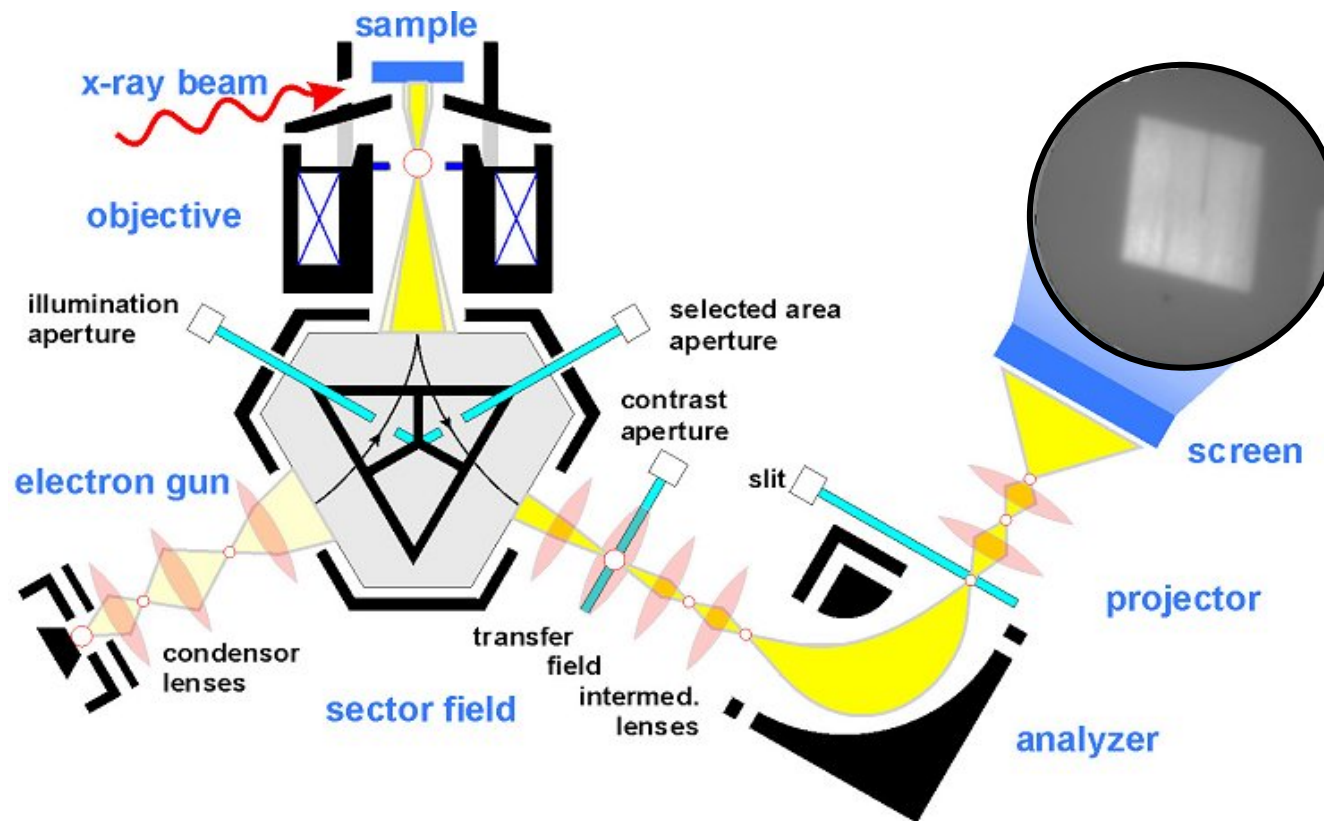
# Why Spectroscopic Microscopy?

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- Lack of information on the oxidation process and on the chemical nature of the grown oxides.
- Information on the uniformity of the grown oxides (electrical and chemical properties).
- Effect of oxidation parameters.
- Lack of reliable microscopic techniques able to perform chemical spectroscopy on such small structures.

# The SPELEEM at Elettra

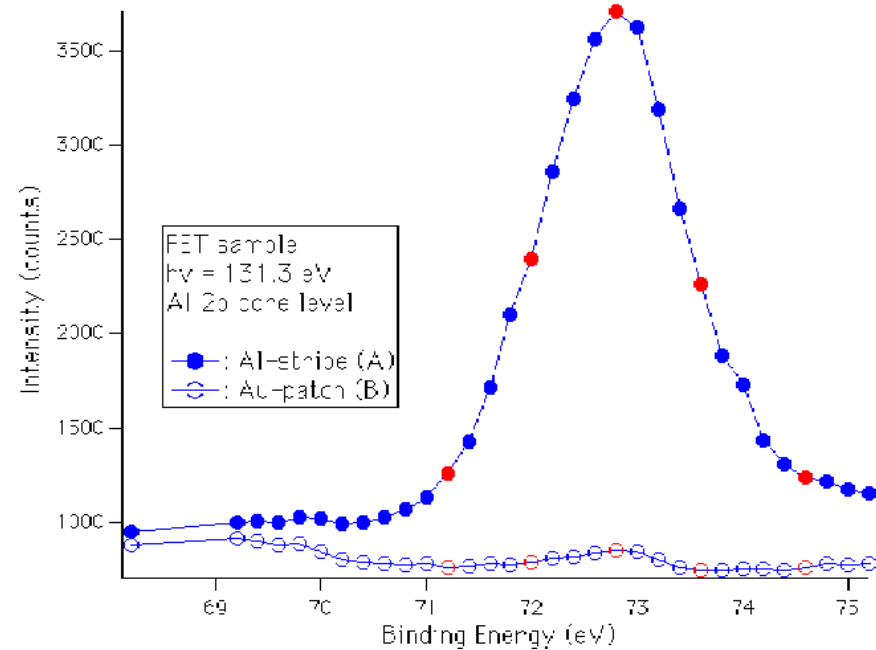
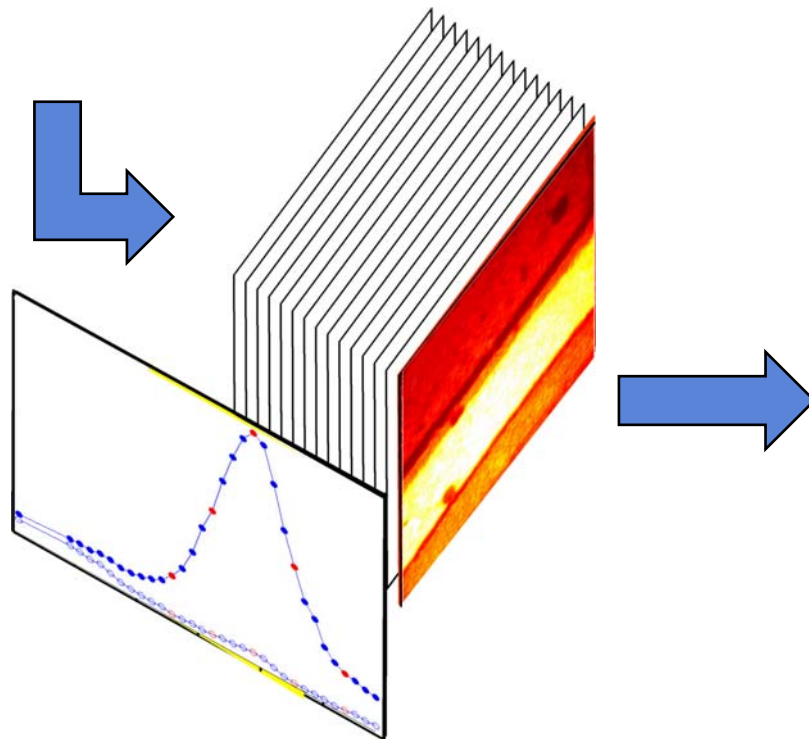
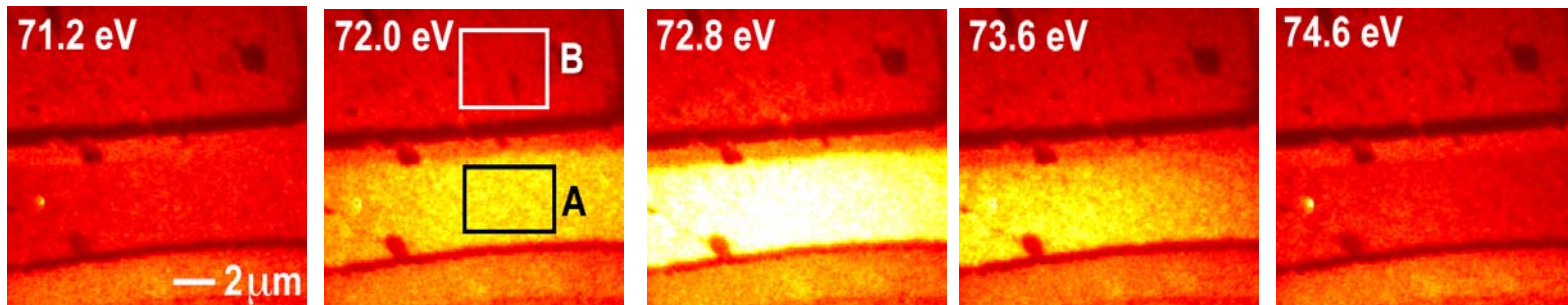
Spectroscopic photoemission and low energy electron microscope



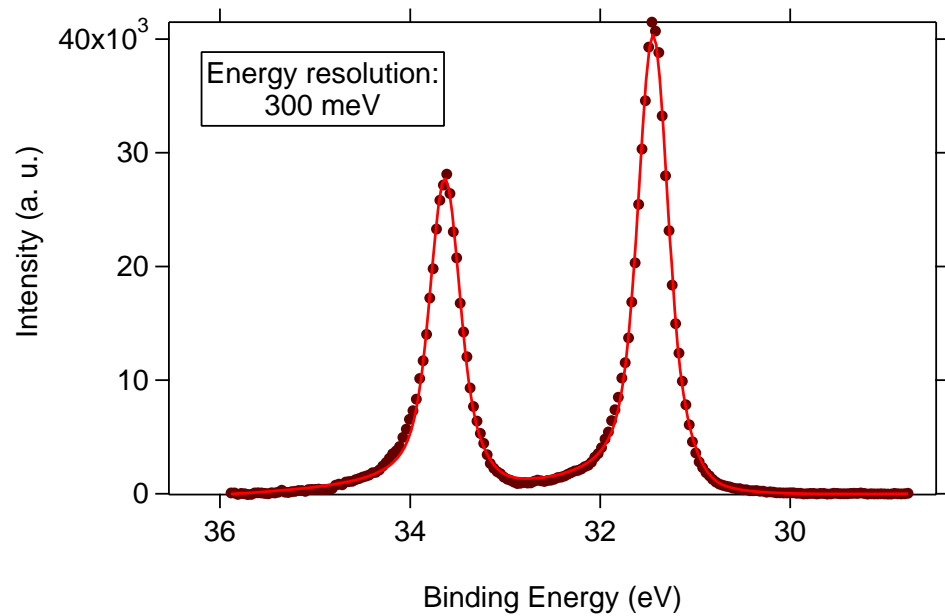
Photon energy range: 20 - 1000 eV (here: 130 eV)

Energy resolution 300 meV, lateral resolution in XPEEM 30 nm

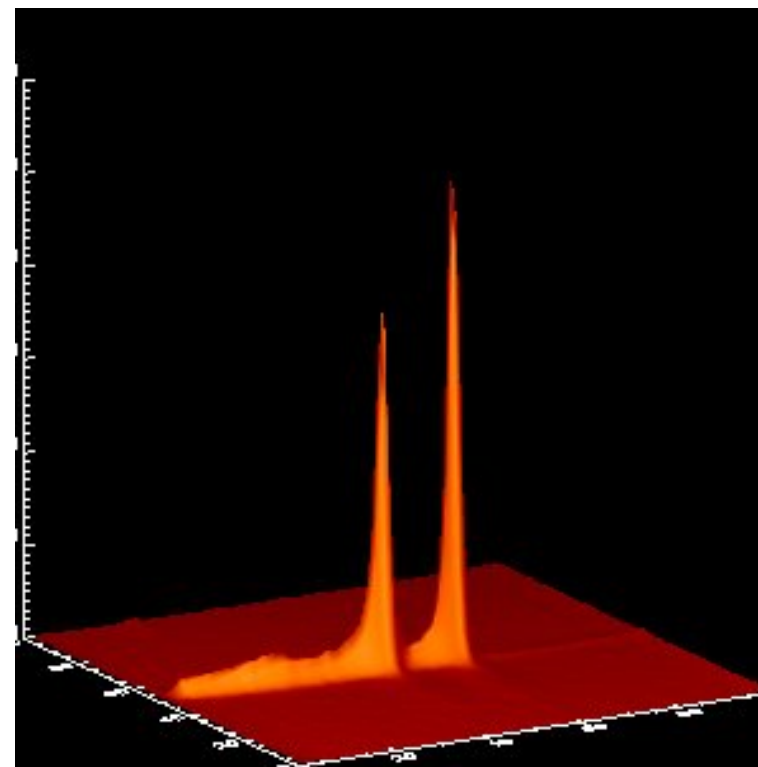
# Spectroscopy With XPEEM



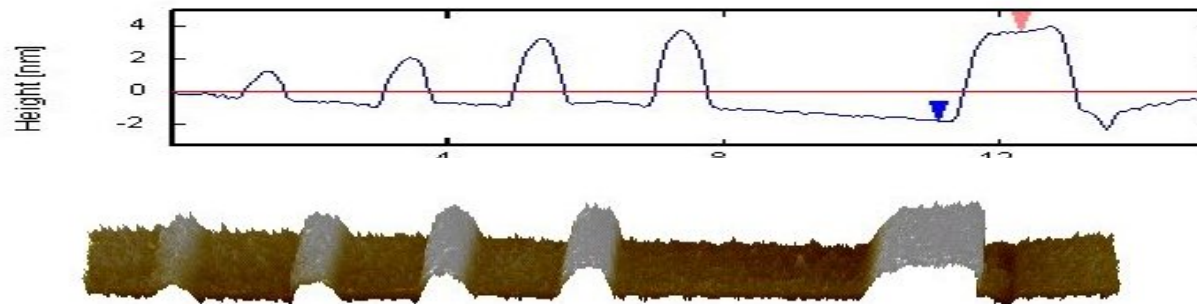
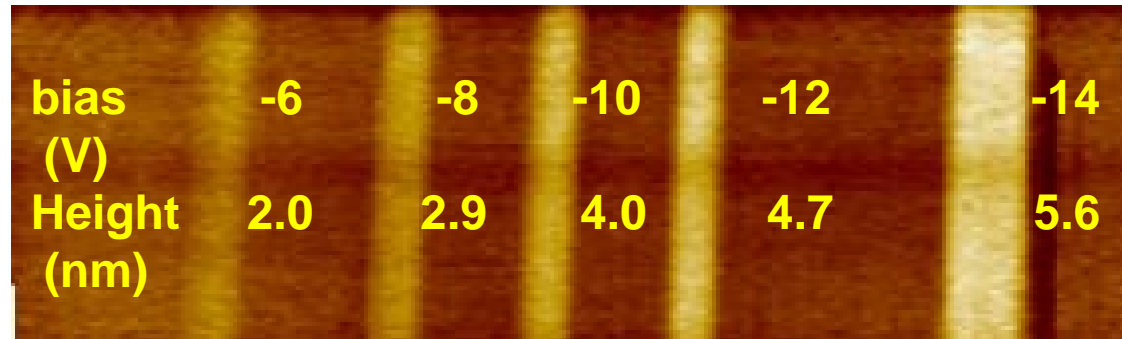
# Dispersive Plane



clean W(110),  
W 4f core level



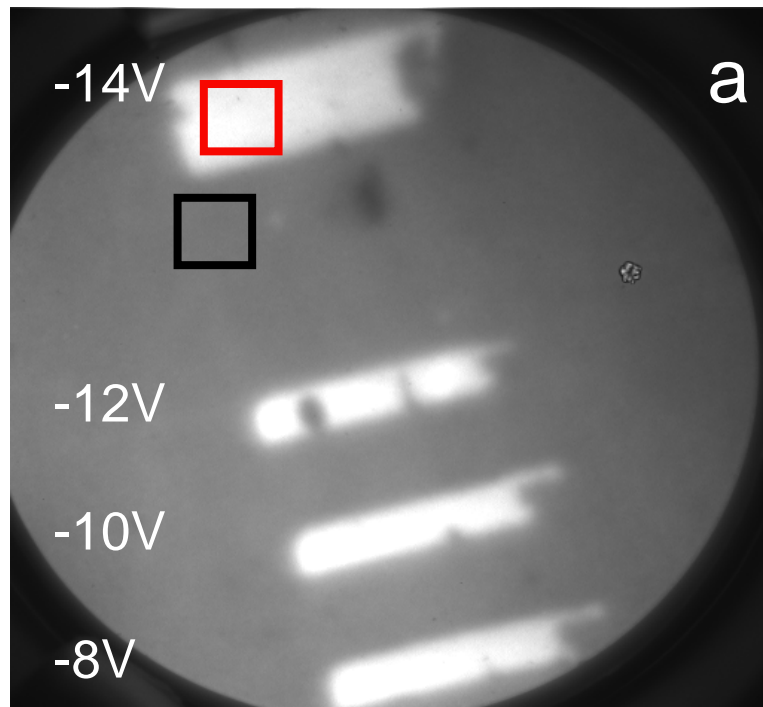
# Si Oxide: Sample



Morphology studied after oxidation by AFM.  
Thickness increases with writing voltage!

# Si Oxide: Image Contrast at Si 2p

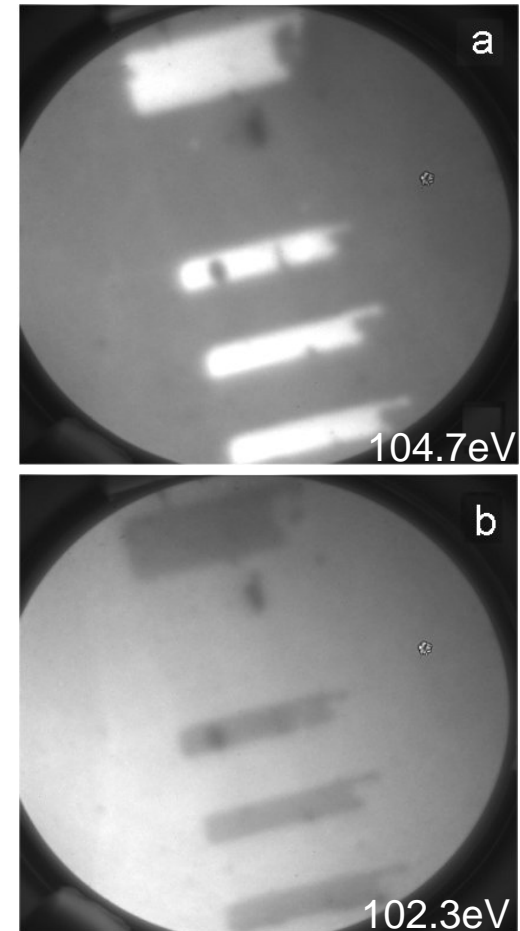
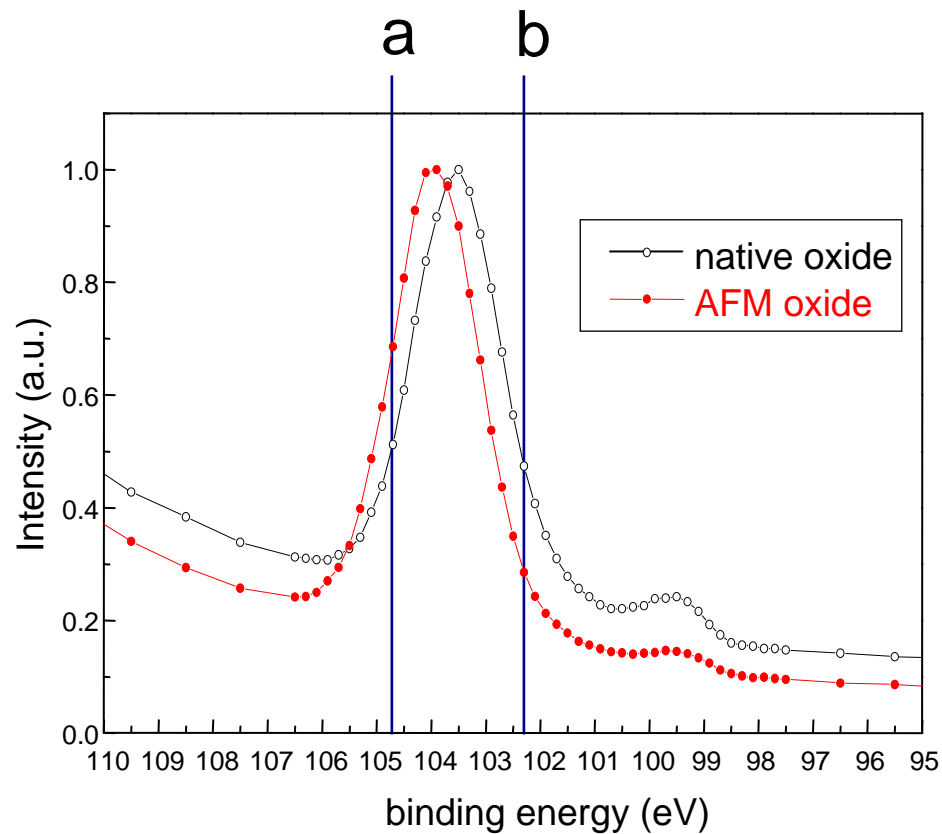
Field of view 12  $\mu\text{m}$ ,  $h\nu = 132.5 \text{ eV}$ , energy resolution: 1 eV



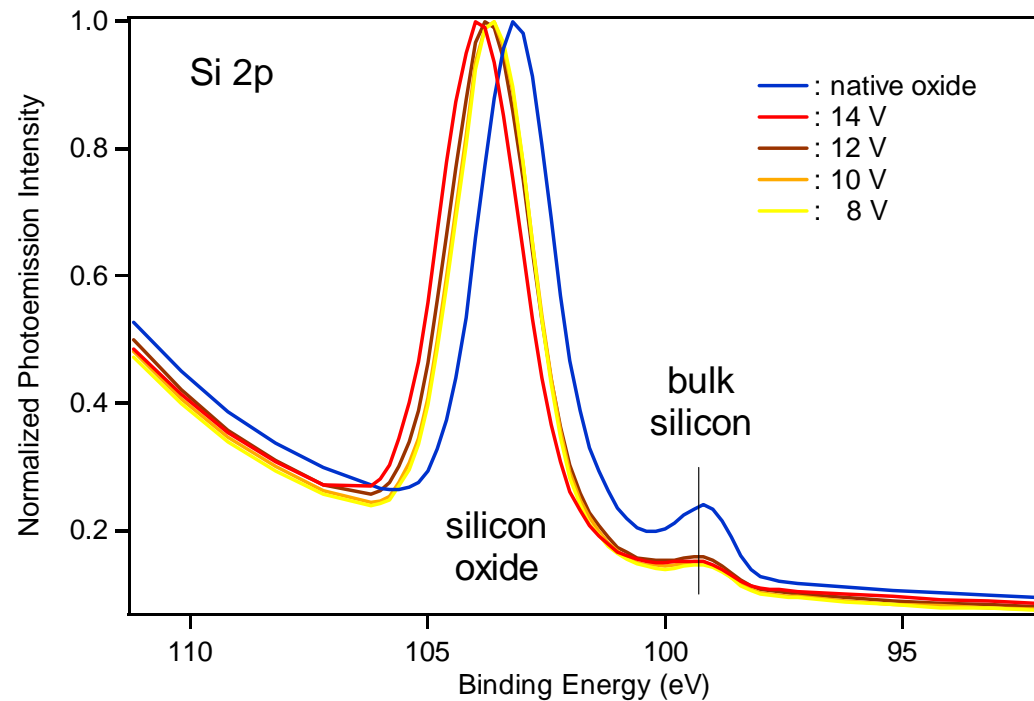
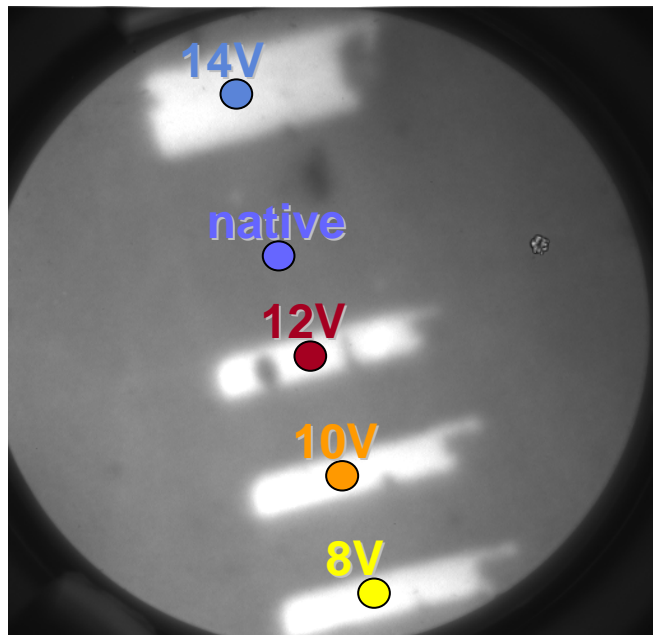
Binding energy 104.7 eV

- Contrast inversion
- Within each stripe: uniform intensity
- Unbiased tip does not induce oxidation

# Si Oxide: Spectroscopy at Si 2p



# Si Oxide: Writing Voltage Effect



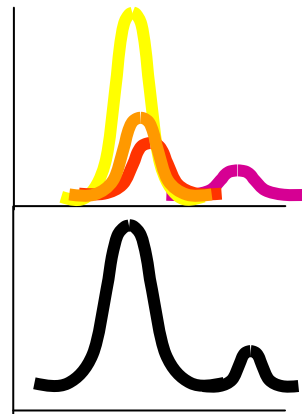
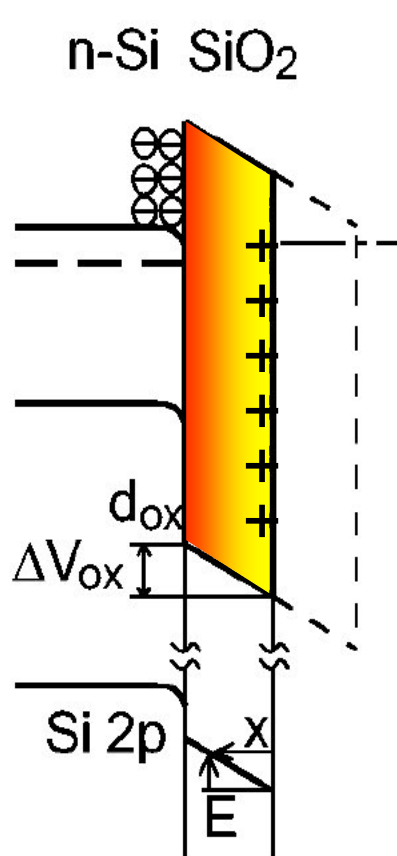
- $\Delta E = 3.97 \text{ eV}$  (native)
- $\Delta E = 4.62 \text{ eV}$  ( $U = 14\text{V}$ )
- $\Delta E = 4.46 \text{ eV}$  ( $U = 12\text{V}$ )
- $\Delta E = 4.41 \text{ eV}$  ( $U = 10\text{V}$ )
- $\Delta E = 4.39 \text{ eV}$  ( $U = 8\text{V}$ )

Shift ( $\text{Si}_{\text{bulk}} - \text{SiO}_x$ ) increases with increasing writing voltage (oxide thickness).

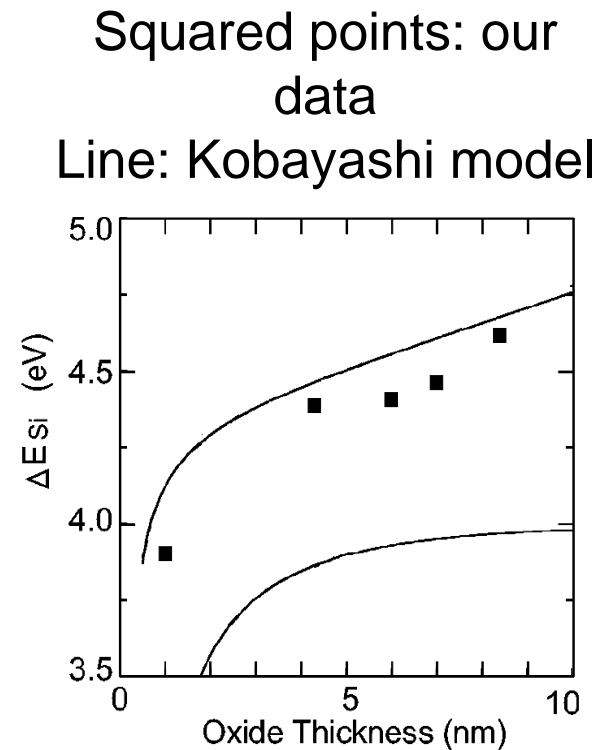


# Si Oxide: Charging Effects

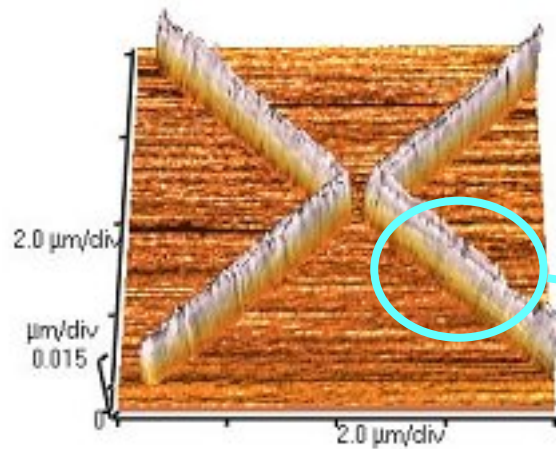
H. Kobayashi, T. Kubota, H. Kawa, Y. Nakato, and M. Nishiyama:  
Appl. Phys. Lett. **73** (1998) 933.



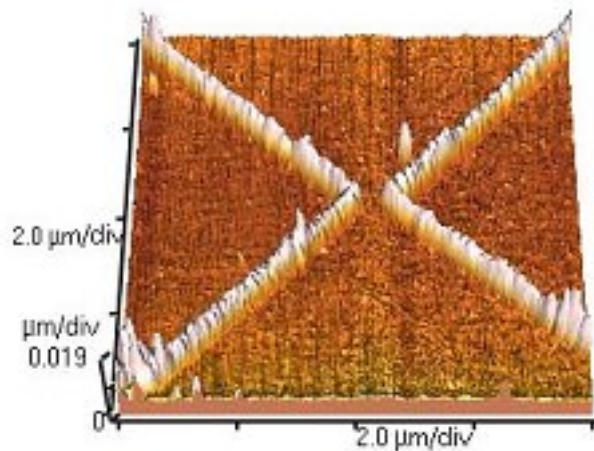
Intensity decreases  
due to escape depth  
effect  
Charged (yellow)  
layer contribution is  
more important



# GaAs Oxide: Photon Exposure



AFM before: height 18nm



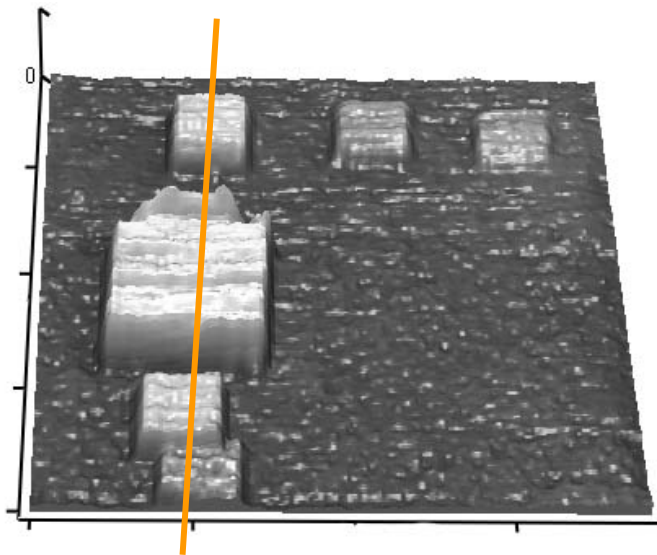
AFM after: height 13nm



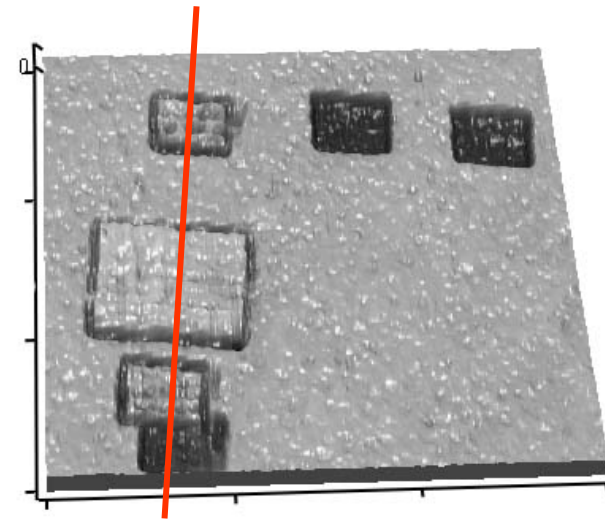
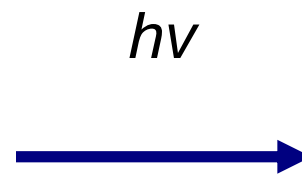
Images taken with secondary electrons

- Photon energy: 125 eV
- Kinetic energy: 4 eV
- Field of view: 10  $\mu\text{m}$
- One image every 2 sec

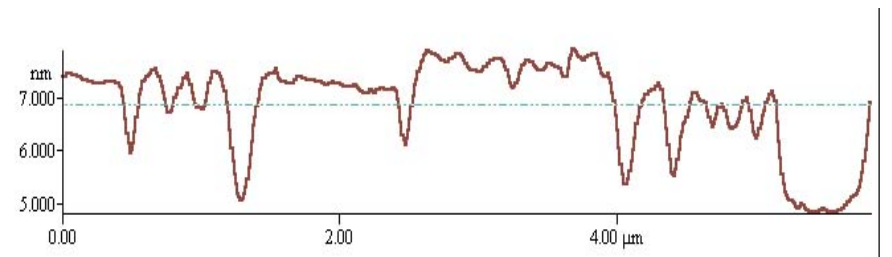
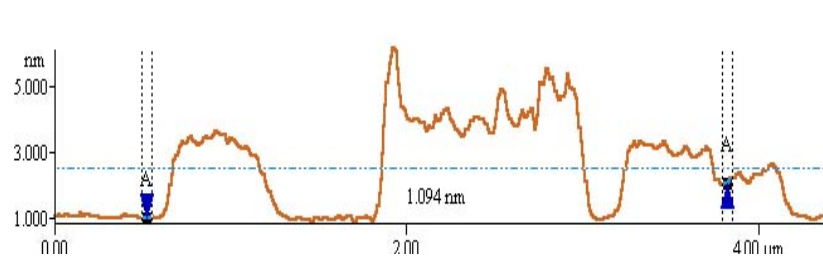
# GaAs Oxide: Desorption



AFM topography taken before photon exposure



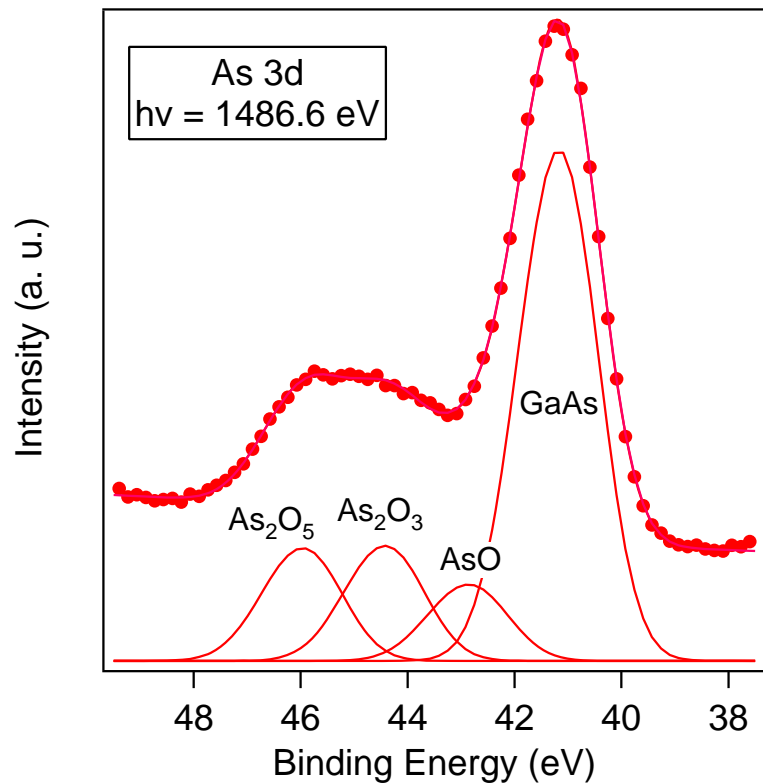
AFM topography of the same region taken after exposure



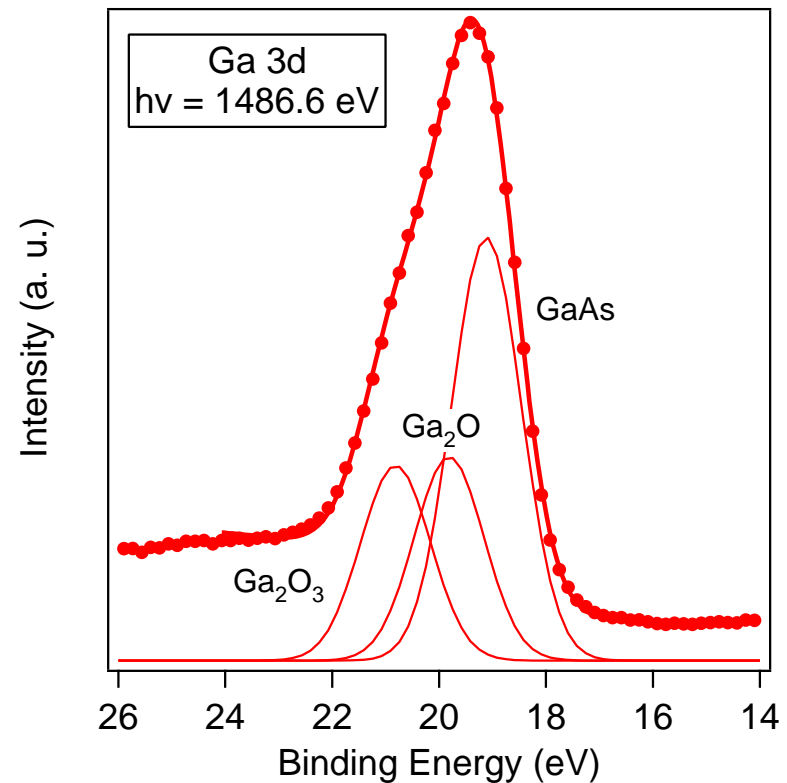
2 - 5 nm of GaAs oxide have been desorbed

# Spectra From GaAs Wafer (XPS)

X-ray Photoelectron Spectroscopy ( $h\nu = 1486.6$  eV) on native GaAs oxide



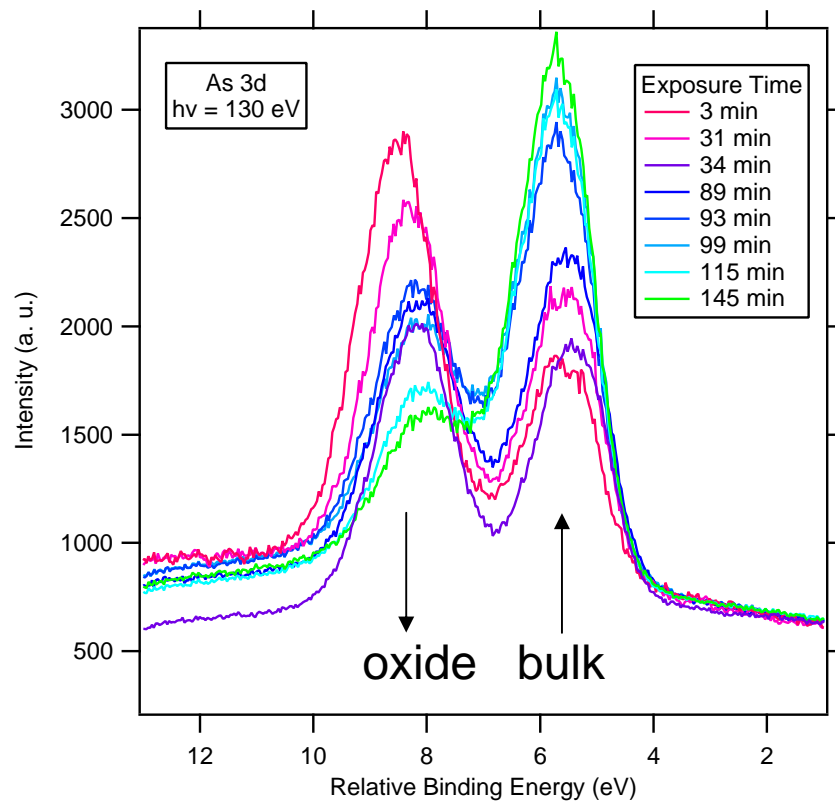
As 3d



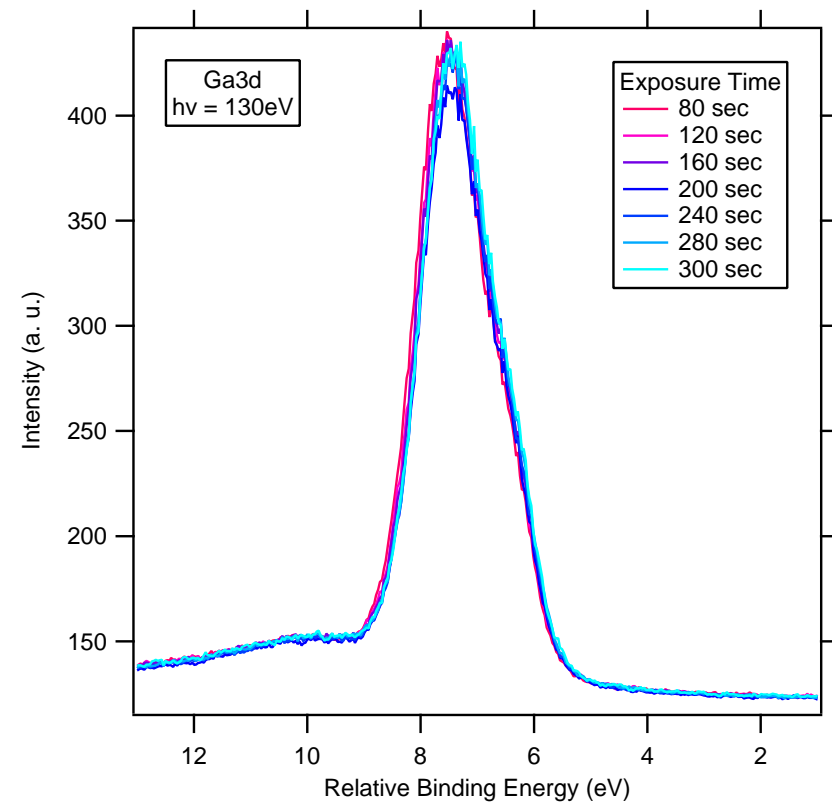
Ga 3d

# Spectra From GaAs Wafer (DP)

Time resolved spectroscopy with SPELEEM using Dispersive Plane ( $h\nu = 130$  eV)



As 3d  
total exposure time: 145 min = 8700 sec



Ga 3d  
300 sec

# Spectra From AFM GaAs Oxide

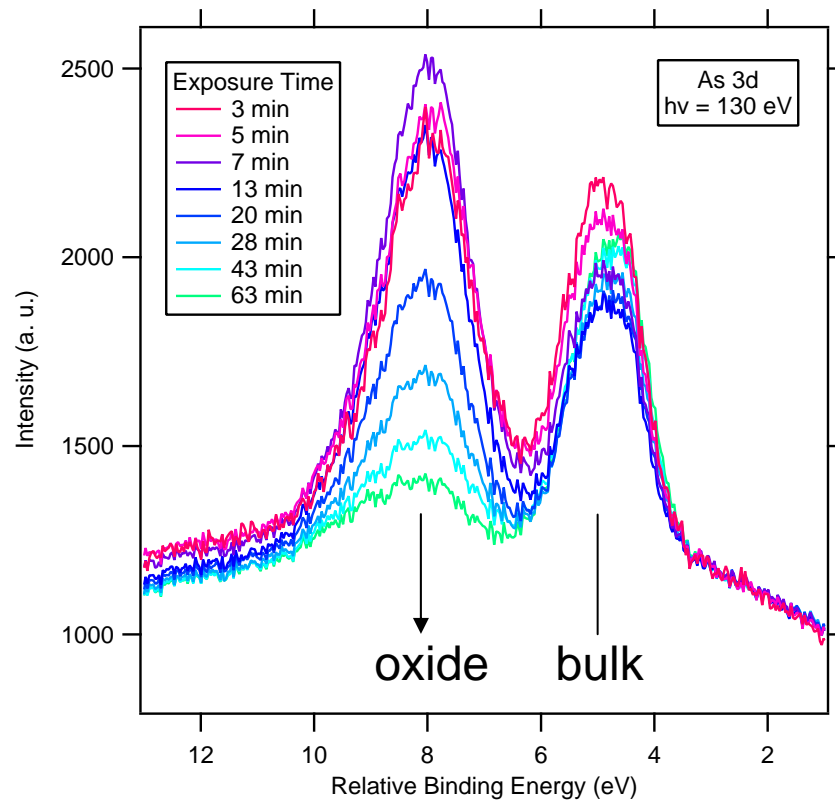
Time resolved spectroscopy with SPELEEM using Dispersive Plane ( $h\nu = 130$  eV)



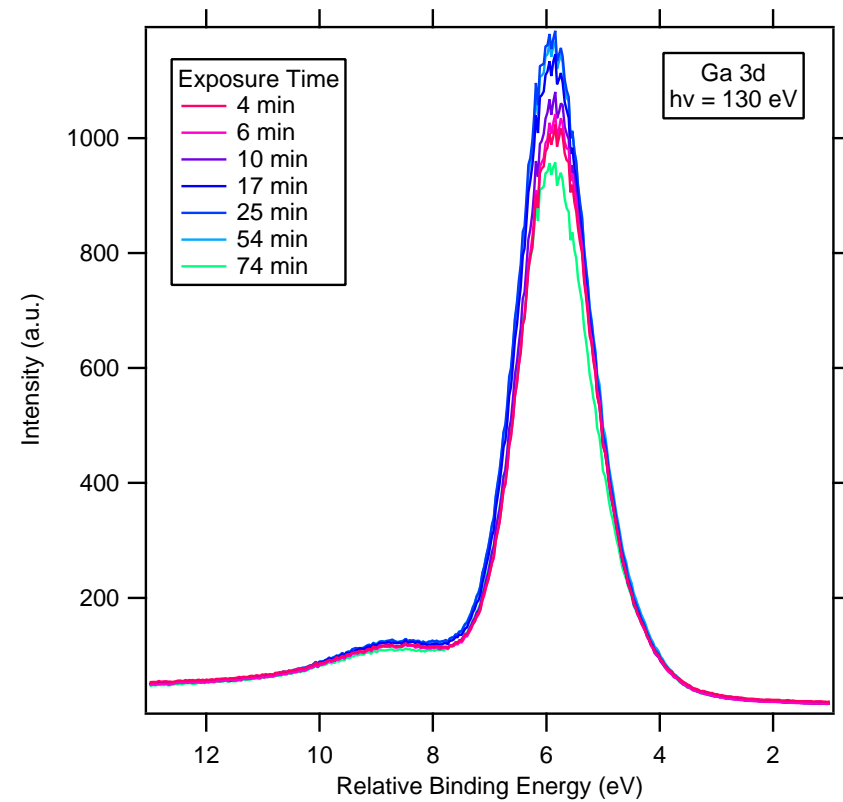
- Sample S03B
- Hole (3,2)
- Writing voltage 15 V
- Structure height 3 nm
- Image taken with secondary electrons:
  - Photon energy: 130 eV
  - Kinetic energy: 0.3 eV
  - Field of view: 10  $\mu\text{m}$

# Spectra From AFM GaAs Oxide

Time resolved spectroscopy with SPELEEM using Dispersive Plane ( $h\nu = 130$  eV)



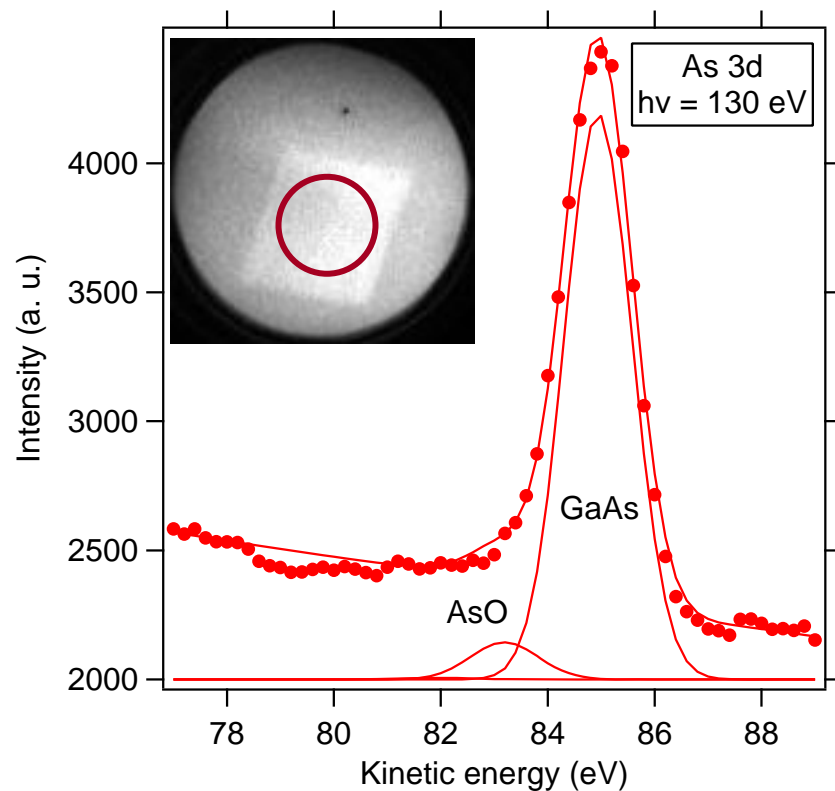
As 3d



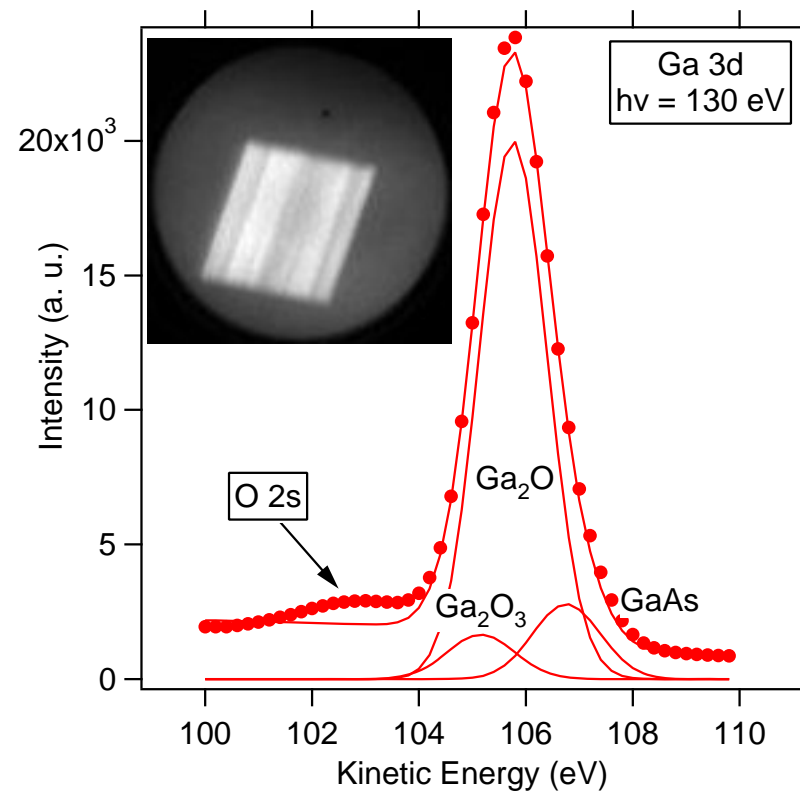
Ga 3d

total exposure time: 76 min

# Spectra From AFM GaAs Oxide



As 3d



Ga 3d

photon energy: 130 eV



# Summary

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- Si oxide:
  - The AFM induced oxidation produces chemically uniform, stoichiometric  $\text{SiO}_2$  with dielectric properties comparable to those of thermal  $\text{SiO}_2$ .
- GaAs oxide:
  - Photon assisted partial desorption of the AFM-grown oxide was observed.
  - All As oxides and the oxygen-rich Ga oxides are desorbed.
  - Photon flux and energy are comparable to those to be used in the next generation lithography at 13 nm.