

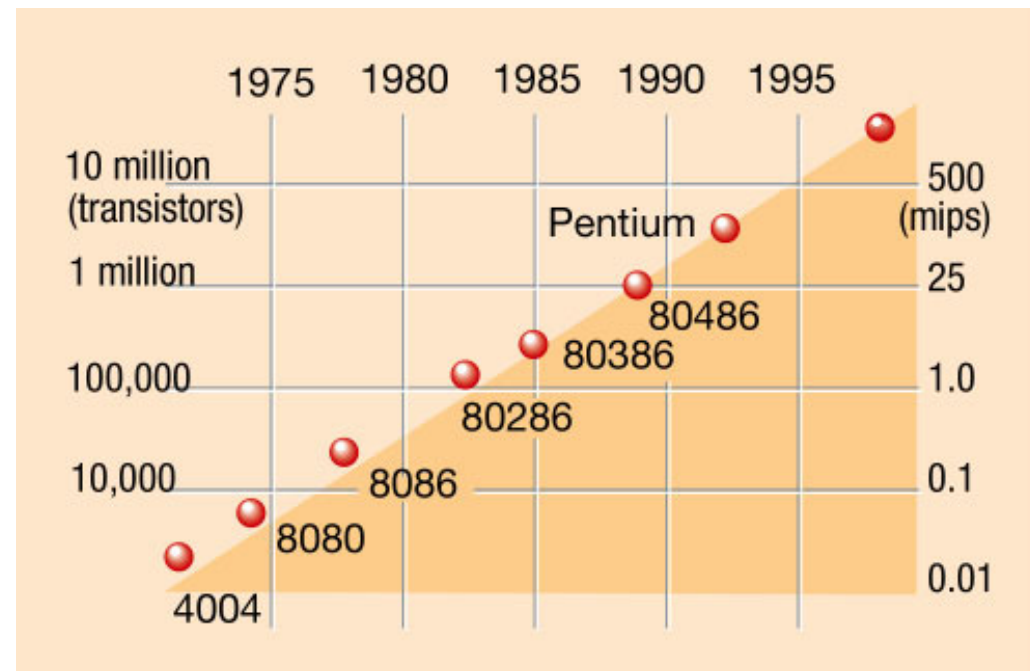
# Nanostructured Materials for Device Applications



Stefan Heun,  
*Laboratorio TASC INFN-CNR,  
Trieste, Italy.*

# Motivation

- Semiconductor Technology: Moore's Law  
(Similar trend for magnetic storage devices)
  - Continuous miniaturization
  - Increasing complexity



Nature **406** (2000) 118.

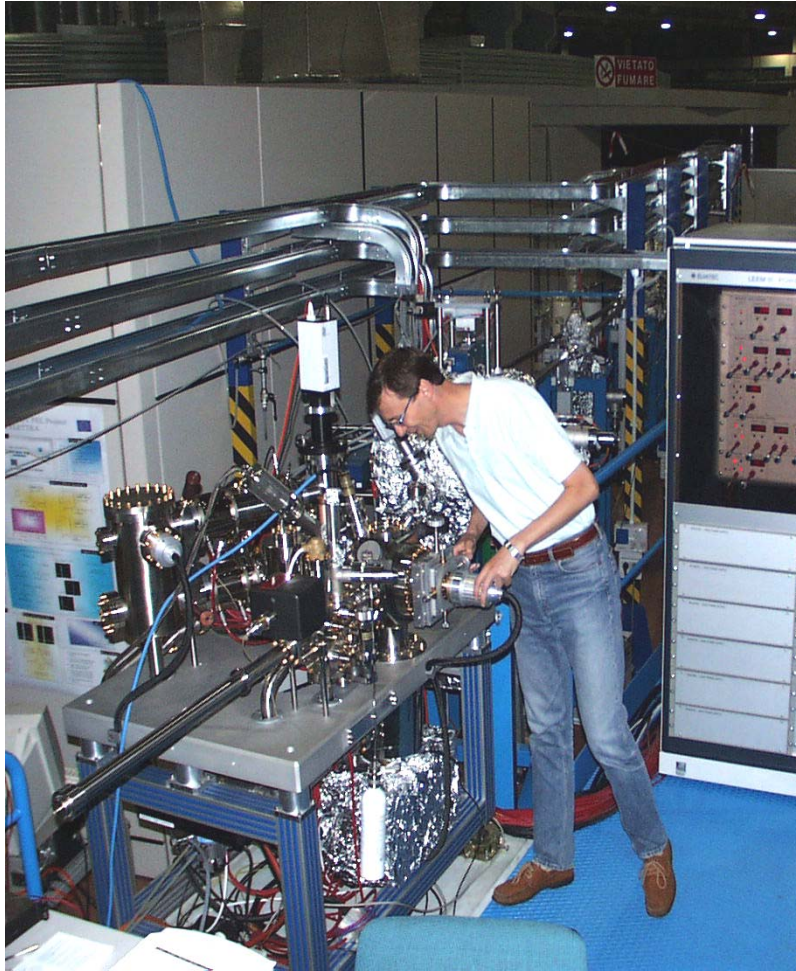
# Motivation

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- Semiconductor Technology: Moore's Law (Similar trend for magnetic storage devices)
  - Continuous miniaturization
  - Increasing complexity
- Need for Analytical Tools with Access to
  - Chemical composition
  - Electronic and magnetic properties
- Lateral Resolution ~ 100 nm
- Delivered by X-ray Spectroscopy (XPS) in combination with X-ray Microscopy (PEEM)

# The SPELEEM at ELETTRA

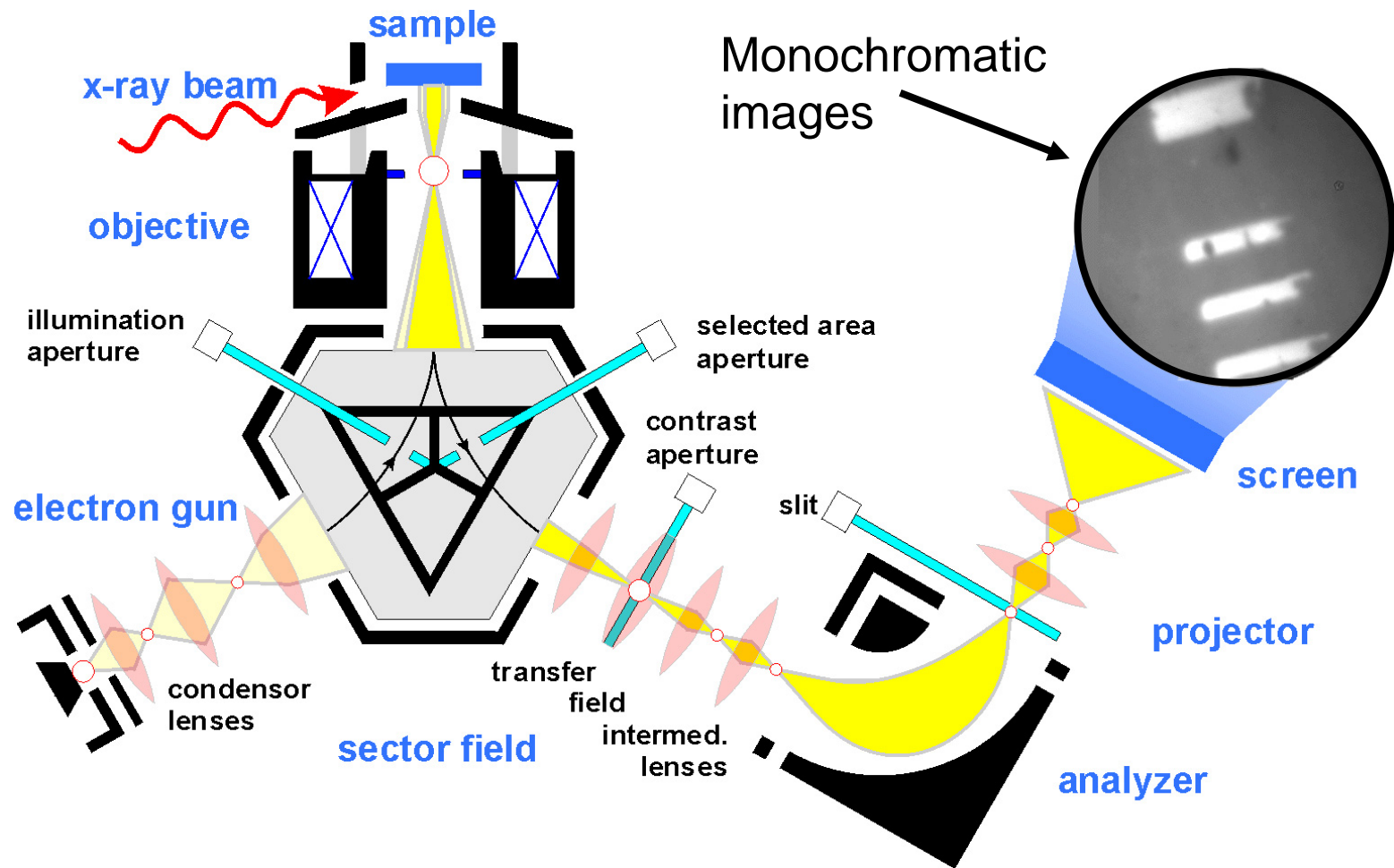
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- ❑ Best energy resolution: 250 meV
- ❑ Best lateral resolution: 25 nm
- ❑ Variable polarization
- ❑ 20 - 1000 eV
- ❑ Photon flux  $10^{13}$  ph/s
- ❑ Small spot ( $2\mu\text{m} \times 25 \mu\text{m}$ )

# The SPELEEM instrument

Spectroscopic Photo-Emission and Low Energy Electron Microscope



# Model Material Systems

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- Semiconductor Quantum Dots (InAs/GaAs, Ge/Si), obtained by self-assembly during MBE growth
- Mesoscopic Devices fabricated by Local Anodic Oxidation Nanolithography with an Atomic Force Microscope
- Suspended individual Single Wall Carbon Nanotubes connecting Si nanostructures

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# InAs/GaAs Quantum Dots

---

- AMD Group @ TASC

- Giorgio Biasiol
- Giovanni Golinelli
- Lucia Sorba

- Nanospectroscopy Beamline @ Elettra

- Andrea Locatelli
- Tevfik Onur Mentesh
- Fangzhun Guo (Spring-8)

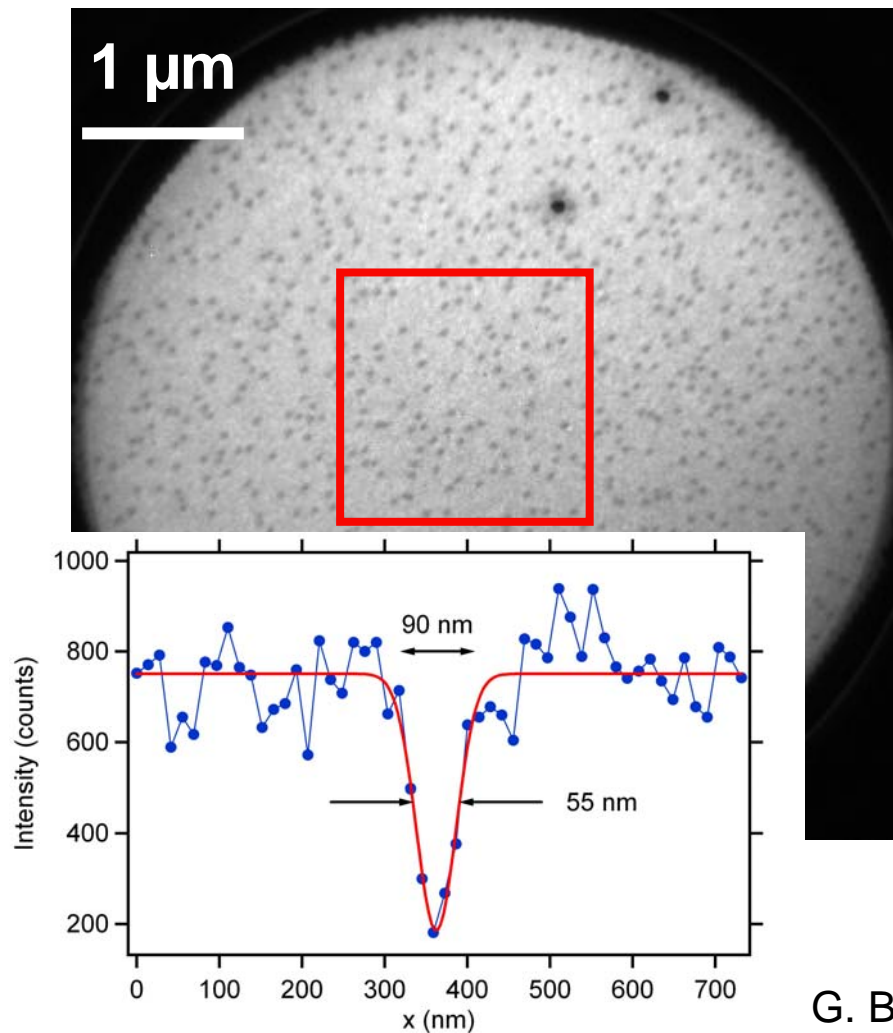


# Motivation

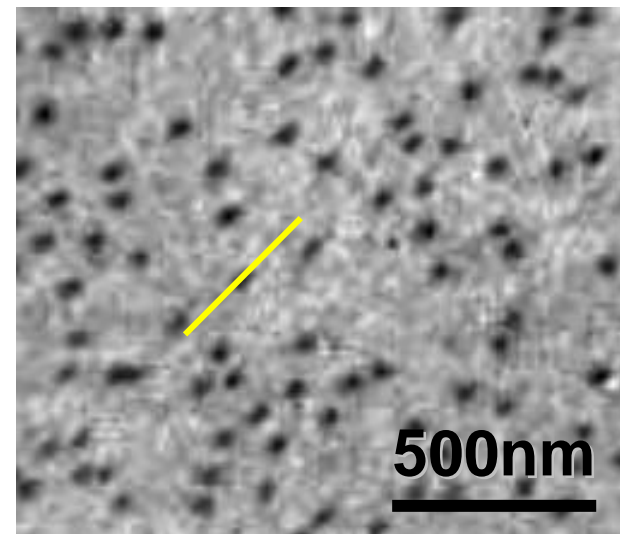
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- Quantum Dot Applications based on their particular electronic properties
- Strain-driven self-assembly (SK-growth)
- Model systems: InAs/GaAs, Ge/Si
- Composition (gradients) within the dot influence energy levels:
  - Intermixing, alloying
  - Shift emission wavelength
  - Introduce anisotropies

# InAs/GaAs Islands (LEEM)

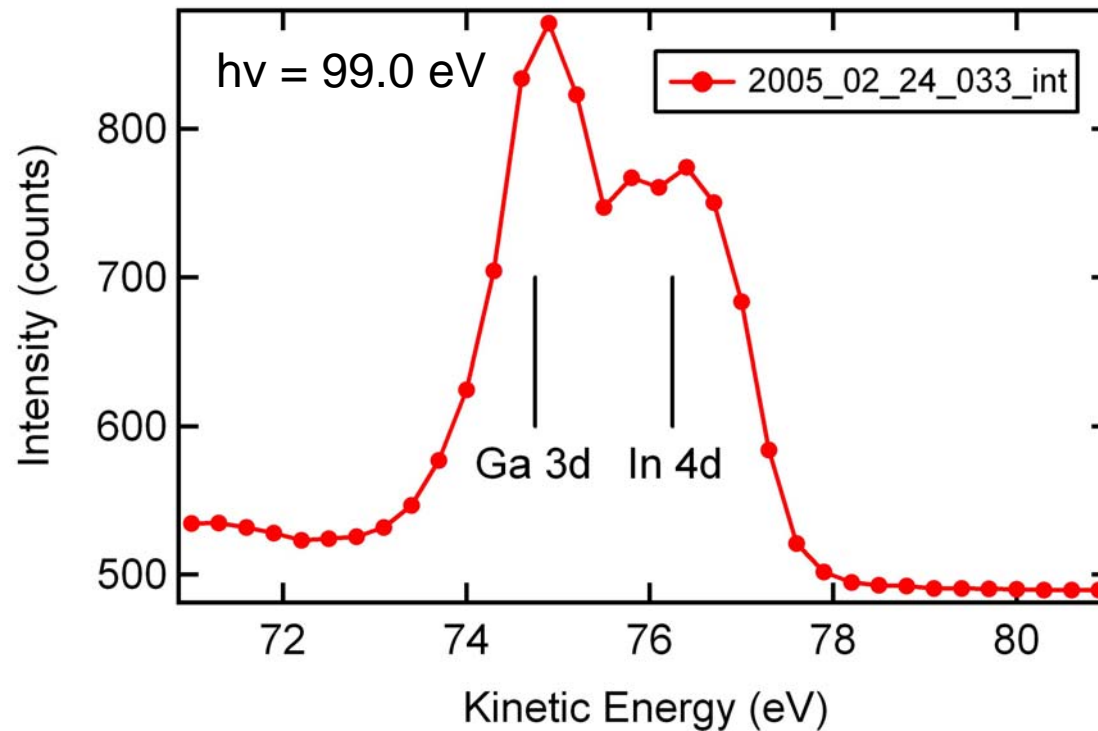


- Electron Microscopy
- LEEM
- 5 μm FOV
- $E_{\text{kin}} = 7.6 \text{ eV}$



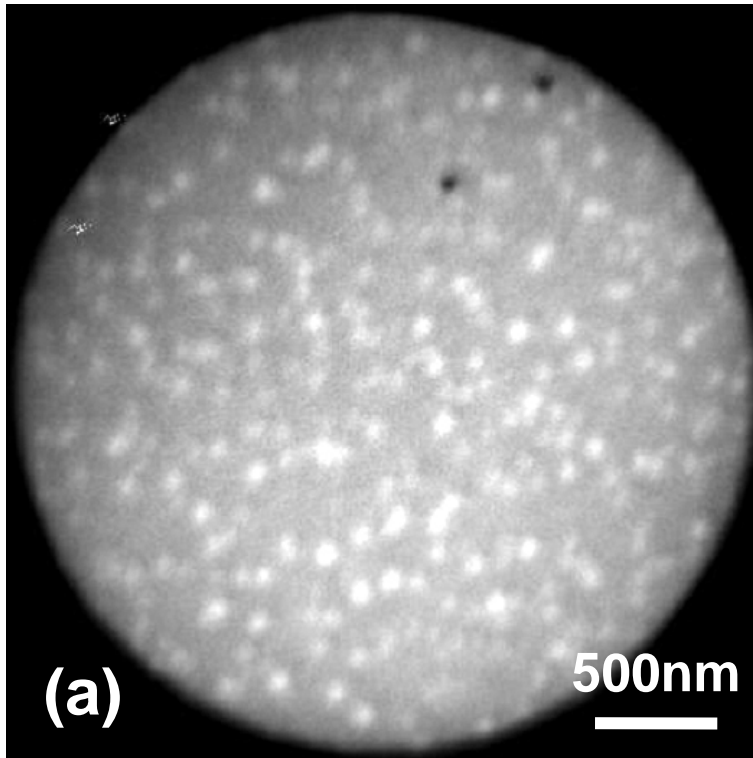
G. Biasiol et al.: Appl. Phys. Lett. **87** (2005) 223106.

# “Integral” Core Level Spectra

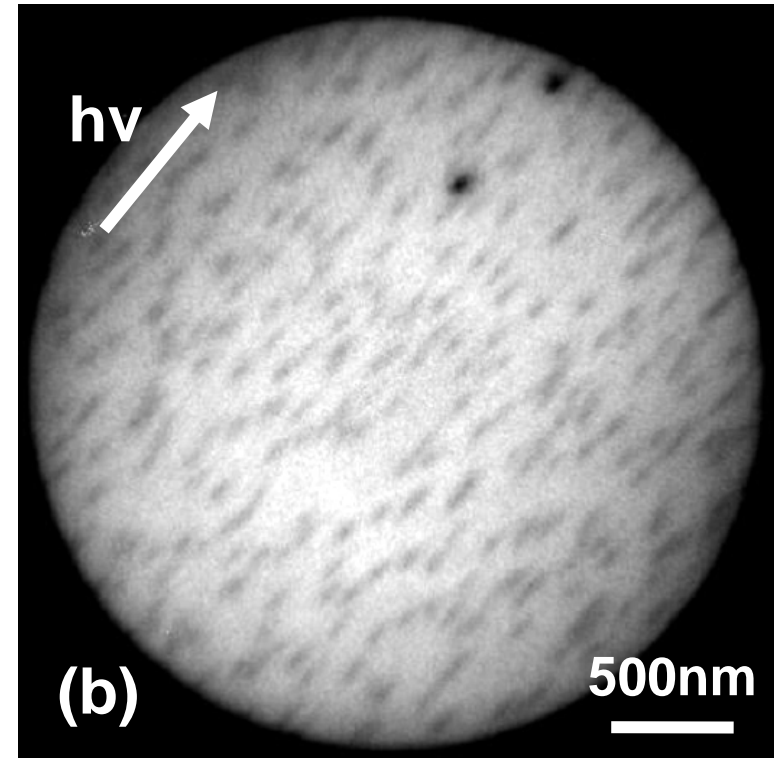


- Spectra taken from a  $1 \mu\text{m} \times 1 \mu\text{m}$  sample area.
- III-V stoichiometry after decapping confirmed.

# XPEEM Core Level Imaging

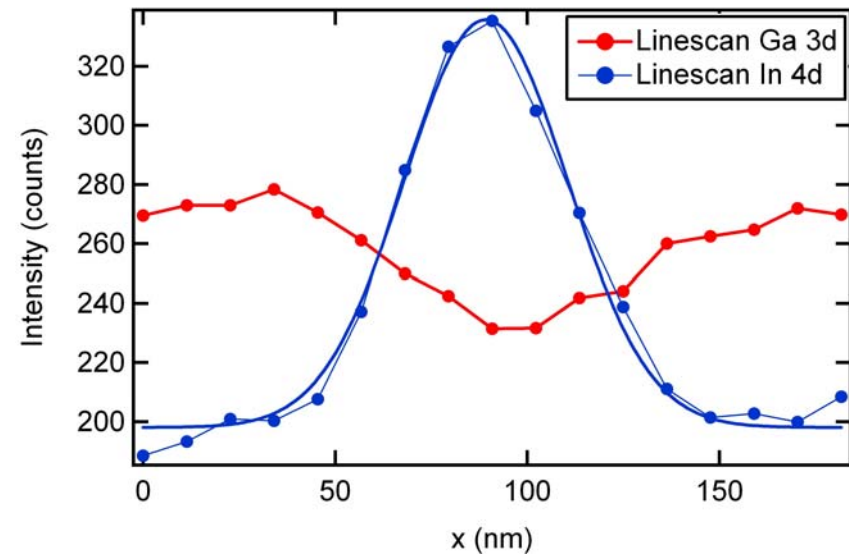
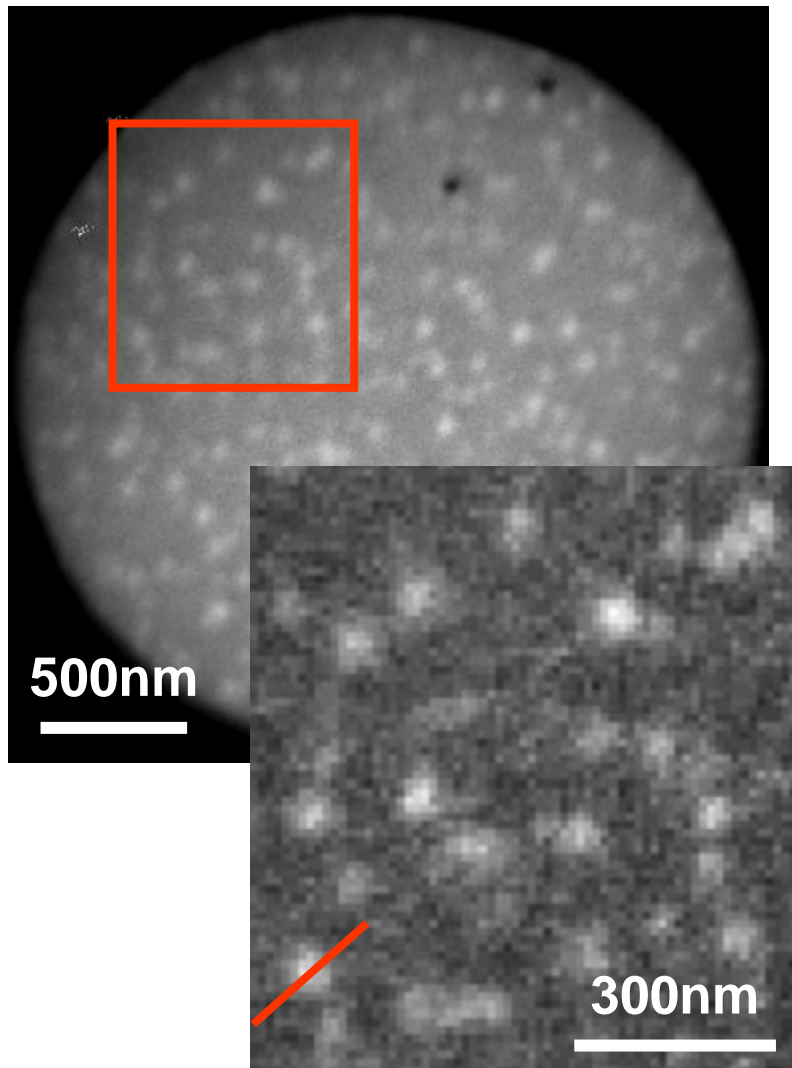


In 4d XPEEM image  
 $h\nu = 99.0 \text{ eV}$ ,  $E_{\text{kin}} = 76.25 \text{ eV}$



Ga 3d XPEEM image  
 $h\nu = 99.0 \text{ eV}$ ,  $E_{\text{kin}} = 74.75 \text{ eV}$

# Island Size in XPEEM

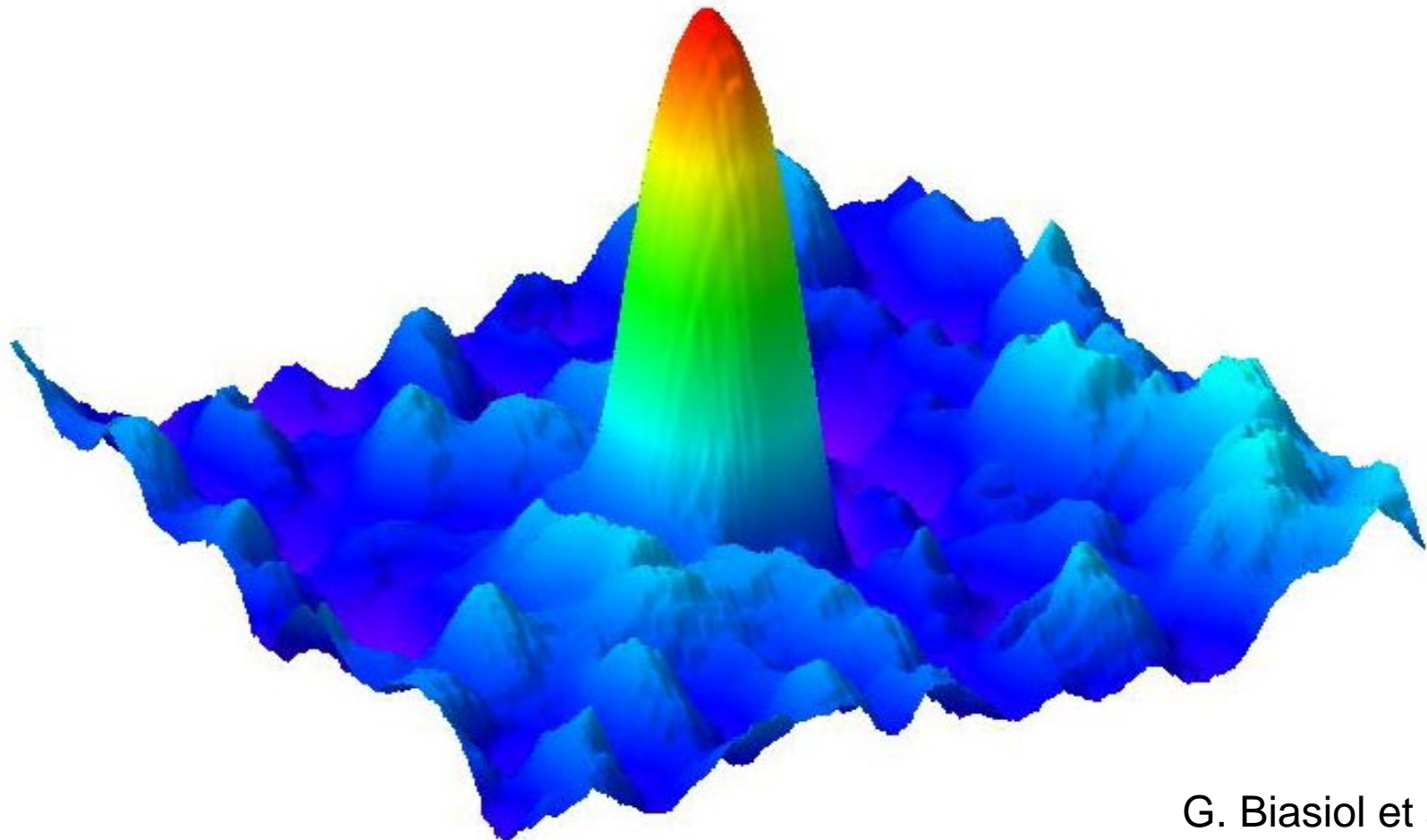


Island Size  $\approx$  60 nm,  
consistent with  
LEEM and  
ex-situ AFM & SEM

G. Biasiol et al.:  
Appl. Phys. Lett. **87** (2005) 223106.

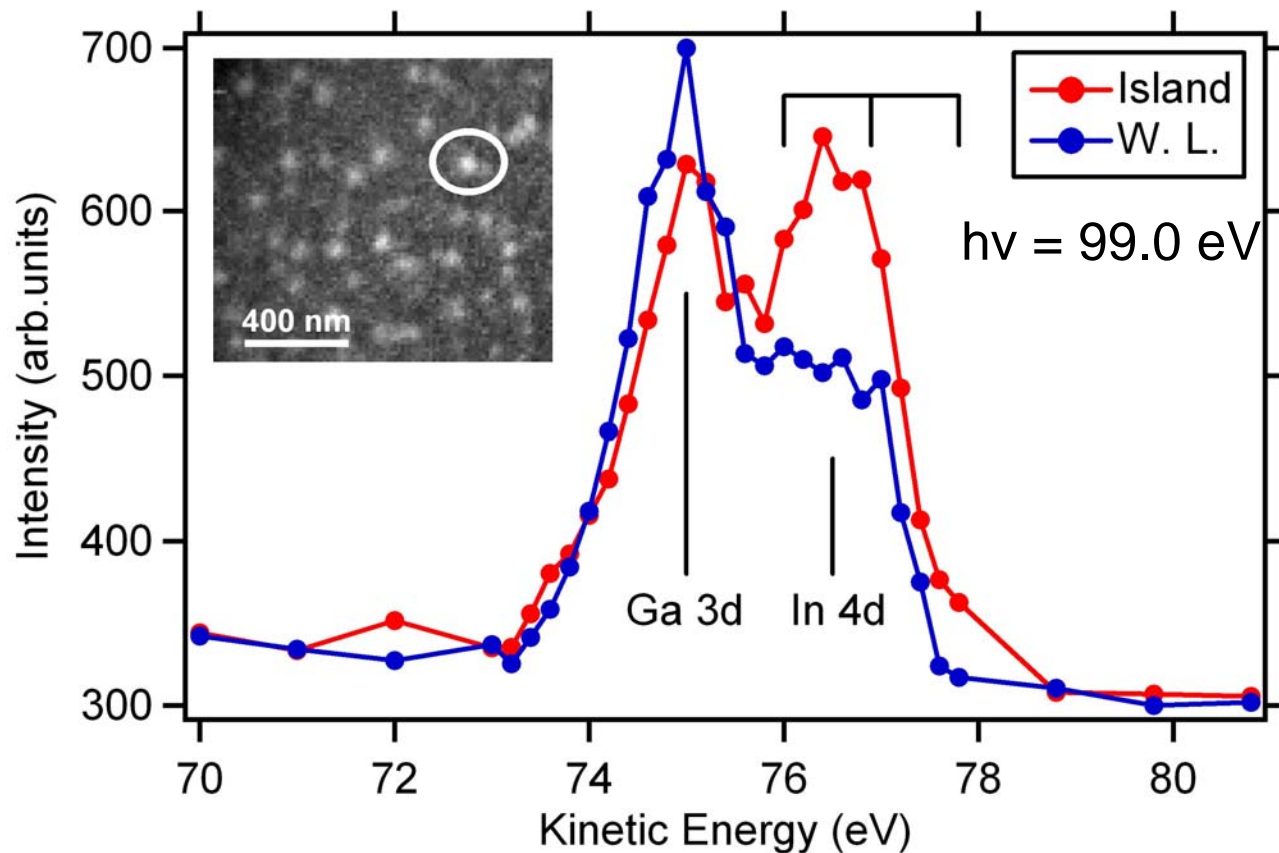
# Island Size in XPEEM

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G. Biasiol et al.:  
Appl. Phys. Lett. **87** (2005) 223106.

# XPEEM Local Spectra

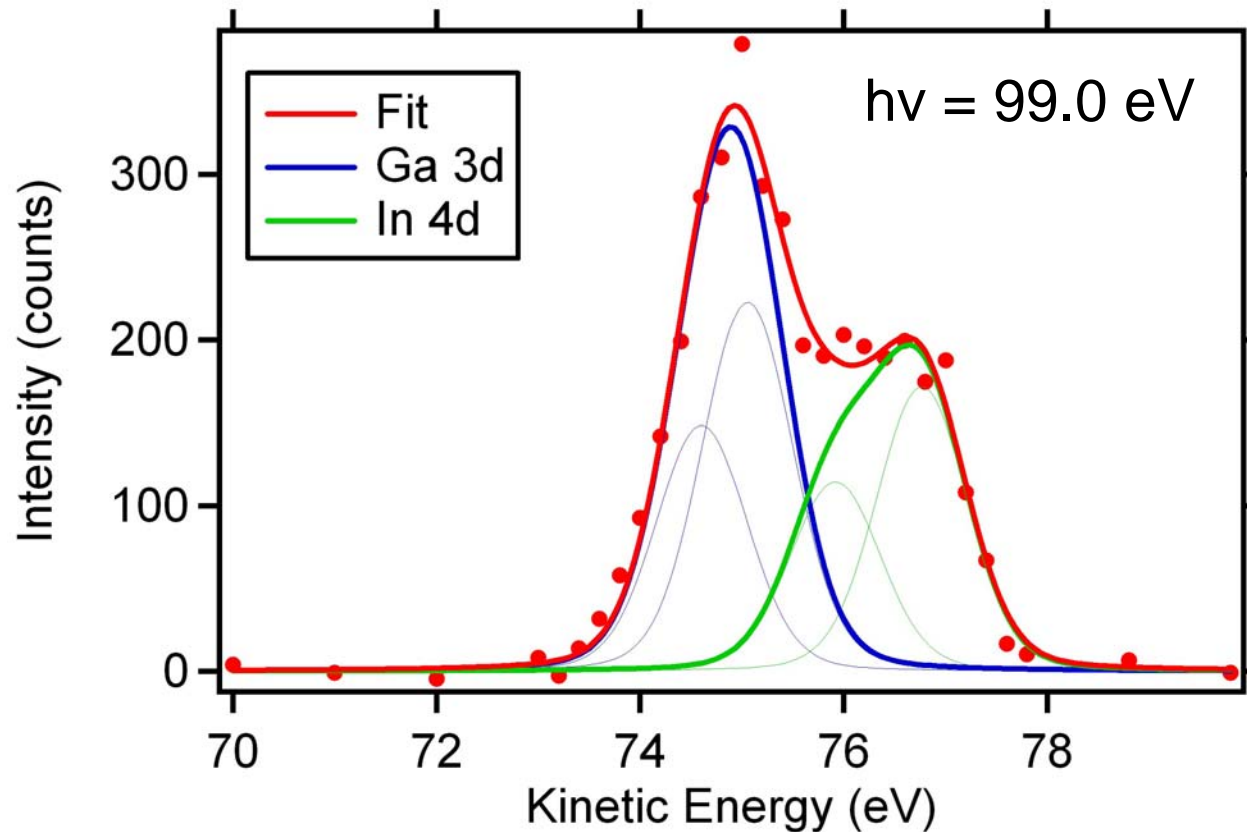


Integration area 25 nm x 25 nm, energy resolution  $\approx 1$  eV

G. Biasiol et al.: Appl. Phys. Lett. **87** (2005) 223106.



# Core Level Line Profile Analysis



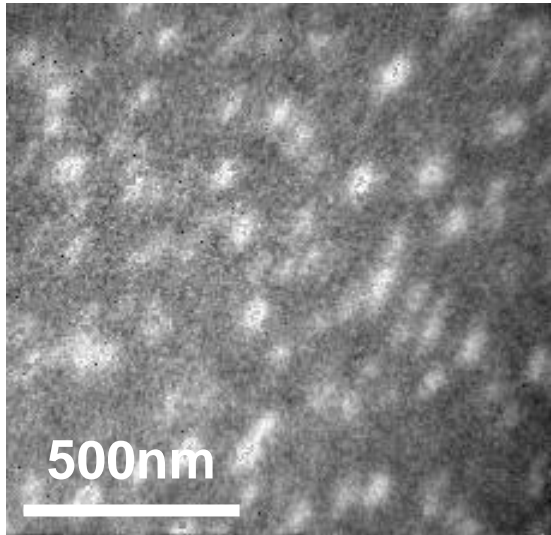
Spectrum from Wetting Layer, Shirley Background subtracted  
Gauss 1 eV, Lor 0.16 eV, BR 1.5, SO: Ga 3d 0.45 eV, In 4d 0.85 eV

G. Biasiol et al.: Appl. Phys. Lett. **87** (2005) 223106.

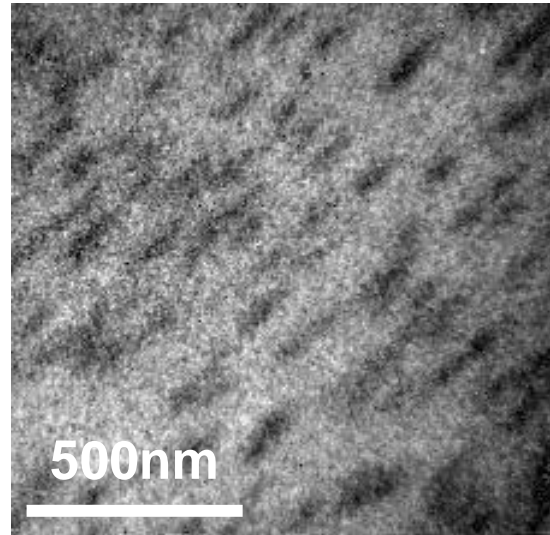


# 2D Fit of XPEEM Data

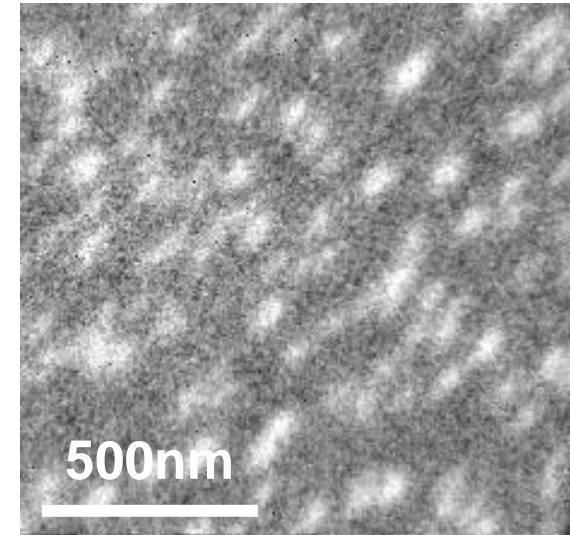
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In 4d peak area  
Min: 220, Max: 520



Ga 3d peak area  
Min: 270, Max: 470



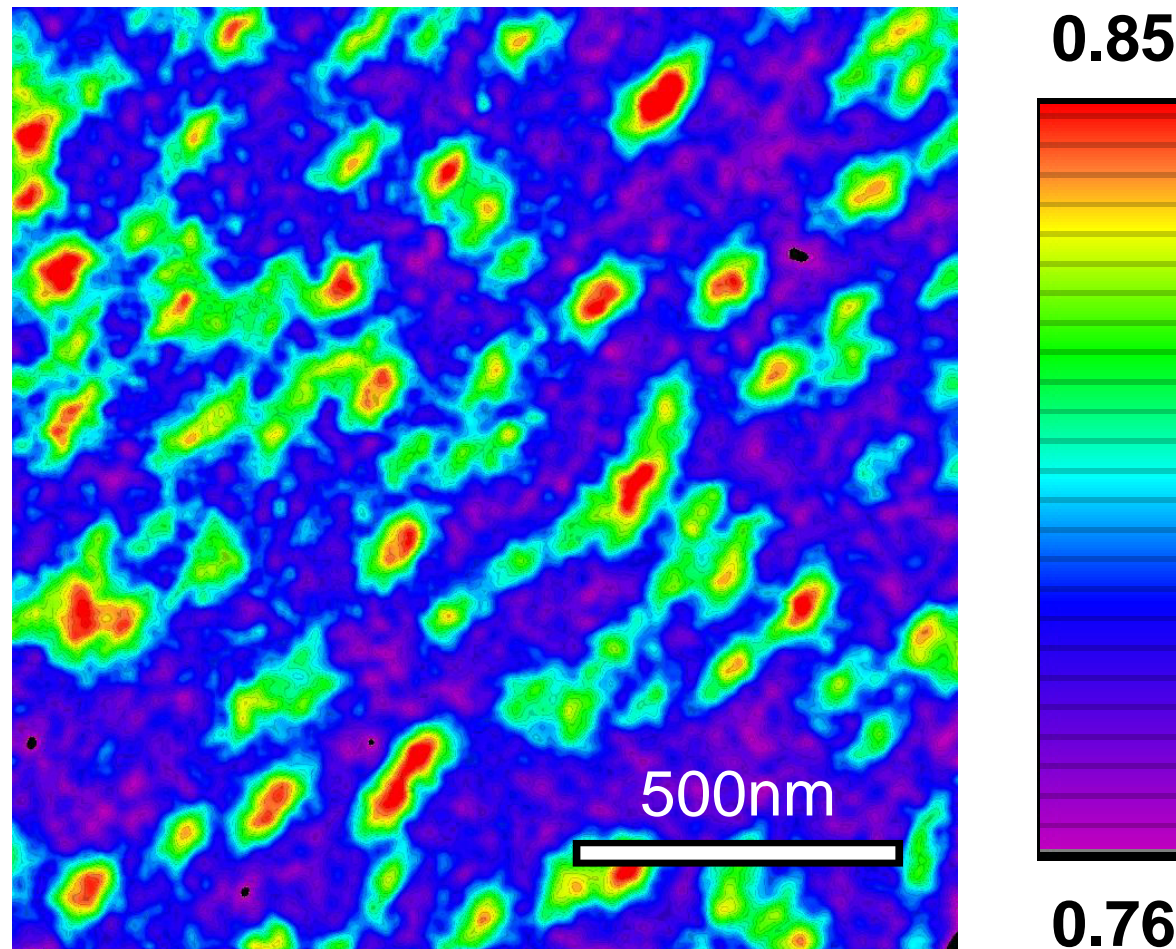
In surface concentration  
**Min: 0.7, Max: 0.86**

Photoionization cross sections: In 4d: 2.2, Ga 3d: 8.5  
In surface concentration =  $\text{In 4d} / (\text{In 4d} + \text{Ga 3d})$

G. Biasiol et al.: Appl. Phys. Lett. **87** (2005) 223106.

# Indium Surface Concentration Map

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G. Biasiol et al.: Appl. Phys. Lett. **87** (2005) 223106.

# Model Material Systems

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# Local Anodic Oxidation on GaAs

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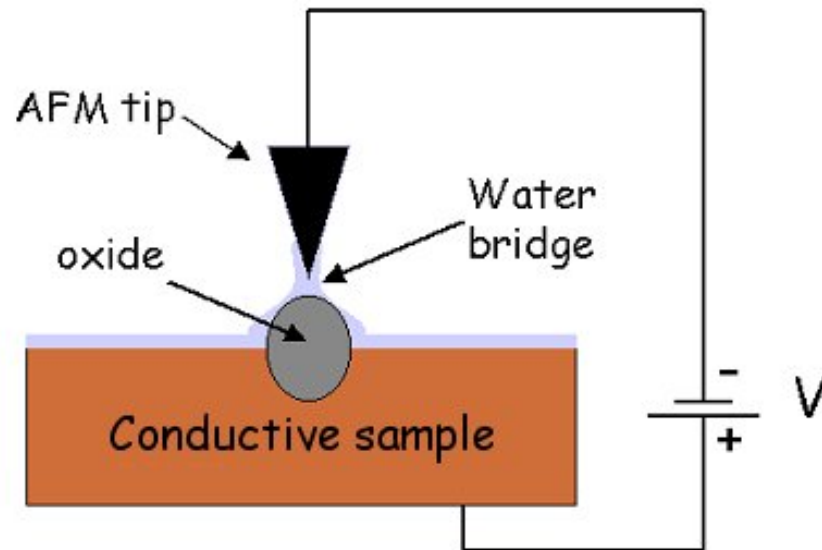
- AMD Group @ TASC

- Giorgio Mori
- Marco Lazzarino
- Daniele Ercolani
- Giorgio Biasiol
- Lucia Sorba

- Nanospectroscopy Beamline @ Elettra

- Andrea Locatelli

# Local Anodic Oxidation (LAO)



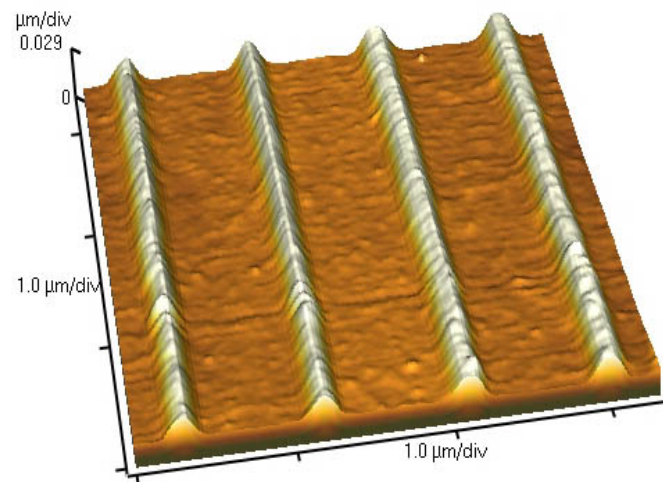
Common model:

- Water electrolysis  
 $\text{H}_2\text{O} \rightarrow \text{H}^+ + \text{OH}^-$ .
- $\text{OH}^-$  groups (or  $\text{O}^-$ ) migrate towards the substrate-oxide interface.
- Oxide penetration induced by the intense local electric field ( $>10^7$  V/cm).

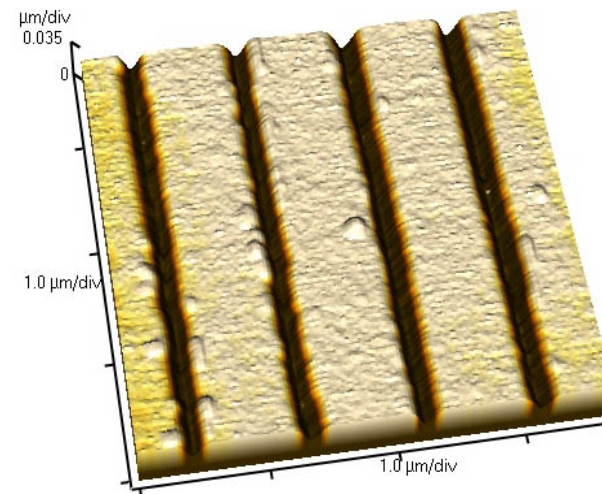
Versatile tool at relatively low cost  
High lateral resolution but small area



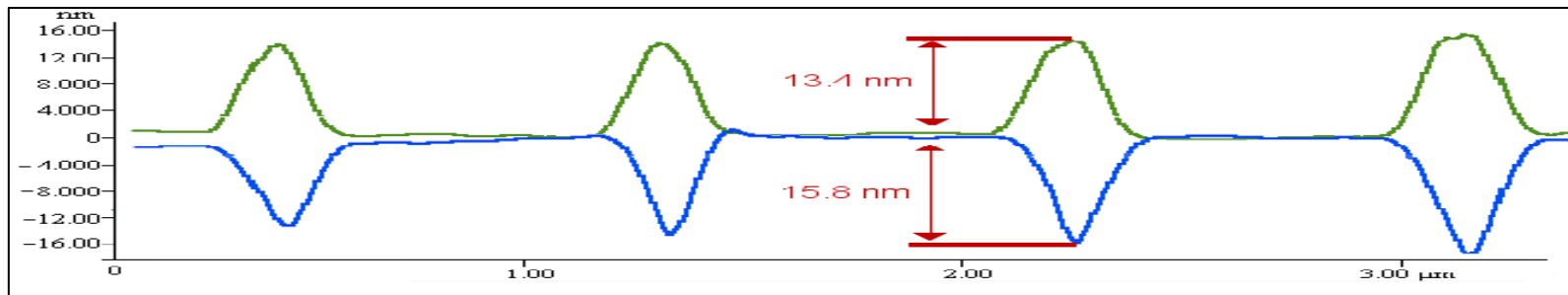
# Local Anodic Oxidation (LAO)



After oxidation



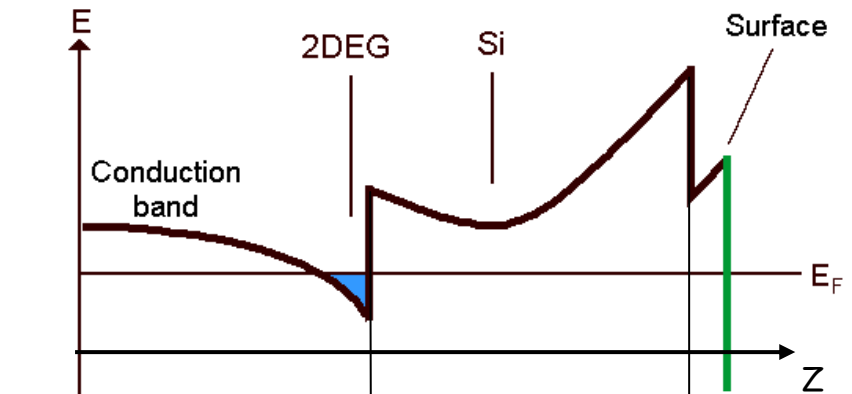
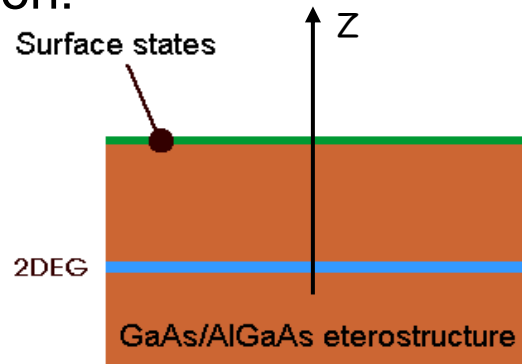
Oxide removal with HF 10%, 30 s



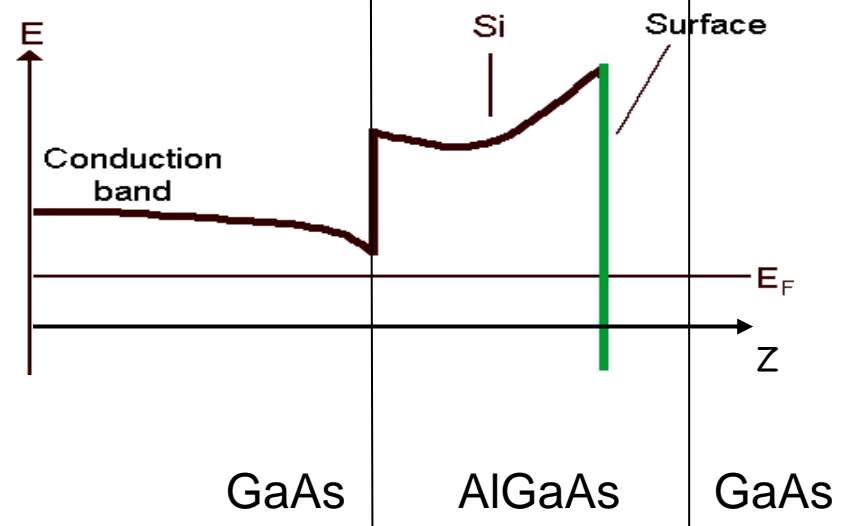
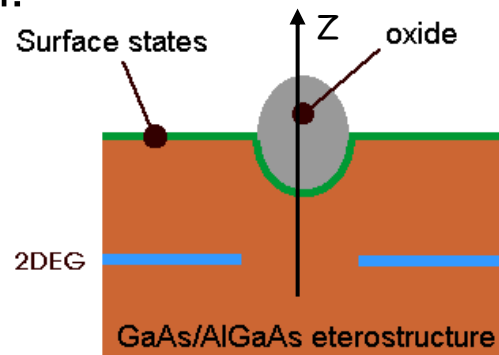
The penetration depth is 1.0-1.5 times the oxide height

# LAO on GaAs/AlGaAs

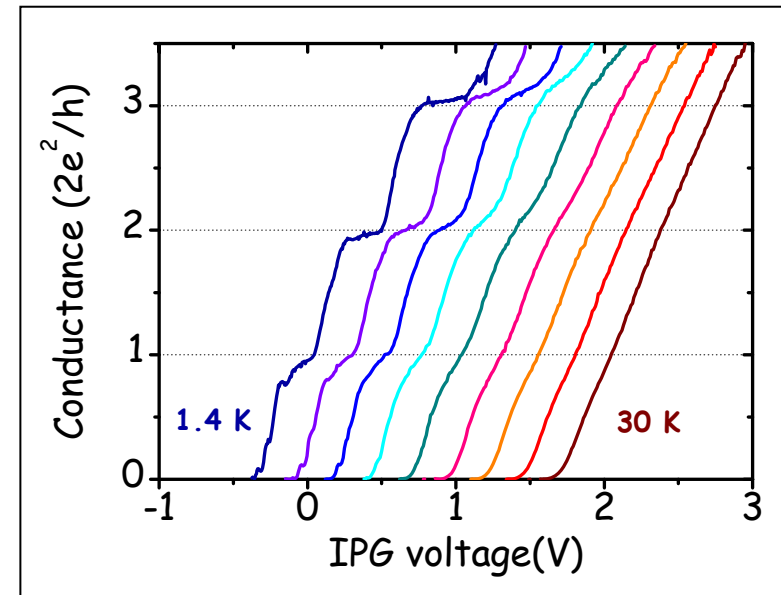
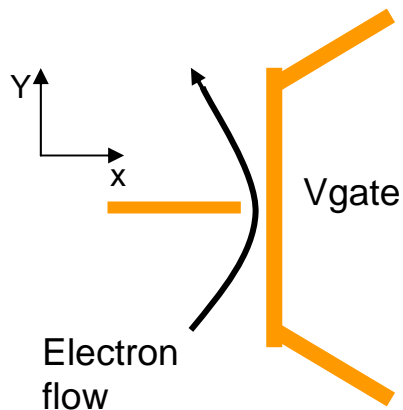
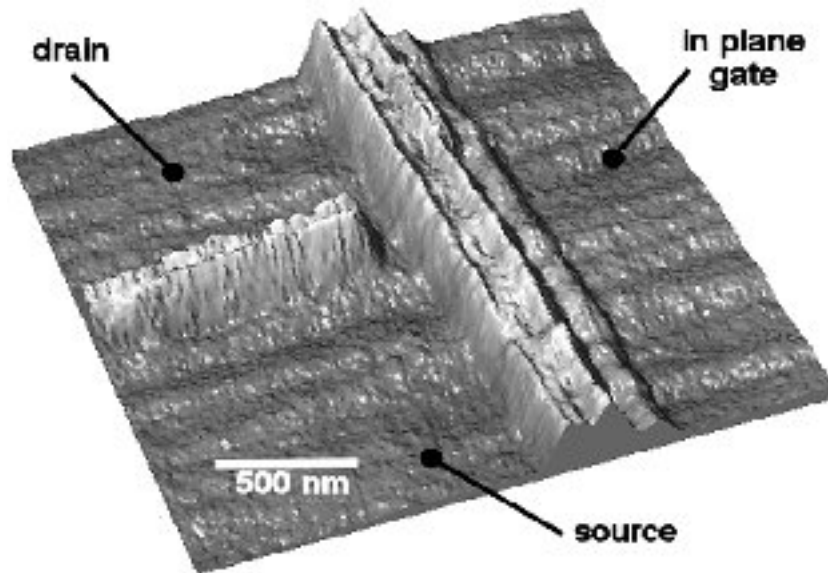
Before oxidation:



After oxidation:



# Quantum Point Contact

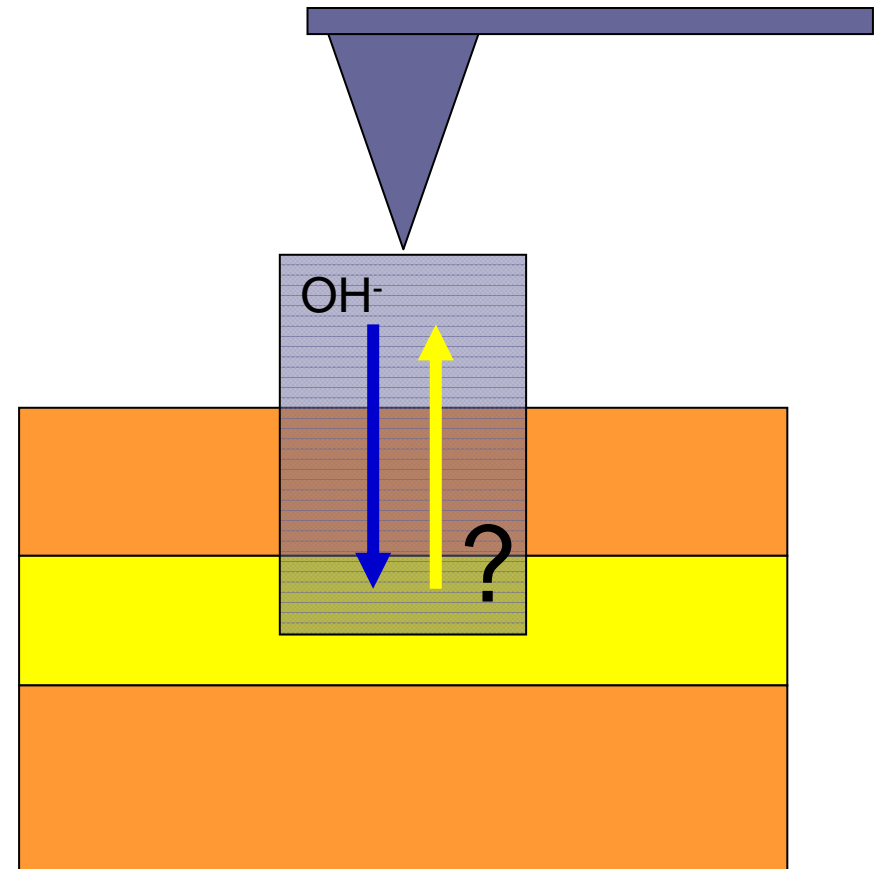
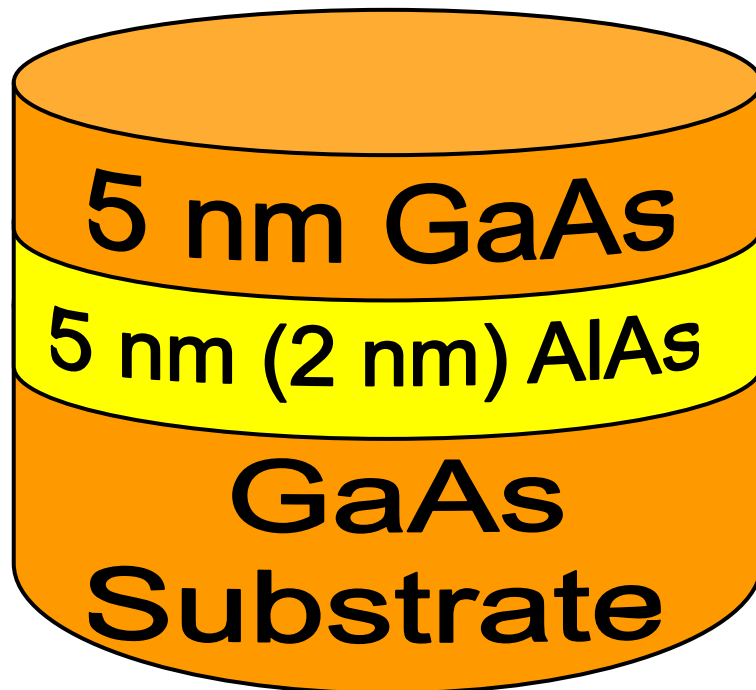


G. Mori et al, J. Vac. Sci. Technol. B **22** (2004) 570.

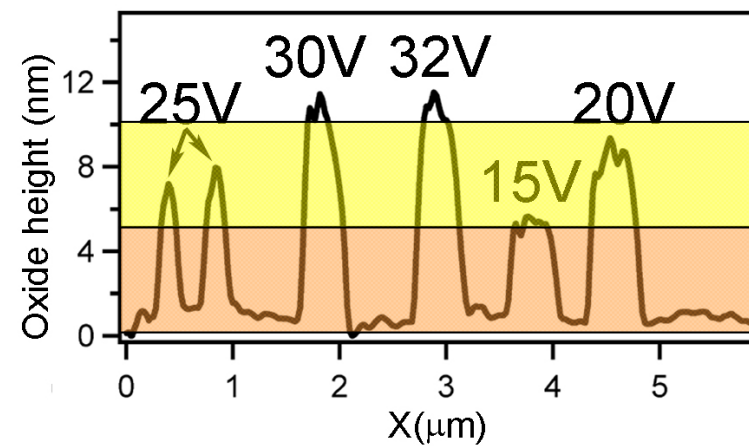
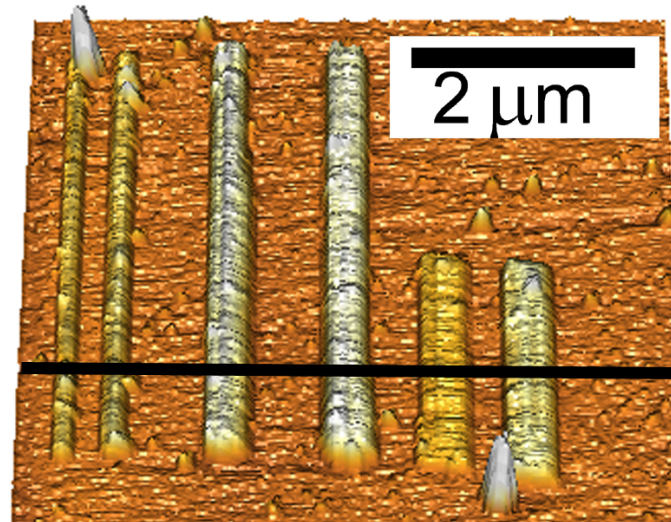


# The dynamics of the LAO process

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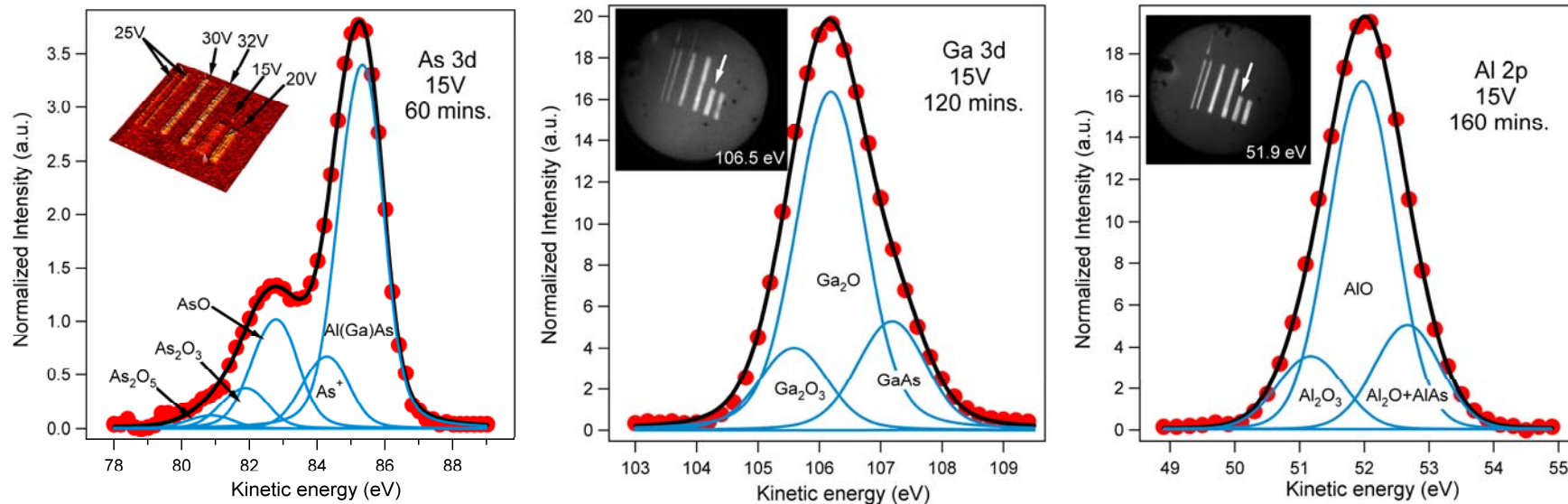
# LAO on III-V Heterostructures



AlAs  
GaAs

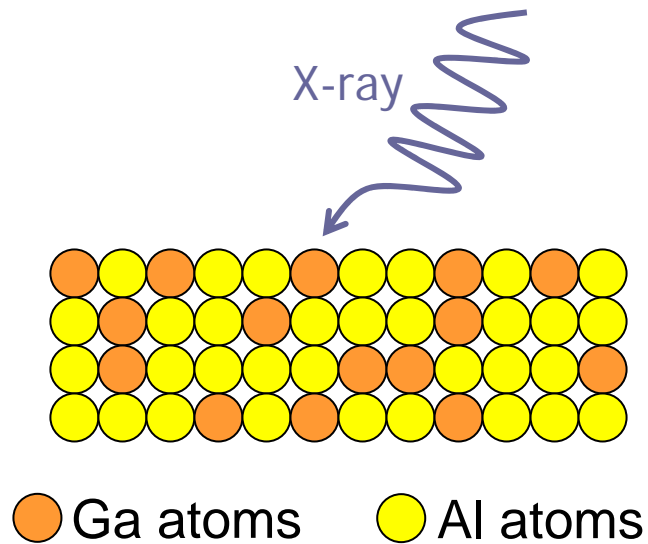
G. Mori et al.: J. Appl. Phys. **98** (2005)114303.

# Chemical composition of LAO oxide

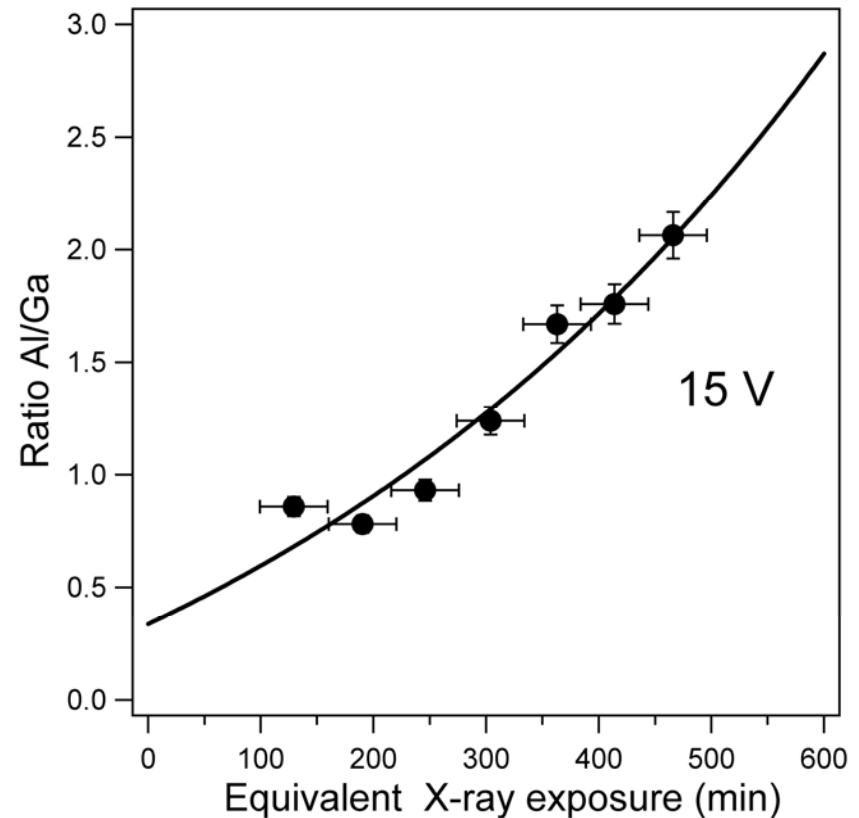


- Aluminium observed at the surface of the LAO oxide
- No Al in the regions not oxidized with LAO
- Time-resolved study: Ga-oxides desorb under x-rays (much faster than Al-oxides)

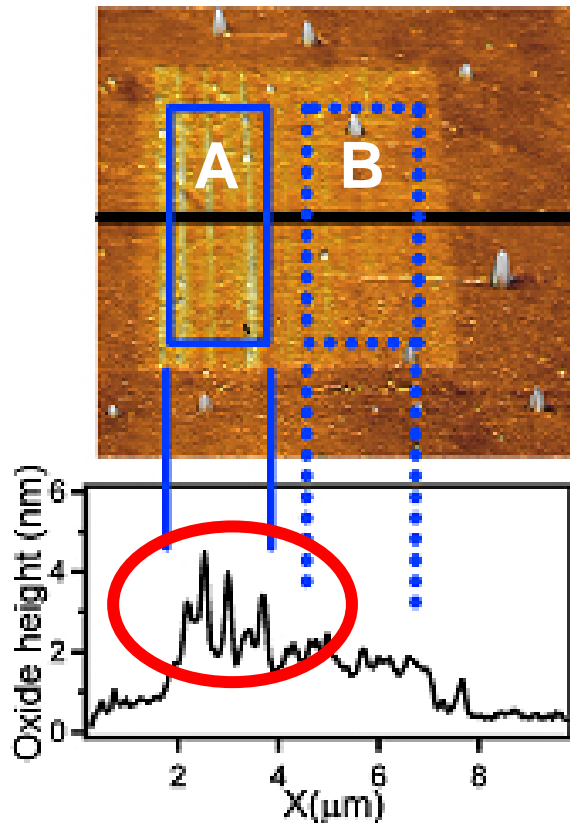
# The effect of X-ray exposure



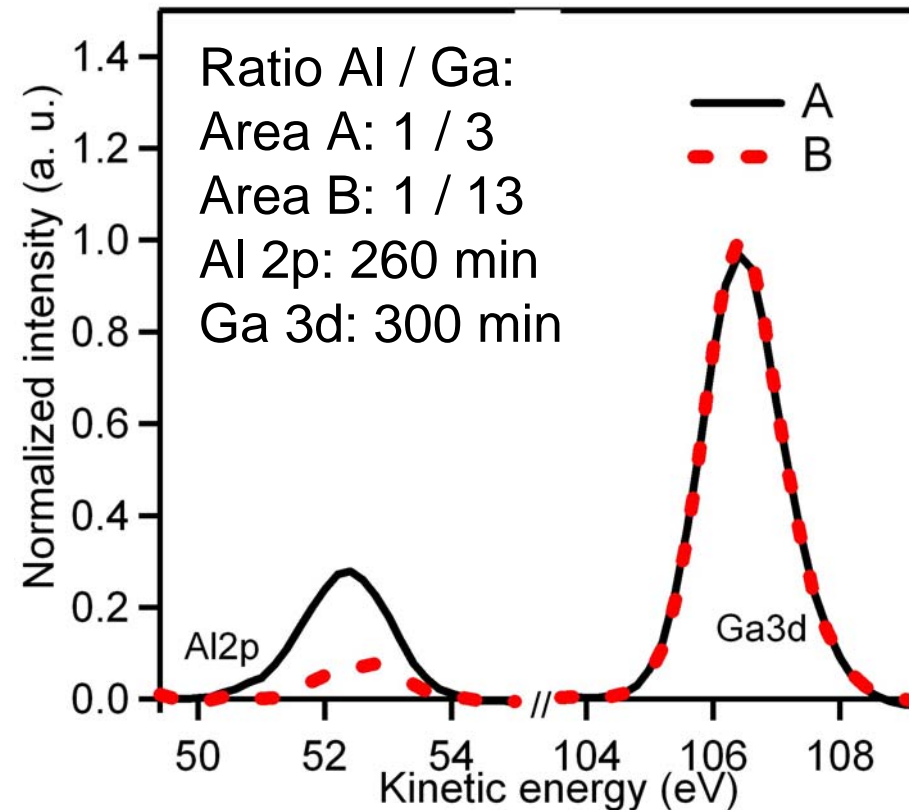
$$N_{Al}(t) \cong N_{Al}^0 + N_{Ga}^0 \cdot \left( 1 - e^{-\frac{t}{\tau_{Ga}}} \right)$$



# Shallow Oxidations



Average oxide height:  
Area A:  $1.9 \pm 0.7$  nm  
Area B:  $1.3 \pm 0.3$  nm

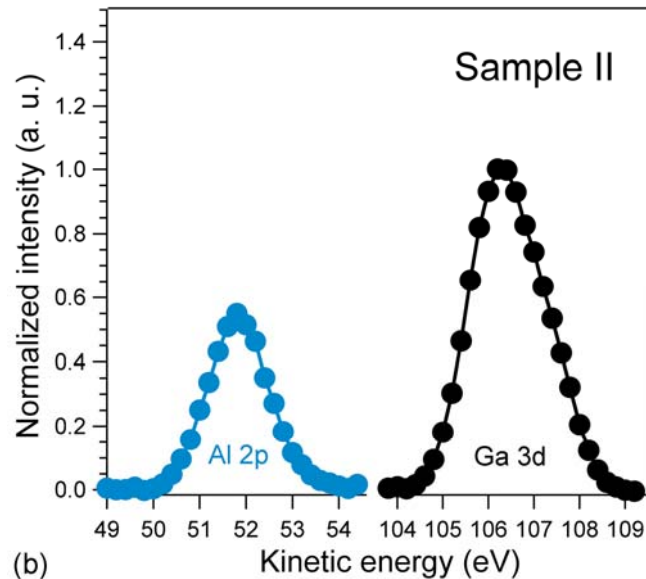


Al content depends strongly on oxide height

G. Mori et al.: J. Appl. Phys. **98** (2005)114303.

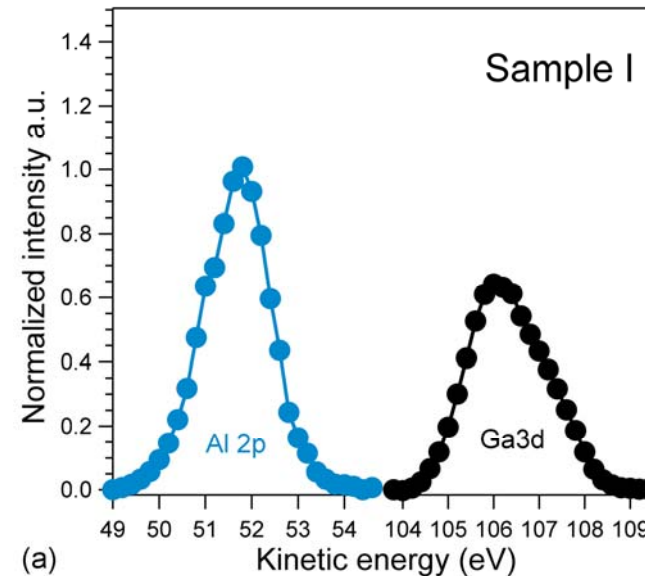
# Variation in AlAs layer thickness

- 2 nm AlAs
- Oxide height  $9.0 \pm 1.0$  nm



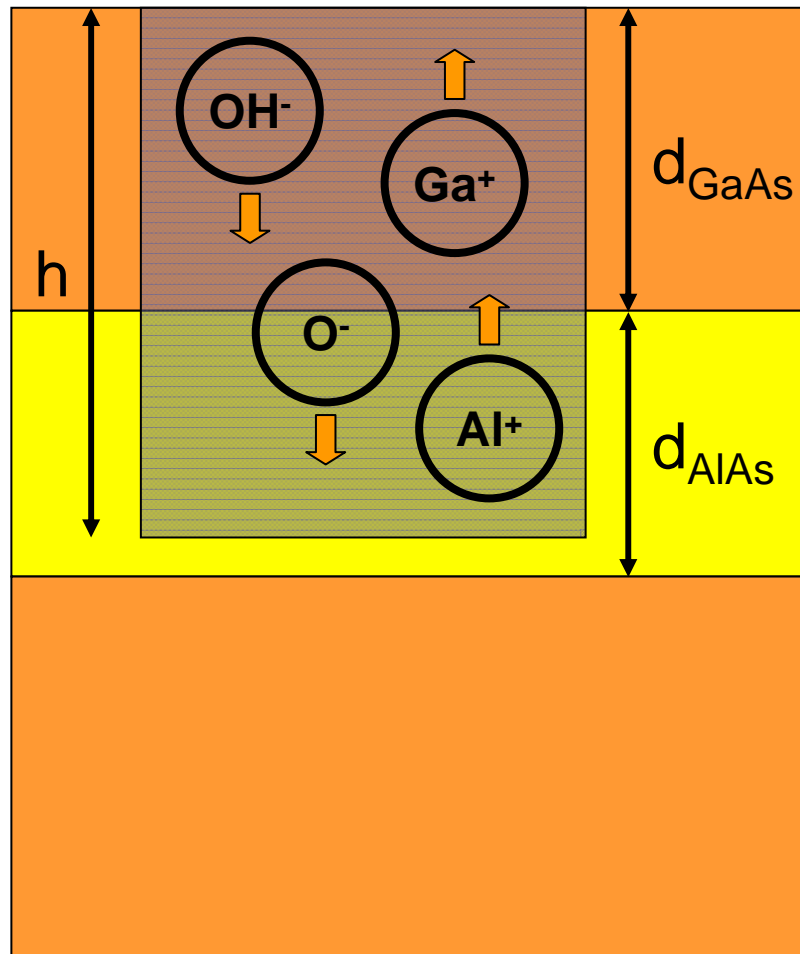
- Ratio Al / Ga: 0.5
- Al 2p: 220 min
- Ga 3d: 300 min

- 5 nm AlAs
- Oxide height  $8.9 \pm 0.9$  nm



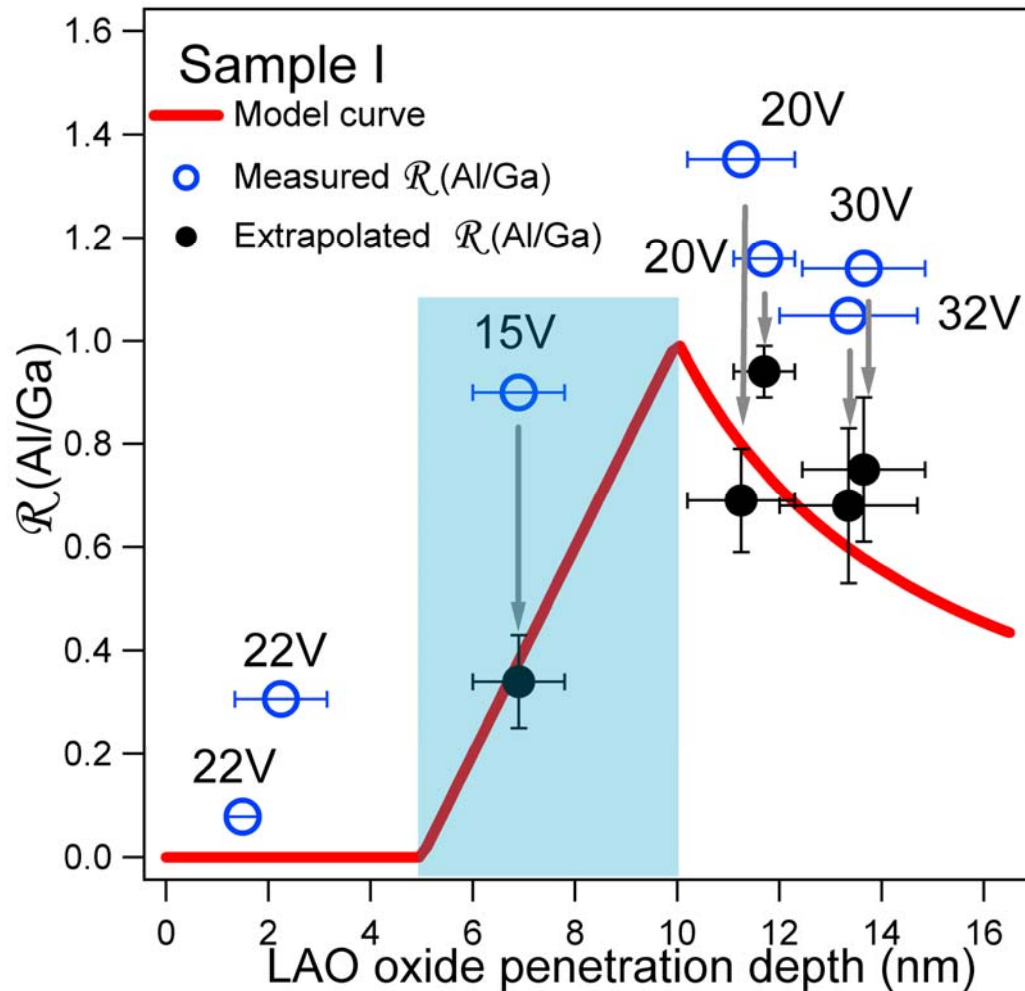
- Ratio Al / Ga: 1.3
- Al 2p: 260 min
- Ga 3d: 230 min

# Refined Model



- Diffusion of oxygen-rich ions plus substrate ions
- Homogeneous mixing of components
- Ratio Al / Ga:
  - 0 for  $h < d_{\text{GaAs}}$
  - $(h - d_{\text{GaAs}}) / d_{\text{GaAs}}$  for  $d_{\text{GaAs}} < h < d_{\text{GaAs}} + d_{\text{AlAs}}$
  - $d_{\text{AlAs}} / (h - d_{\text{AlAs}})$  for  $h > d_{\text{GaAs}} + d_{\text{AlAs}}$

# Comparison with Experiment



G. Mori et al.: J. Appl. Phys. **98** (2005)114303.



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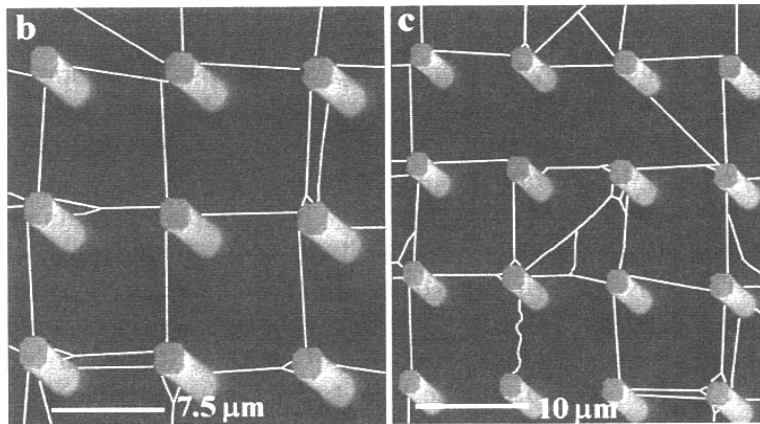
# Suspended Carbon Nanotubes

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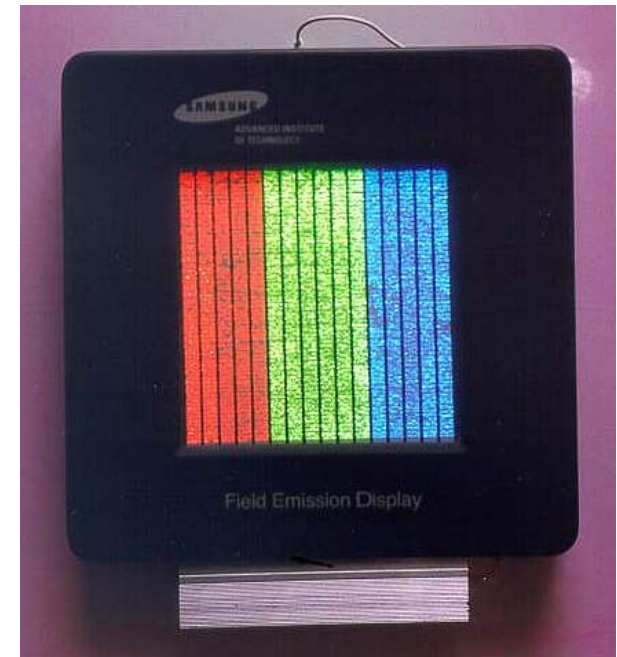
- NTT Basic Research Laboratories
  - Satoru Suzuki
  - Yoshio Watanabe
  - Yoshikazu Homma
  - Shin-ya Fukuba
- Nanospectroscopy Beamline @ Elettra
  - Andrea Locatelli

# Motivation

- Carbon Nanotube Applications:
  - Field effect transistor
  - Field emission displays
  - Interconnects



Adv. Mat. **12** (2000) 890



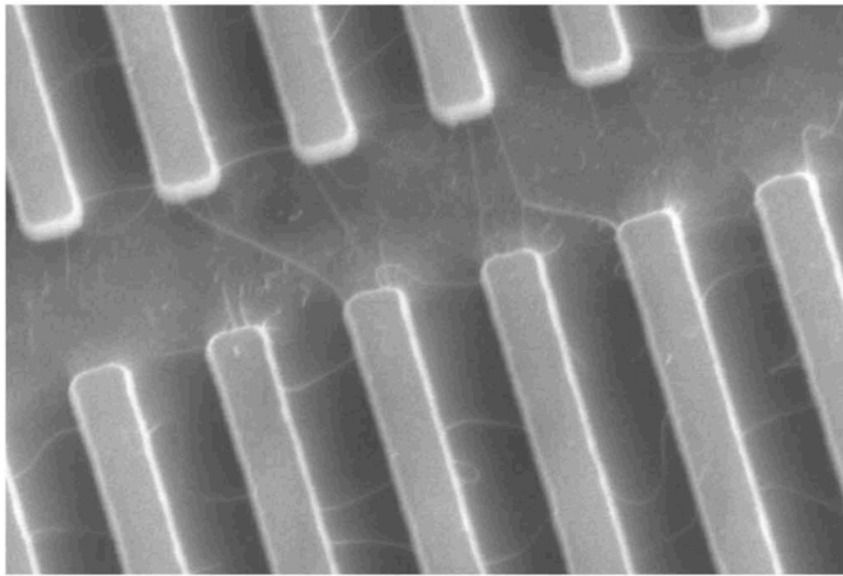
APL **75** (1999) 3129

- Work function and electronic properties of these CNT close to the Fermi energy are important

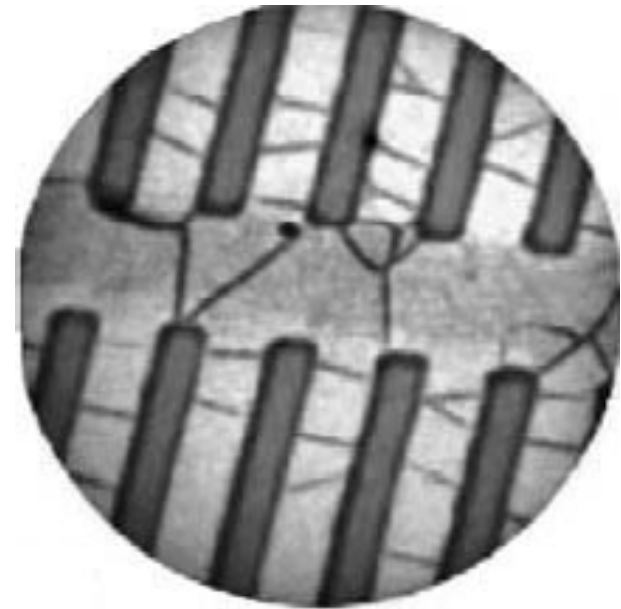
# Samples for SPELEEM studies

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- Si line pattern, pitch 1  $\mu\text{m}$ , height 500 nm
- SWNT grown by CVD at 900°C (Fe catalyst)
- SWNT diameter 1-3 nm

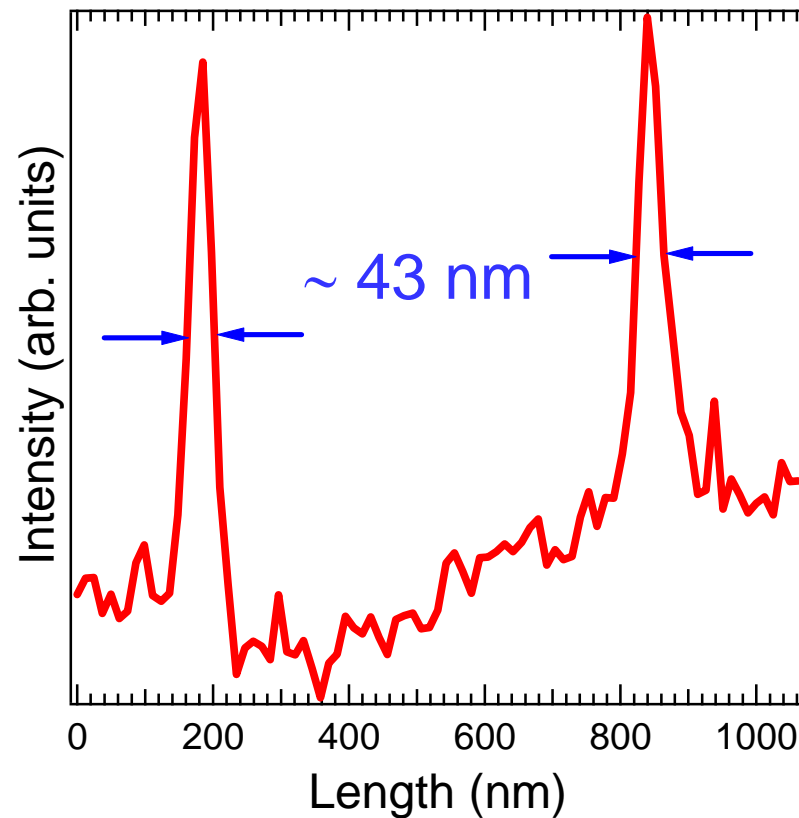
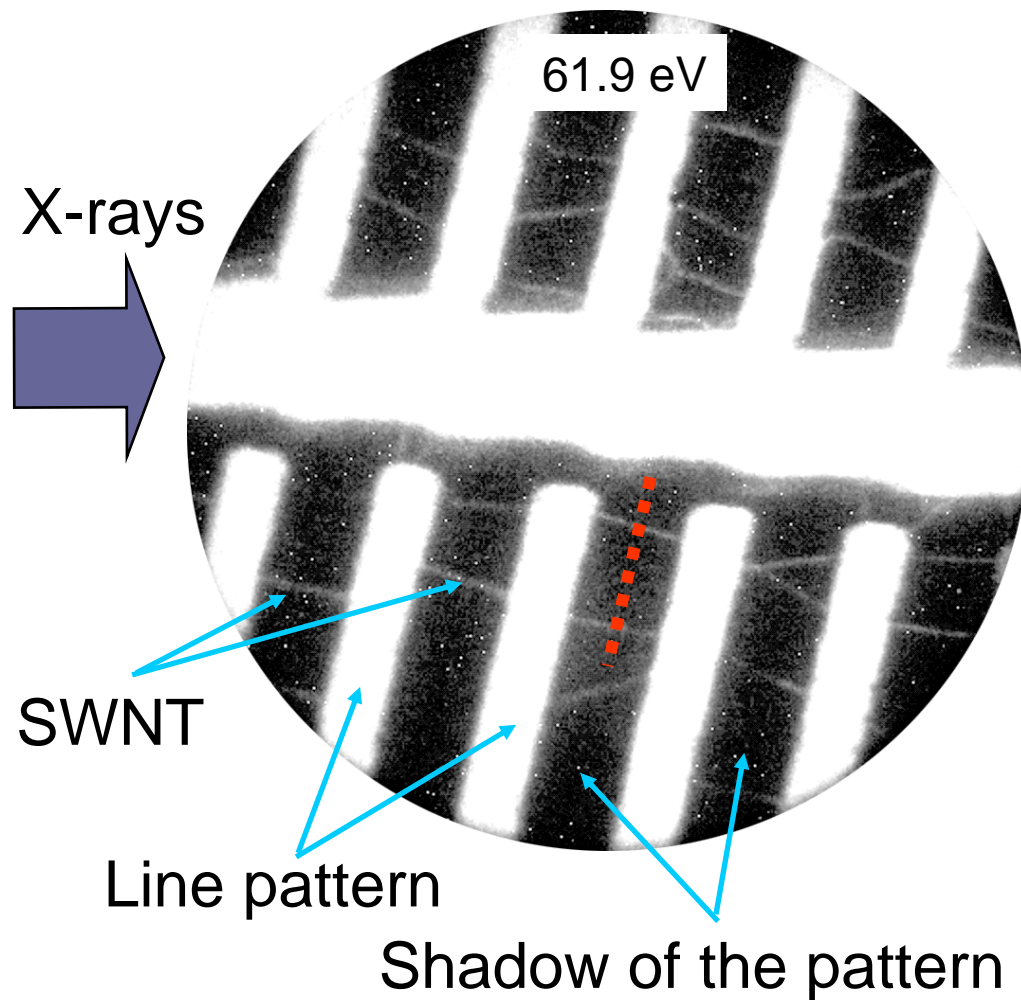


SEM



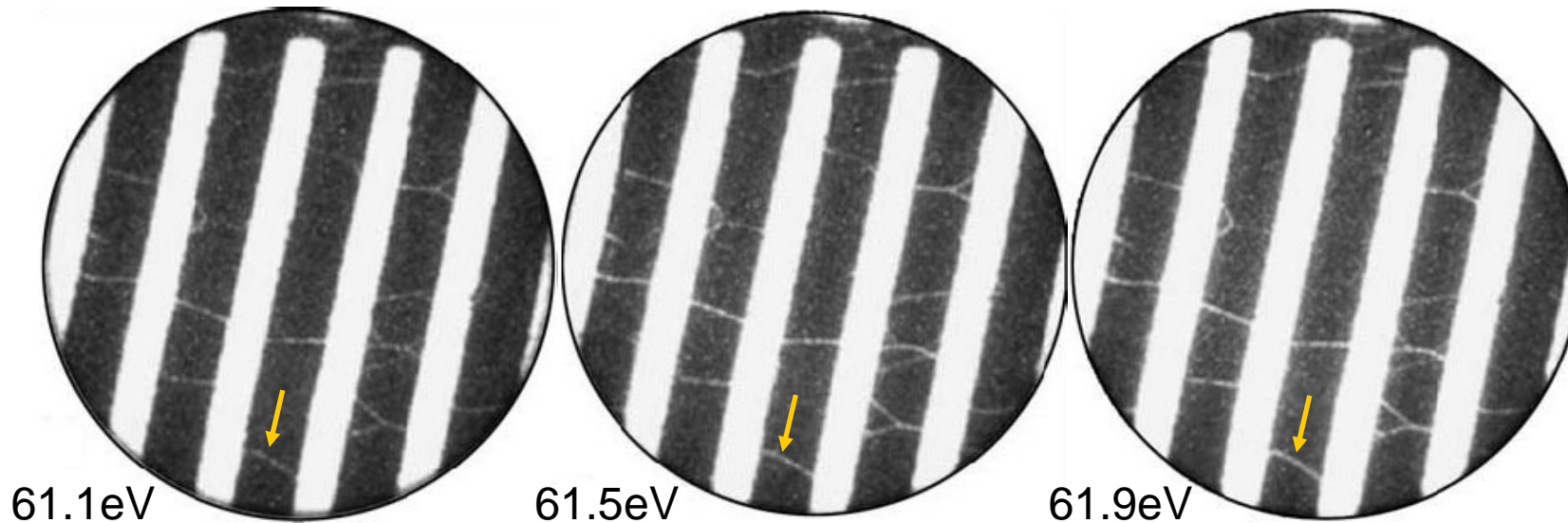
LEEM

# C 1s core level image

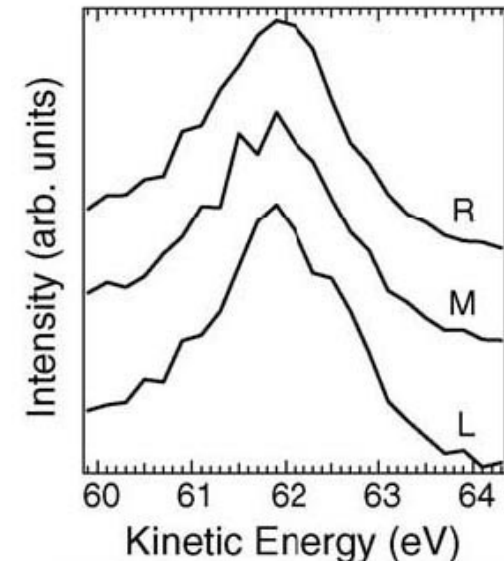


Photoelectron intensity profile along the broken line.

# C 1s core level spectra



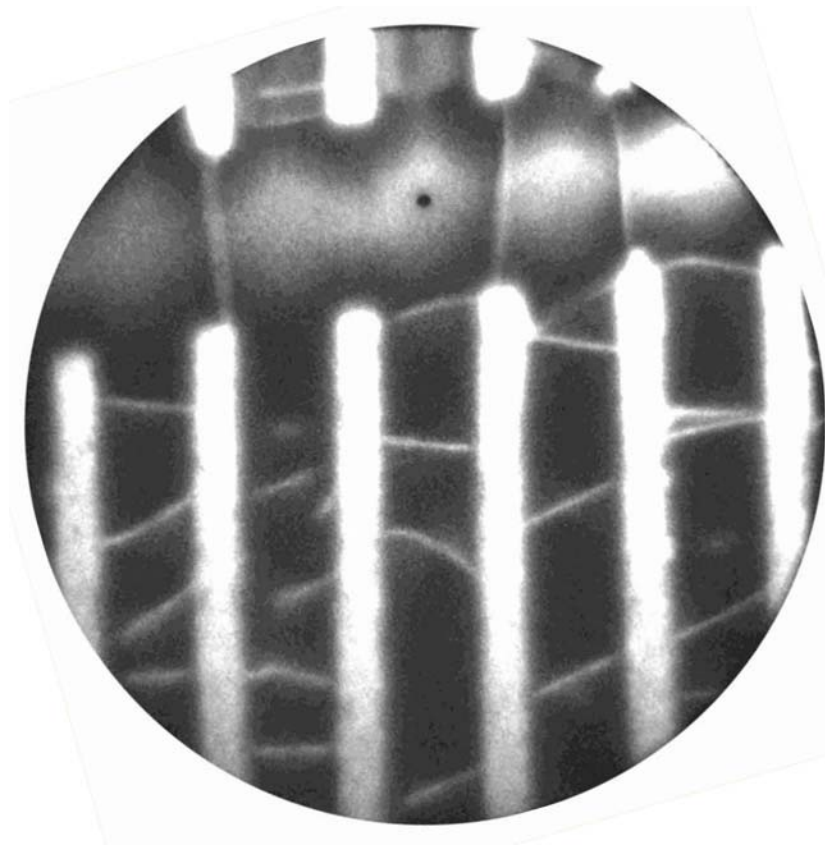
- No band bending along tube axis
- Depletion width < 40 nm



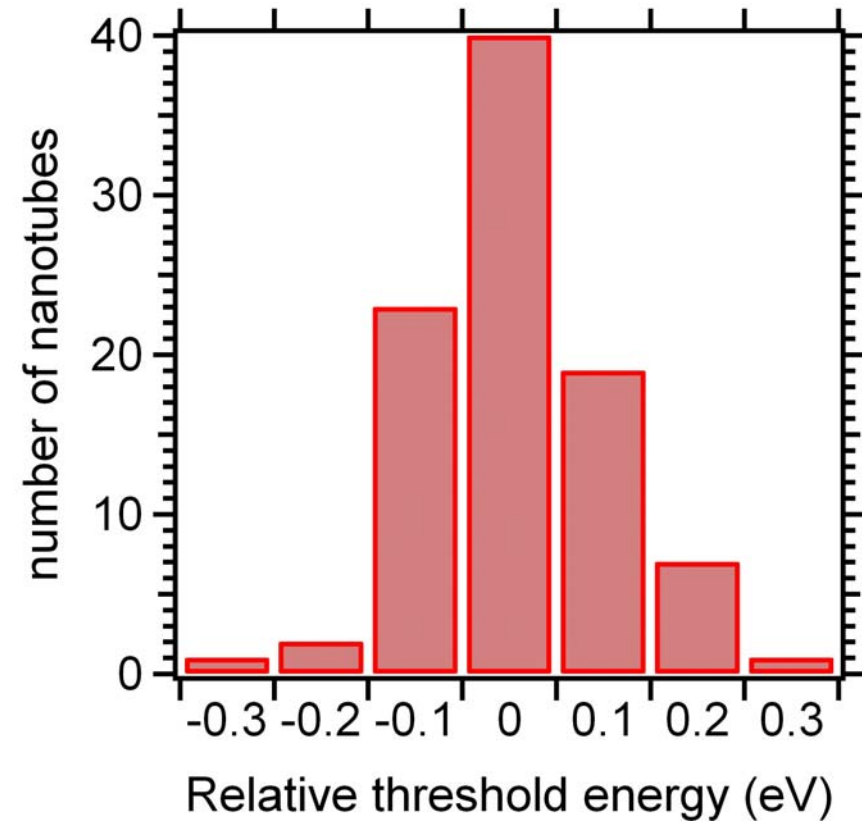
S. Suzuki et al.: J. Electron Spectrosc.  
Relat. Phenom. **144-147** (2005) 357.



# CNT work function



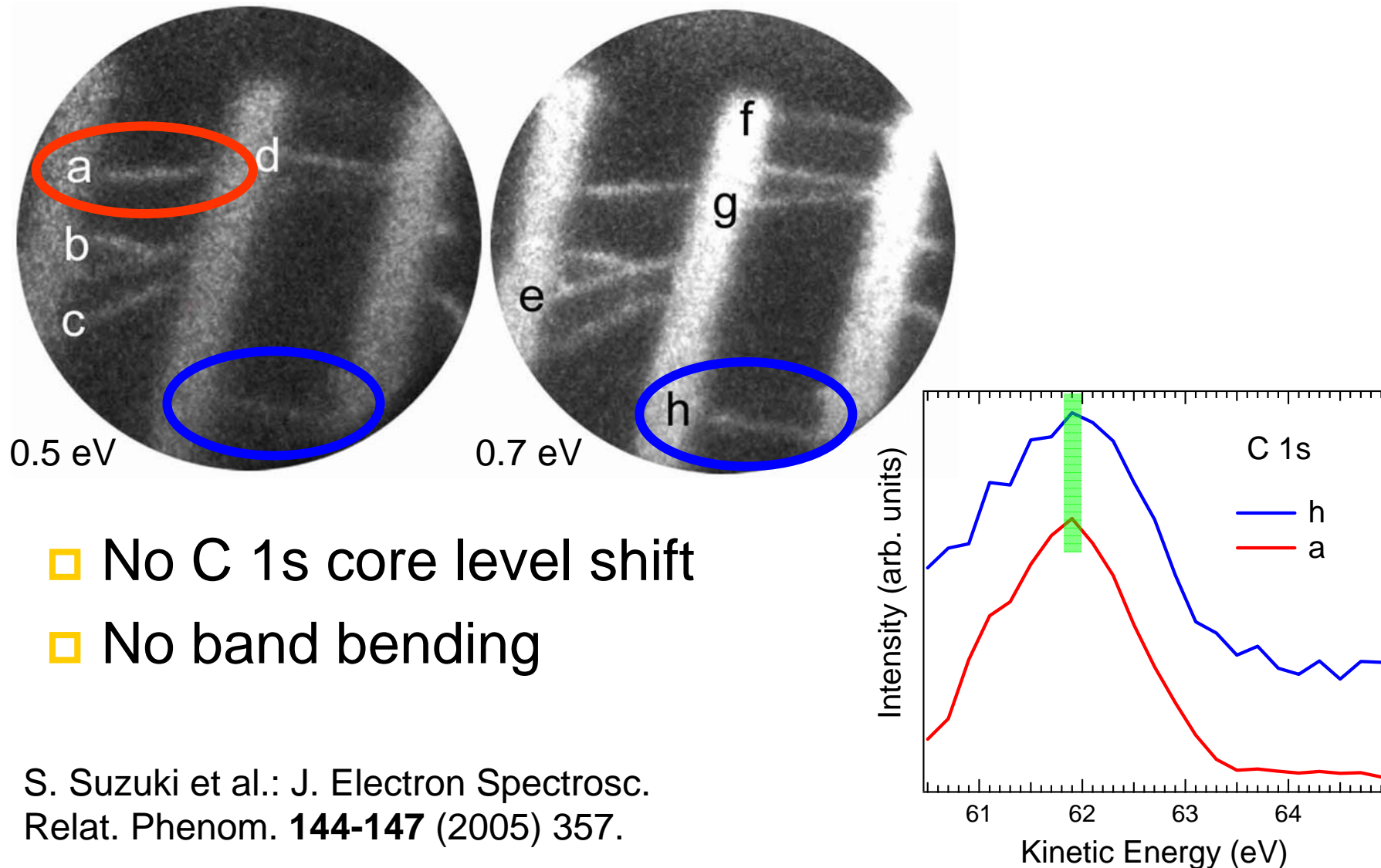
- Secondary electron PEEM image



- Work function distribution of 93 SWNT

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

# Secondary electron PEEM images





# Conclusions

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- X-ray microscopy in combination with x-ray spectroscopy provides information on:
  - Elemental composition
  - Sample chemistry
  - Electronic properties
  - Magnetic properties
- Today's x-ray spectromicroscopes offer
  - Some ten nanometer lateral resolution
  - Sufficient energy resolution (some 100 meV)
- Ideal tools for analysis of nanostructures