Spectromicroscopic investigations of nanostructured materials for device applications

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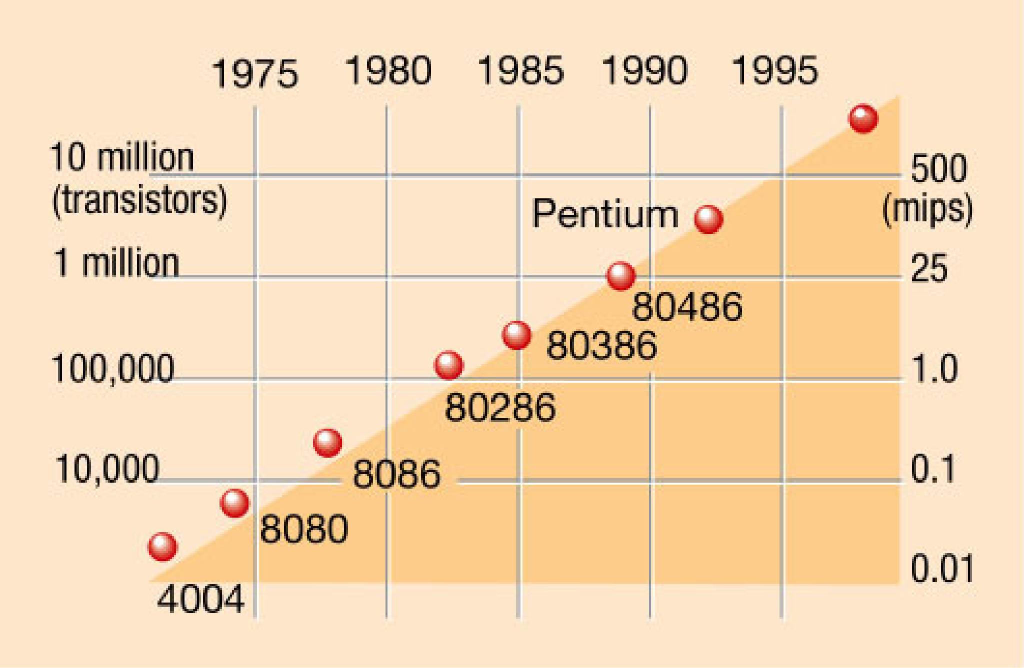
Motivation

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



Nature **406** (2000) 118.

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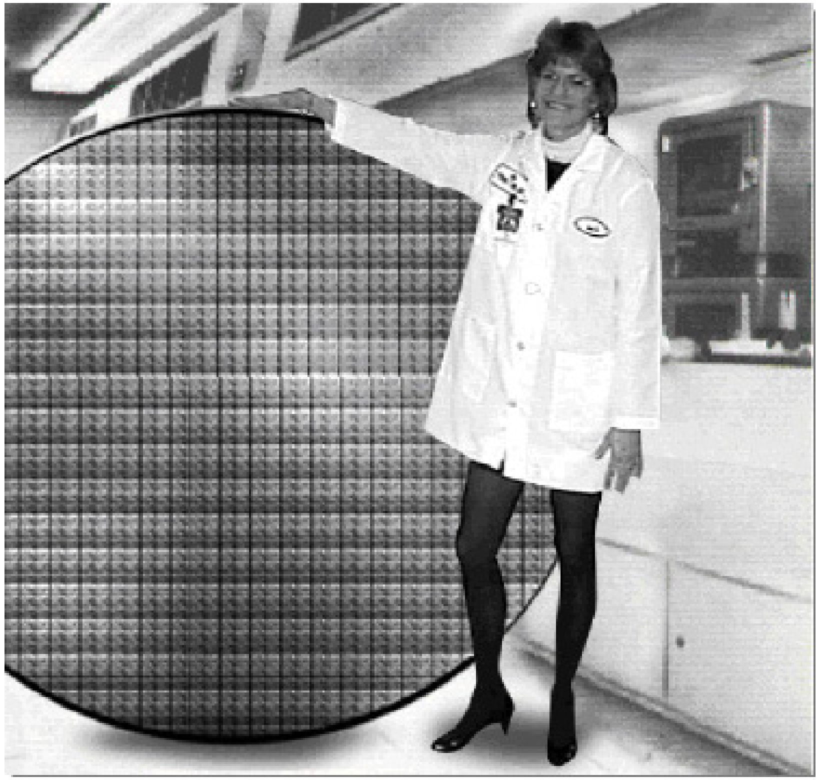
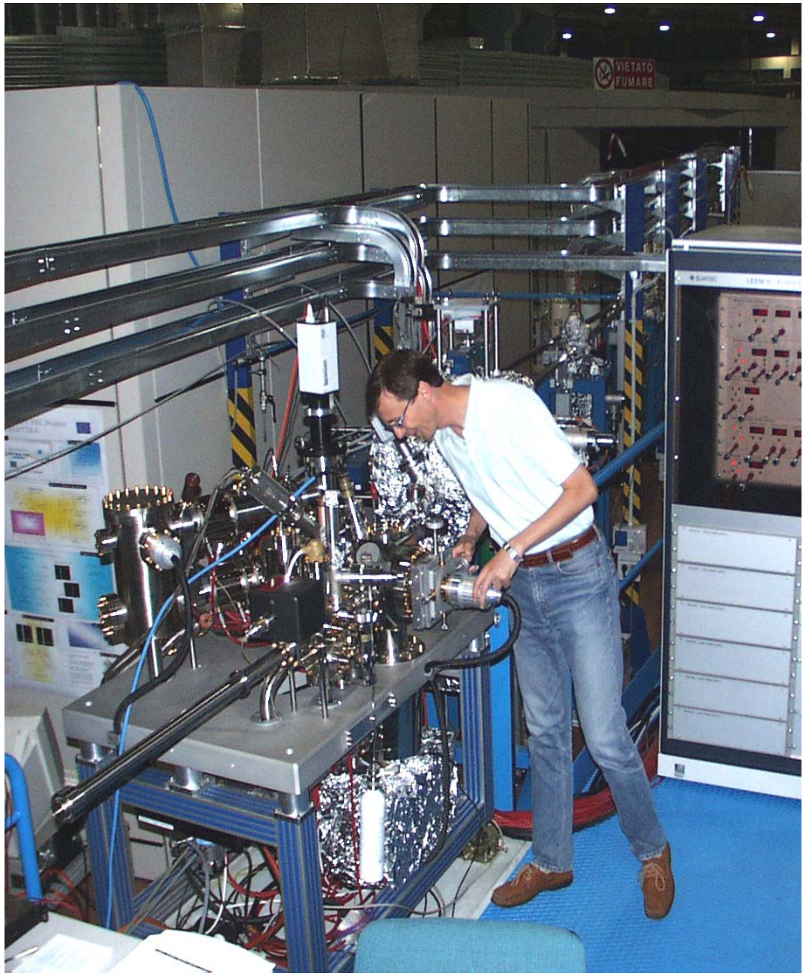


Motivation

- 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

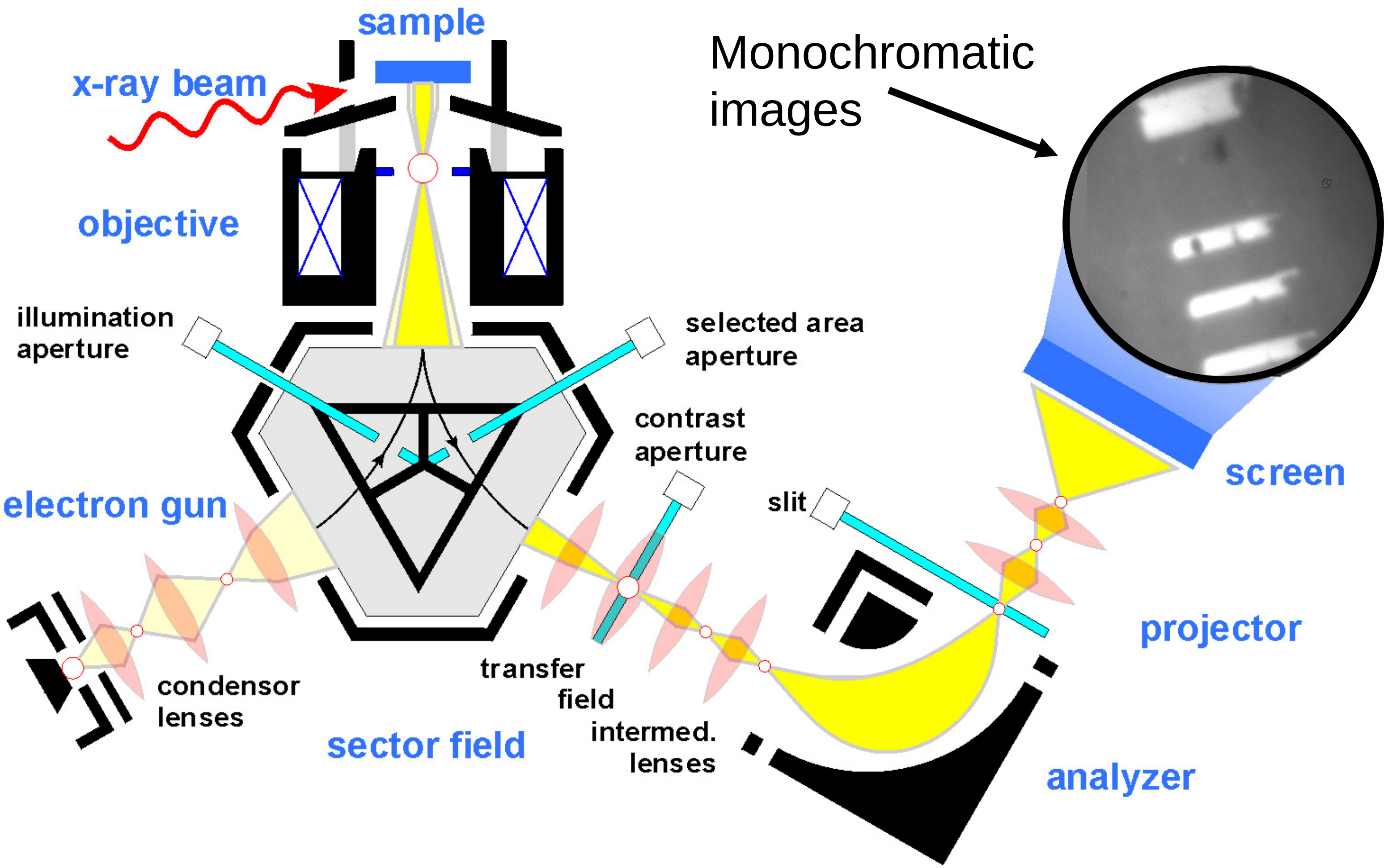


- Best energy resolution: 250 meV
- Best lateral resolution: 25 nm

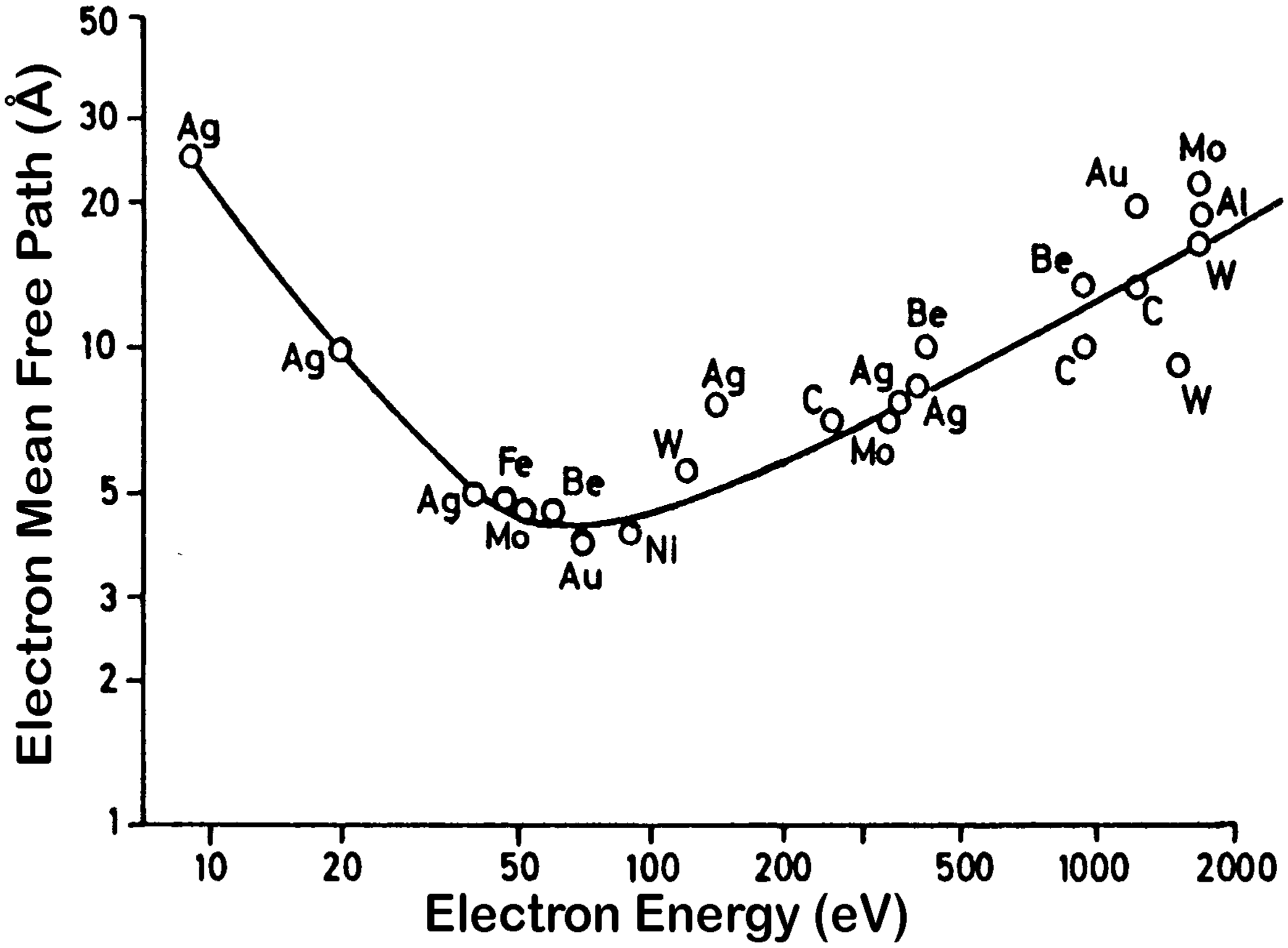
- Variable polarization
- 20 - 1000 eV
- Photon flux 10^{13} ph/s
- Small spot (2µm x 25 µm)

The SPELEEM instrument

Spectroscopic Photo-Emission and Low Energy Electron Microscope



Photoelectron Mean Free Path



M. P. Seah and W. A. Dench: Surf. Interface Anal. **1** (1979) 2.

Model Material Systems

- 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

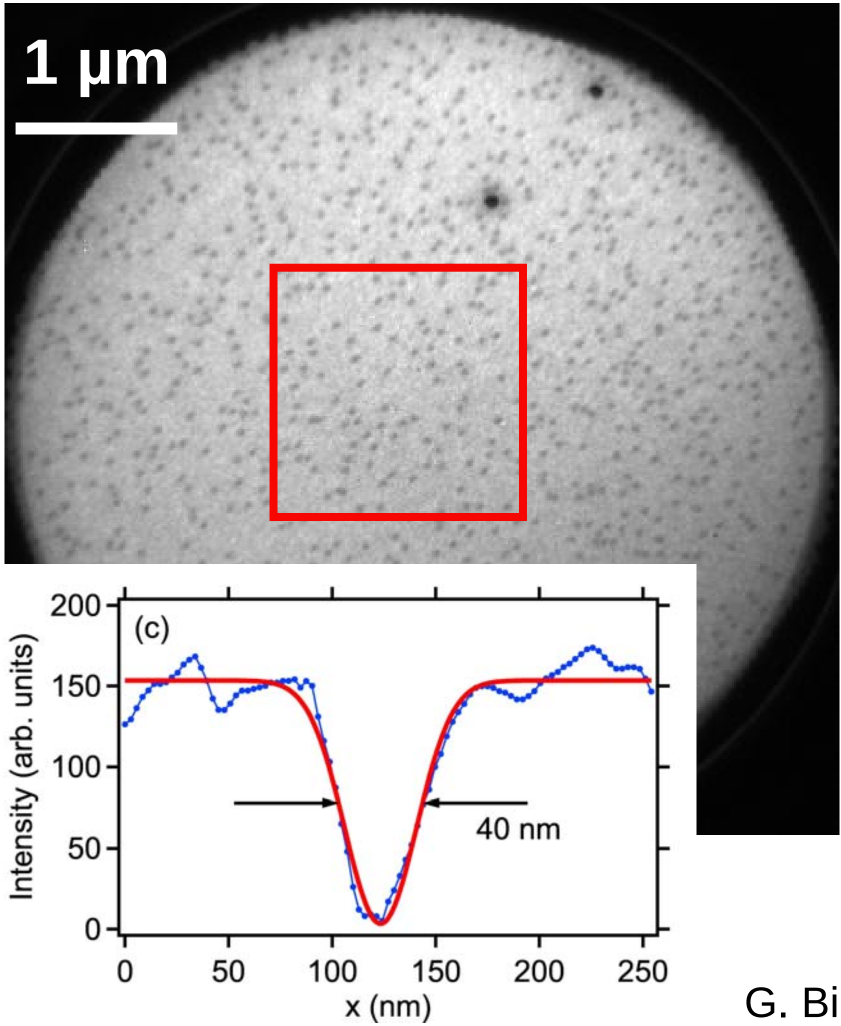
- AMD Group @ TASC
  - Giorgio Biasiol
  - Giovanni Golinelli
  - Lucia Sorba

- Nanospectroscopy Beamline @ Elettra
  - Andrea Locatelli
  - Tevfik Onur Mentes
  - Fangzhun Guo (Spring-8)

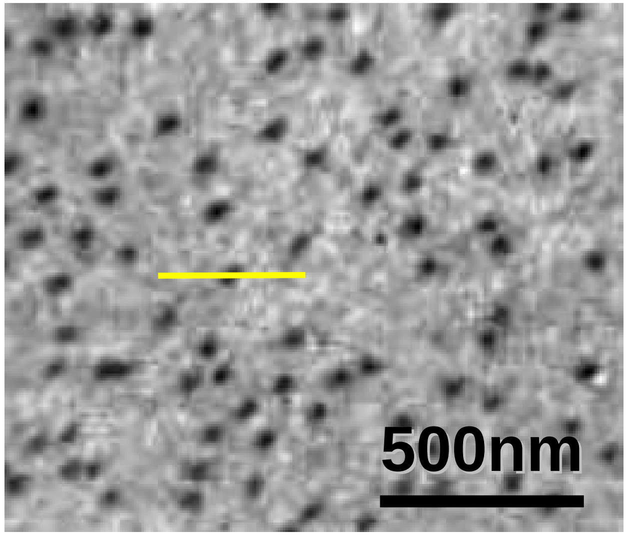
Motivation

- Quantum Dot Applications based on their particular electronic properties (confinement)
- Strain-driven self-assembly (SK-growth)
- Model systems: InAs/GaAs, Ge/Si
- Intermixing and alloying allow for partial strain relaxation
- Composition (gradients) within the dot influence energy levels and shift the emission wavelength

InAs/GaAs Islands (LEEM)

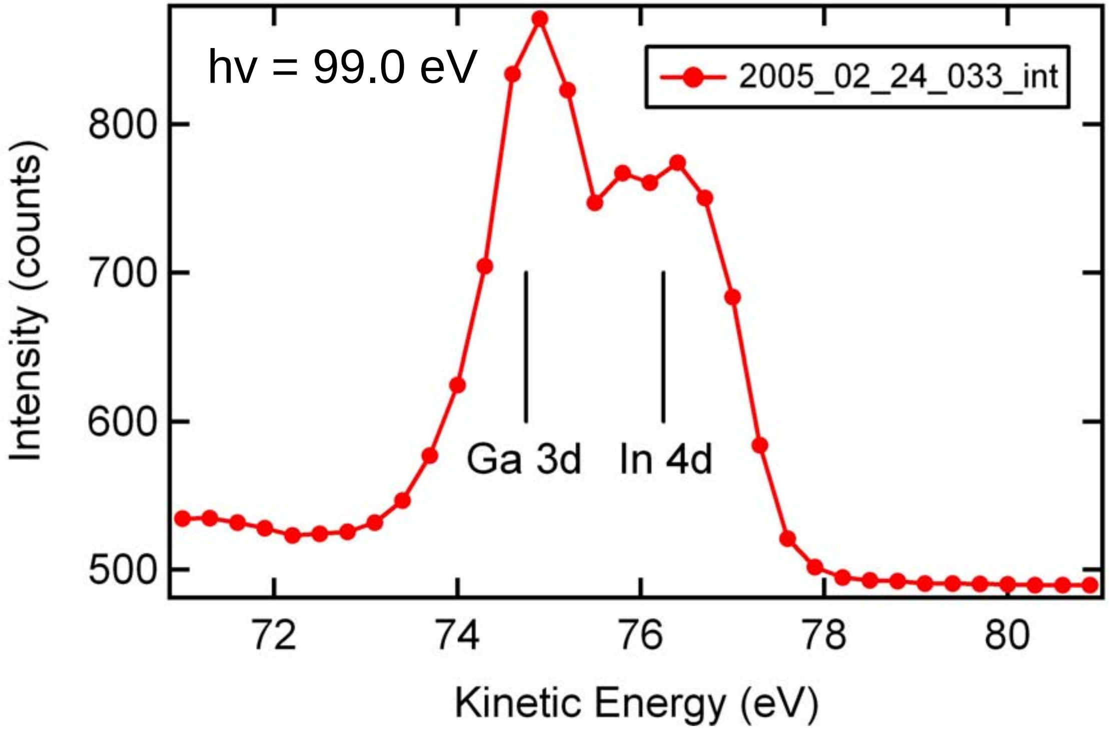


- Electron Microscopy
- LEEM
- 5 µm FOV
- E_{kin} = 7.6 eV



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

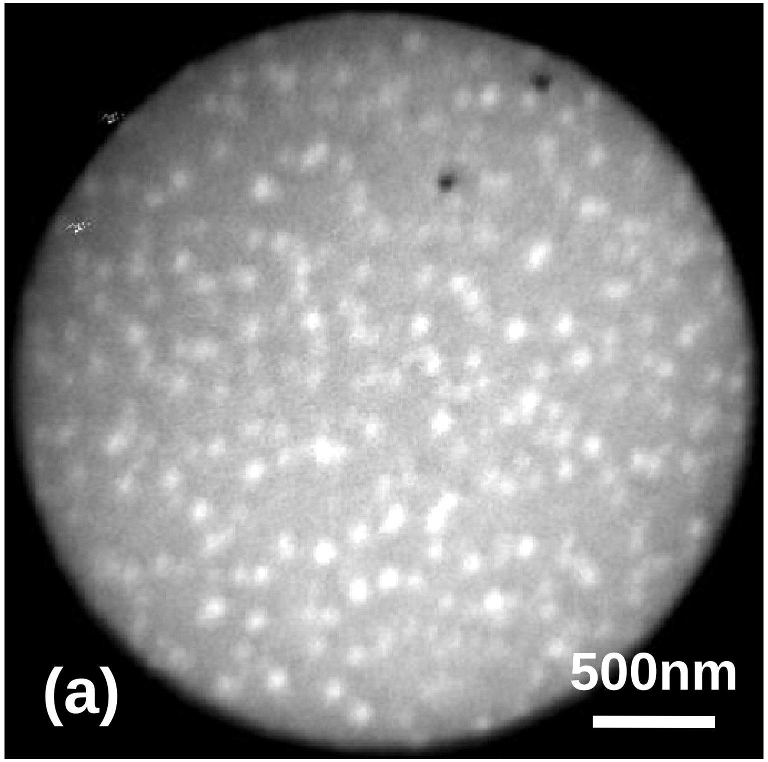
“Integral” Core Level Spectra



- Spectra taken from a 1 µm x 1 µm sample area.
- III-V stoichiometry after decapping confirmed.

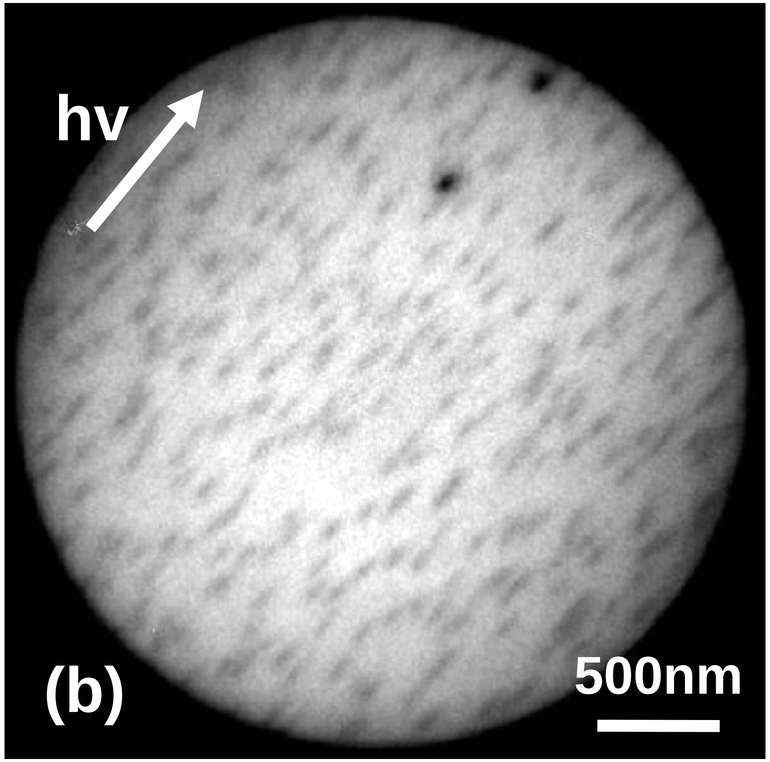
G. Biasiol et al.: Appl. Phys. Lett. **87** (2005) 223106.

XPEEM Core Level Imaging



In 4d XPEEM image

hv = 99.0 eV, E_{kin} = 76.25 eV

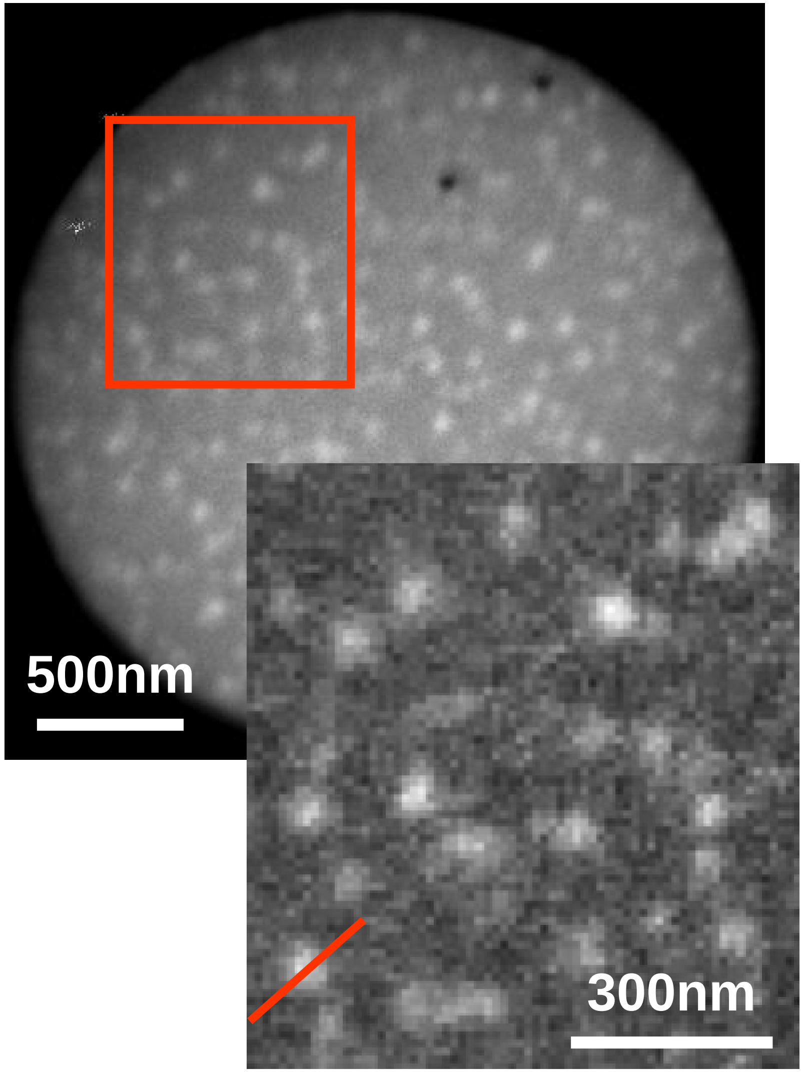


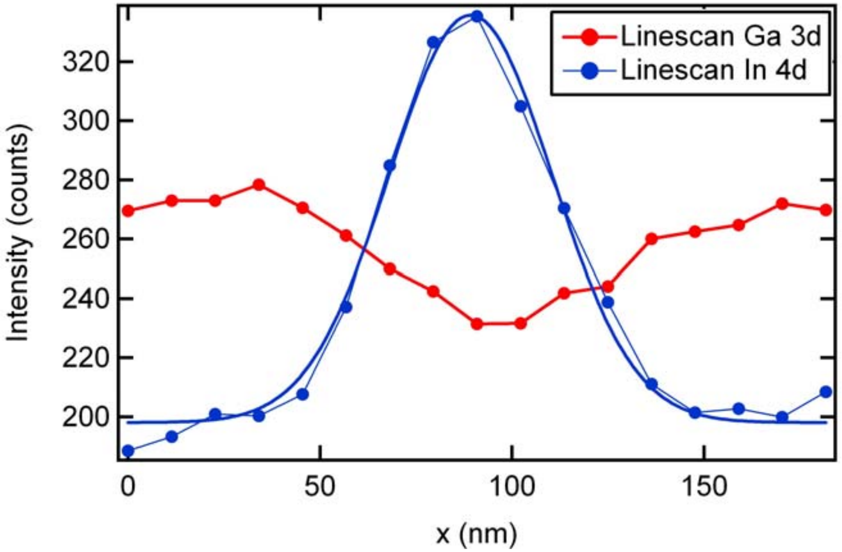
Ga 3d XPEEM image

hv = 99.0 eV, E_{kin} = 74.75 eV

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

Island Size in XPEEM

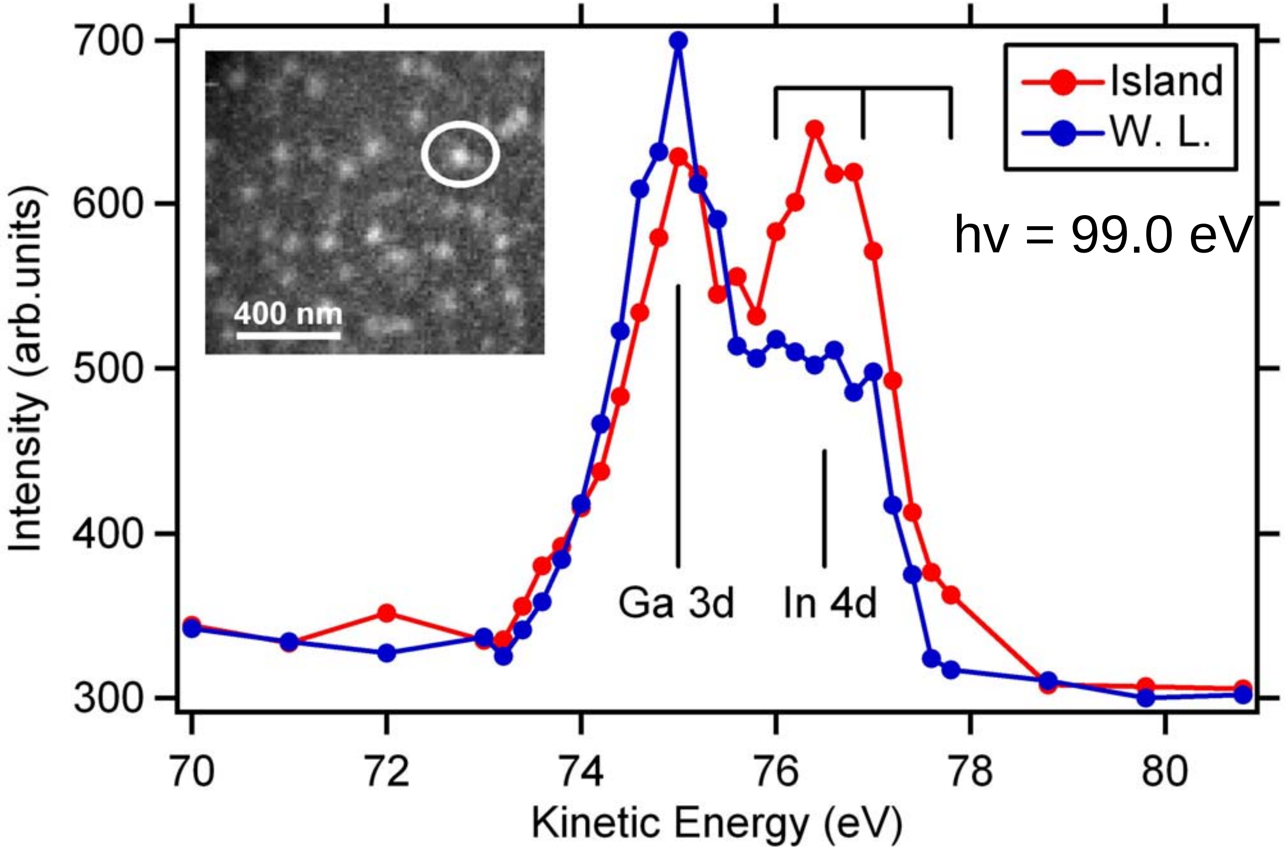




Island Size ≈ 60 nm,
consistent with
LEEM and TEM

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

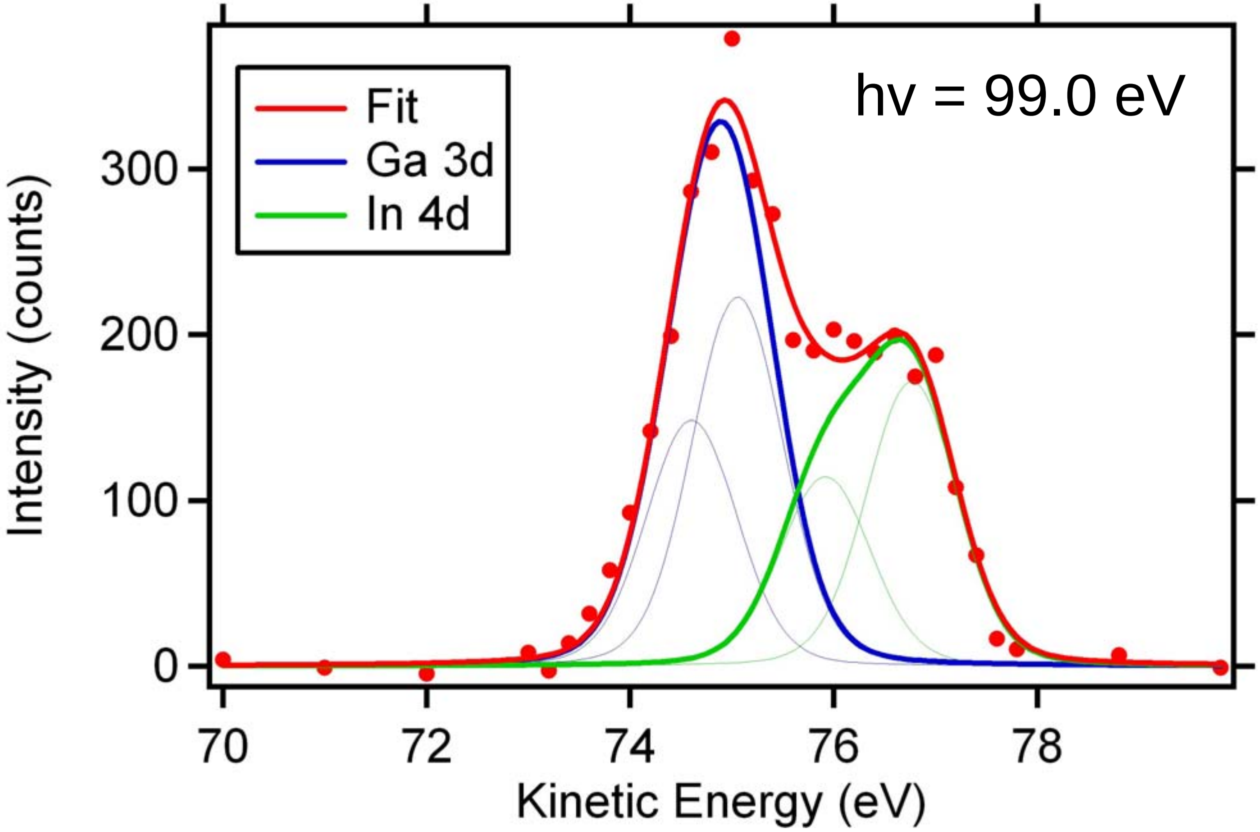
XPEEM Local Spectra



Integration area 25 nm x 25 nm, energy resolution ≈ 1 eV

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

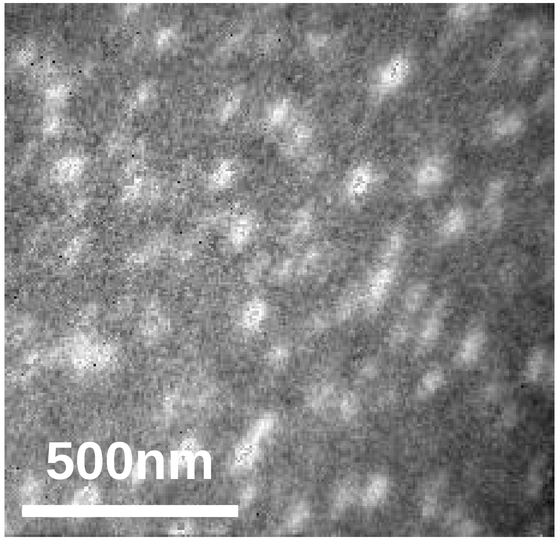
Core Level Line Profile Analysis



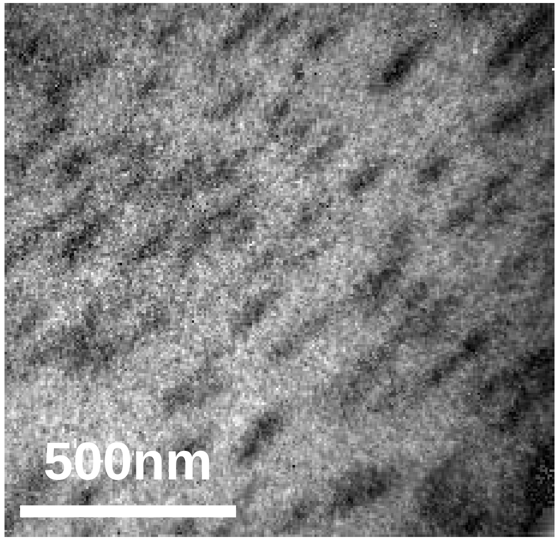
Spectrum from Wetting Layer, Shirley Background subtracted
Gauss 1 eV, Lor 0.16 eV, BR 1.5, SO: Ga 3d 0.45 eV, In 4d 0.85 eV

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

2D Fit of XPEEM Data



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



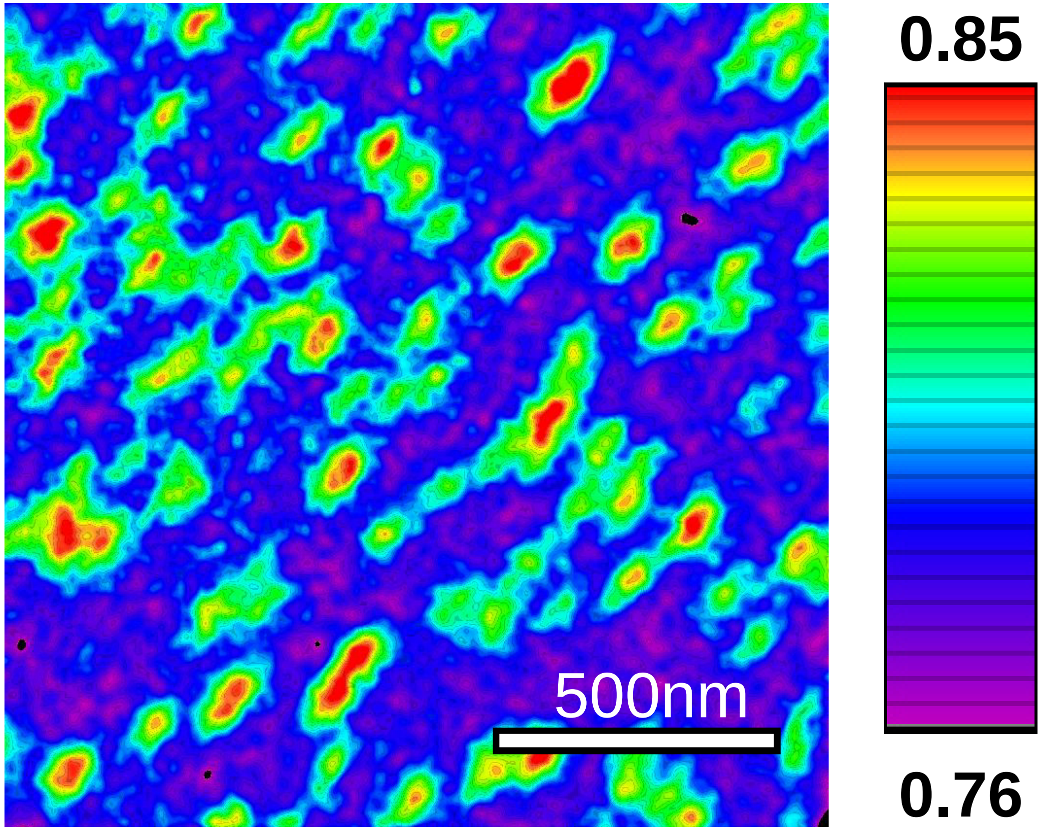
Ga 3d peak area
Min: 270, Max: 470

Ratio of Number of Atoms: $$\frac{n_{In}}{n_{Ga}} = \frac{I_{In}}{I_{Ga}} \cdot \frac{\sigma_{Ga}}{\sigma_{In}}$$

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

Indium Surface Concentration Map

$$\frac{n_{In}}{n_{tot}} = \frac{I_{In}\sigma_{Ga}}{I_{In}\sigma_{Ga} + I_{Ga}\sigma_{In}}$$



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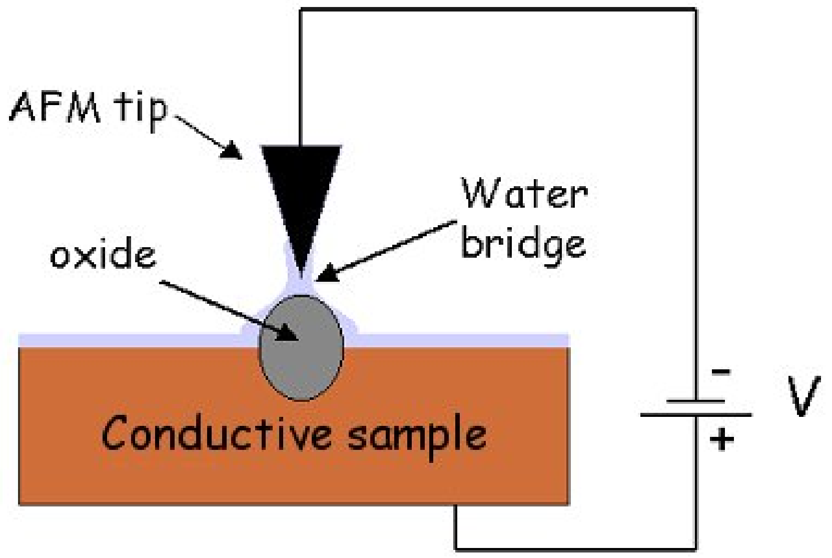
Local Anodic Oxidation on GaAs

- AMD Group @ TASC
  - Giorgio Mori
  - Marco Lazzarino
  - Daniele Ercolani
  - Giorgio Biasiol
  - Lucia Sorba

- Nanospectroscopy Beamline @ Elettra
  - Andrea Locatelli

Local Anodic Oxidation (LAO)



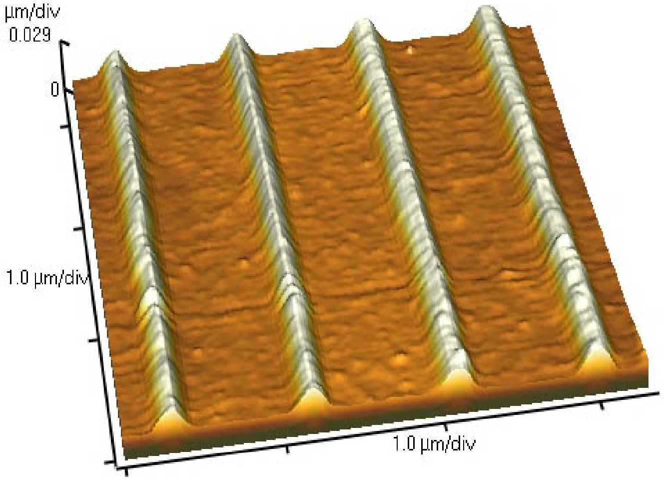
Common model:

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

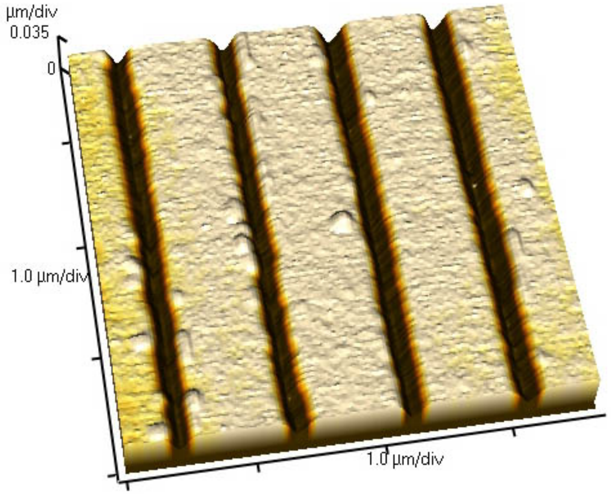
Versatile tool at relatively low cost

High lateral resolution but small area

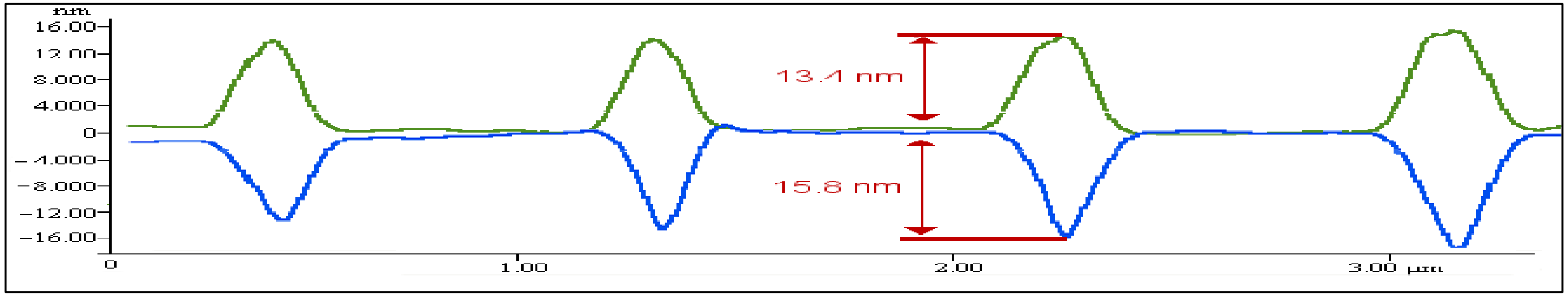
Local Anodic Oxidation (LAO)



After oxidation



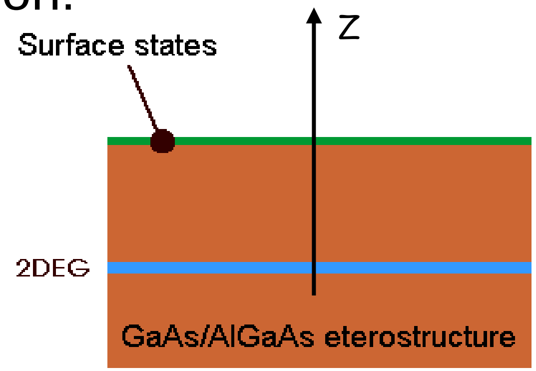
Oxide removal with HF 10%, 30 s



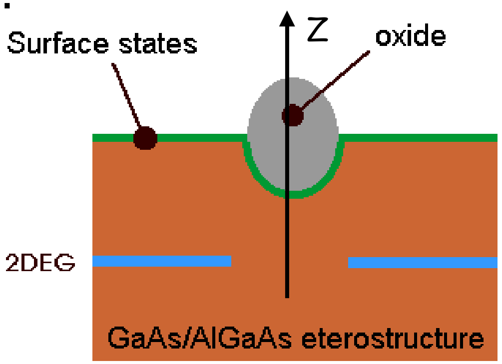
The penetration depth is 1.0-1.5 times the oxide height

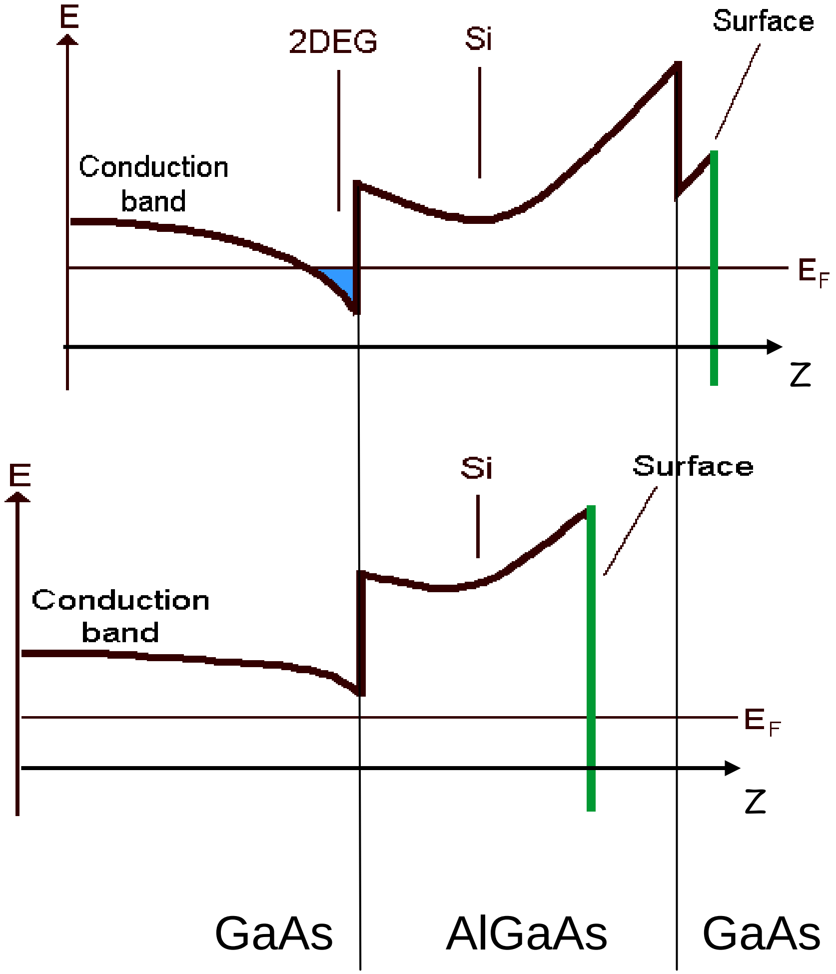
LAO on GaAs/AlGaAs

Before oxidation:

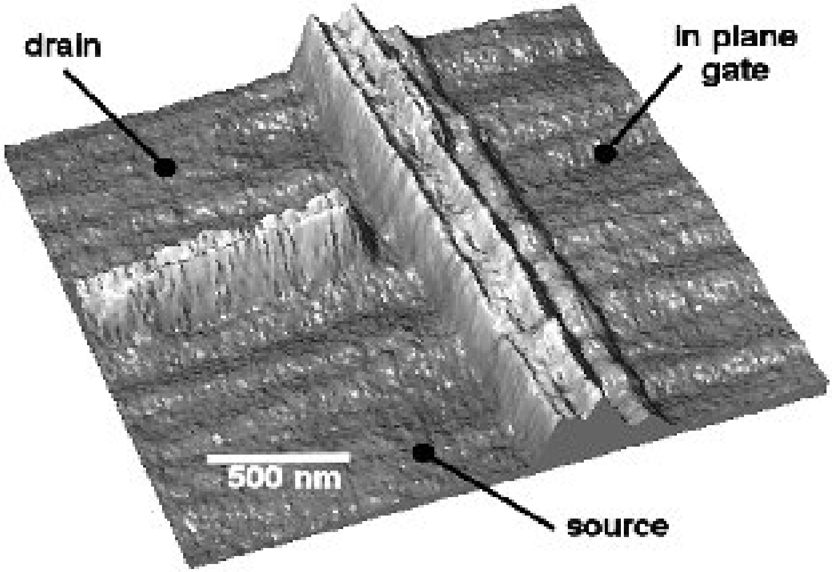


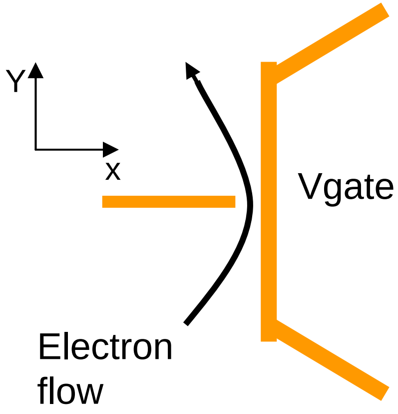
After oxidation:

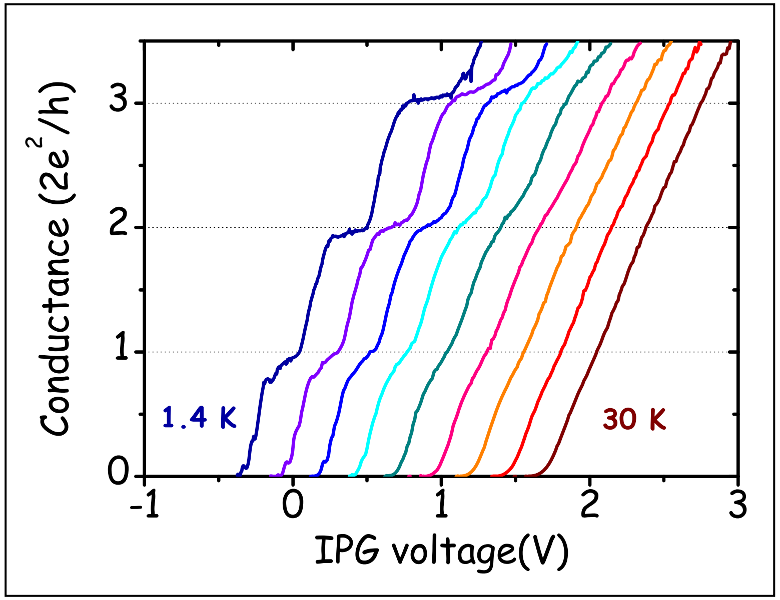




Quantum Point Contact

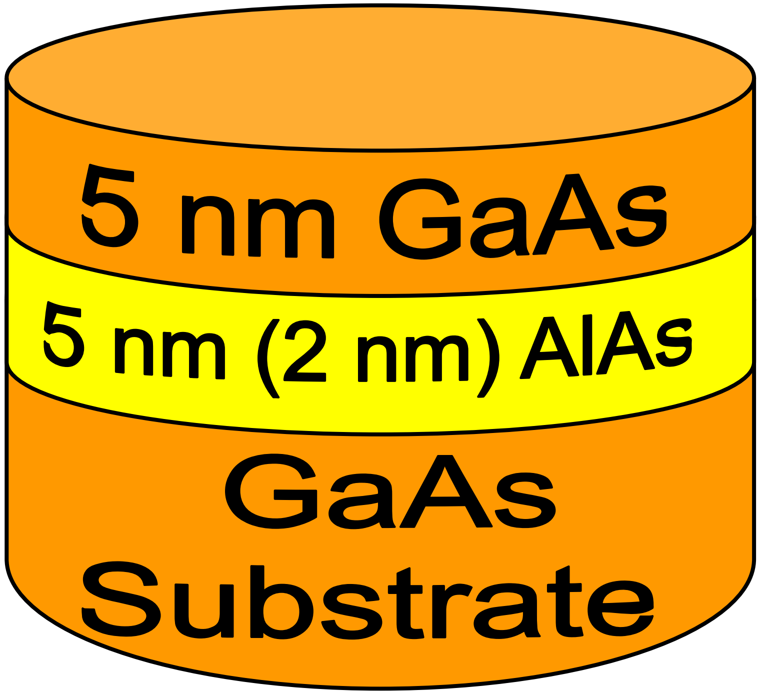






G. Mori et al, J. Vac. Sci. Technol. B **22** (2004) 570.

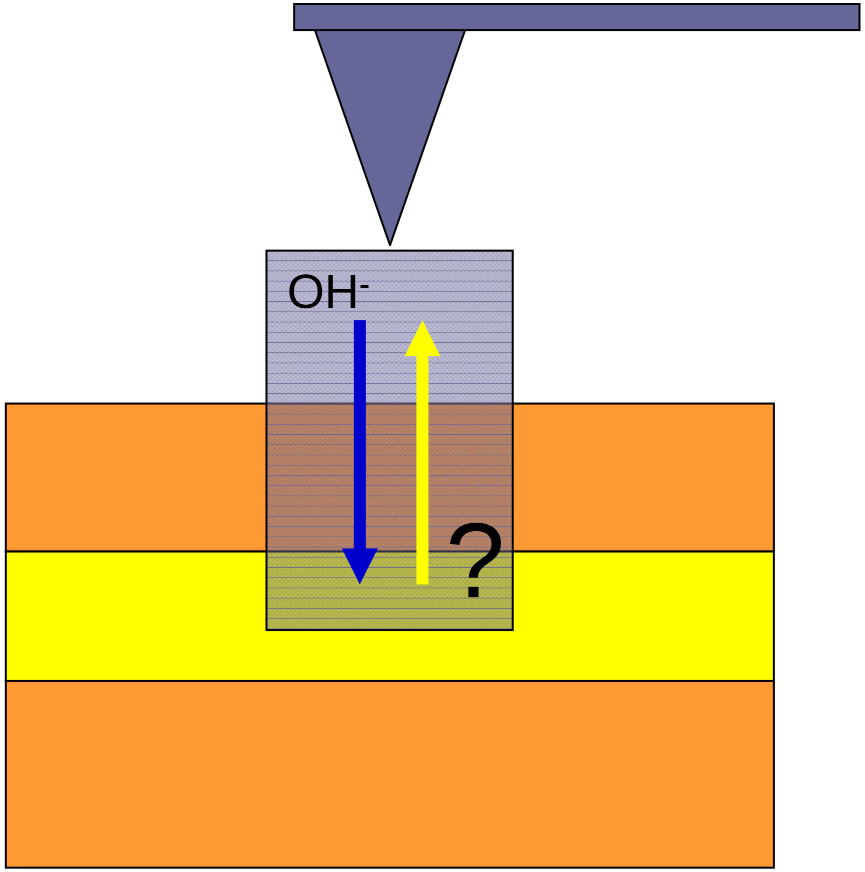
The dynamics of the LAO process



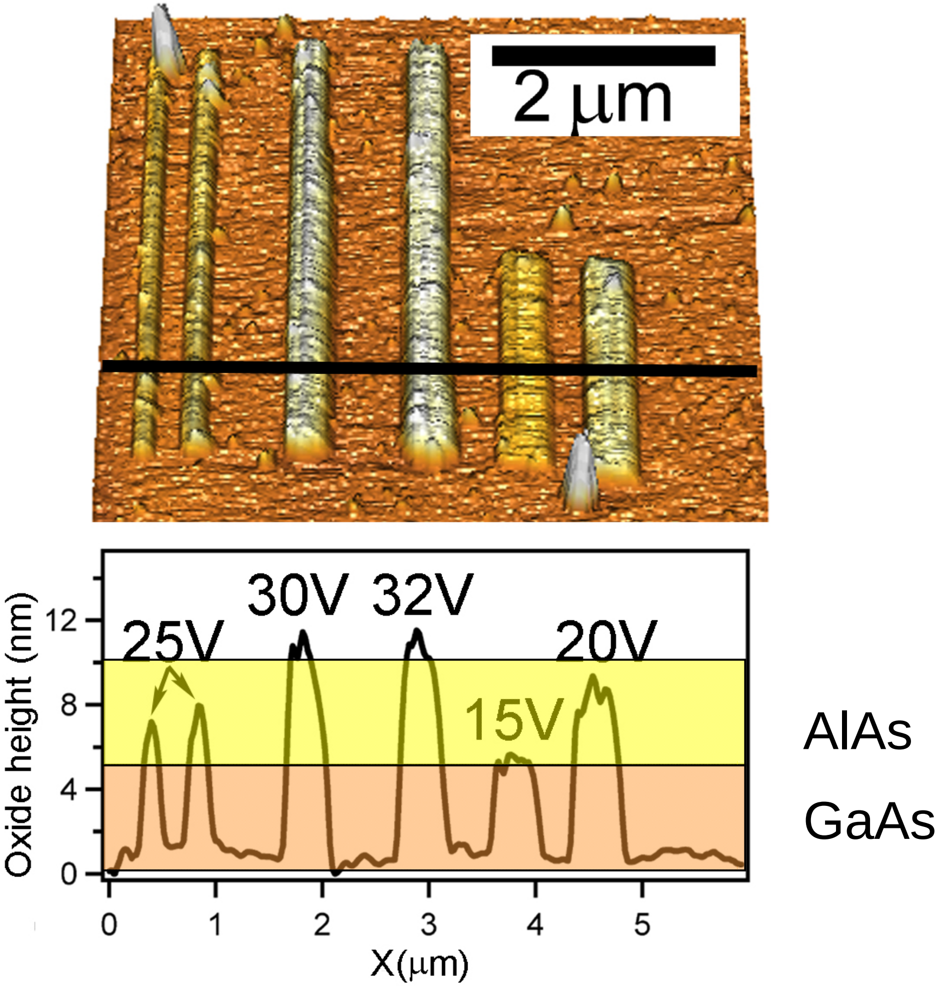
5 nm GaAs

5 nm (2 nm) AlAs

GaAs Substrate

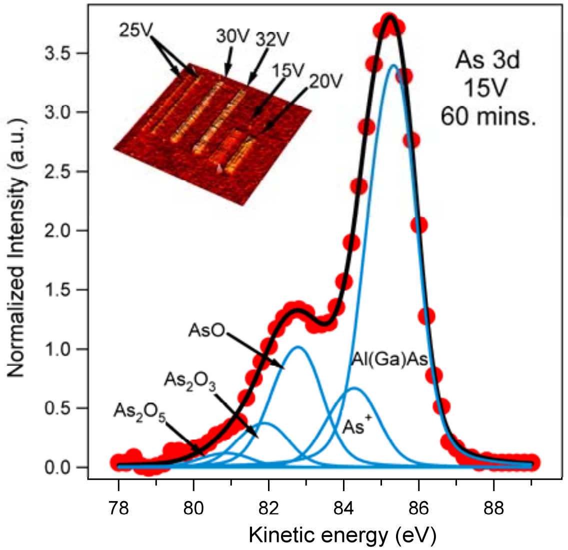


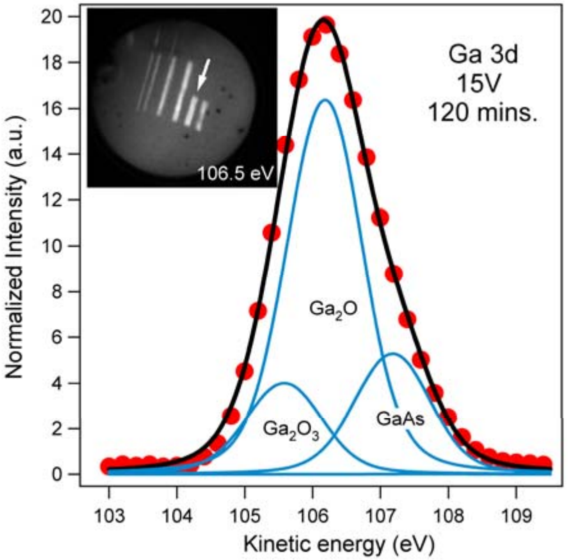
LAO on III-V Heterostructures

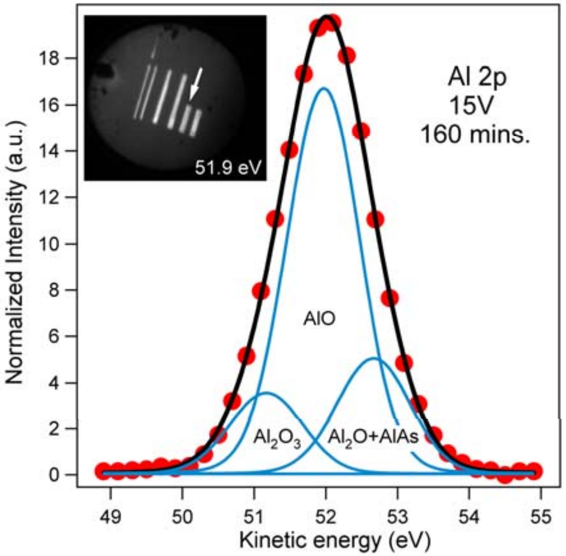


G. Mori et al.: J. Appl. Phys. **98** (2005)114303.

Chemical composition of LAO oxide



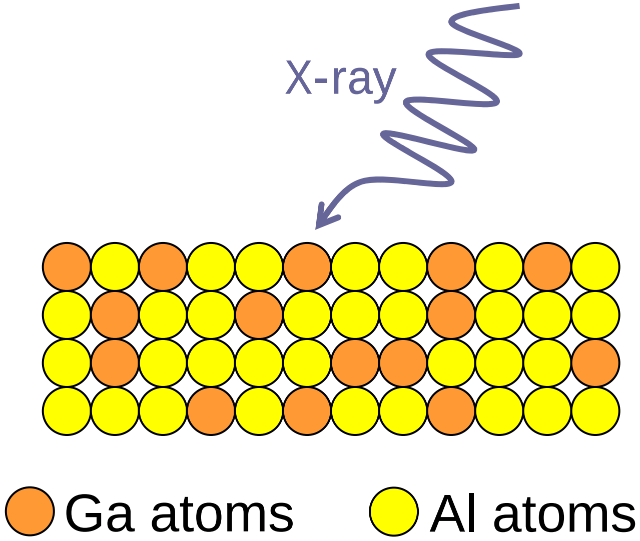




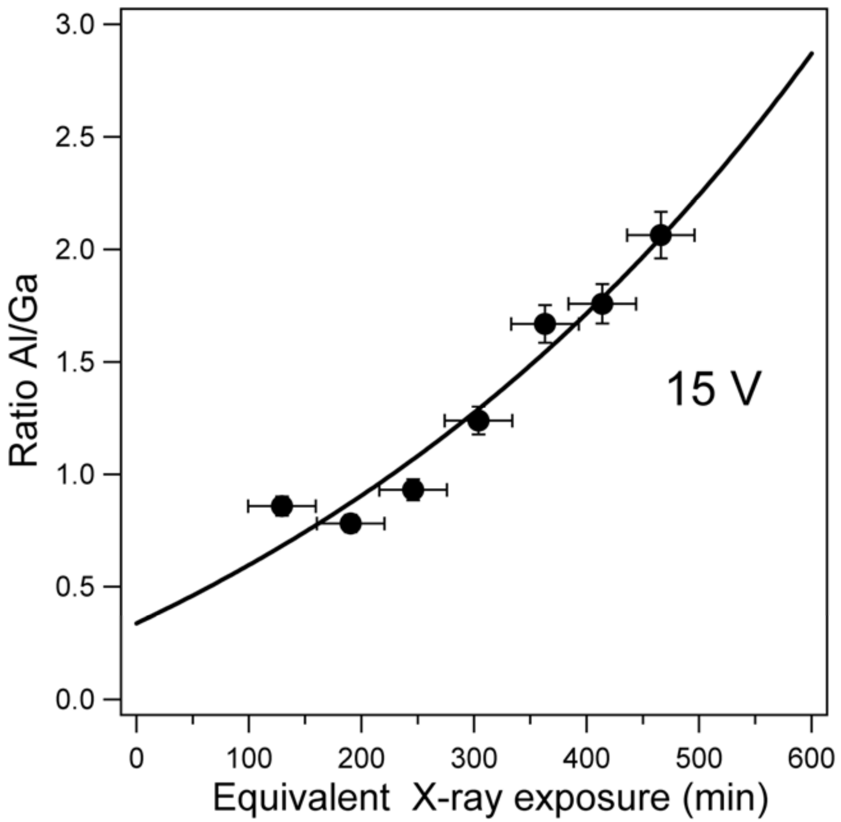
- Aluminium observed at the surface of the LAO oxide
- No Al in the regions not oxidized with LAO
- Time-resolved study: Ga-oxides desorb under x-rays (much faster than Al-oxides)

G. Mori et al.: Nucl. Instr. and Meth. B, in press.

The effect of X-ray exposure



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



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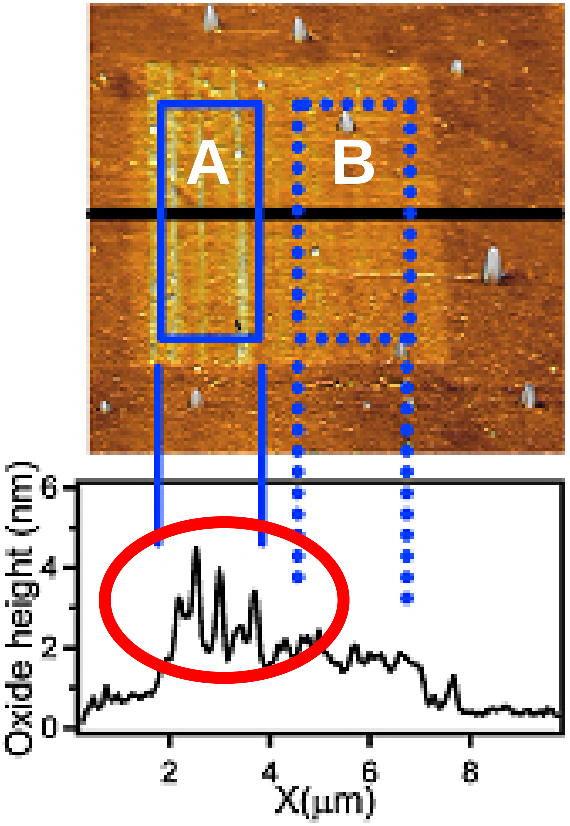
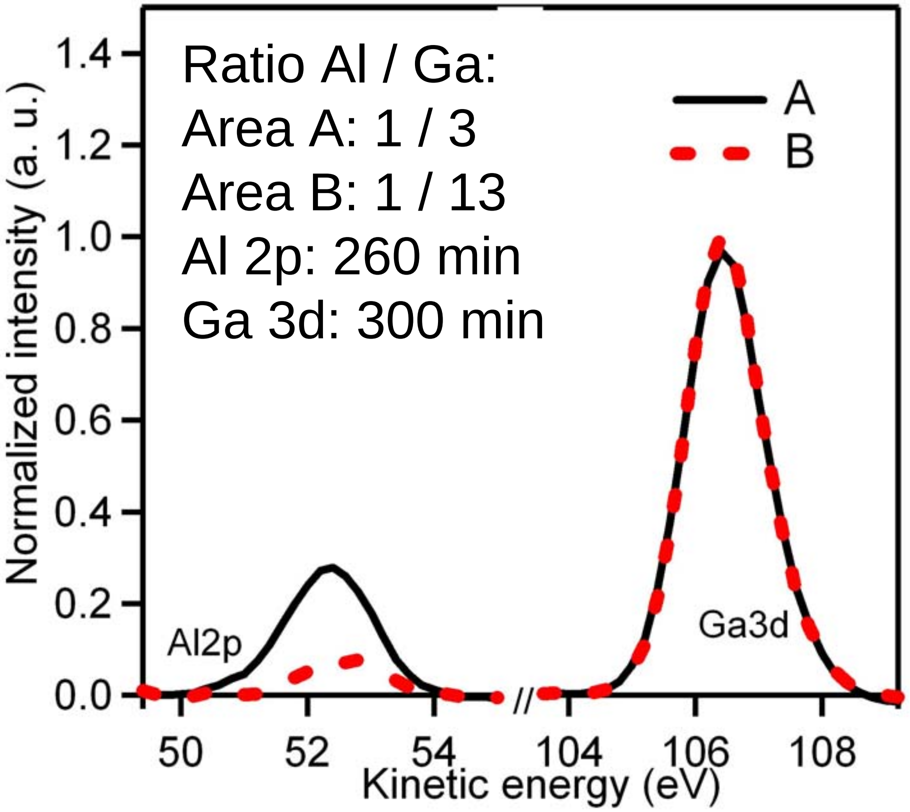
Shallow Oxidations



Average oxide height:
Area A: 1.9 ± 0.7 nm
Area B: 1.3 ± 0.3 nm

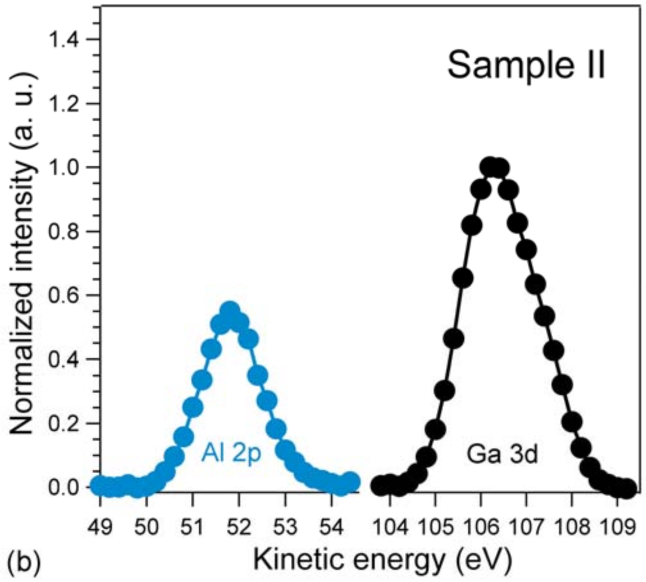

Ratio Al / Ga:
Area A: 1 / 3
Area B: 1 / 13
Al 2p: 260 min
Ga 3d: 300 min

Al content depends strongly on oxide height

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

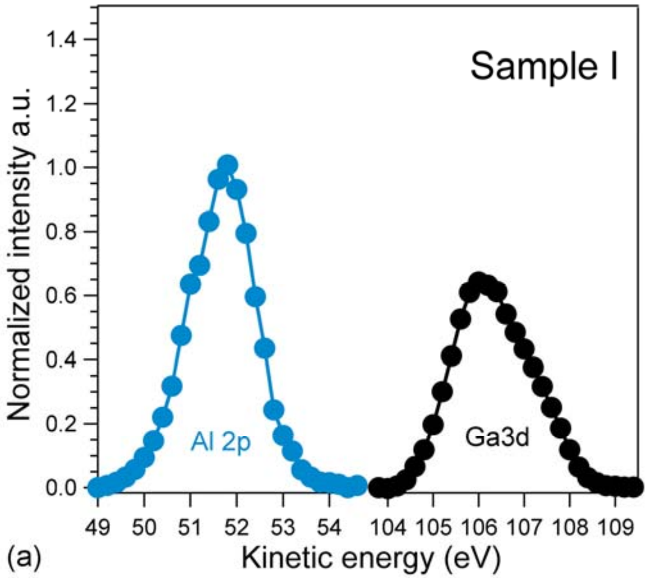
Variation in AlAs layer thickness

- 2 nm AlAs
- Oxide height 9.0 ± 1.0 nm



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

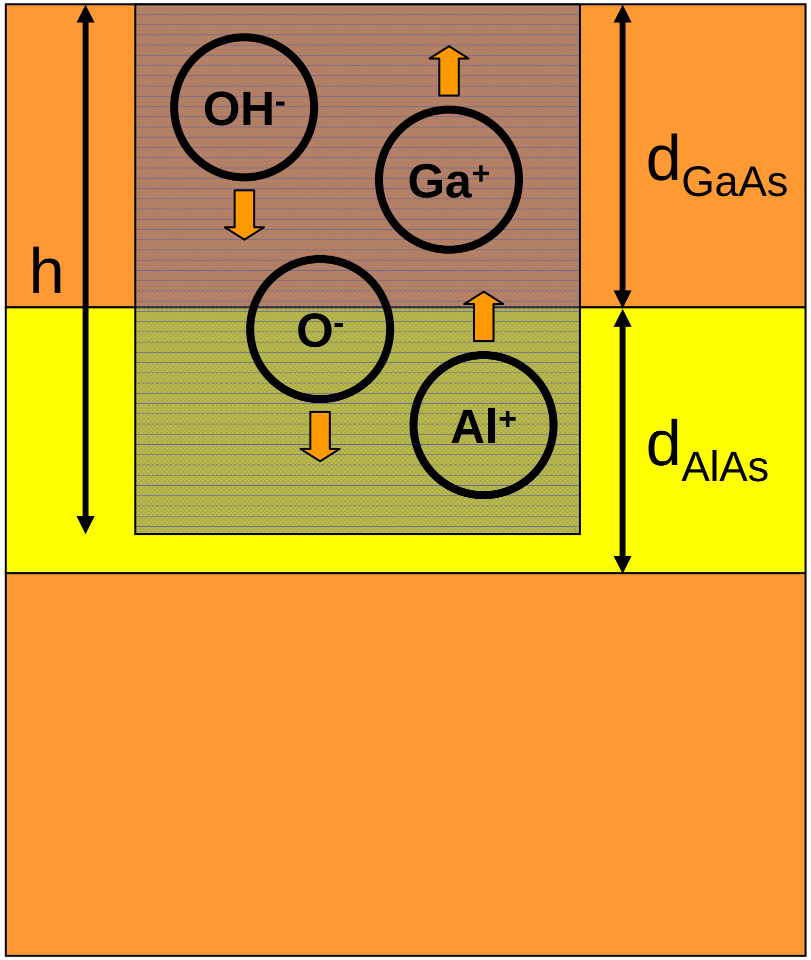
- 5 nm AlAs
- Oxide height 8.9 ± 0.9 nm



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

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

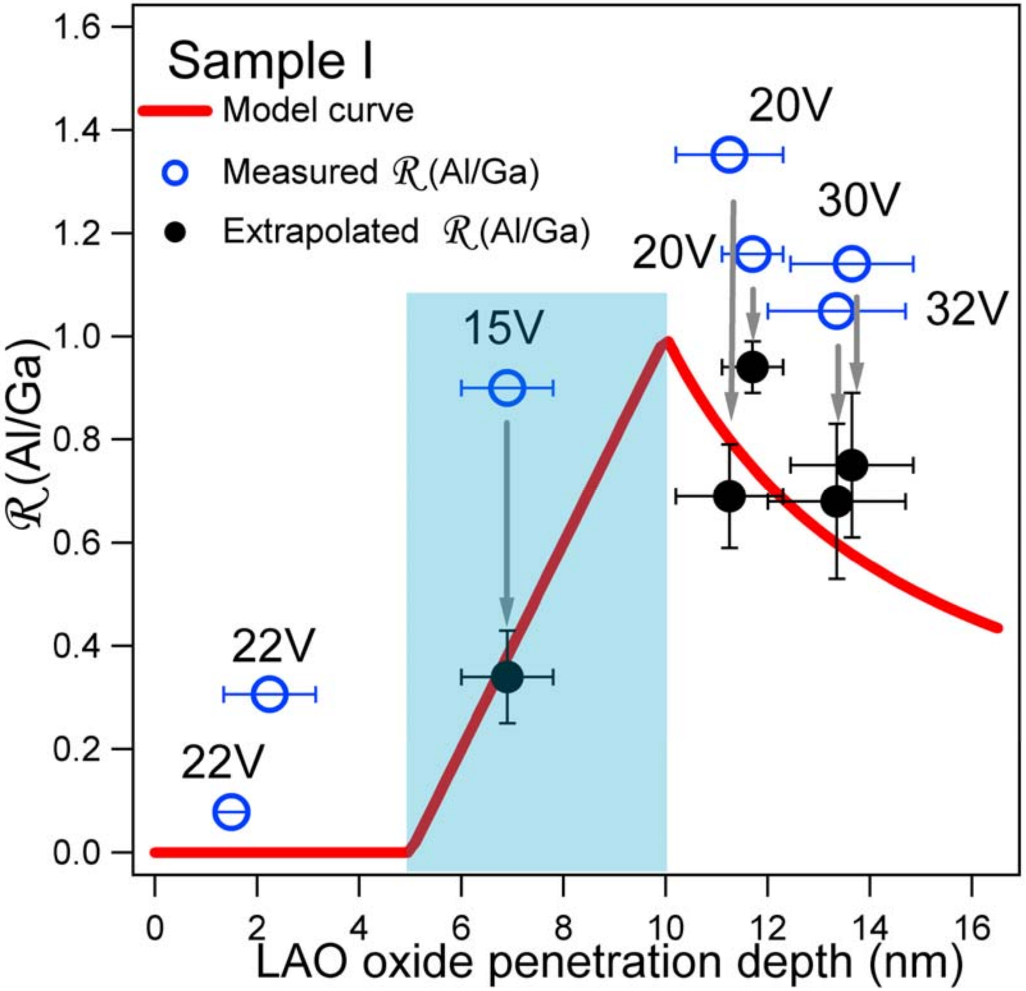
Refined Model



- 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 $h > d_{GaAs} + d_{AlAs}$

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

Comparison with Experiment



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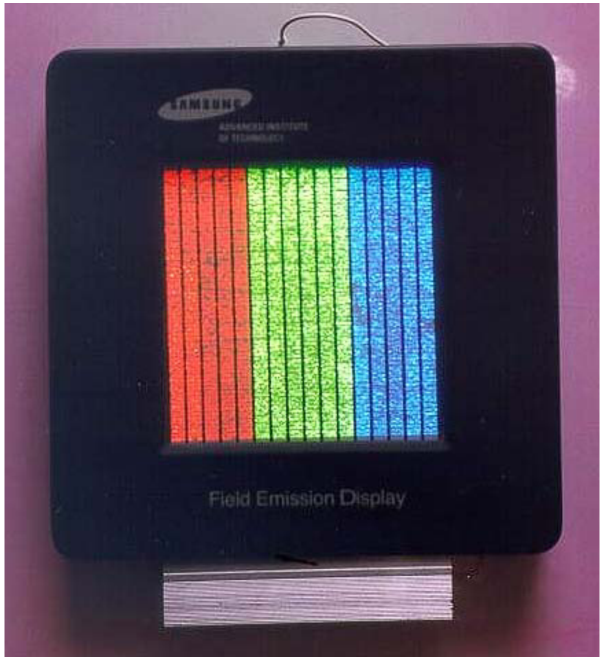
Suspended Carbon Nanotubes

- NTT Basic Research Laboratories
  - Satoru Suzuki
  - Yoshio Watanabe
  - Yoshikazu Homma
  - Shin-ya Fukuba

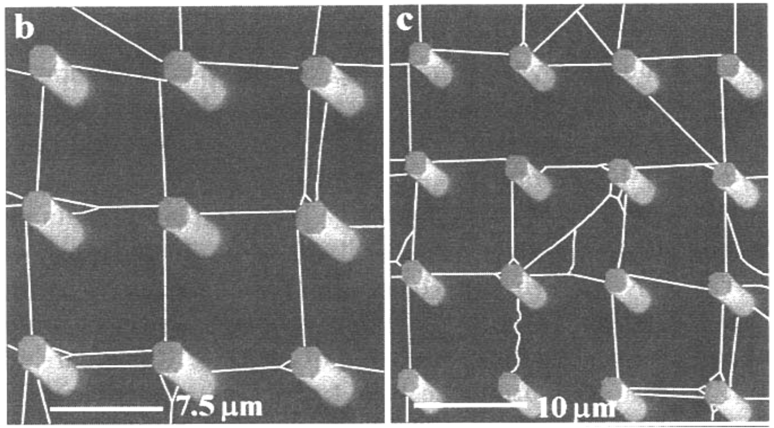
- Nanospectroscopy Beamline @ Elettra
  - Andrea Locatelli

Motivation

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



APL **75** (1999) 3129

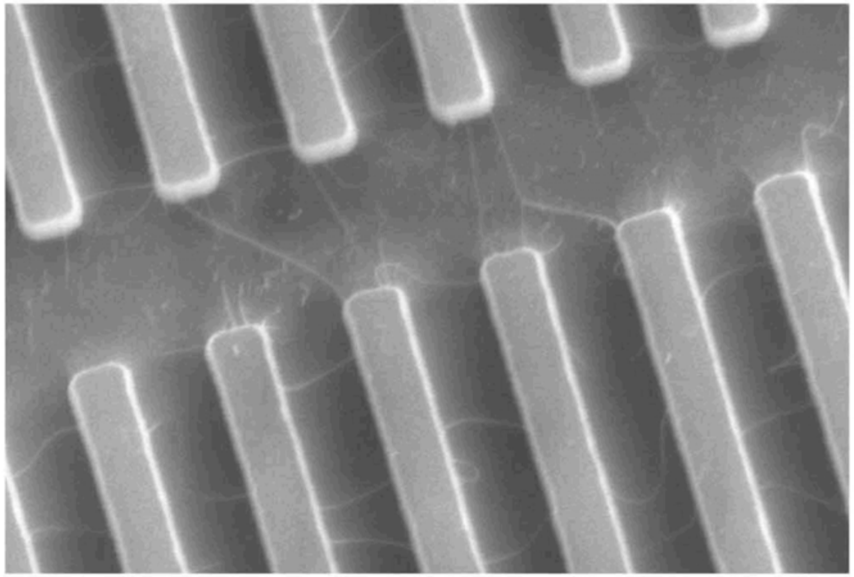


Adv. Mat. **12** (2000) 890

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

Samples for SPELEEM studies

- Si line pattern, pitch 1 µm, height 500 nm
- SWNT grown by CVD at 900°C (Fe catalyst)
- SWNT diameter 1-3 nm

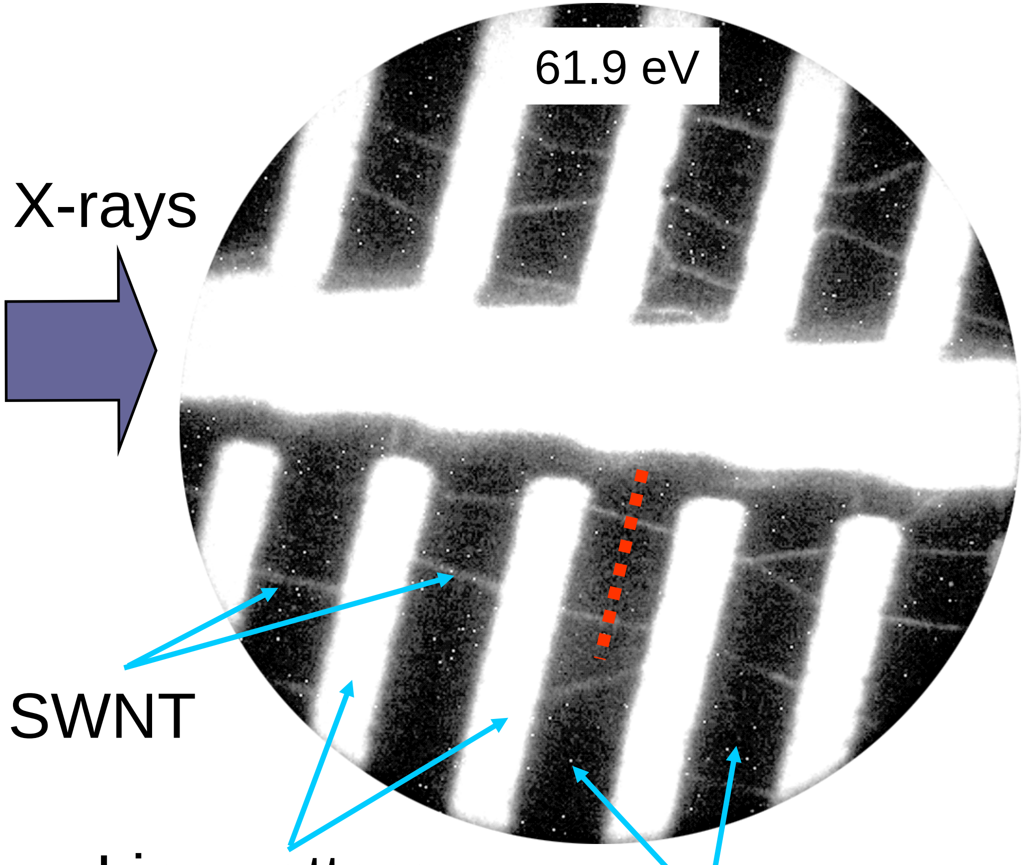


SEM

LEEM

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

C 1s core level image

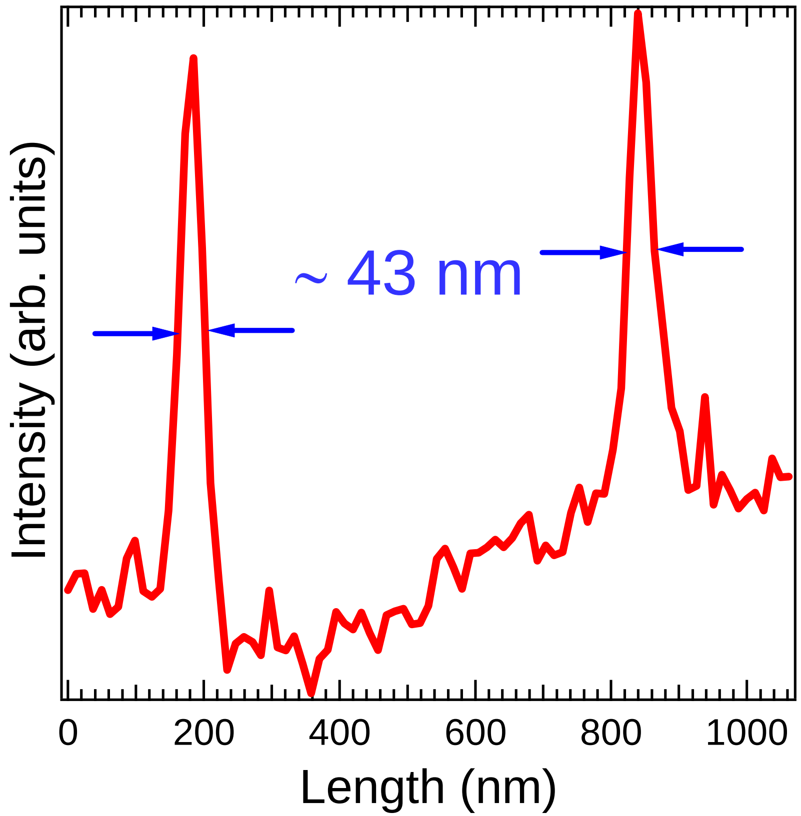


X-rays

SWNT

Line pattern

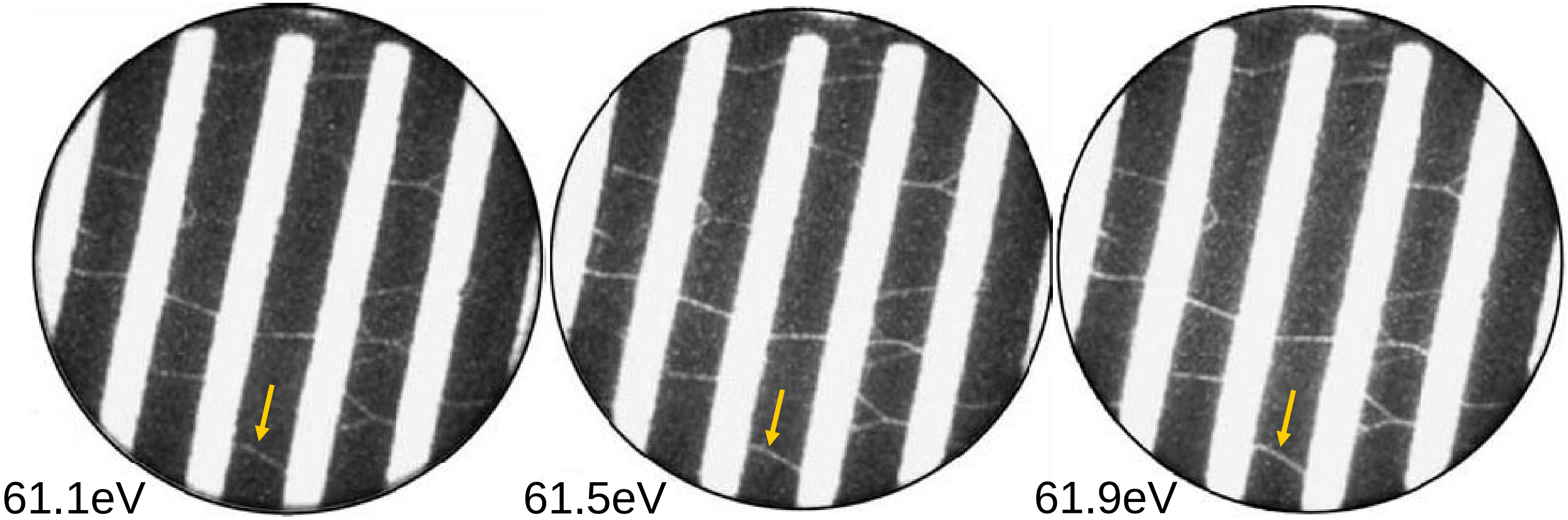
Shadow of the pattern



Photoelectron intensity profile along the broken line.

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

C 1s core level spectra



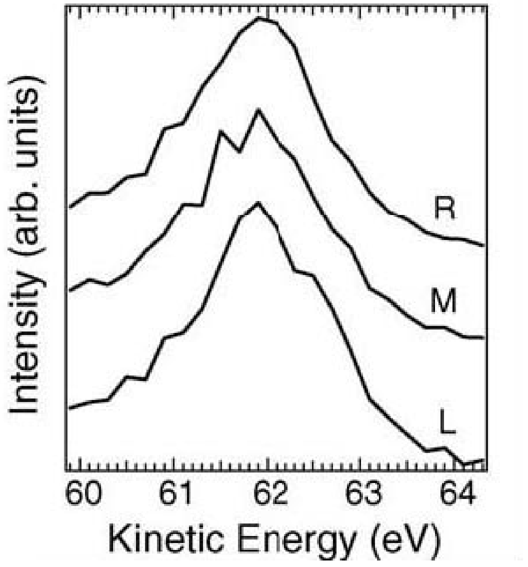
61.1eV

61.5eV

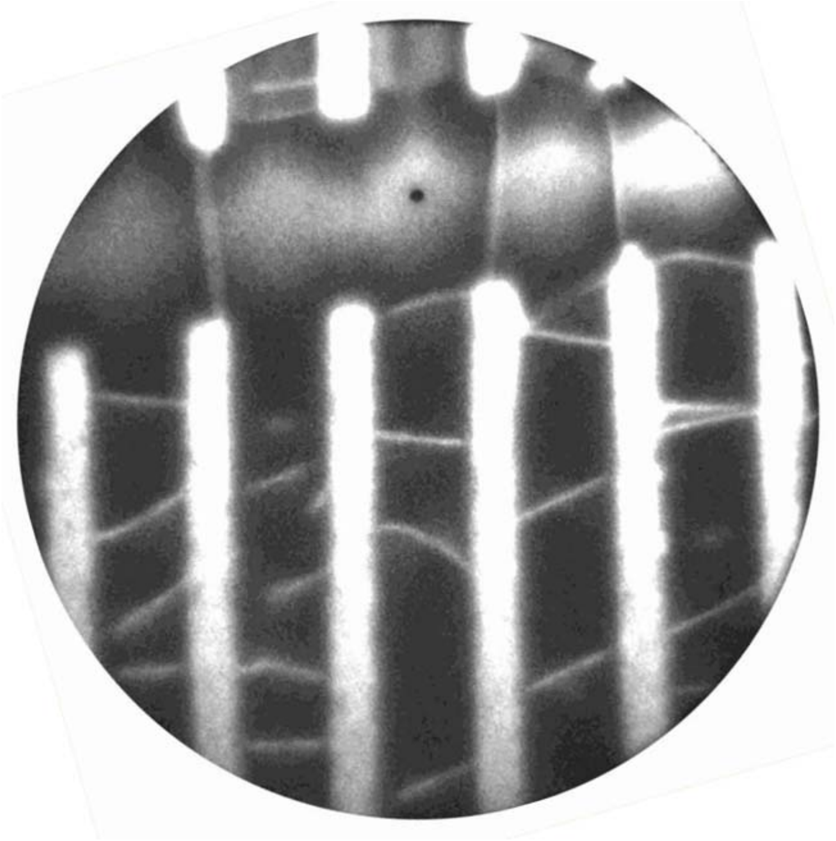
61.9eV

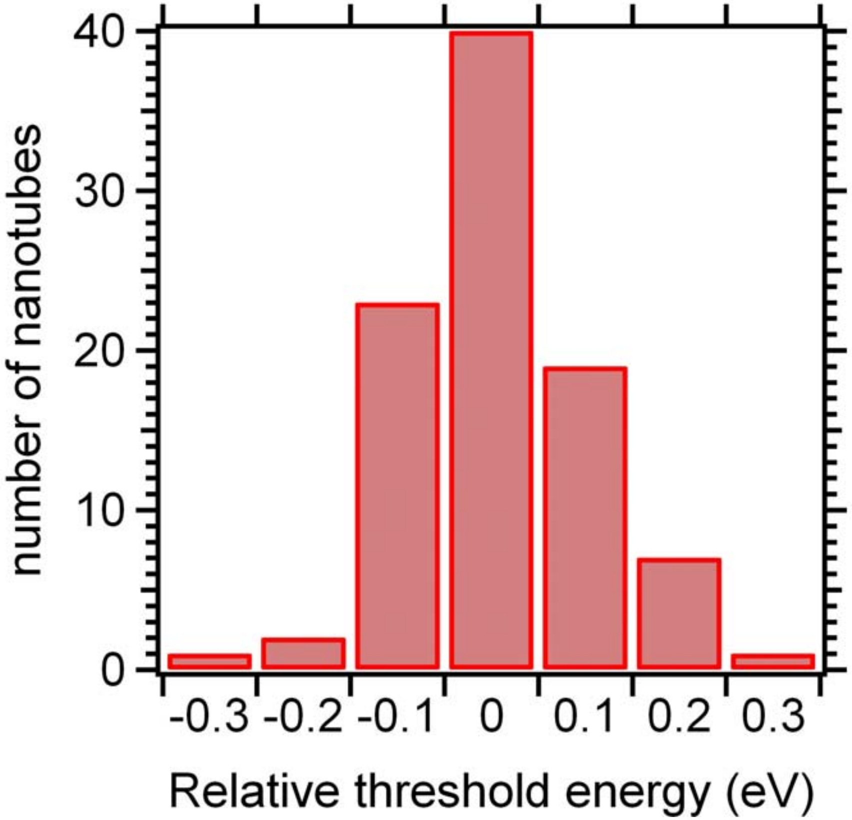
- No band bending along tube axis
- Depletion width < 40 nm

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



CNT work function

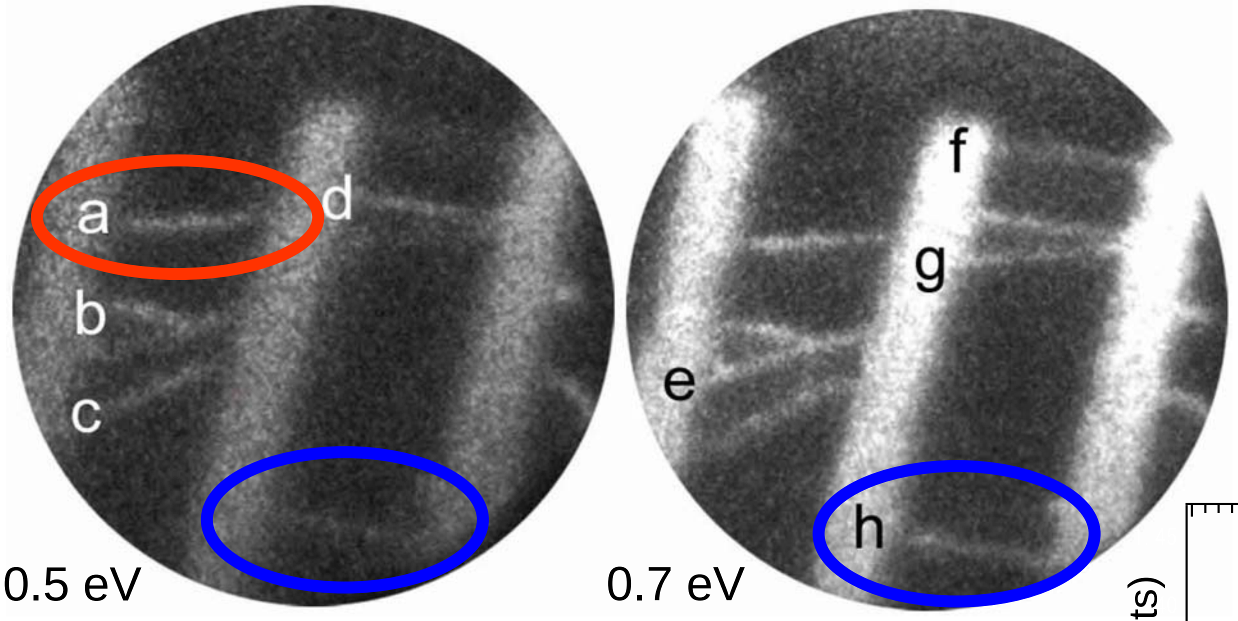


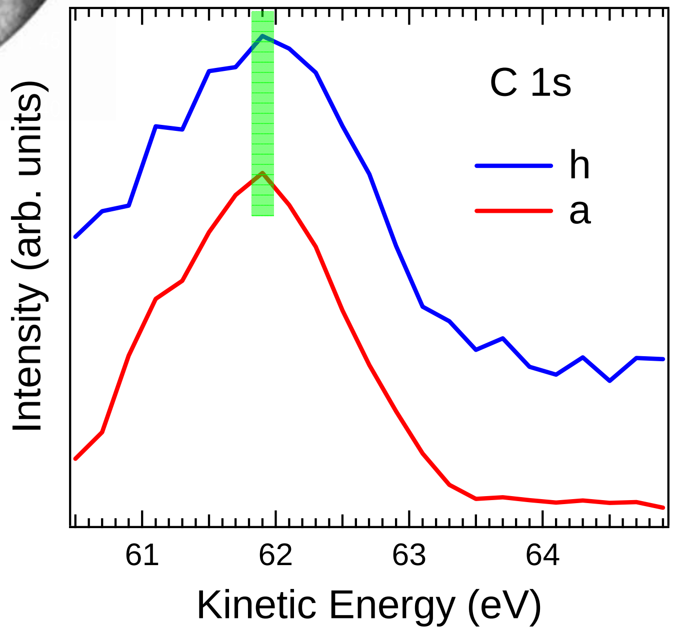


- Secondary electron PEEM image
- Work function distribution of 93 SWNT

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

Secondary electron PEEM images





- No C 1s core level shift
- No band bending

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

Conclusions

- 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