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UHV experiments

Luca Gregoratti

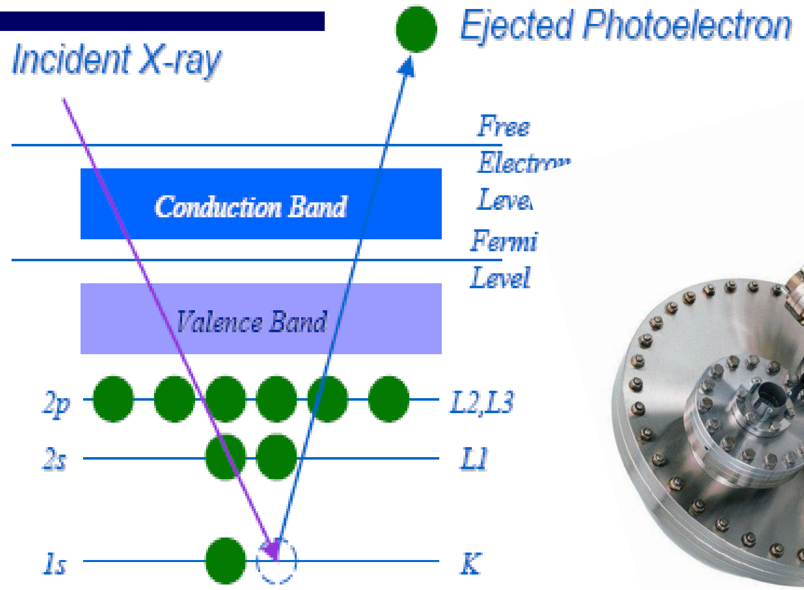
Coordinators of the

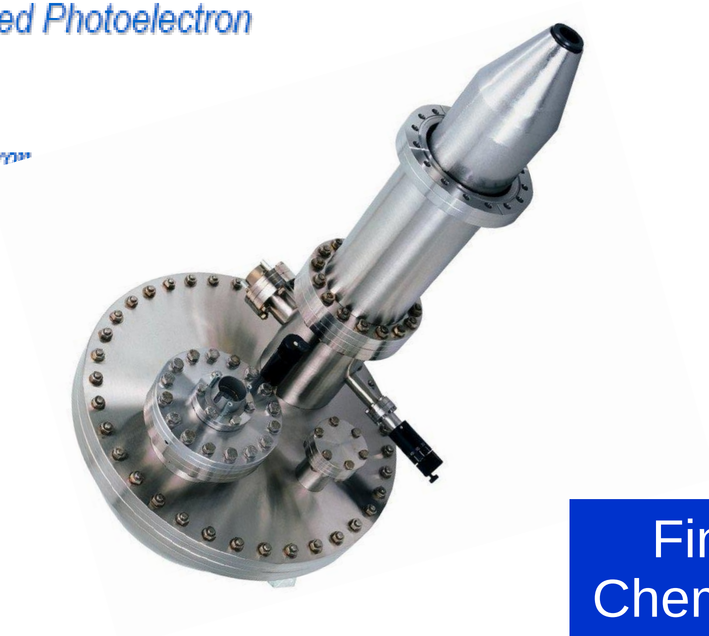
Microscopy/Diffraction Beamlines Groups

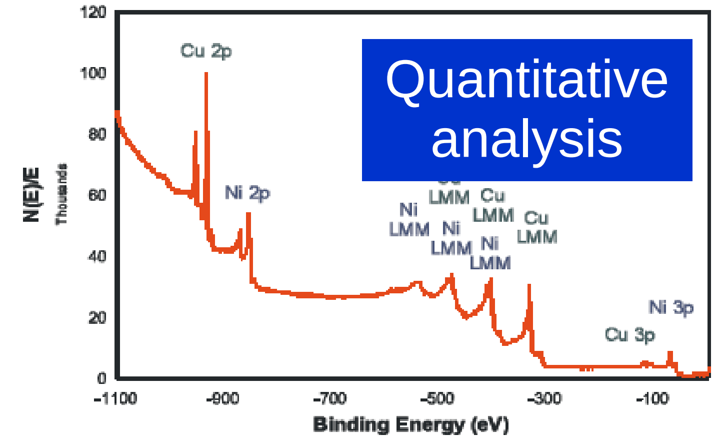
Outline

- The surface science case in research
  (e.g. catalysis, interfaces, sensors).
- Photoemission spectroscopy and microscopy.
- Instrumentation requirements.
- Examples and technologies for the future

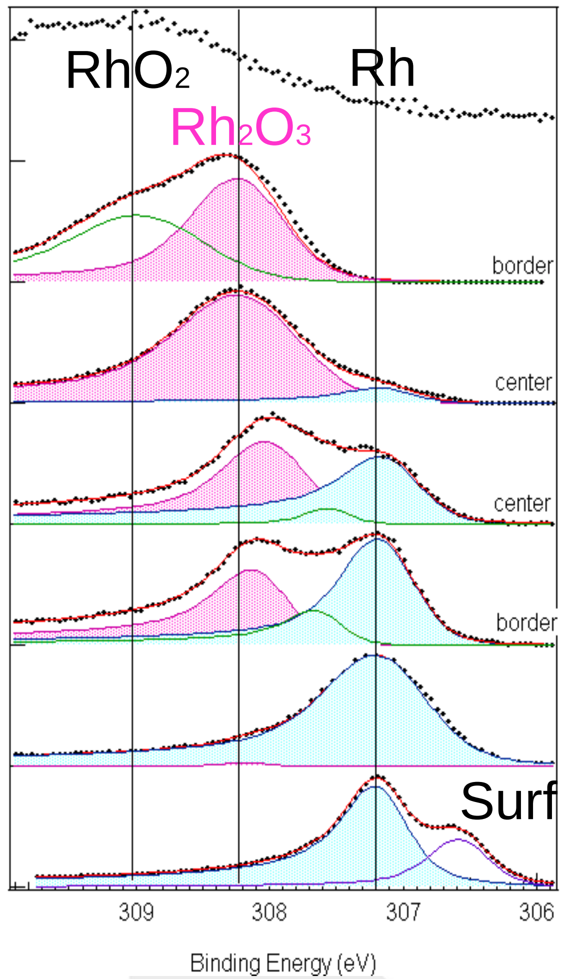
The Photoelectric Process





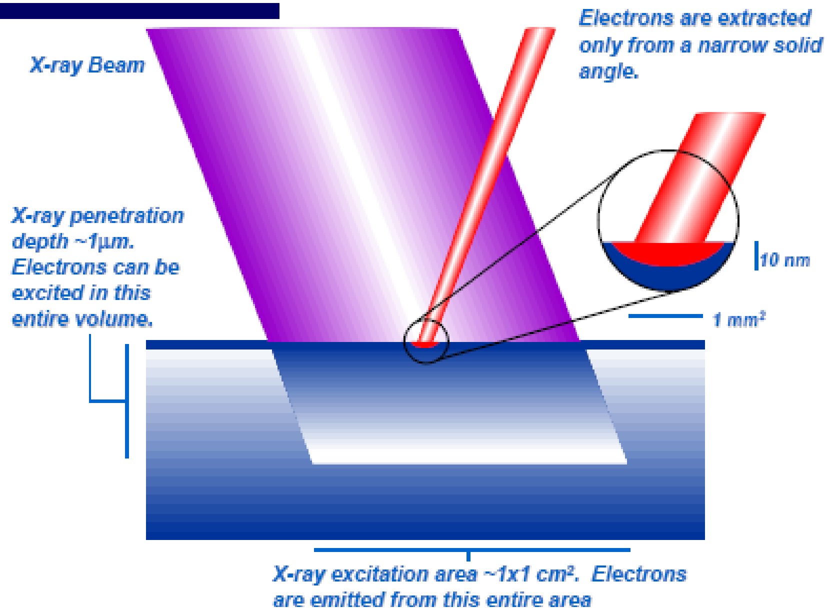


Fine Chemical analysis

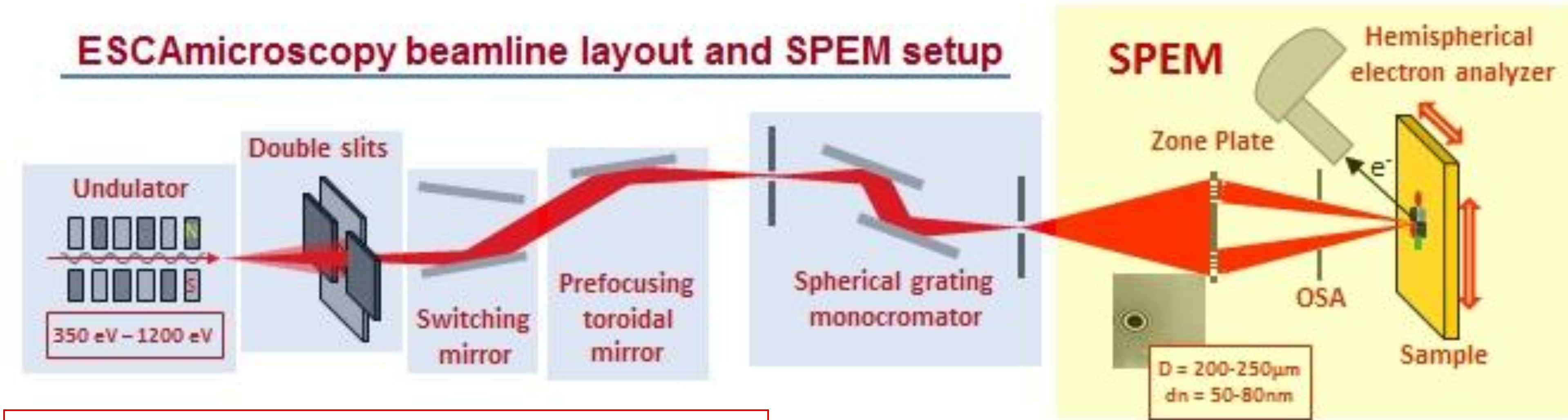


X-ray Photoelectron Spectroscopy

Small Area Detection

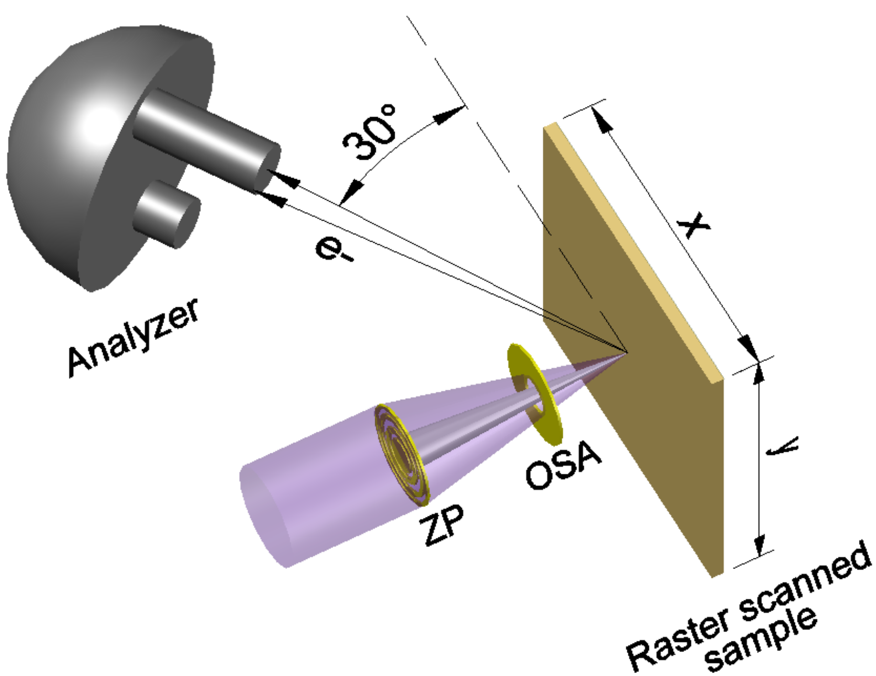


Escamicroscopy - SPEM layout



Milestones

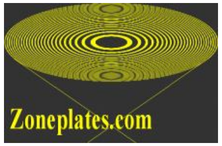
- 1995:first user
- 2000-2004: new micros/prep chambers



- Linearly Polarised Undulator
- Photon energy range: 350 – 1200 eV
- SGM monochromator equipped with 2 gratings for low and high photon energy

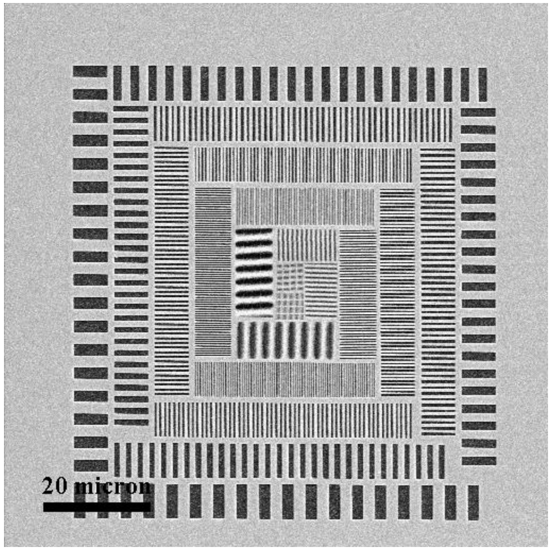
Spatial resolution

Zone plate used: xradia insight in 3D



Best ZP: D=200 μm, dr=50 nm

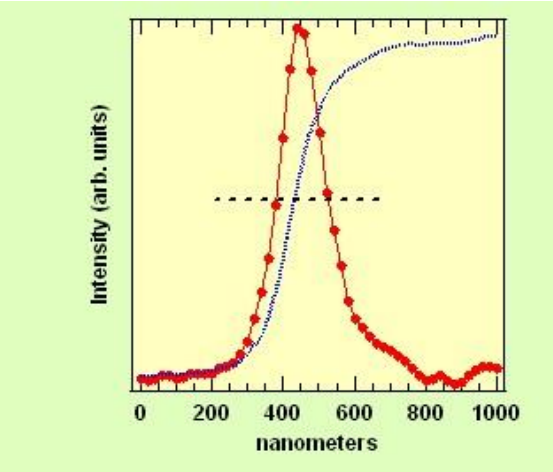
Other sizes: D=250 μm, dr=100nm
D=250 μm, dr=80nm



50 nm test object

Spectromicroscopy:
real beam size and shape

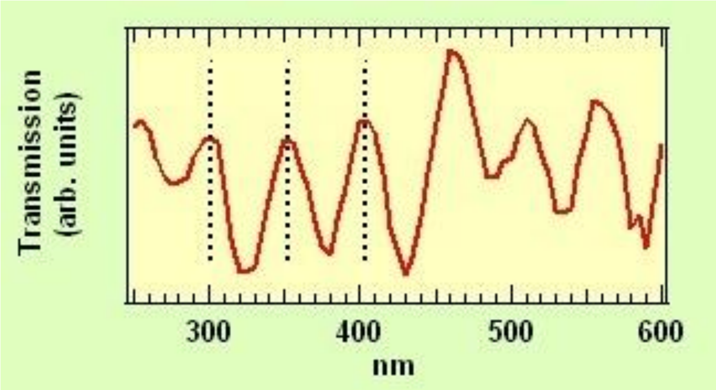
135 nm (SPEM)



Imaging:
effectice resolution

< 50 nm (SPEM)

Horizontal scan



Chemical imaging

Chemical inhomogeneity

Ni islands on Si

Image on Ni

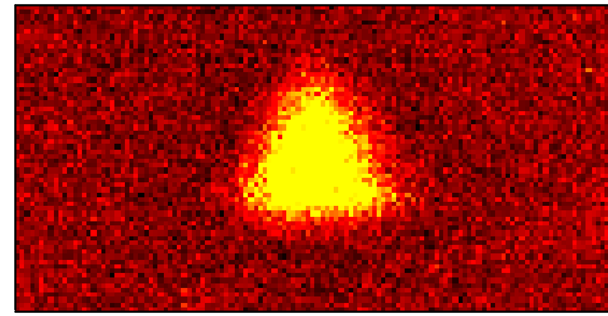
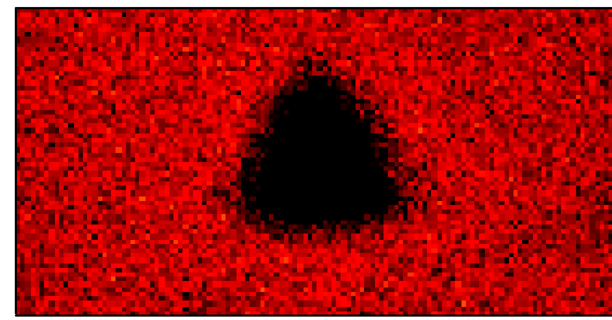


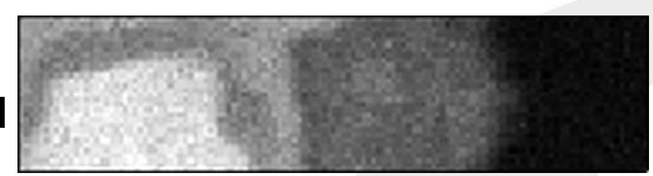
Image on Si



Au patch on Rh(110)

Image on Rh

Image on Au



Degradation of light emitting diodes

(in collaboration with P. Melpignano CRP, R. Zamboni CNR-ISMN)

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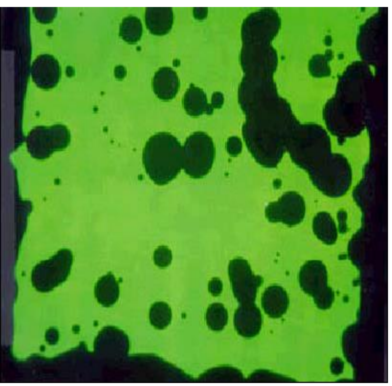
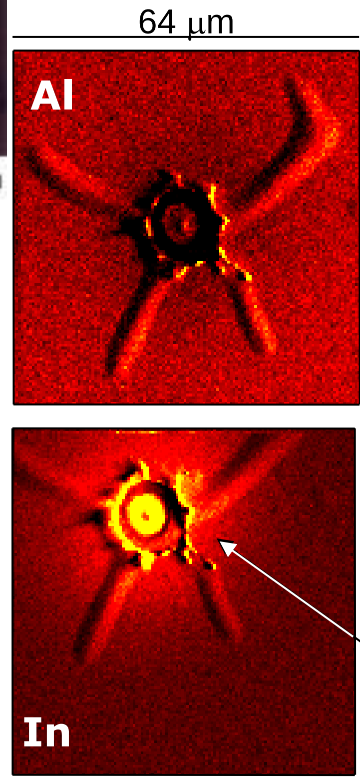
OLED Display Screen (from Universal Display Corp)

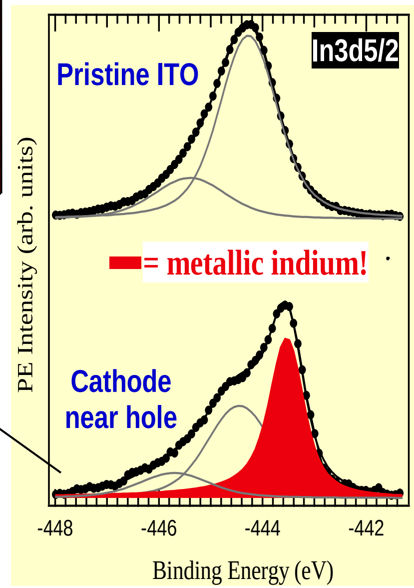


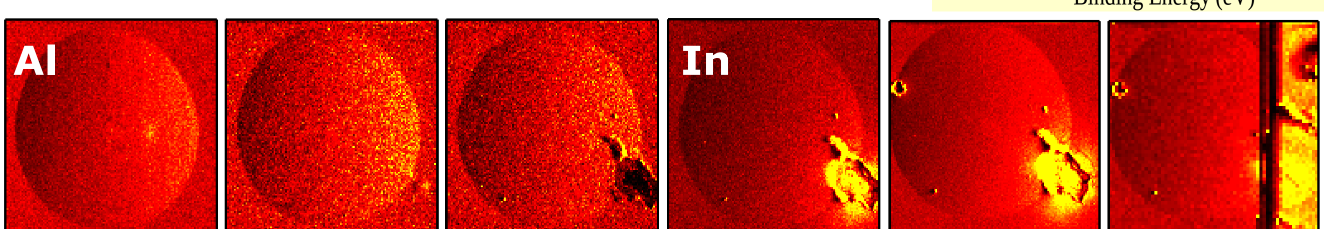
Dark spot in OLED

OLED exposed to atmospheric moisture: failure due to light emission



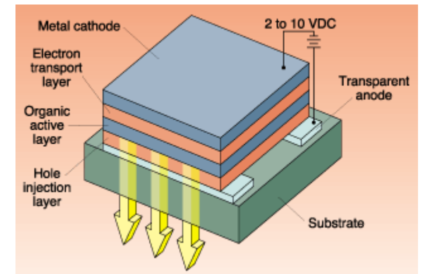
•Decomposition of ITO

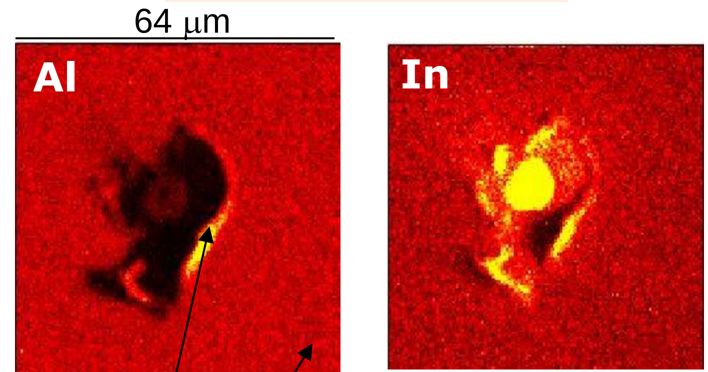


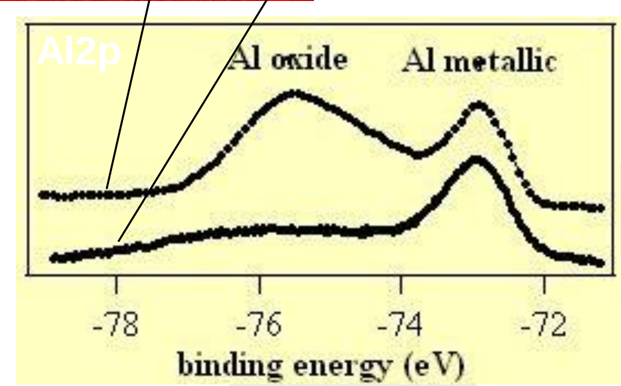


increasing voltage and operating time

"Clean" experiment: OLED growth and operated in the SPEM (UHV ambient) : failure due to light emission







P. Melpignano et al. Appl. Phys. Lett. 86, 041105 (2005),

S. Gardonio et al. Org. Electr. 8 (1), 37-43, (2007)

Operating SOFC: mass transport

(in collaboration with M. Backhaus- Corning Inc. - USA)

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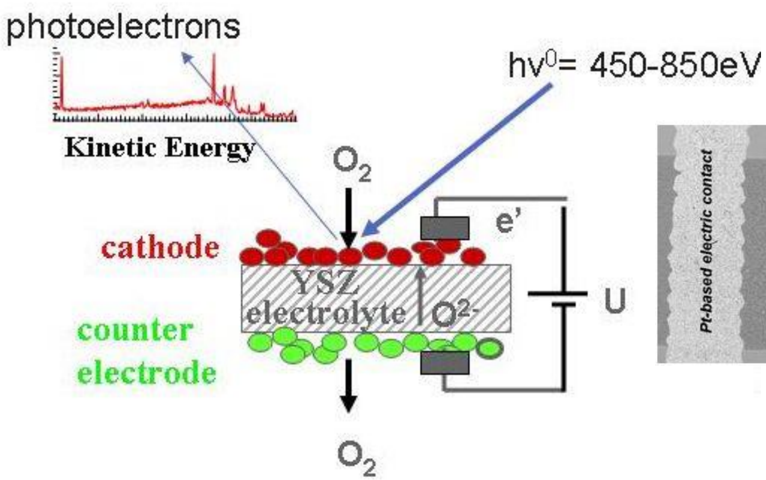
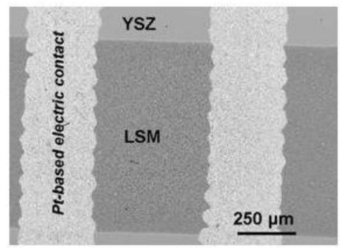
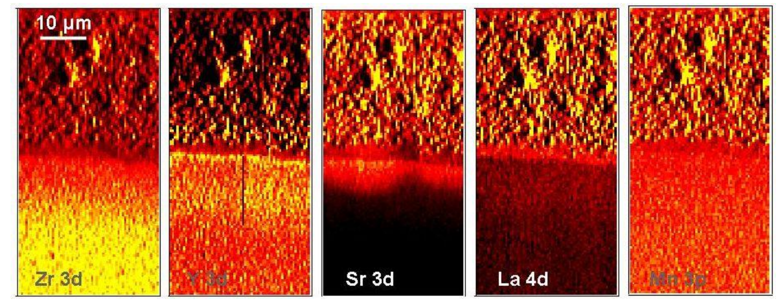




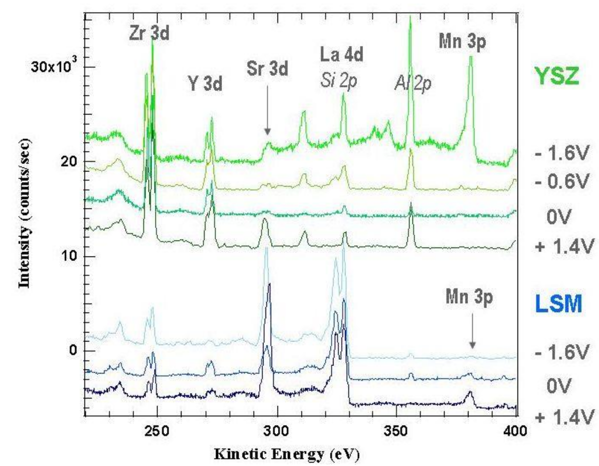
Strongly constraining experimental setup

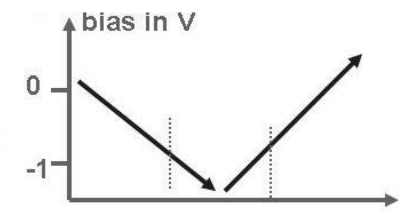
- Real samples
- High T = 650-700° C
- pO_2=1x10^{-6} mbar
- Applied potentials
  -2V<U<+2V
- Surface sensitive technique
- High lateral resolution

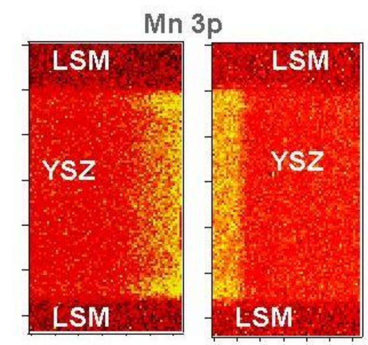
Elemental distribution at electrolyte/LSM interface



Surface composition change with bias





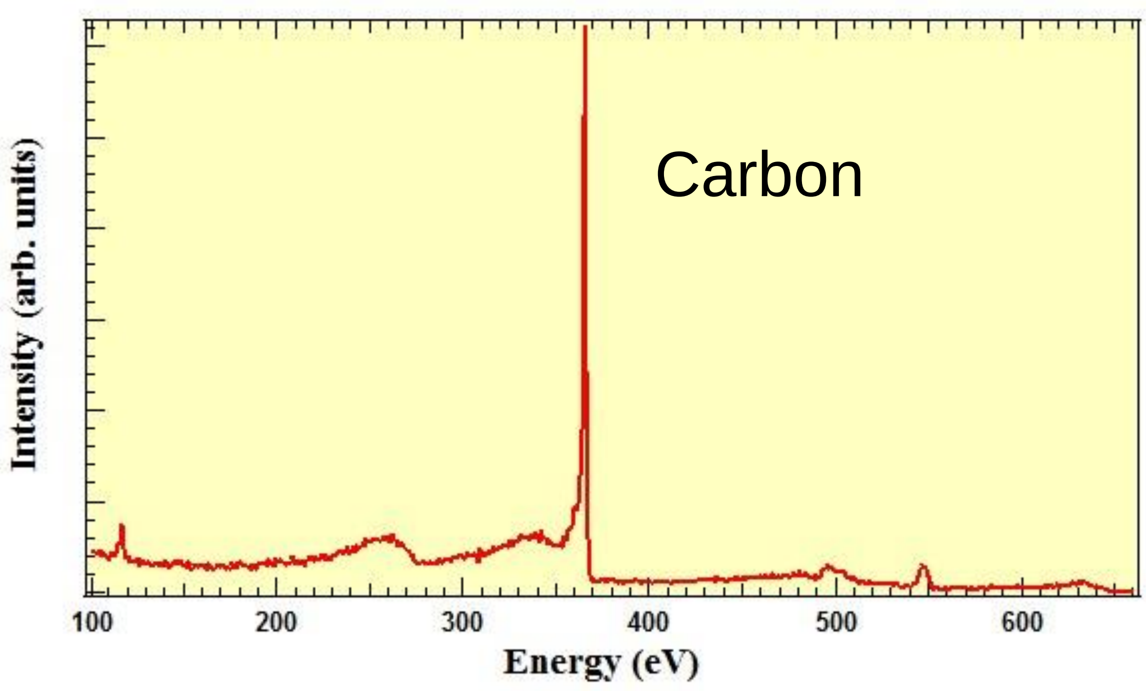


Observation and explanation of electrochemical cathode activation

- Strong current increase under negative bias when Mn spreads on electrolyte
- Mn2+ electrolyte surface enrichment→electrolyte surface conductivity → direct oxygen incorporation into electrolyte
- Oxygen incorporation extends under bias from TPB to the entire electrolyte surface

M. Backhaus et al. Solid State Ionics 179 (2008) 891–895 , M. Backhaus et al. Advances in Solid Oxide Fuel Cells III 28 (4), 2007.

For many samples/experiments a CLEAN surface is crucial



How fast clean surfaces get dirty

Pressure (Torr)	Time to produce 1 ML (seconds)
760	3.44×10^{-9}
1	2.61×10^{-6}
1×10^{-3}	2.61×10^{-3}
1×10^{-6}	2.61
1×10^{-9}	2.61×10^{3}
1×10^{-11}	2.61×10^{5}

→ Samples from air must be cleaned before measurement !!

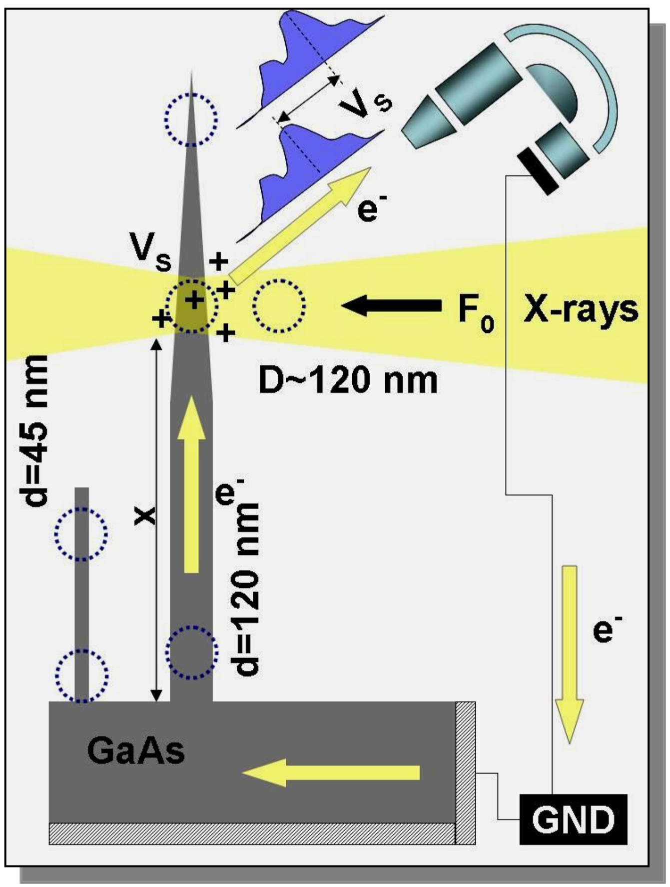
- Annealing
- O (2), H (2), bath
- Sputtering
- Vacuum exposure
- Capping

→ Materials and equipment

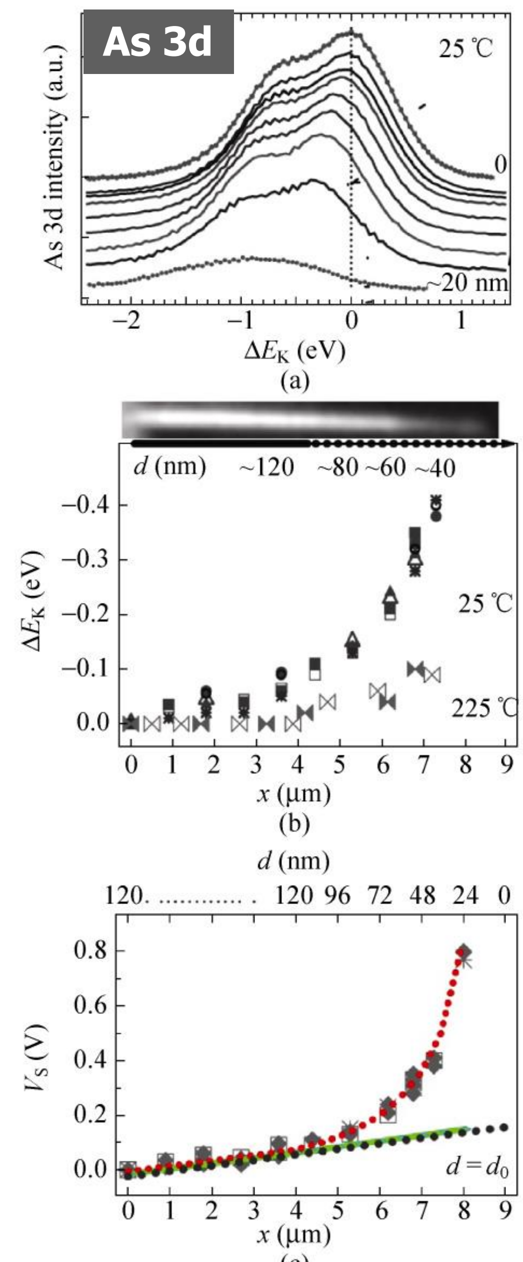
Contactless monitoring of the diameter-dependent conductivity of GaAs nanowires

(in collaboration with S. Rubini – CNR-IOM Laboratory - Italy)

D<3nm



D=120nm



Debye lengths~*d*

Influence of size (temperature and surface treatment) on the conductance of individual low-doped GaAs NWs can be addressed and quantified by contactless measurements of the photon beam-induced surface potential along a NW axis using photoelectron microspectroscopy

F. Jabeen et al. *Nano Res.* 2010, 3(9): 676–684

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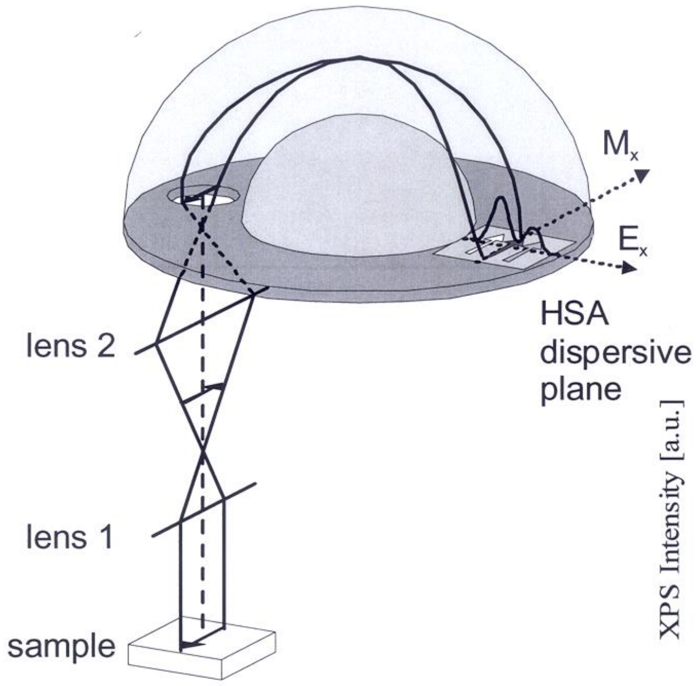
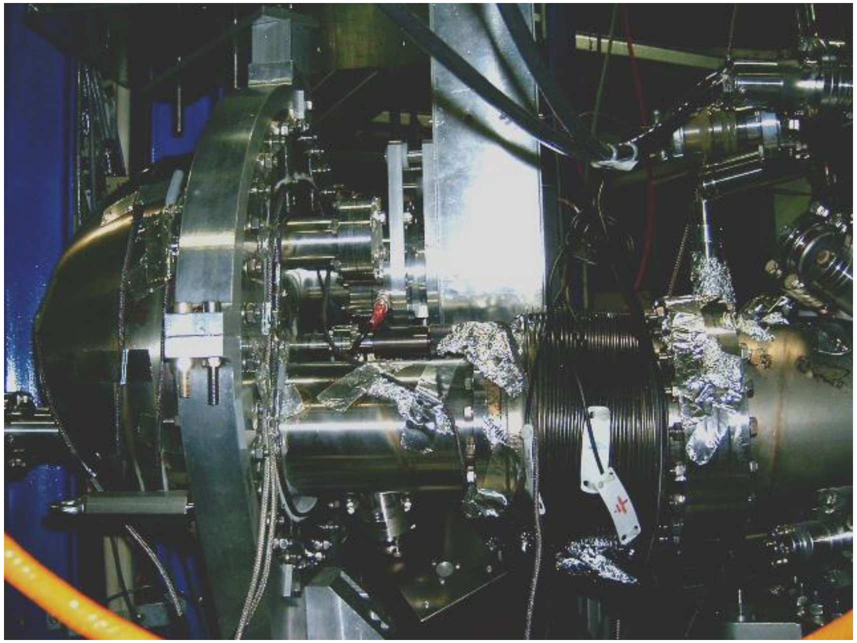
New approaches for UHV environments

Electron analyzers

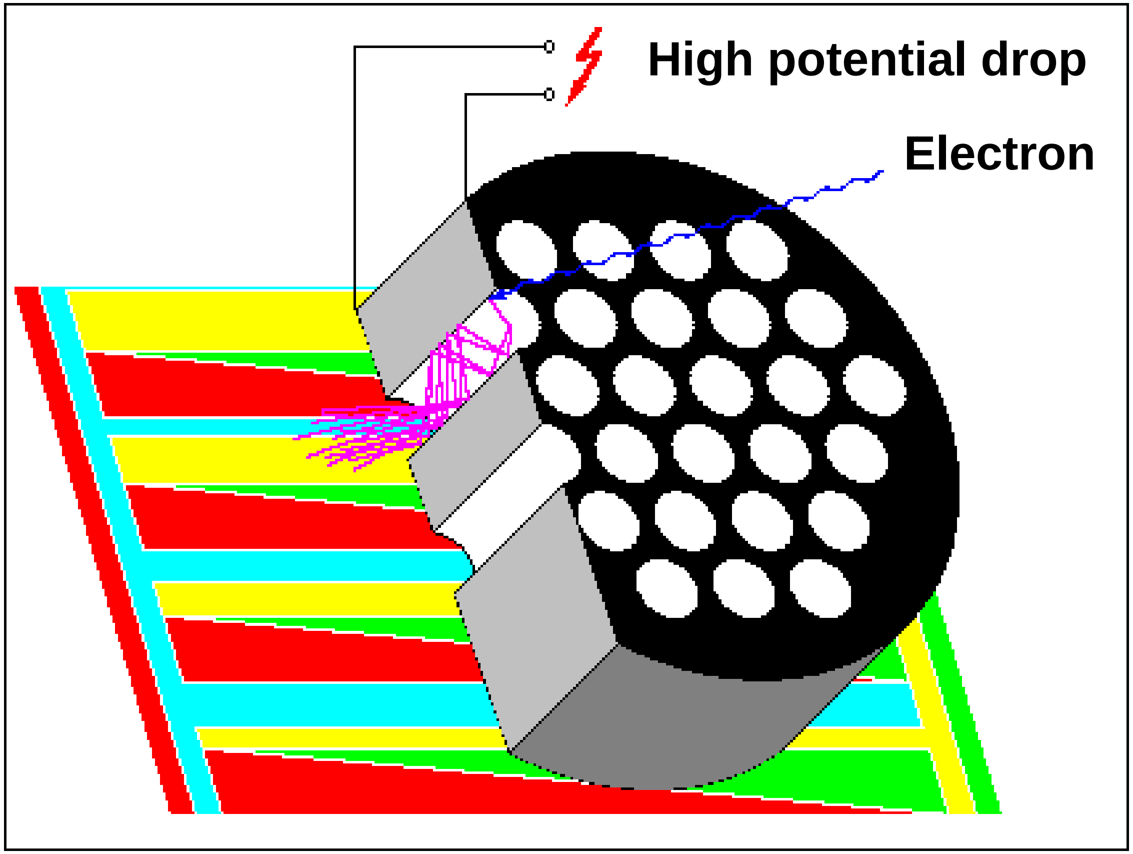
- The most used type of electron analyzer is the Hemispherical Electron Analyzer (HEA)
- Due to geometrical constrains the detection in mainly grazing

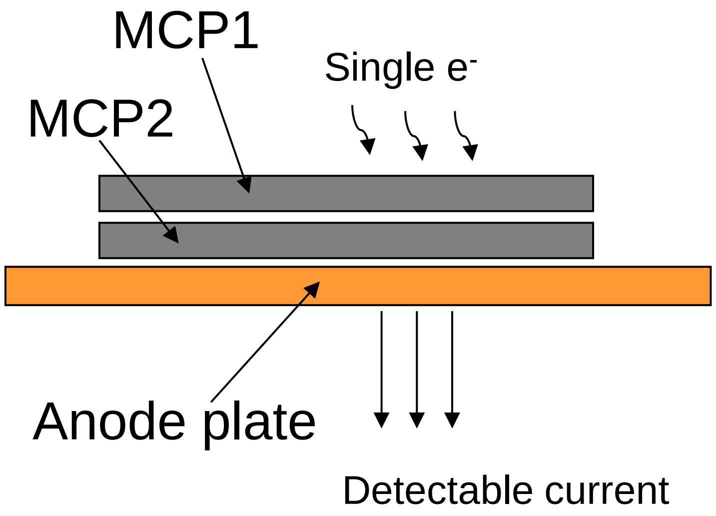


Electron analyzer of the SPEM



Electron detectors based on micro-channel plates





The Microchannel Plate (MCP) consists of millions of very-thin, conductive glass capillaries (4 to 25 micro meters in diameter) fused together and sliced into a thin plate. Each capillary or channel works as an independent secondary-electron multiplier to form a two-dimensional secondary-electron multiplier.

Environmental cell with graphene oxide windows
(in collaboration with A. Kolmakov – Souther Illinois Uni. - USA)

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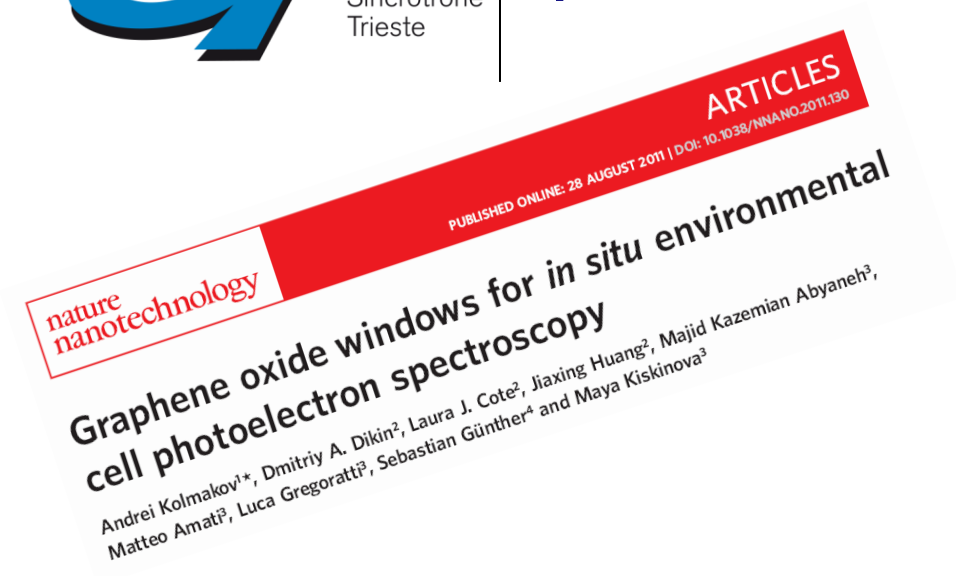

nature nanotechnology

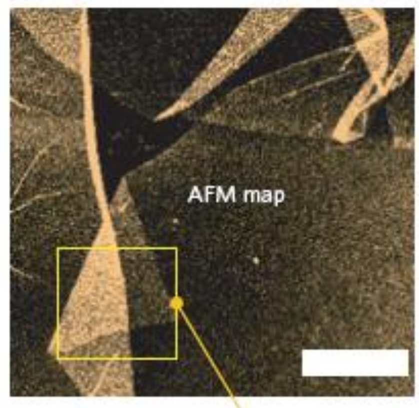
ARTICLES

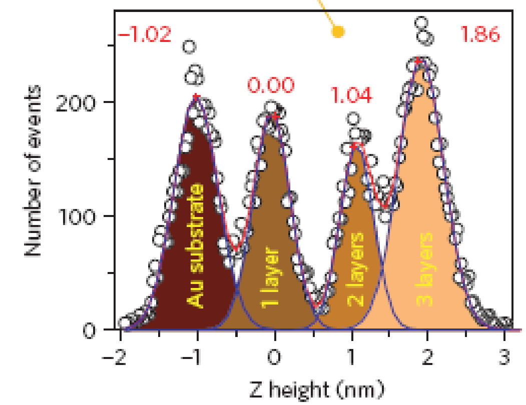
PUBLISHED ONLINE: 28 AUGUST 2011 | DOI: 10.1038/NNANO.2011.130

Graphene oxide windows for in situ environmental cell photoelectron spectroscopy

Andrei Kolmakov[1]*, Dmitriy A. Dikin[2], Laura J. Cote[2], Jiaxing Huang[2], Majid Kazemian Abyaneh[3], Matteo Amati[3], Luca Gregoratti[3], Sebastian Günther[4] and Maya Kiskinova[3]

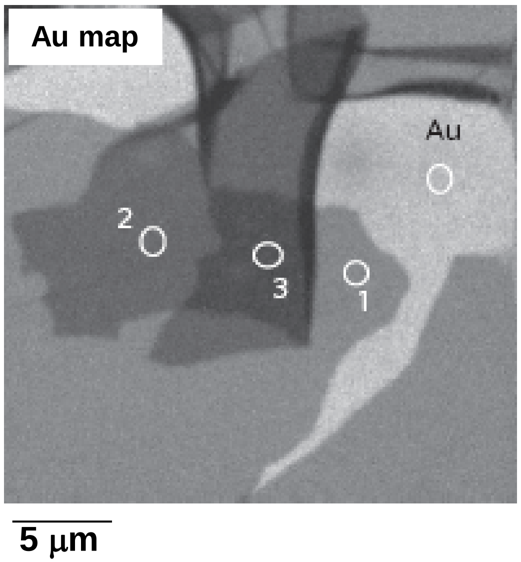
Graphene/Au: AFM

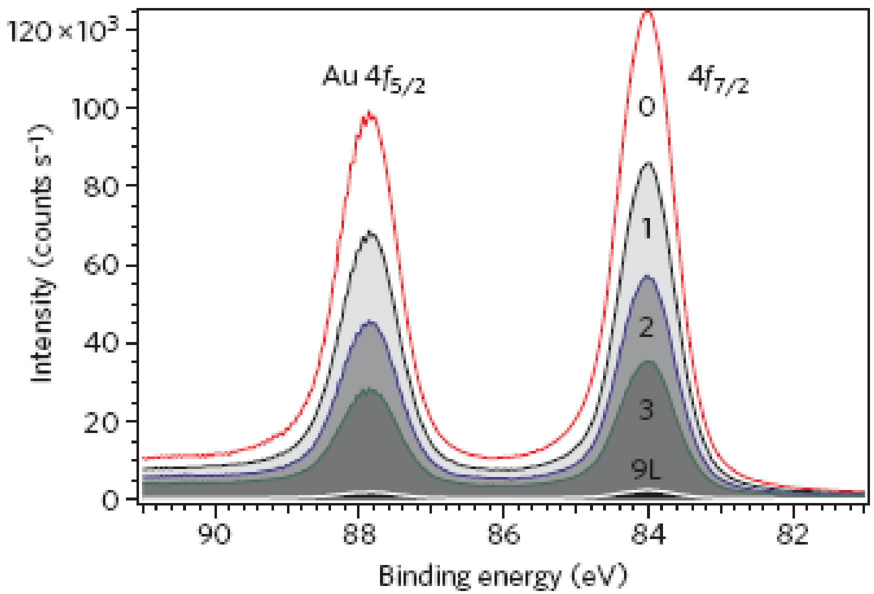


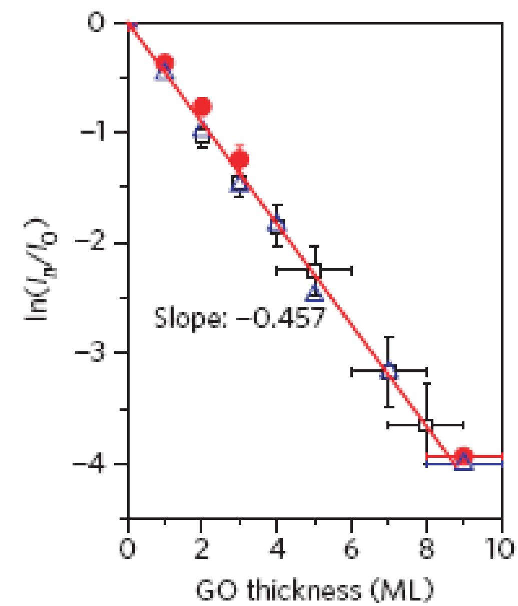


Graphene/Au: SPEM

Graphene layers are transparent to photoelectrons







A. Kolmakov et al. *Nature Nanotechnology* 6, 651–657 (2011)

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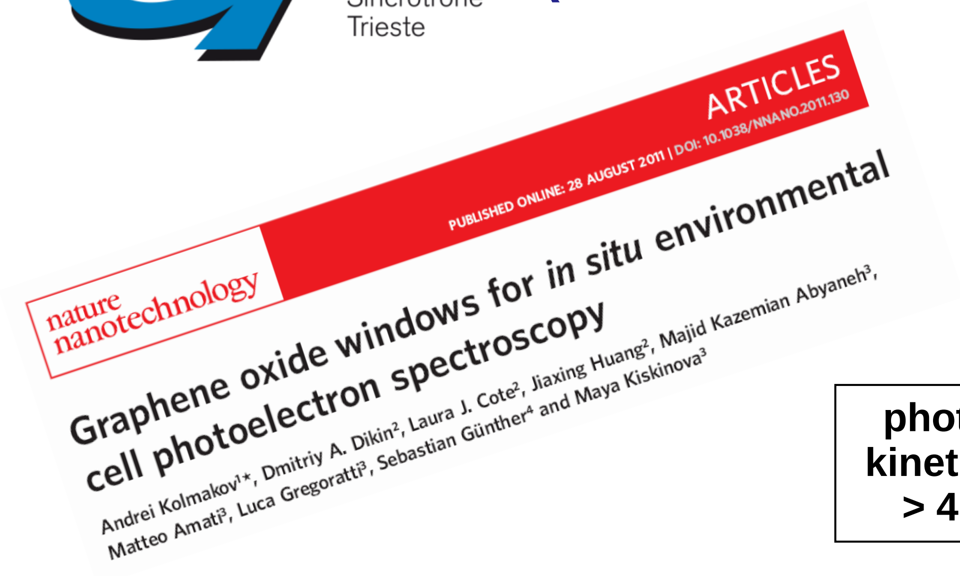
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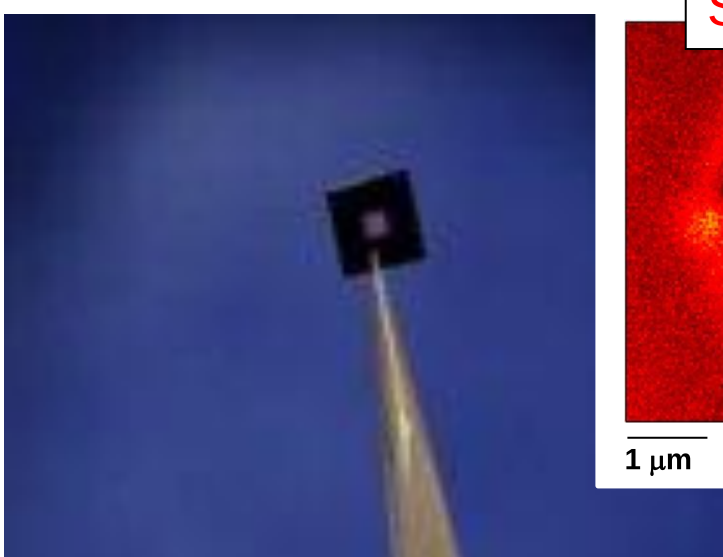
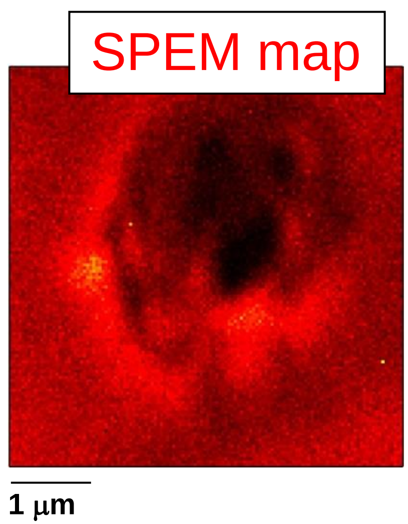
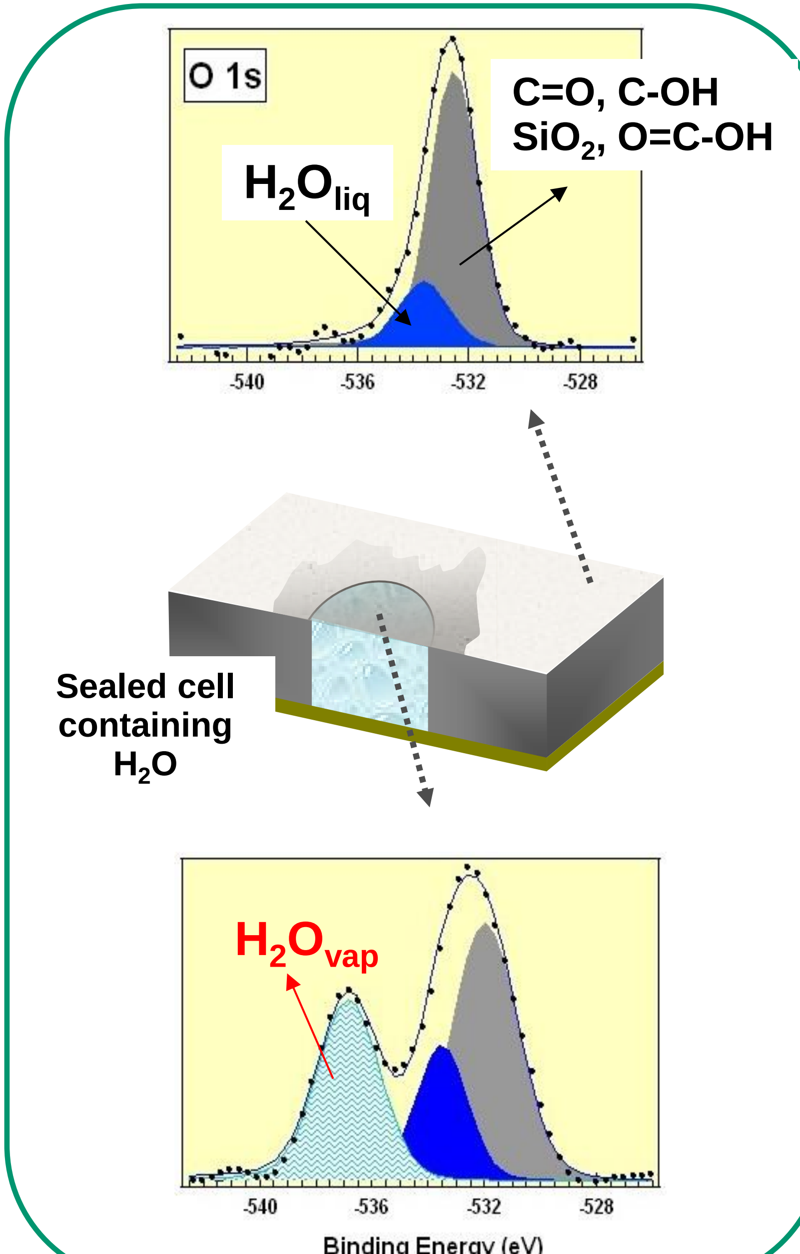
photoelectron kinetic energies > 450–500 eV

- **low-cost, single-use environmental cells**
- **(near) compatible with commercial X-ray and Auger**







A. Kolmakov et al. *Nature Nanotechnology* 6, 651–657 (2011) and *Nanoscale* 2014, DOI: 10.1039/C4NR03561E

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