The Abdus Salam
International Centre for Theoretical Physics

United Nations
Educational, Scientific and
Cultural Organization

IAEA
International Atomic Energy Agency

1936-26

Advanced School on Synchrotron and Free Electron Laser Sources and their Multidisciplinary Applications

7 - 25 April 2008

Scanning Photoemission Microscopy: Instrumentation and Methodology

Luca Gregoratti
Sincrotrone Trieste SCpA

Strada Costiera 11, 34014 Trieste, Italy - Tel. +39 040 2240 111; Fax +39 040 224 163 - sci_info@ictp.it, www.ictp.it

Scanning Photoemission Microscopy: Instrumentation and Methodology

Luca Gregoratti

Sincrotrone Trieste SCpA, Area Science Park, SS14-Km163.5, 34012 Trieste, Italy

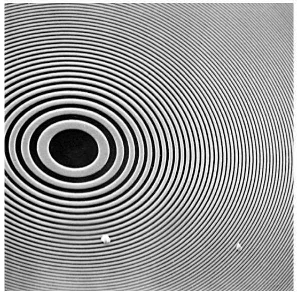
email: luca.gregoratti@elettra.trieste.it

Scanning

SPEM

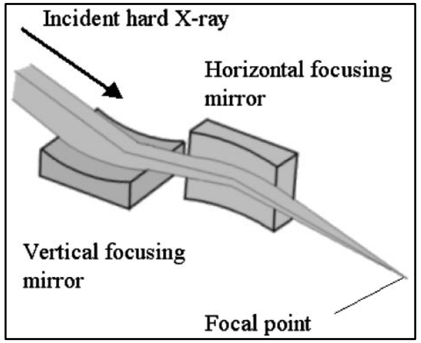
Zone Plates lenses

- **Elettra**
- **ALS**
- **SRRC**
- **PAL**



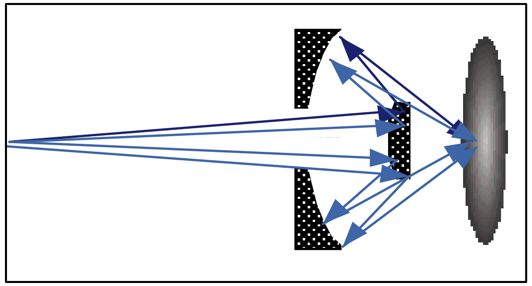
Focusing mirrors

Kirkpatrick-Baez



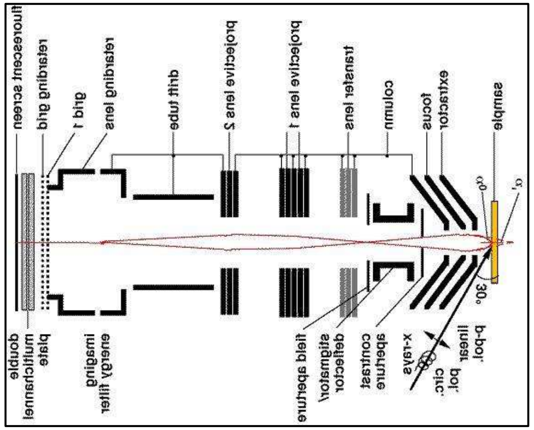
Schwarzschild mirrors

- **Elettra**
- **Madison**

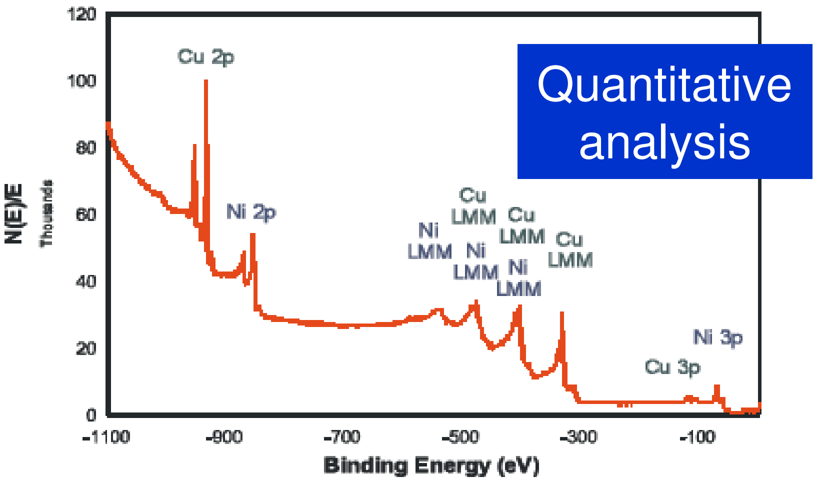


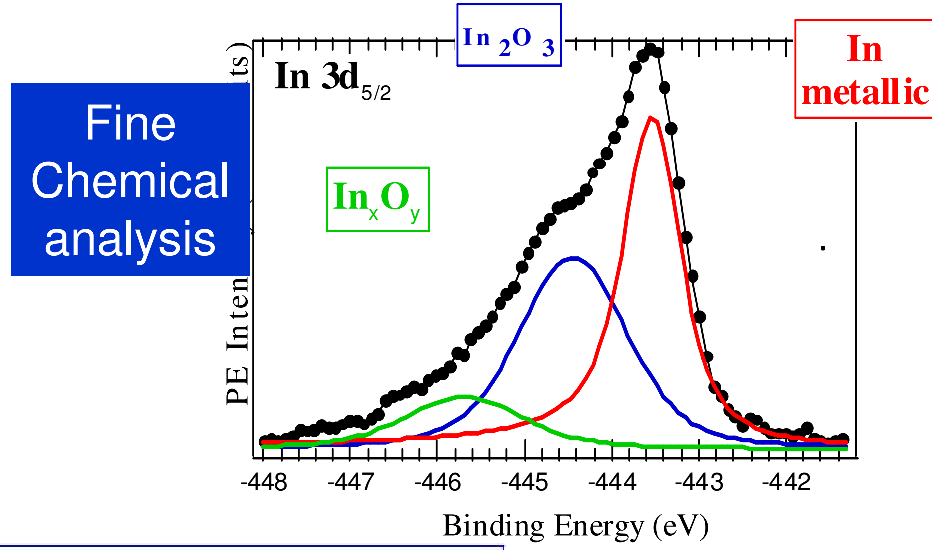
No scanning

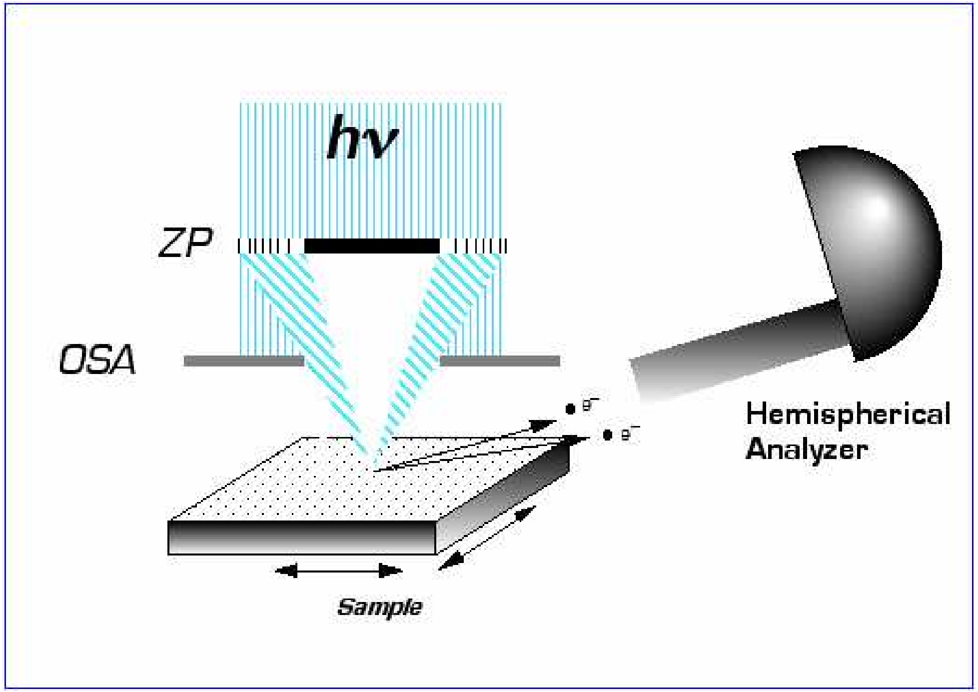
XPEEM (XPEEM, SPELEEM)



- **Elettra**
- **SLS**
- **MAX-lab**
- **BESSY**
- **Madison**
- **Spring8**


Quantitative analysis
N(E)/E
Thousands
120
100
80
60
40
20
0
-1100
-900
-700
-500
-300
-100
Binding Energy (eV)
Cu 2p
Ni 2p
Ni LMM
Cu LMM
Ni LMM
Cu LMM
Ni LMM
Cu LMM
Ni 3p
Cu 3p


Fine Chemical analysis
In 3d5/2
In2O3
In metallic
InxOy
PE Inten
-448
-447
-446
-445
-444
-443
-442
Binding Energy (eV)

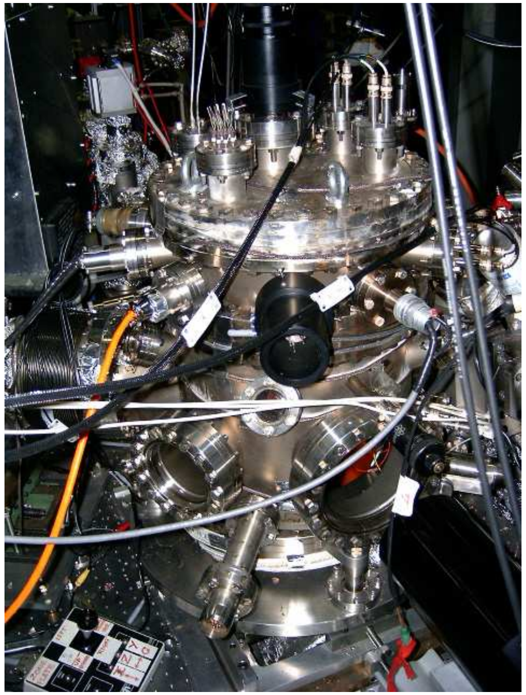

hν
ZP
OSA
Hemispherical Analyzer
Sample

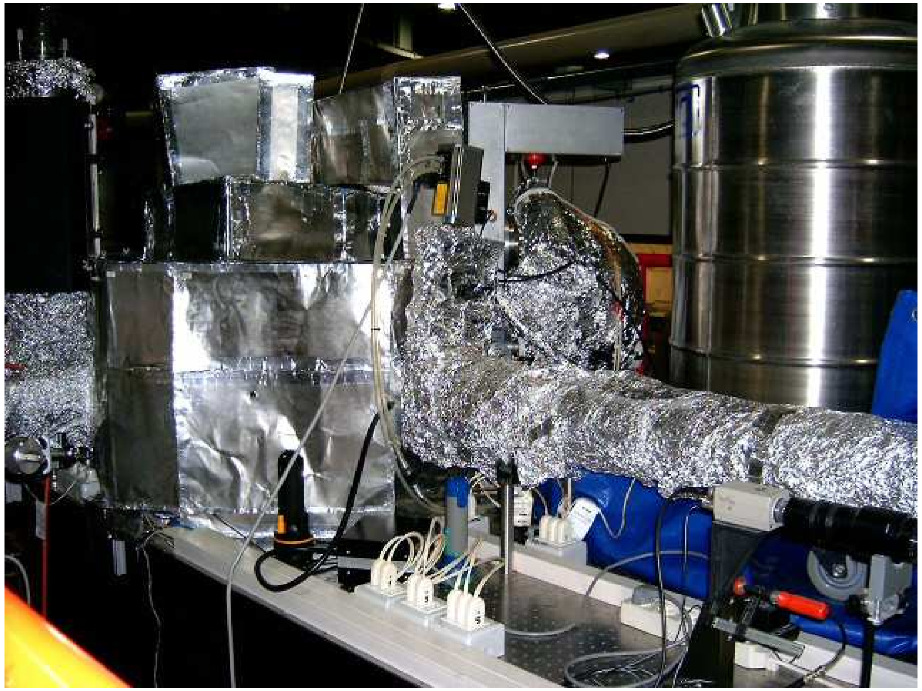
Outline:
Ingredients of a Scanning Photoemission Microscope (SPEM) based on Zone Plates

- Vacuum chambers
- Sample and optics manipulators
- Sample holders
- Electron analyzers
- Electron detectors

Vacuum chambers

- No standard geometry
- Dimensions depends mainly from the size of the manipulators
- Large flanges for the manipulators (>CF200)
- Geometry limits the possibility of in-situ experiments





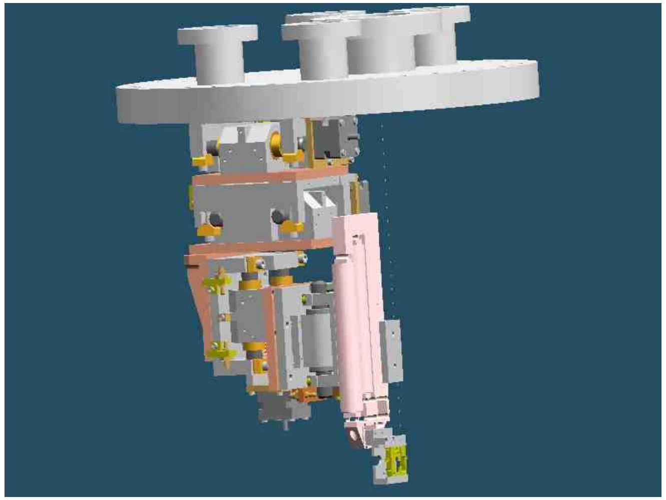
Manipulators

Sample

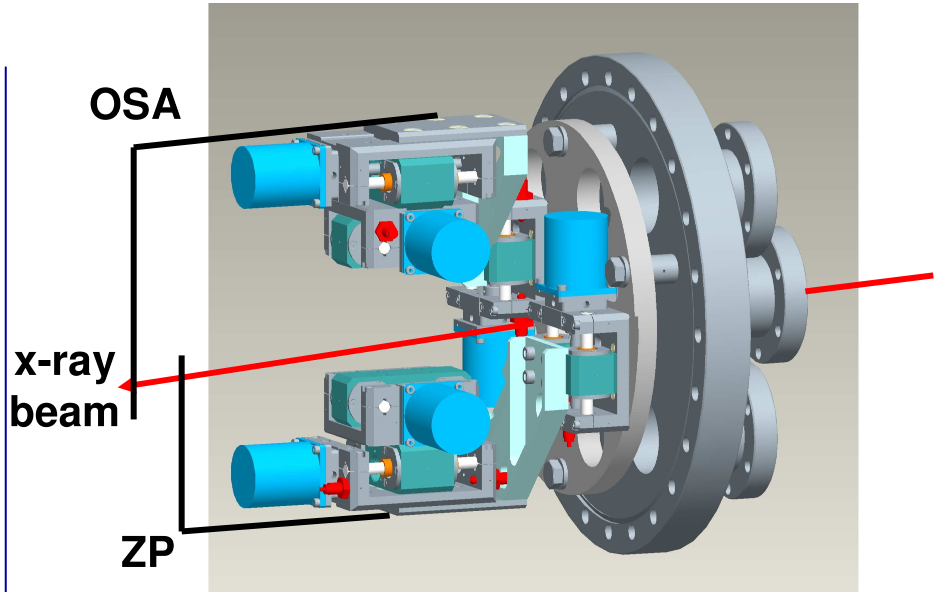
- Large scanning range (>1mm) with large steps (1-100 μm)
- Small scanning range (<3mm) with small steps (10-50 nm)
- The most common choice is to use two kind of motors: stepper (for large scans) and piezo (for small scans)
- Compact design to improve the stability

Optics (ZP+OSA)

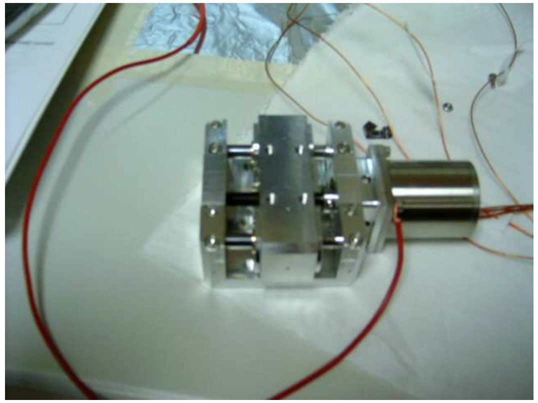
- 6-axis needed: 3 for the ZP and 3 for the OSA
- Typical range: 10 - 15 mm
- Movement resolution of 1-3 μm
- Only one type of motors needed (stepper or inchworm)
- Compact design to improve the stability



x-y piezo stages

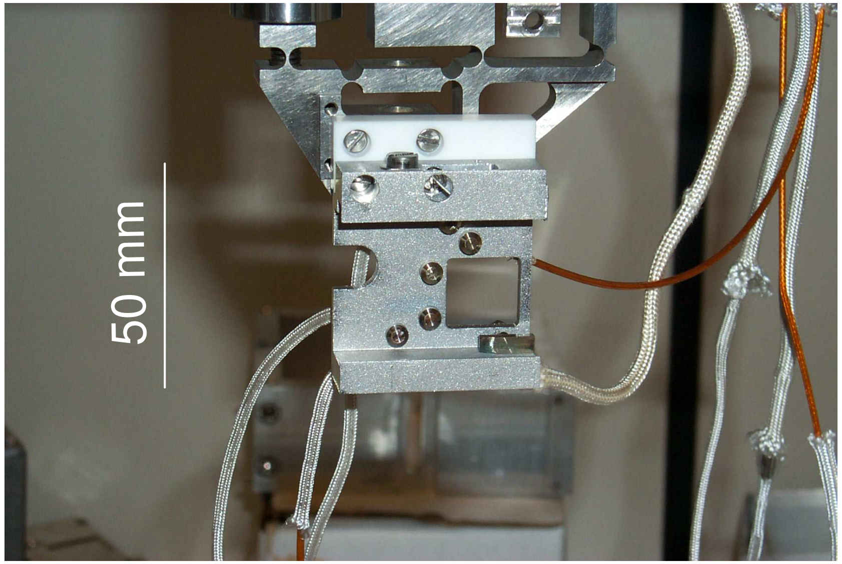


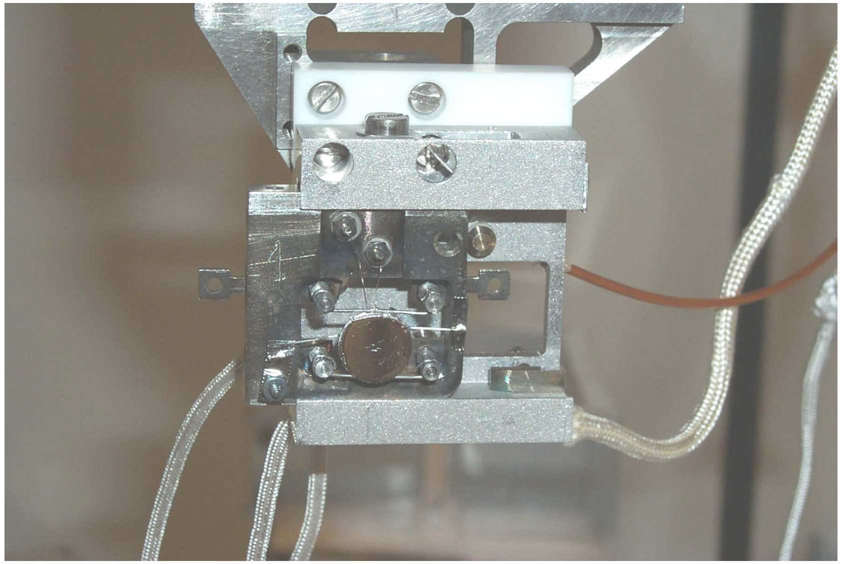
1 axis coarse translation stage



Sample holders

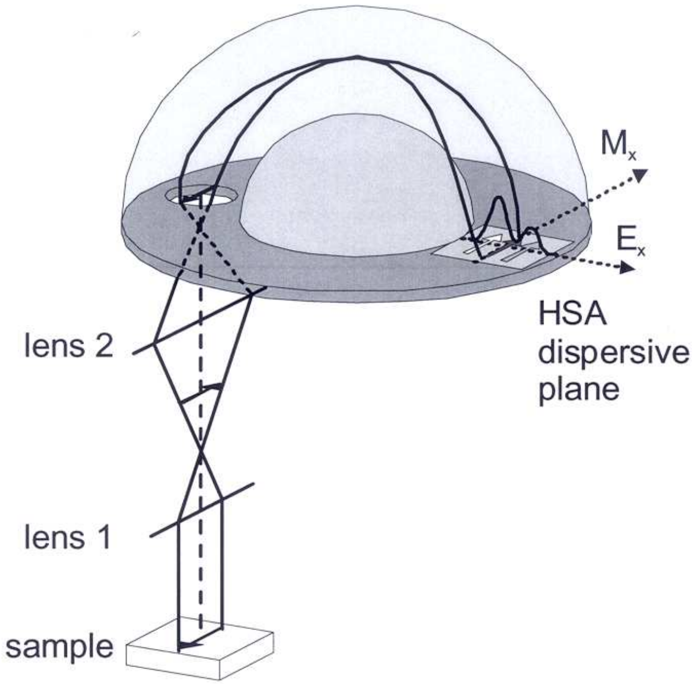
- Cabling used for the contacts (heating, grounding, potentials, etc.) must not interfere with the scanning motion.
- Cooling needs special design
- In most of the cases sample holders are home designed (or modified from standard designs)



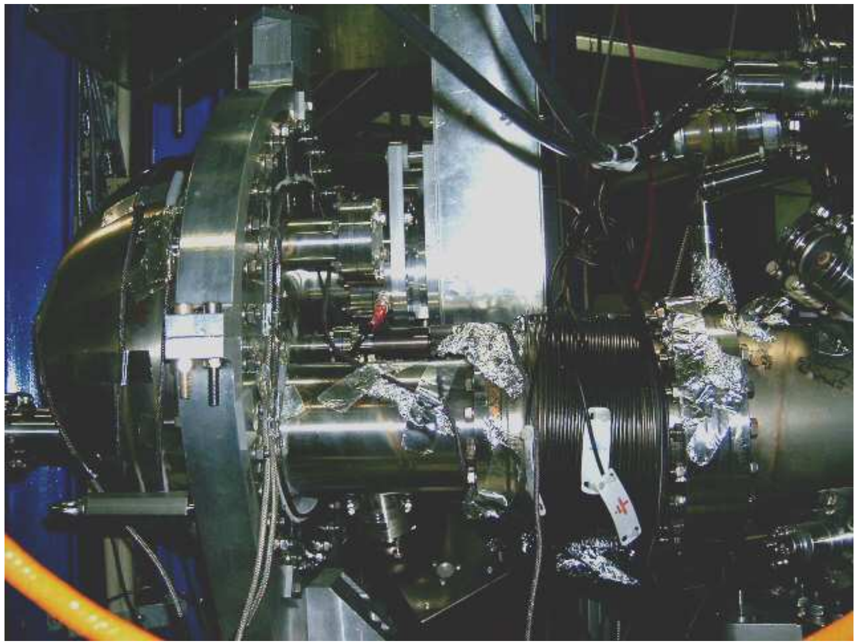


Electron analyzers

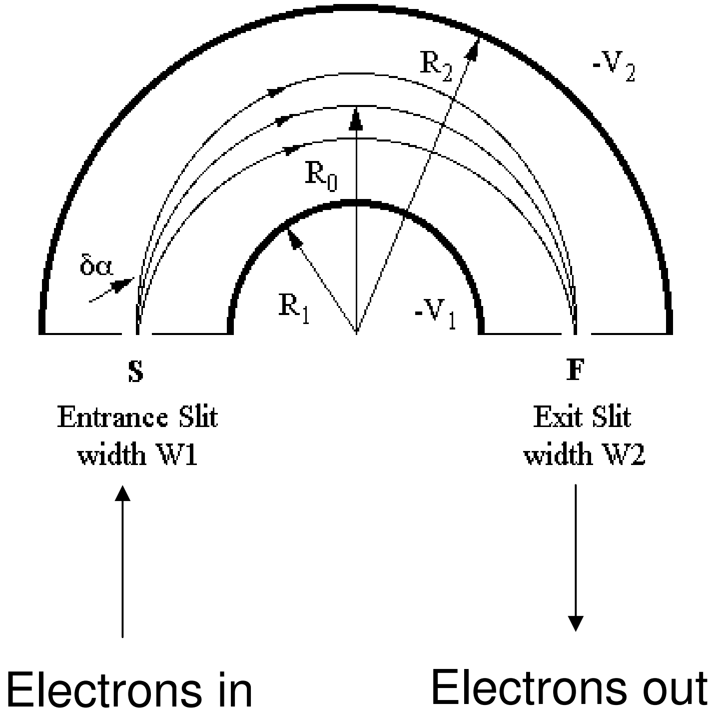
- The most used type of electron analyzer is the Hemispherical Electron Analyzer (HEA)
- Due to geometrical constrains the detection in mainly grazing



Electron analyzer of the SPEM



Potential along the median surface: $V_0 = \frac{V_1 R_1 + V_2 R_2}{2R_0}$



Tangential injection

$$V_1 = V_0 \frac{R_2}{R_1} \qquad V_2 = V_0 \frac{R_1}{R_2}$$

$$E_0 = eV_0$$

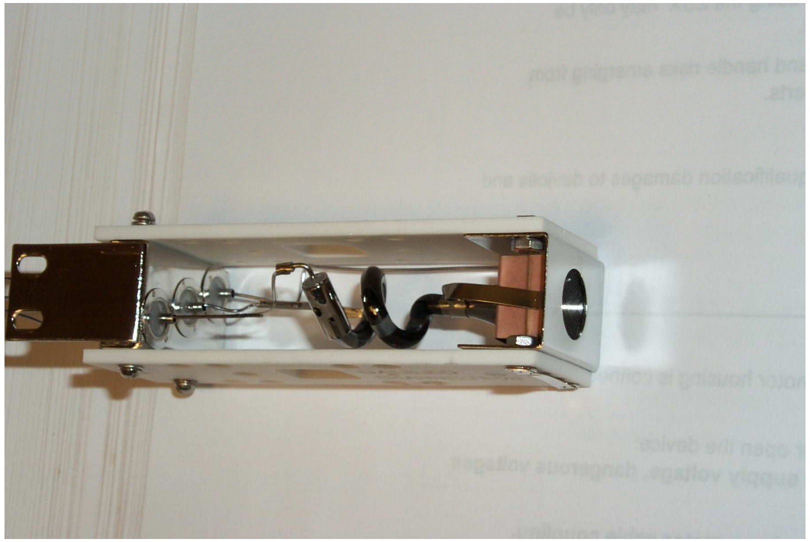
Angled injection

$$\Delta R = 2 R_0 \left[\frac{\Delta E}{E} - (\delta\alpha)^2 \right]$$

Electron detectors

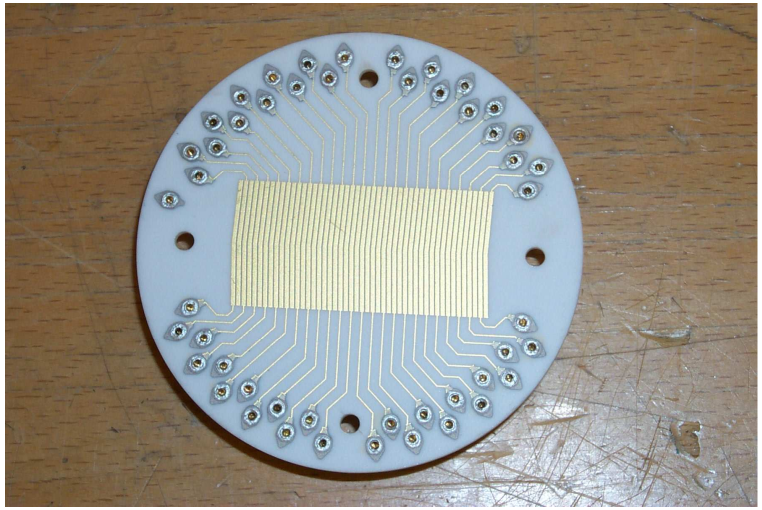
Single channel

- Single channeltron
- Single Au plated anode
- Not very diffused

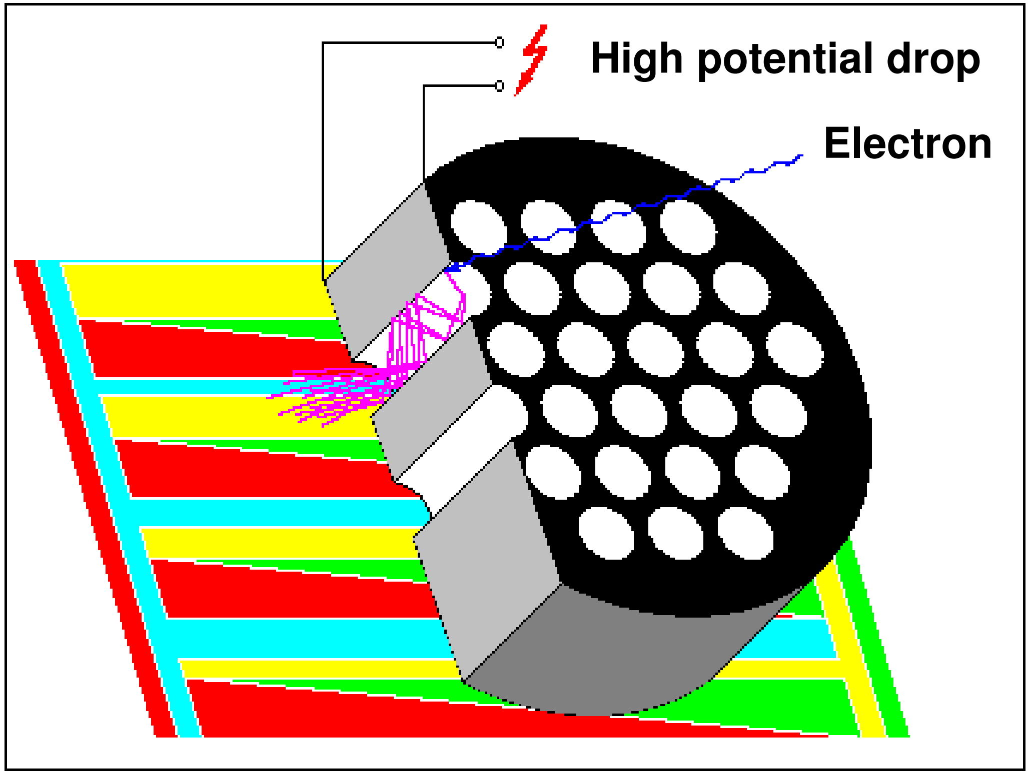


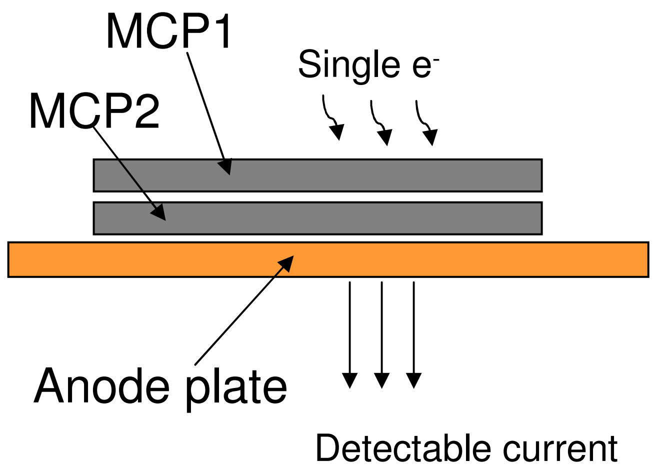
Multi channel

- Array of channeltron (low number of channels)
- Multi Au plated anodes (100 channels)
- 2D-CCD detectors

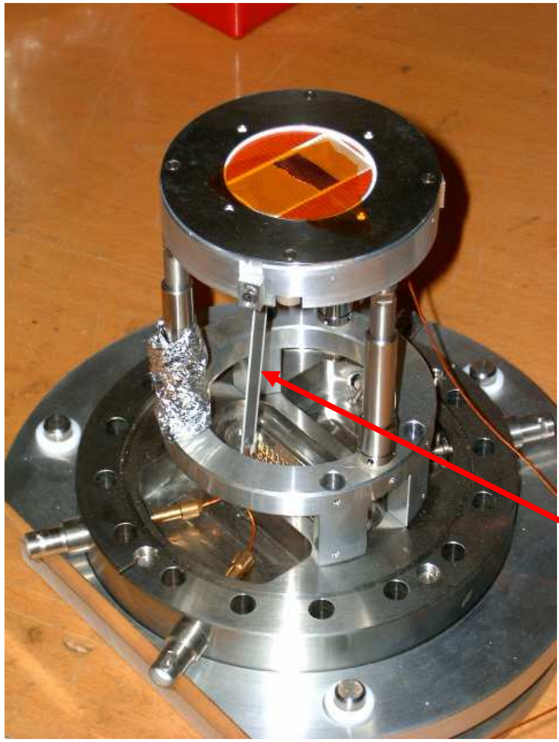


Electron detectors based on micro channel plates

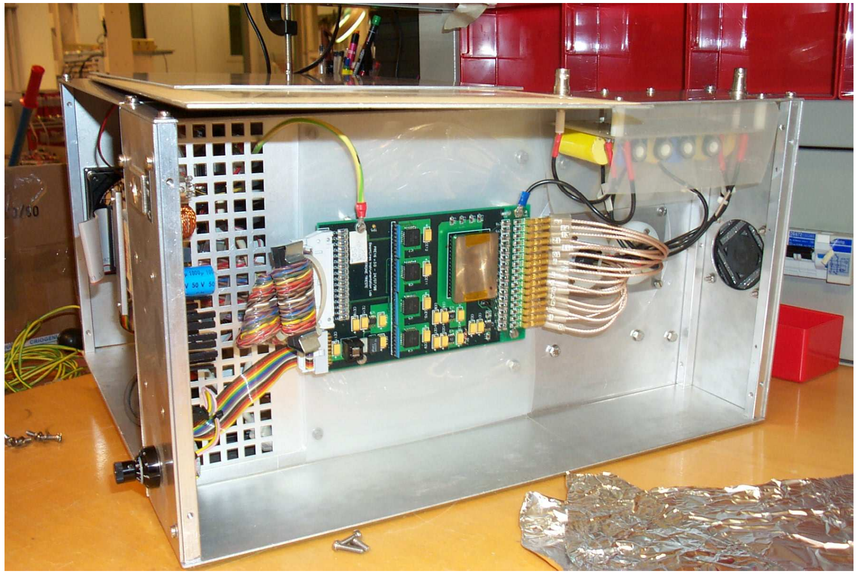


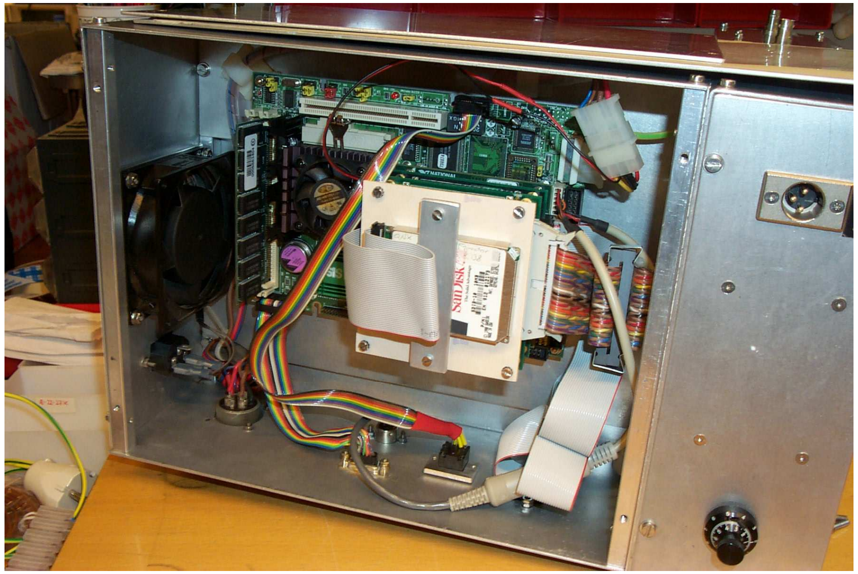


The Microchannel Plate (MCP) consists of millions of very-thin, conductive glass capillaries (4 to 25 micro meters in diameter) fused together and sliced into a thin plate. Each capillary or channel works as an independent secondary-electron multiplier to form a two-dimensional secondary-electron multiplier.



Vacuum compatible condensor

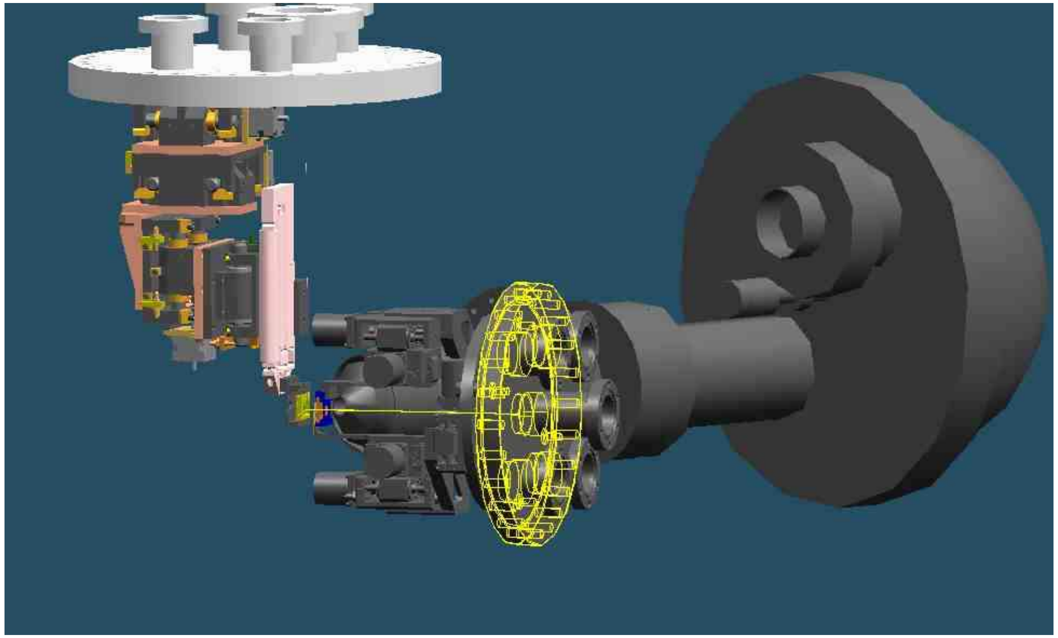


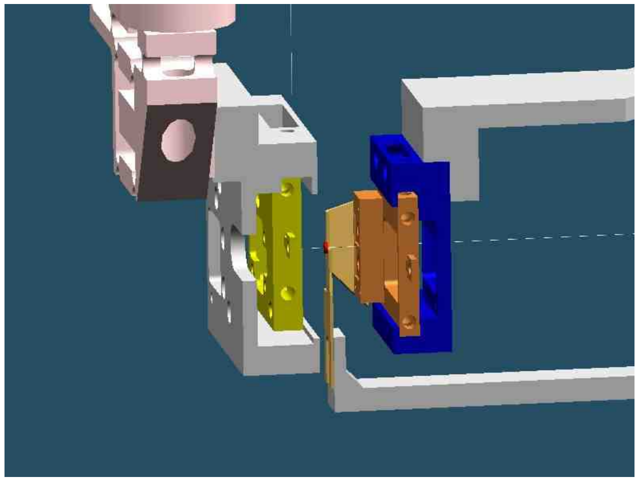


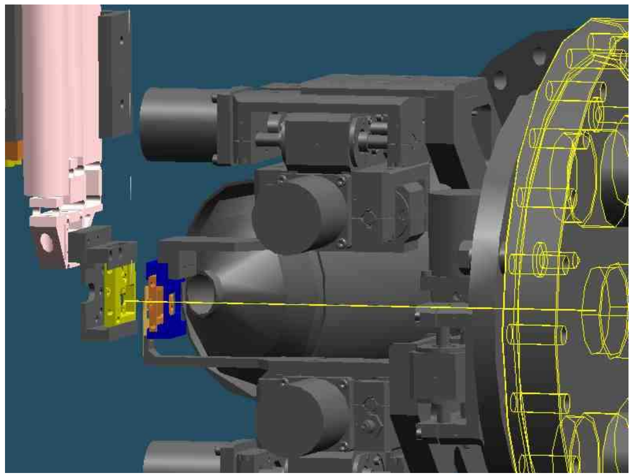
Electron Detector Electronics

- Discriminators
- Preamplifiers
- Counters

Final layout of the experimental chamber

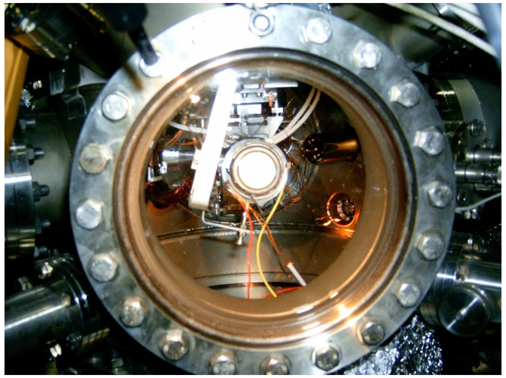


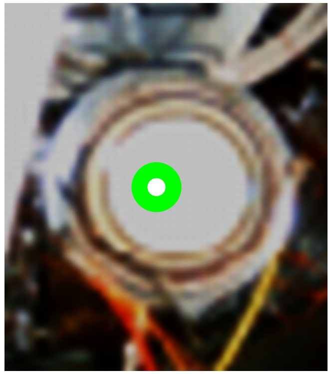


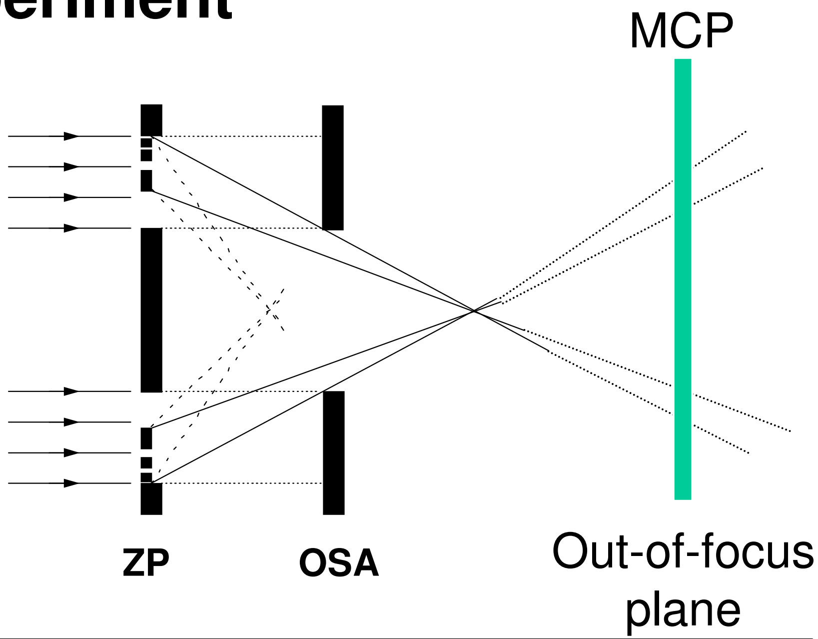


Start-up of an experiment

1. Optics alignment



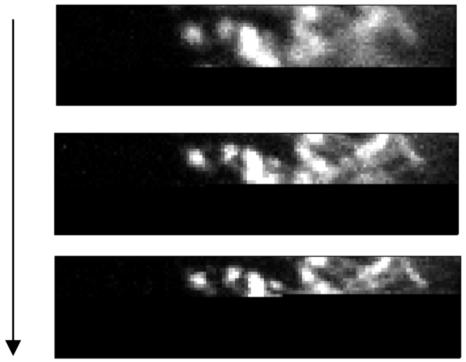


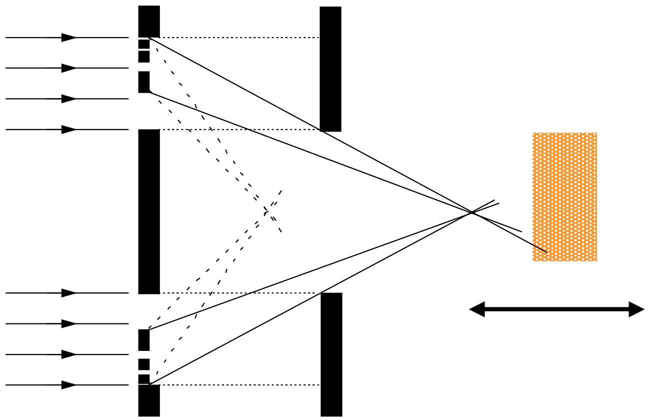


2. Sample on the x-ray focus

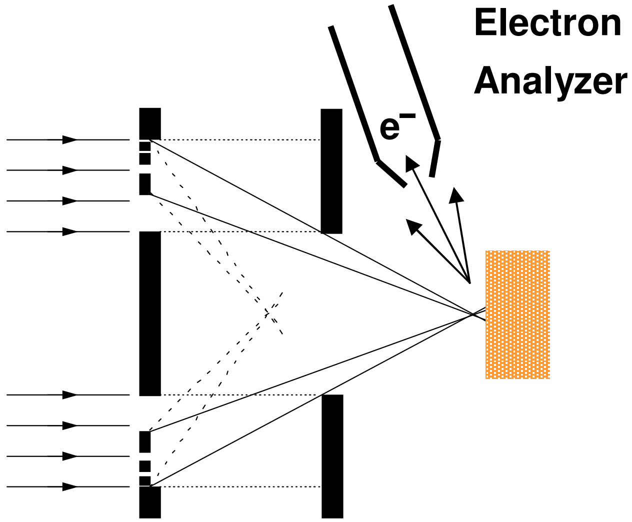
$$DOF = \frac{\delta r}{D} f_m$$

Typical: 5-15μm





3. Analyzer adjustment



4. Data acquisition

- Images: electron analyzer set to a fixed energy and sample rastered
- Photoemission Spectra: sample fixed and energies scanned

Image analysis

- Nature of the contrast in the images
- Getting the chemical information out of the artefacts
- Multichannel detection

• Chemical inhomogeneity

Ni islands on Si

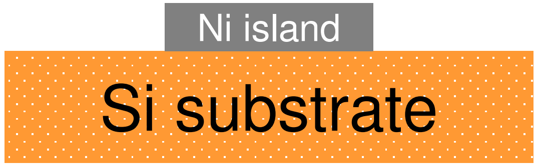
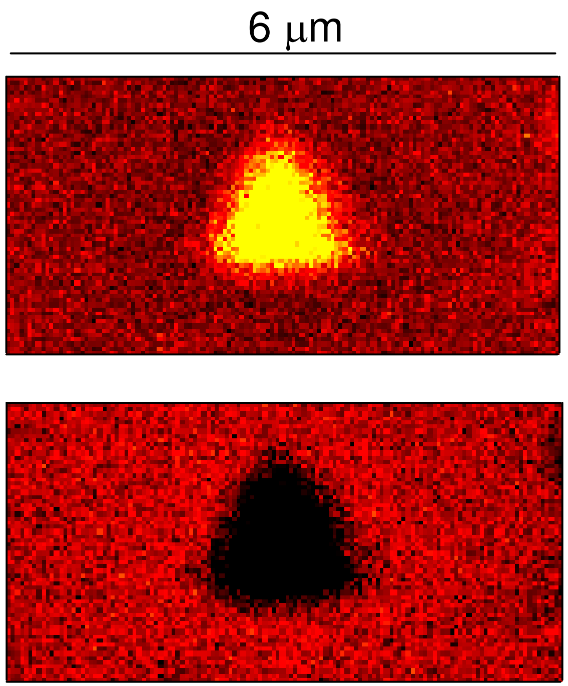


Image on Ni

Image on Si



Au patch on Rh(110)

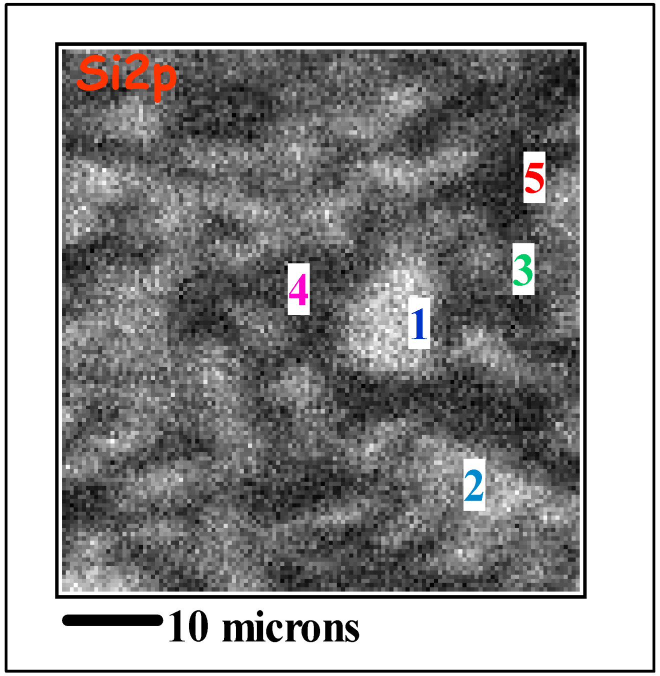
Image on Rh

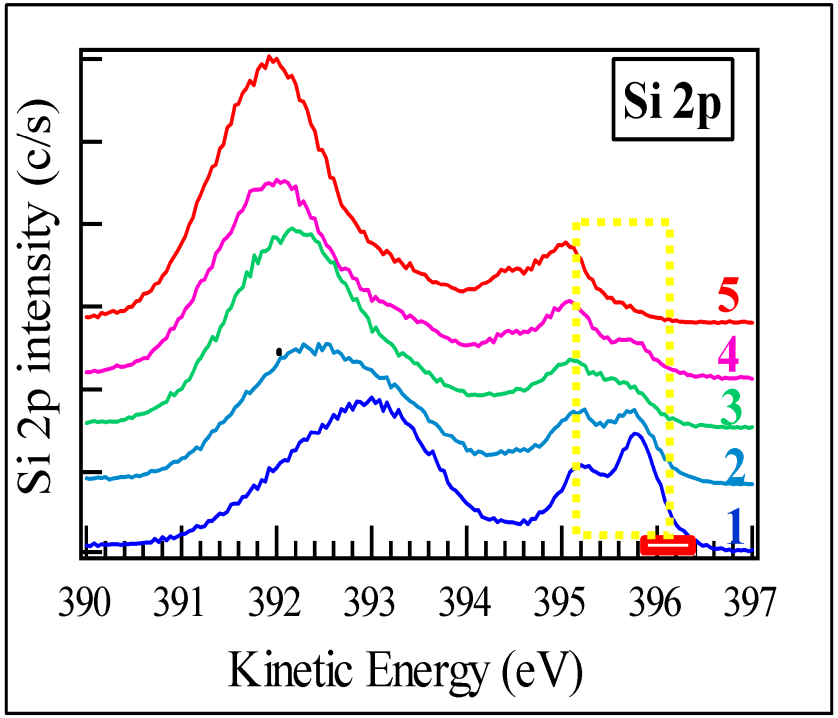
Image on Au

- Topography

- Other sources of contrast

charging





Getting the chemical information out of the artefacts

Artefacts

1. Topography
2. Beam induced effects:
   - C deposition (residual gases)
   - O_2 reduction
   - Charging
3. Background level

How to remove the topographic contribution

$$I = \frac{I_{peak} - I_{bkg}}{I_{peak} + I_{bkg}}$$

$$I = \frac{I_{peak} - I_{bkg}}{I_{bkg}}$$

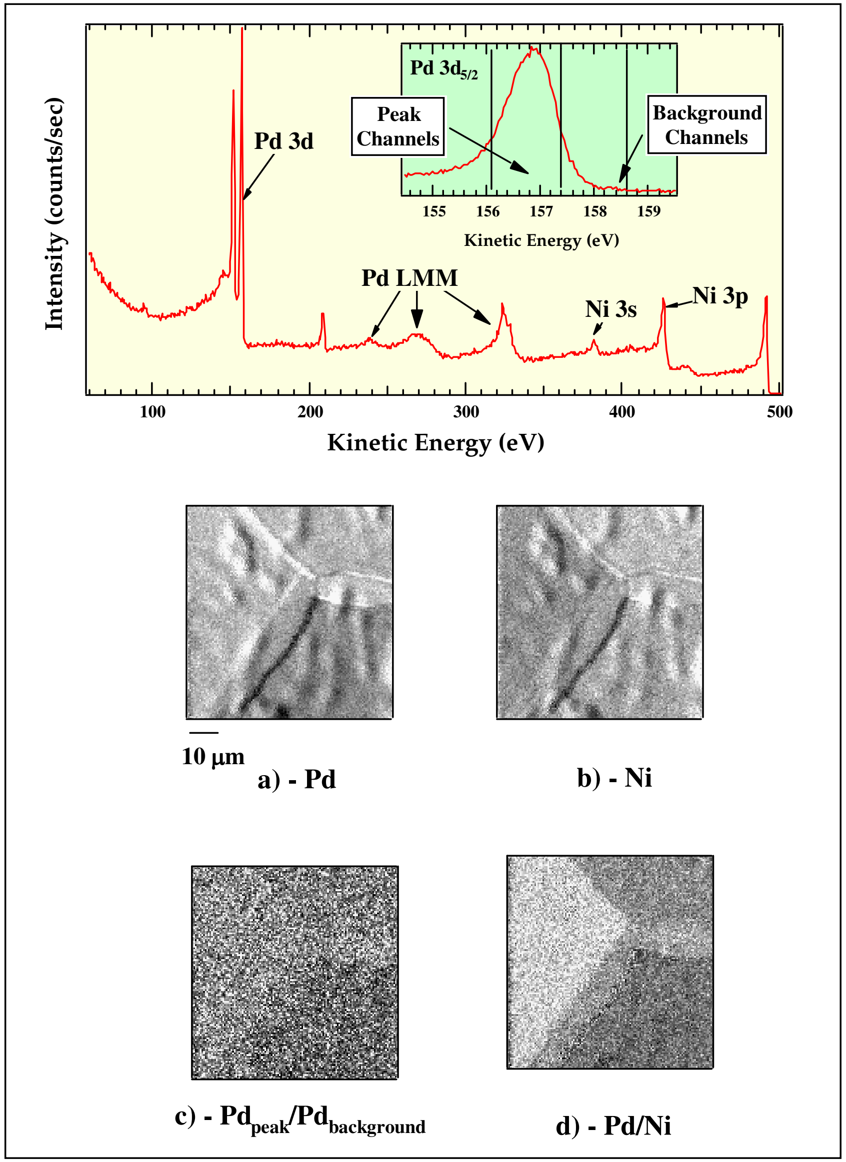
$$I = \frac{I_{peak}}{I_{bkg}}$$

sometimes only:

$$\frac{I_{peak1}}{I_{peak2}}$$

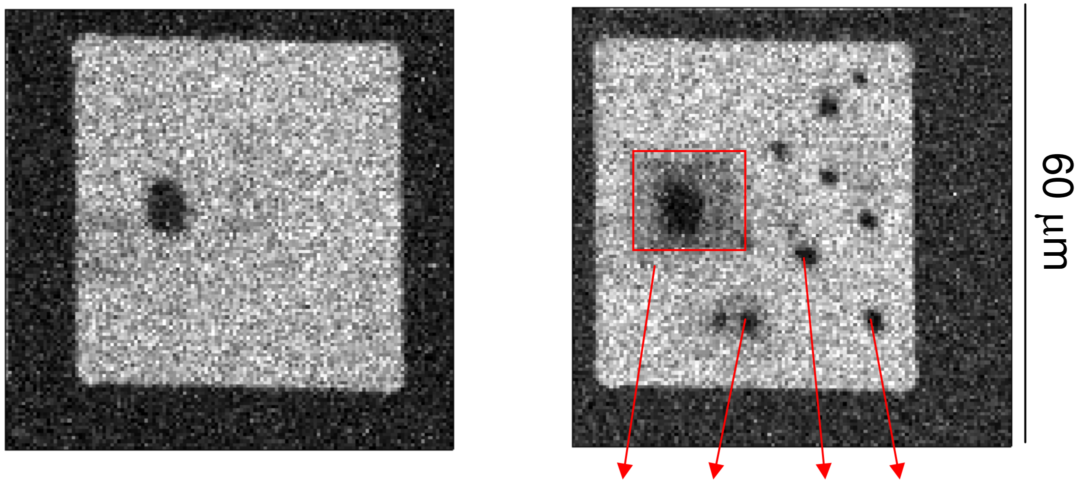
Which I_{bkg} ?

- $I_{bkg\ left}$
- $I_{bkg\ right}$
- $I_{bkg\ (left+right)}$
- *secondaries*



C growth

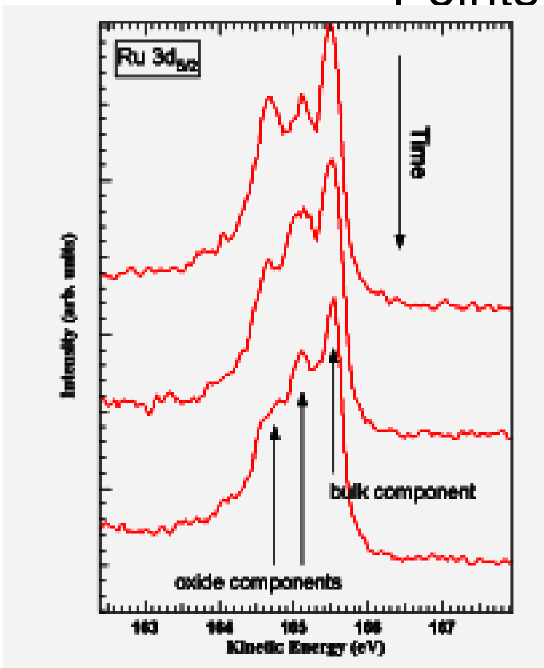
SiO_x sample
Si2p maps



Points irradiated (>10 min)

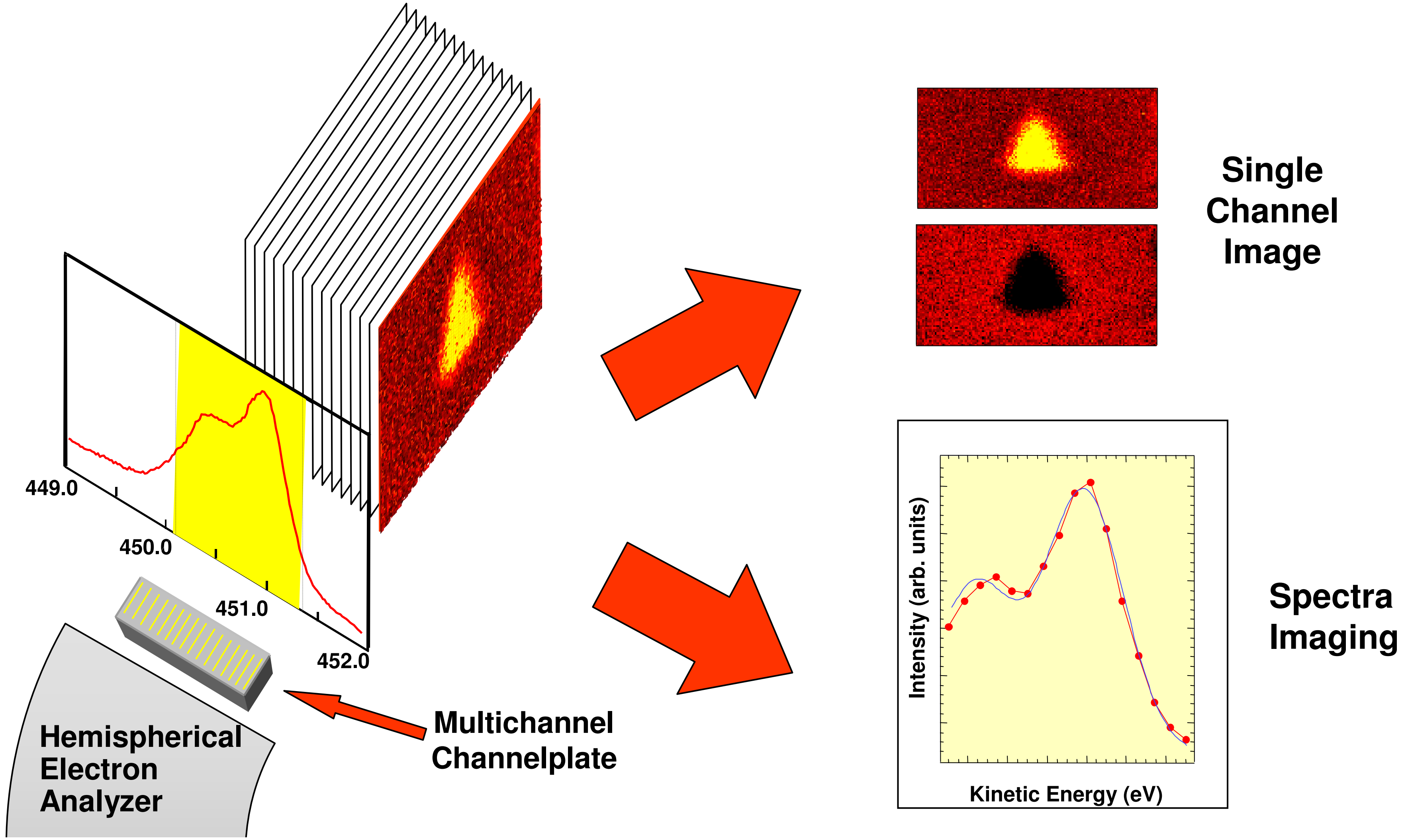
O_2 reduction

RuO_x sample



Each spectrum every 1 min

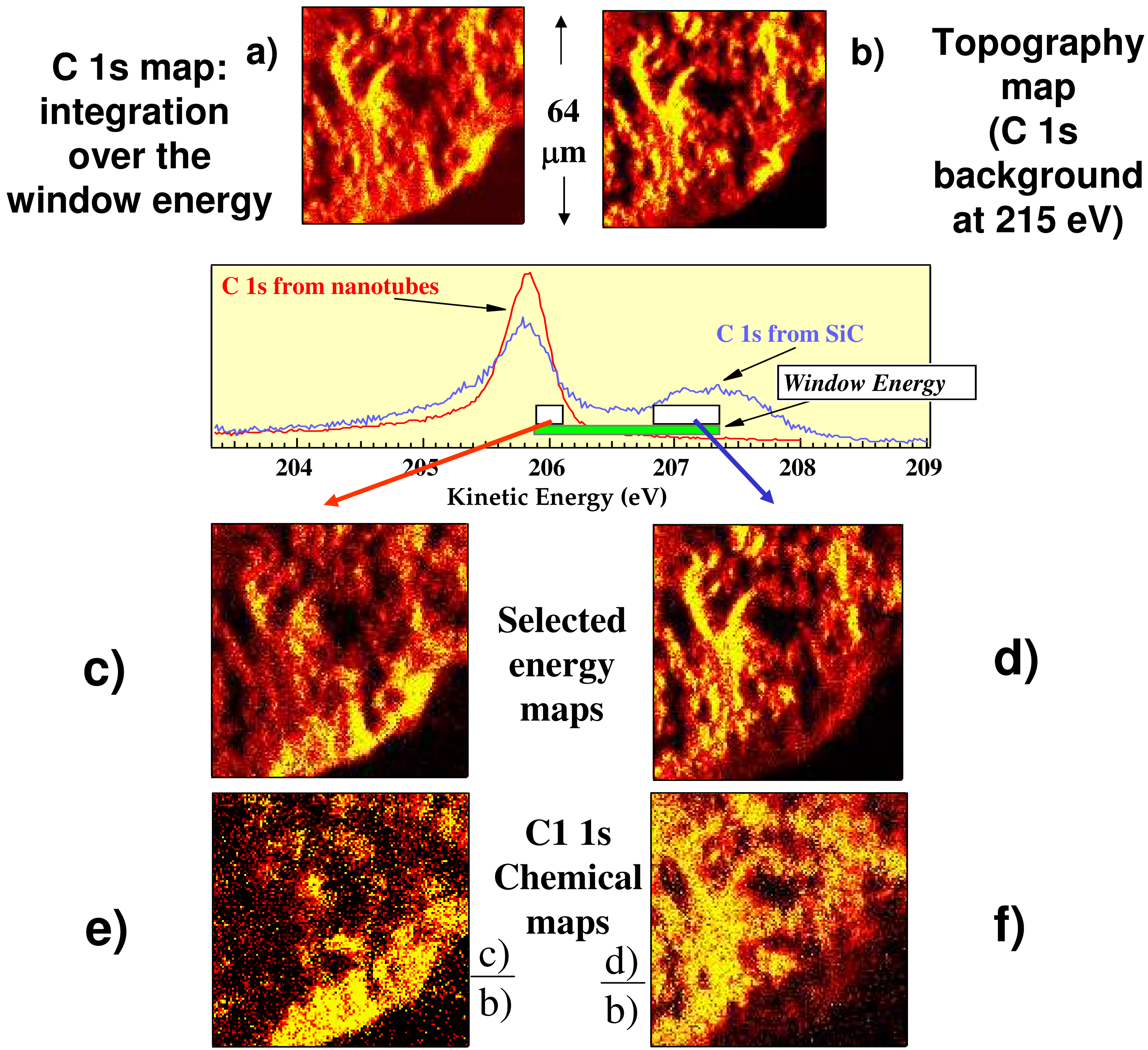
Multichannel detection



Single Channel Analysis

Carbon nanotubes on SiC

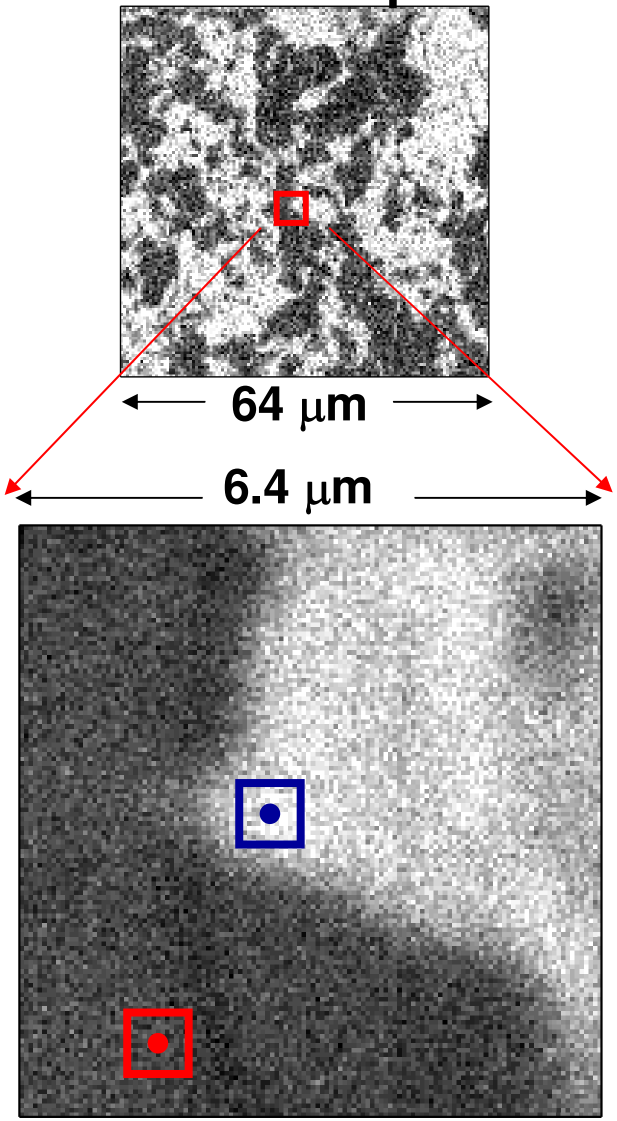
R. Larciprete – Enea - Italy



Spectra Imaging

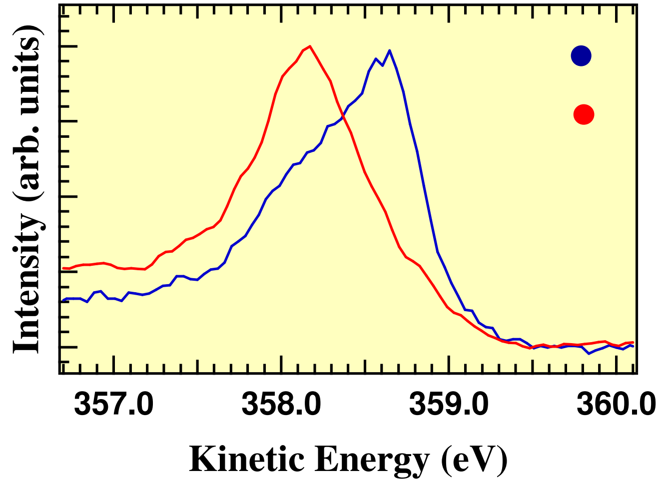
Ru(0001) oxidation
H. Conrad – FHI - Germany

Ru 3d maps

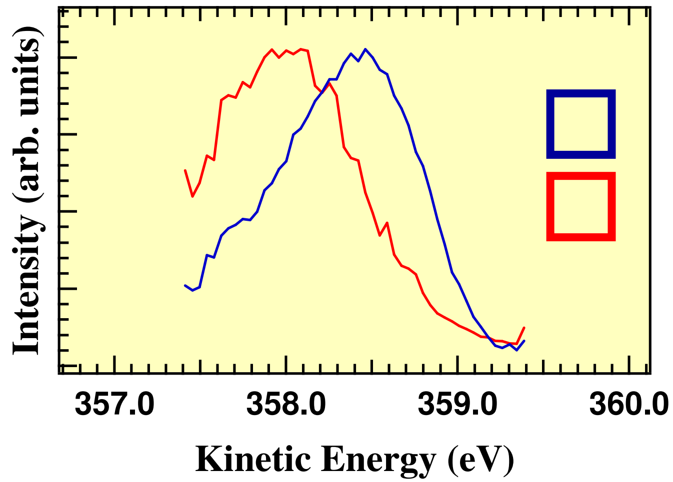


Ru $3d_{5/2}$ spectra

Conventional Scanning Spectroscopy (48 points - 70 sec)



Inherent Dispersion Energy Spectroscopy (48 points - 10 sec)



References

S. Guenther, B. Kaulich, L. Gregoratti, M. Kiskinova, “Photoelectron Microscopy and Applications in Surface and Material Science”, PROG SURF SCI, 70-, pp. 187-74 (2002).

A.W. Potts, G.R. Morrison, L. Gregoratti, A. Barinov, B. Kaulich, M. Kiskinova, “The exploitation of multichannel detection in scanning photoemission microscopy”, SURF REV LETT, 9-2, pp. 705-8 (2002).

L. Gregoratti, A. Barinov, E. Benfatto, G. Cautero, C. Fava, P. Lacovig, D. Lonza, M. Kiskinova, R. Tommasini, S. Mahl, “48-Channel electron detector for photoemission spectroscopy and microscopy”, REV SCI INSTRUM, 75-1, pp. 68-4 (2004).