Microspectroscopy and Spectromicroscopy

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Microspectroscopy and Spectromicroscopy

Part I:

- **Motivation**
- **concepts to achieve spatial resolution**
- **XPS and XAS concept for X-ray microscopy**
- **Focussing devices for X-rays**
- **STXM versus TXM**
- **SPEM**

Why microscopy with x-rays or electrons?

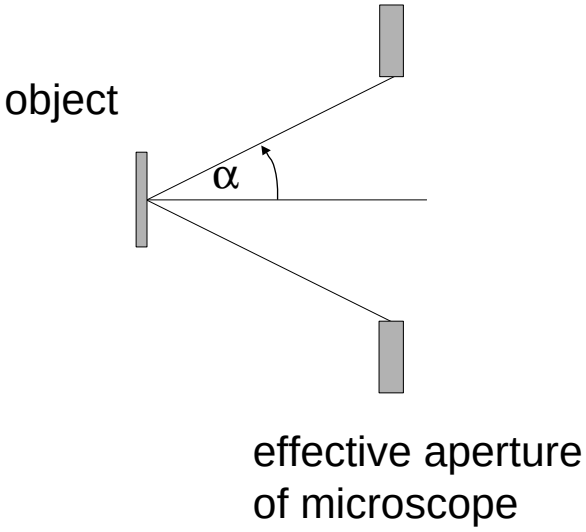
Diffraction limit: $r_d = 0.61 \frac{\lambda}{NA}$

$NA = n \sin(\alpha)$

typical NA = 10^{-1} - 10^{-3}

photons $\lambda = \frac{1240}{E[eV]}[nm]$

electrons $\lambda = 1.23 \frac{1}{\sqrt{E[eV]}}[nm]$



- **high lateral resolution** (10 nm range possible, TEM sub nm range)
- **transparency** of matter

Why Microspectroscopy and Spectromicroscopy?

- **elemental resolution:**

  quantify composition of sample with lateral resolution

- **chemical information:**

  characterize chemical environment

- **further information on:**

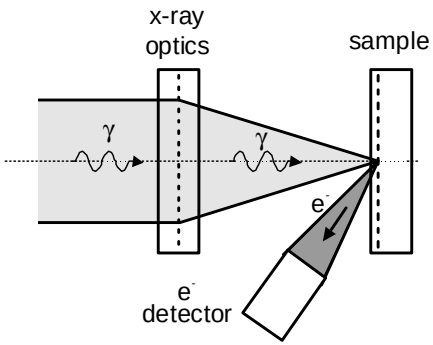
  Magnetism
  different type of bonding (e.g. π, σ bonds)
  orbital alignment
  electronic structure

- **combination with other microscopy techniques**

Concepts to achieve spatial resolution

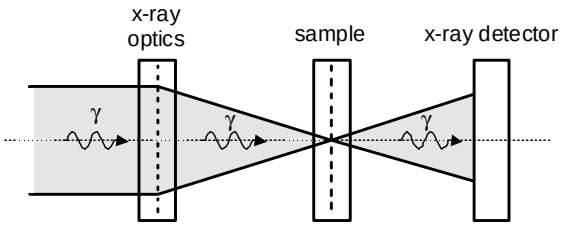
a) µ-spot illumination

1)



SPEM

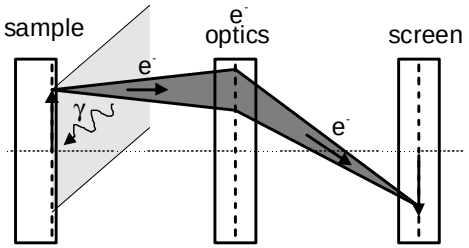
2)



STXM

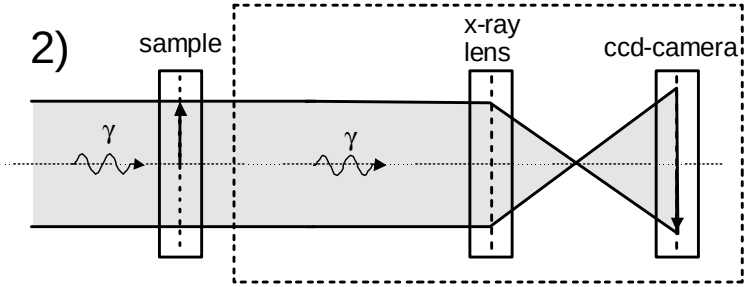
b) homogeneous illumination + imaging system

1)

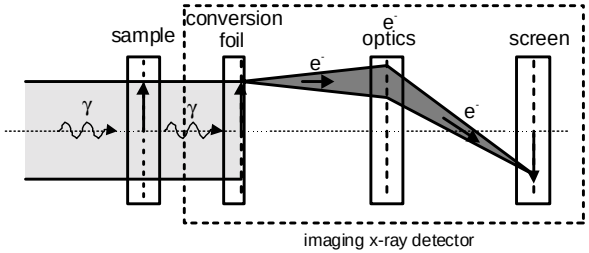


X-PEEM

2)



TXM

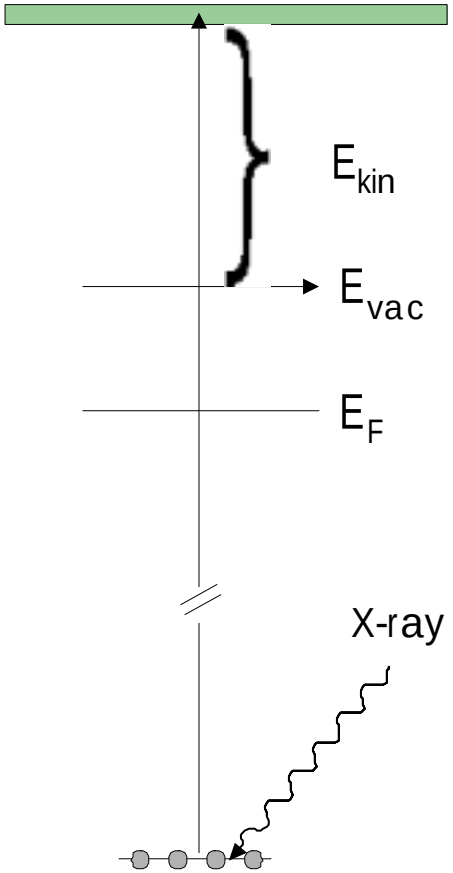


Concepts of Spectromicroscopy: Detection mode

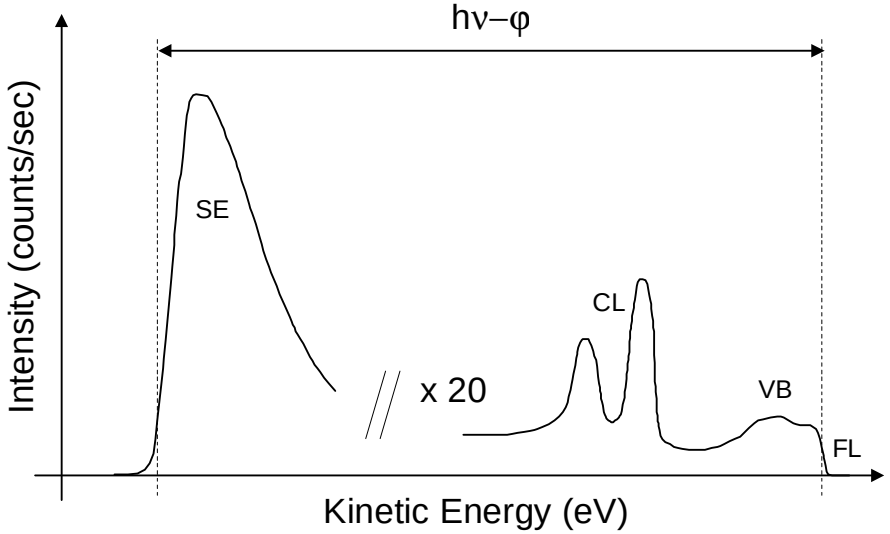
detection	microscope	sensitive to	sample thicknes required	depth information
photon in / electron out	SPEM imaging XPS X-PEEM	XPS	thick possible	few atomic layers
photon in / electron out	SPEM X-PEEM	XAS	thick possible	5-50 nm
photon in / photon out	STXM, TXM conversion microscopy	XAS	thin (< several µm)	bulk
photon in / flourescence out	not considered here			
photon in / ion out	not considered here			

Concepts of Spectromicroscopy: XPS-mode

photon in / electron out + energy filtering of electrons => XPS - mode



photoelectrons of certain kinetic energy used for imaging



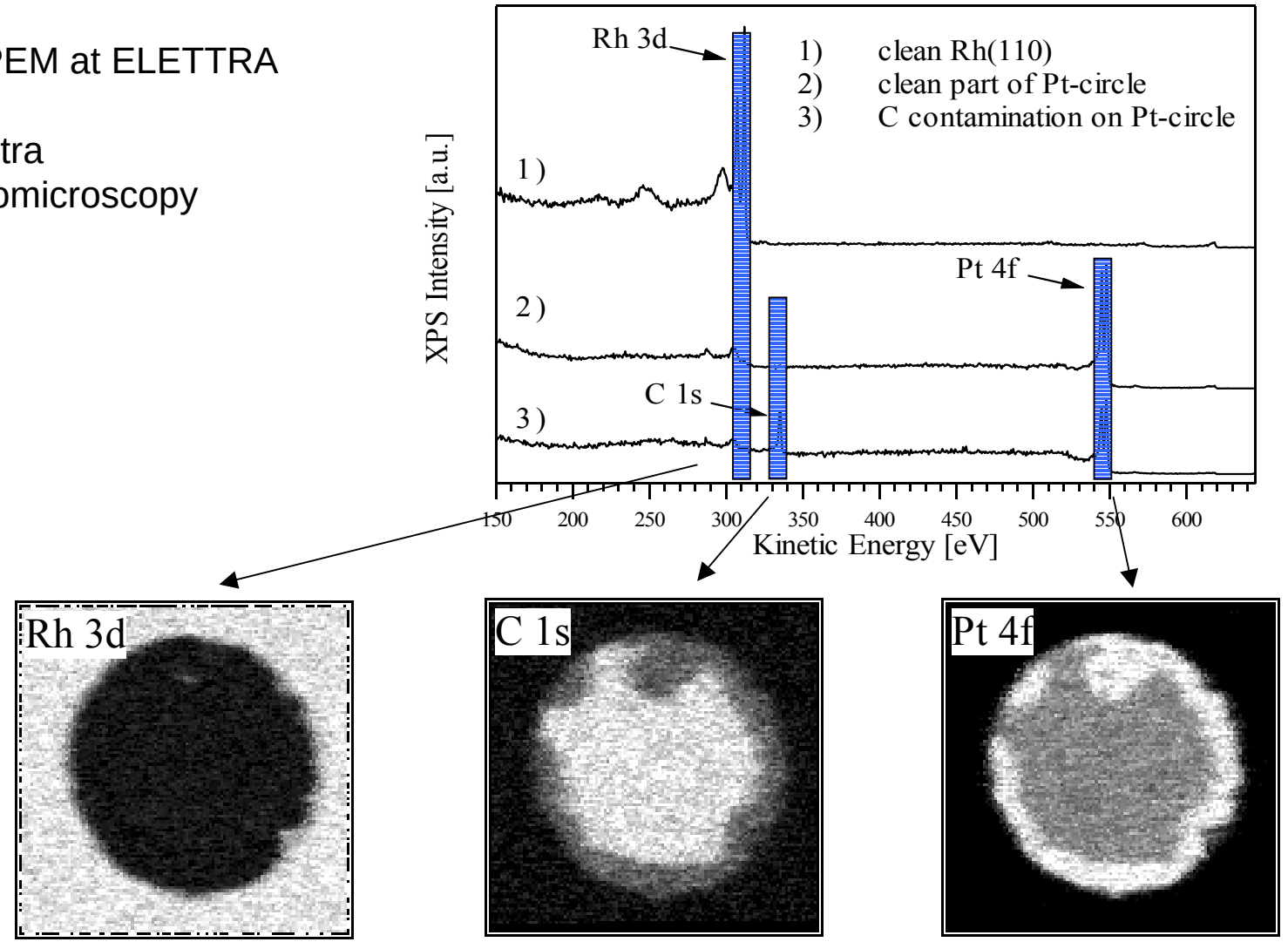
- **high pass filtering:** easier to realize, but noisy
- **bandpass filtering:** better used

- short escape length of e: **probe a few layers only**

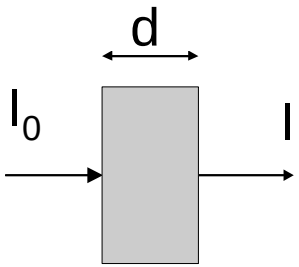
Concepts of Spectromicroscopy: XPS-mode

e.g. SPEM at ELETTRA

μ-Spectra
Spectromicroscopy

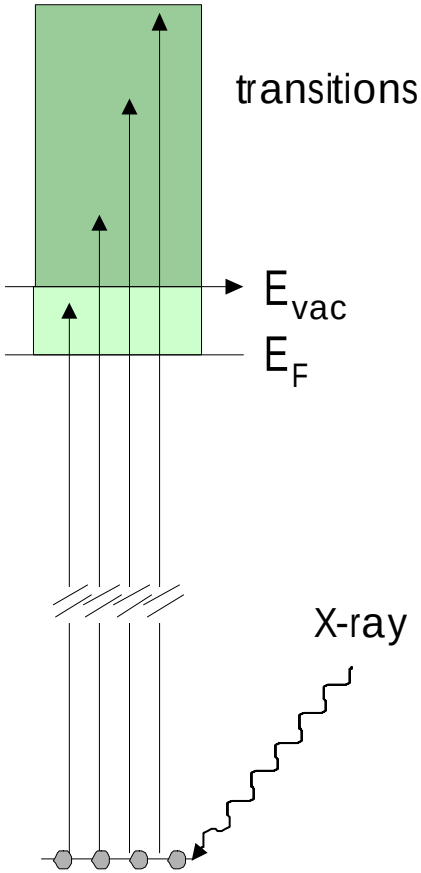


Concepts of Spectromicroscopy: XAS - mode



$$\frac{I}{I_0} = e^{-\mu d}$$

μ: absorption coefficient



detection of **transmitted light: STXM /TXM (bulk sensitive)**

or

detection of **total or secondary electron yield: SPEM / XPEEM (sensitive down to 5-50 nm)**

Concepts of Spectromicroscopy: XAS - mode

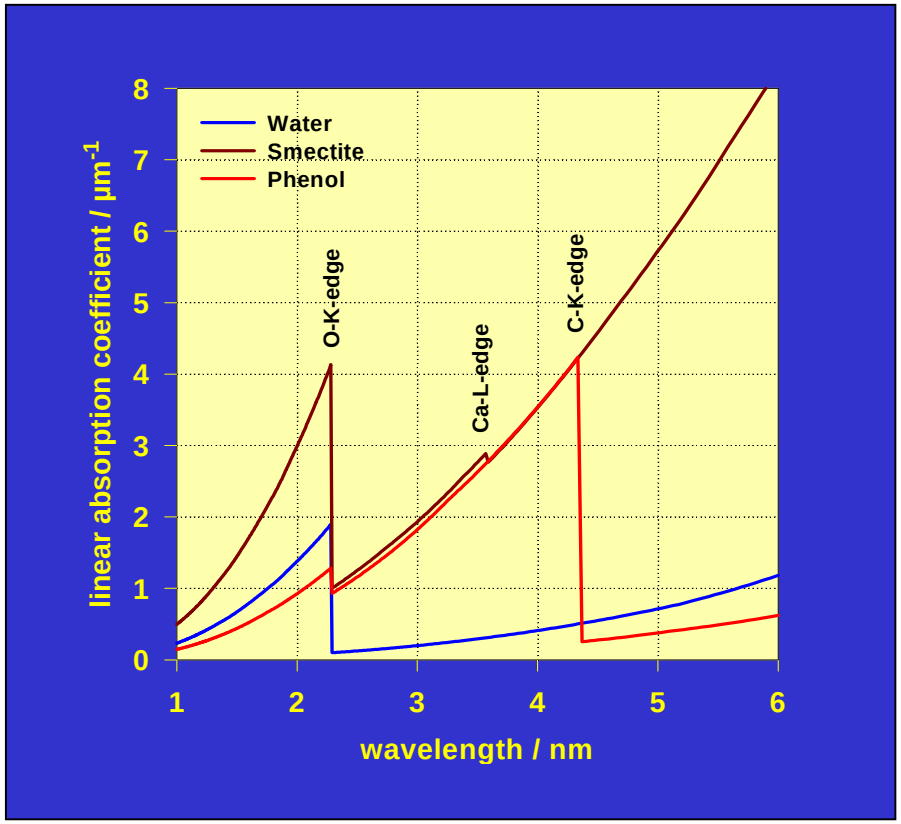
Tuning of photon energy through and absorption edge

water window

chemical contrast for N,C,O
(biology polymers)

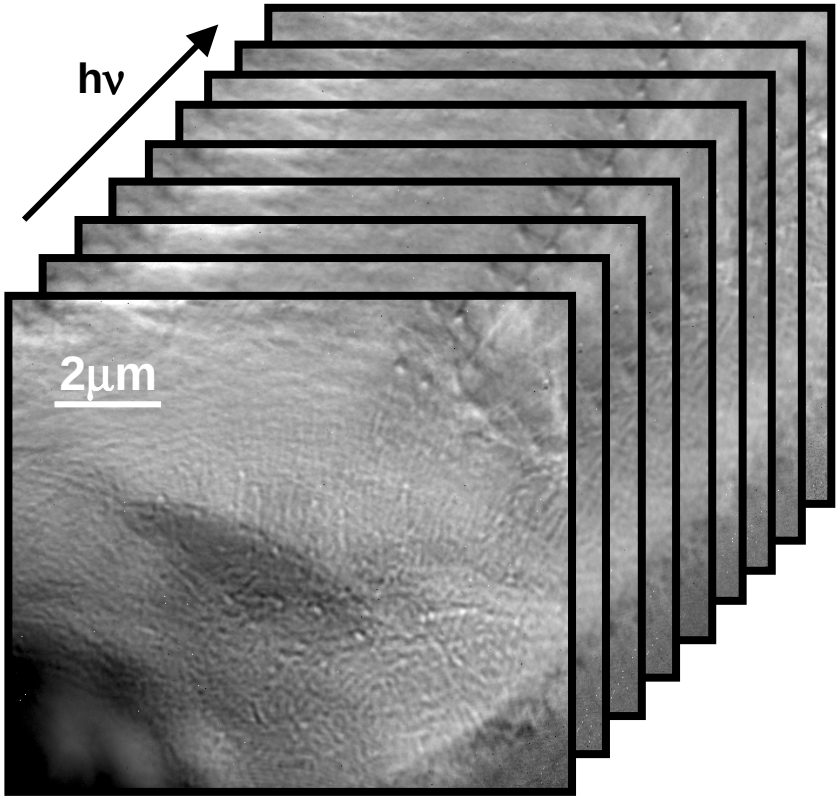
higher energy

good for higher Z-materials



Concepts of Spectromicroscopy: XAS - mode

Image stack

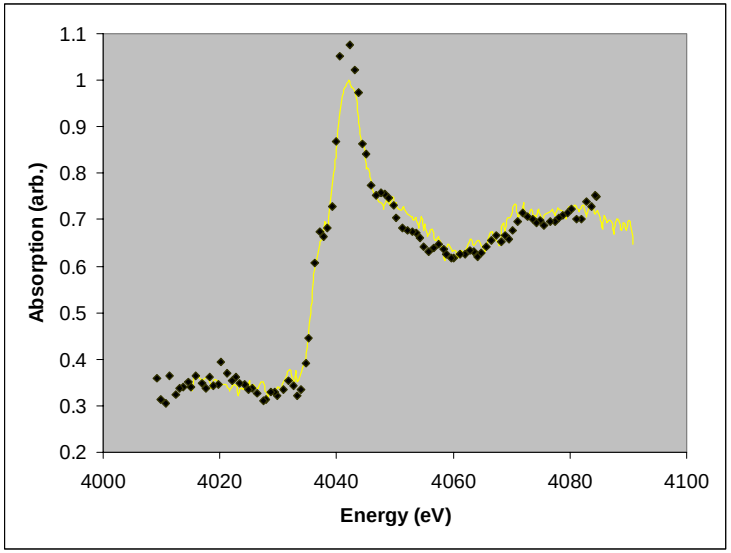


Trabecular bone of a mouse femur sample (10μm thick); Image field is 27 x 21 μm^2

Mineralized area in trabecular bone tissue near Ca K-edge

M.Salome et al. X-Ray Microscopy, AIP Proc. 507 (2000) 178-183

μ-spectrum

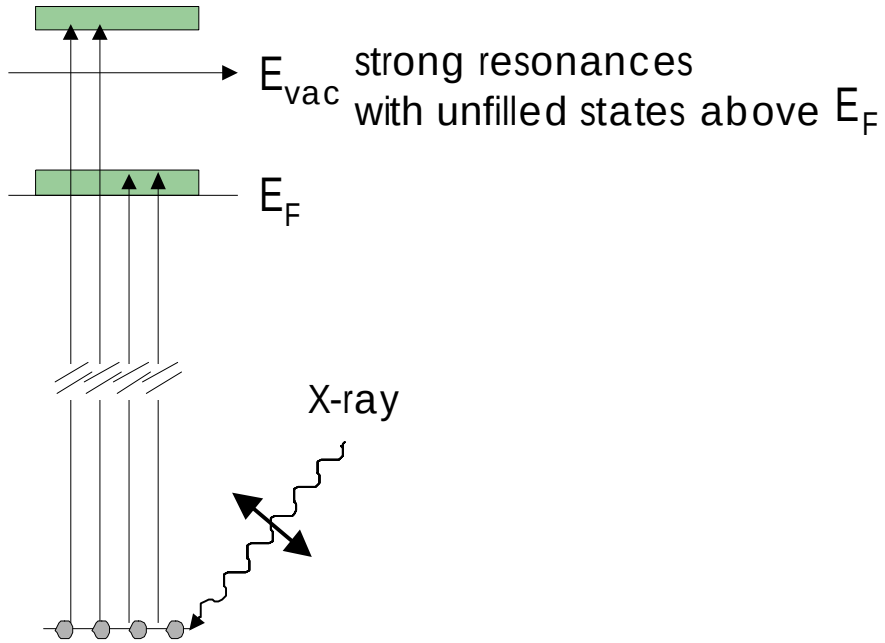


Hydroxy-apatite spectrum recovered from a stack of 200 images

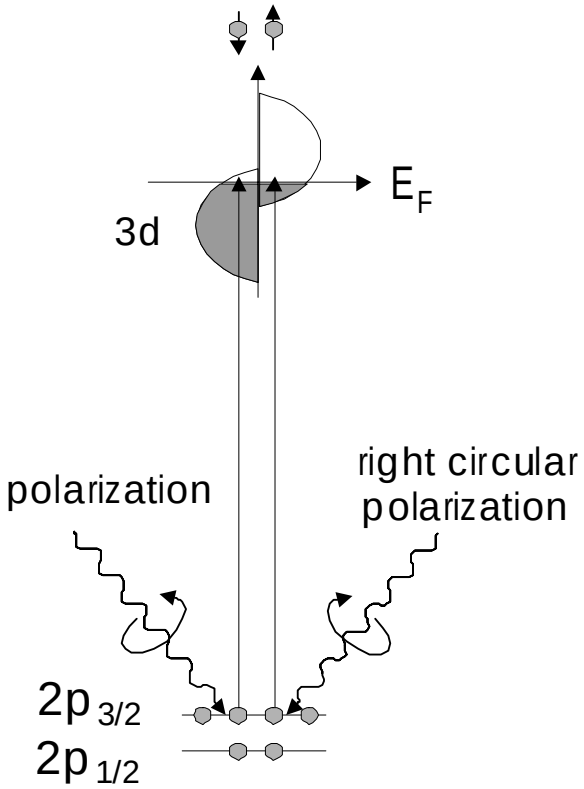
Concepts of Spectromicroscopy: XAS - mode

XANES: tuning on molecular orbitals
XMLD: imaging antiferromagnets, XMCD: imaging ferromagnets

XAS-XANES, XMLD



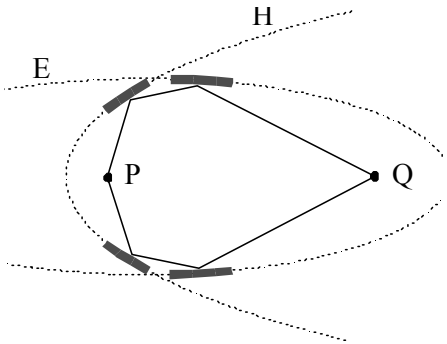
XMCD



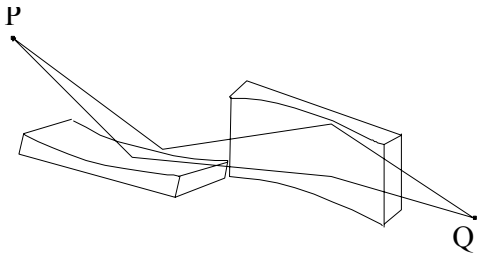
Focusing devices for x-ray point sources

Mirrors at grazing incidence

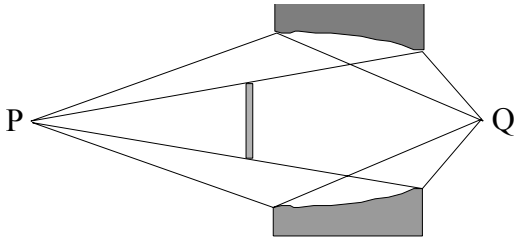
- working in total reflection
- straight forward **energy tunability**
- typical **lateral resolution ≤ 1 μm**



Wolter type mirror



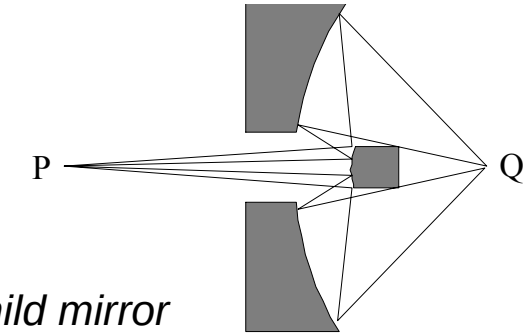
Kirkpatrick-Baez configuration



ellipsoidal mirror

Mirrors at non grazing incidence

- use of **multilayer coatings** to achieve reflectivity
- no **energy tunability**
- **lateral resolution d: 0.1 μm < d < 1 μm**



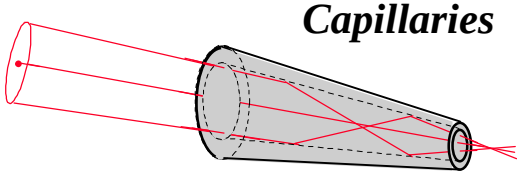
Schwarzschild mirror

Focusing devices for x-ray point sources

Other focusing devices for high energy applications

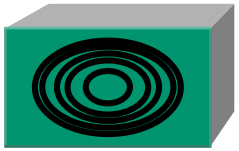
e.g. at ESRF

Capillaries



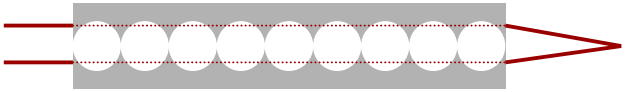
3x3μm² & 10^{11}ph/s
8 keV < E < 18 keV

Bragg-Fresnel lens



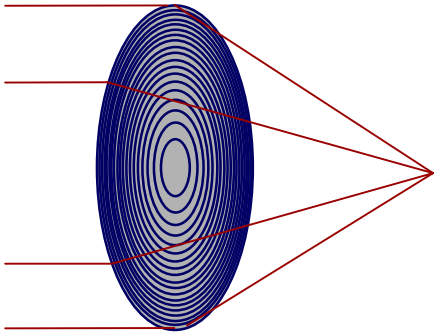
1x10μm² & 10^{10}ph/s-Si(111)
4 keV < E < 70 keV

Refractive lens (Tomi lens)



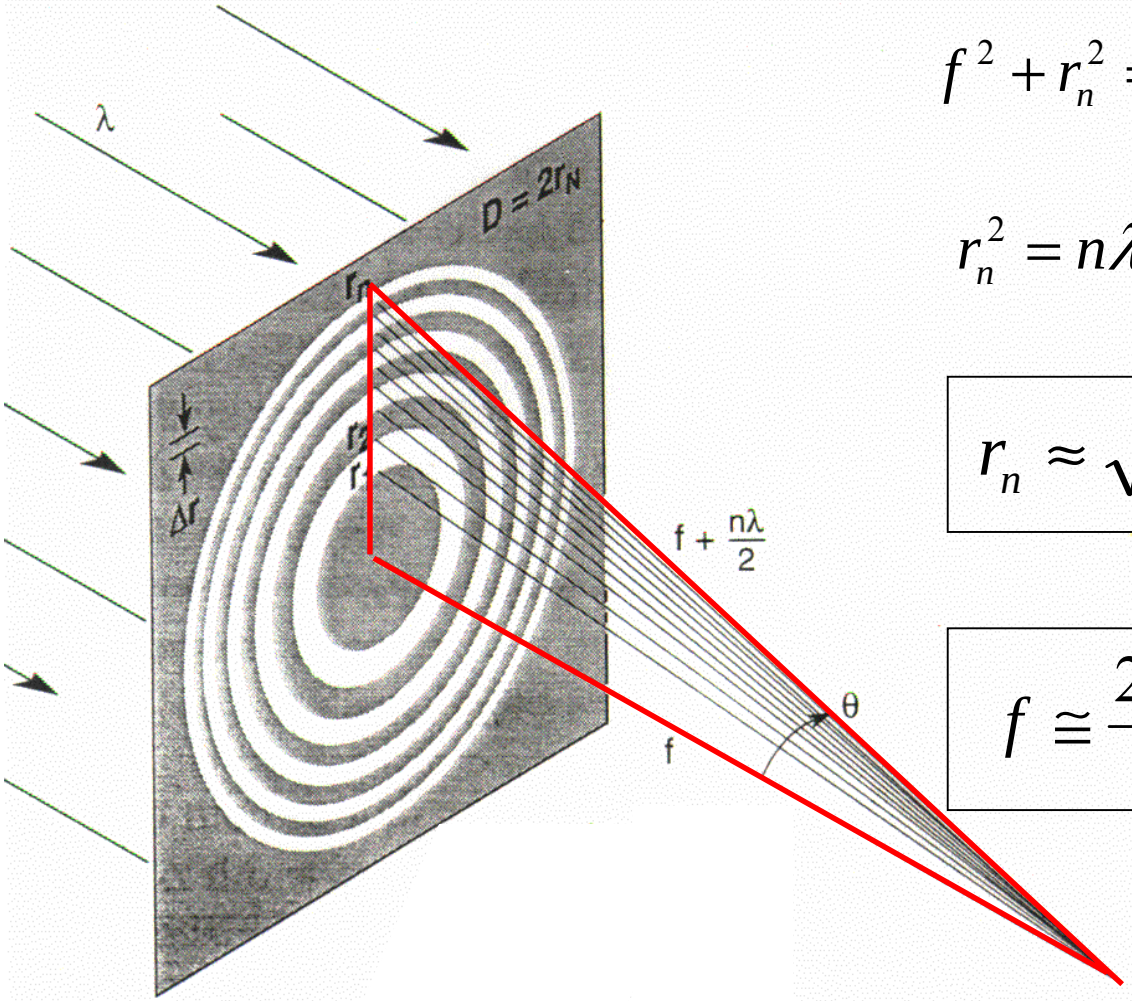
1x5μm² & 10^{9}ph/s - Si(111)
4 keV < E < 70 keV

Currently highest lateral resolution with Fresnel lenses



Fresnel lens

Focusing devices for x-ray point sources: Fresnel lenses



$$f^2 + r_n^2 = (f + \frac{n\lambda}{2})^2$$

$$r_n^2 = n\lambda f + \frac{n^2\lambda^2}{4} \quad if \quad f >> \frac{n\lambda}{4}$$

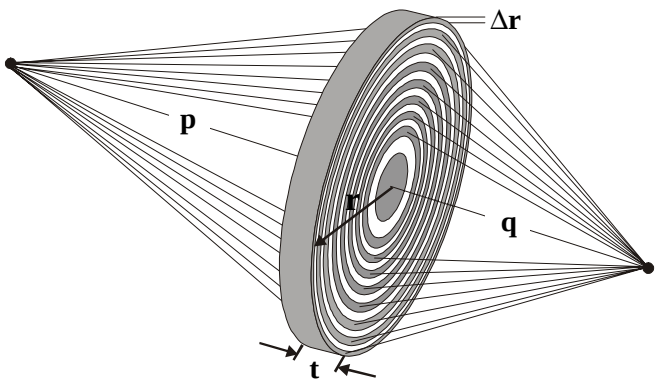
$$r_n \approx \sqrt{n\lambda f}$$

$$\Delta r = r_n - r_{n-1}$$

$$f \cong \frac{2r\Delta r}{\lambda} = \frac{2r\Delta r[nm]}{1240} E[eV]$$

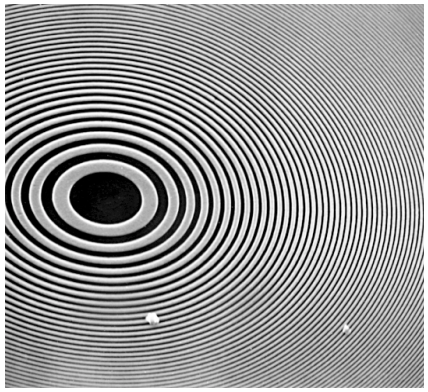
e.g. 2r = 100 μm Δr=100 nm, E= 500 eV => f = 8 mm

Focusing devices for x-ray point sources: Fresnel lenses

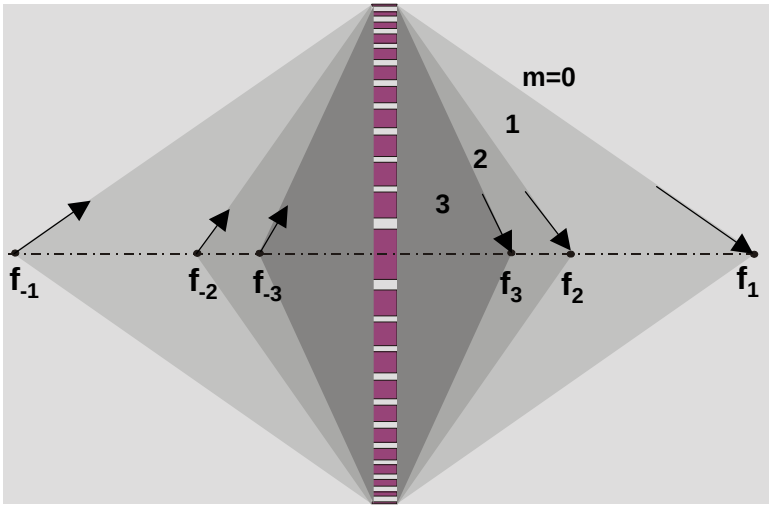


analogue: **if n > 100**

$$\frac{1}{f} = \frac{1}{p} + \frac{1}{q}$$



higher orders:



$$\frac{\text{Intensity diffracted in order m}}{\text{Intensity incident on the zone plate}} \sim \frac{1}{m^2}$$

=> **work only in 1st order diffraction**

Depht of focus: $$DOF = \pm \frac{2(\Delta r)^2}{\lambda}$$

e.g. Δr=100 nm, E= 500 eV => DOF = 8 μm

Focusing devices for x-ray point sources: Fresnel lenses

Spatial resolution δ in 1st order:

$$\delta = \sqrt{\delta^2_r + \delta^2_i + \delta^2_c}$$

δ_r: intrinsic ZP resolution

$\delta_r = 1.22 \times \Delta r_n$

(Rayleigh criterion)

=> **small outermost zone**,

δ_i: demagnified source

$\delta_i = \sigma \bullet q / p$

σ: source size
p: source-ZP distance
q: ZP-object distance

small source size,

δ_c: chromatic aberration

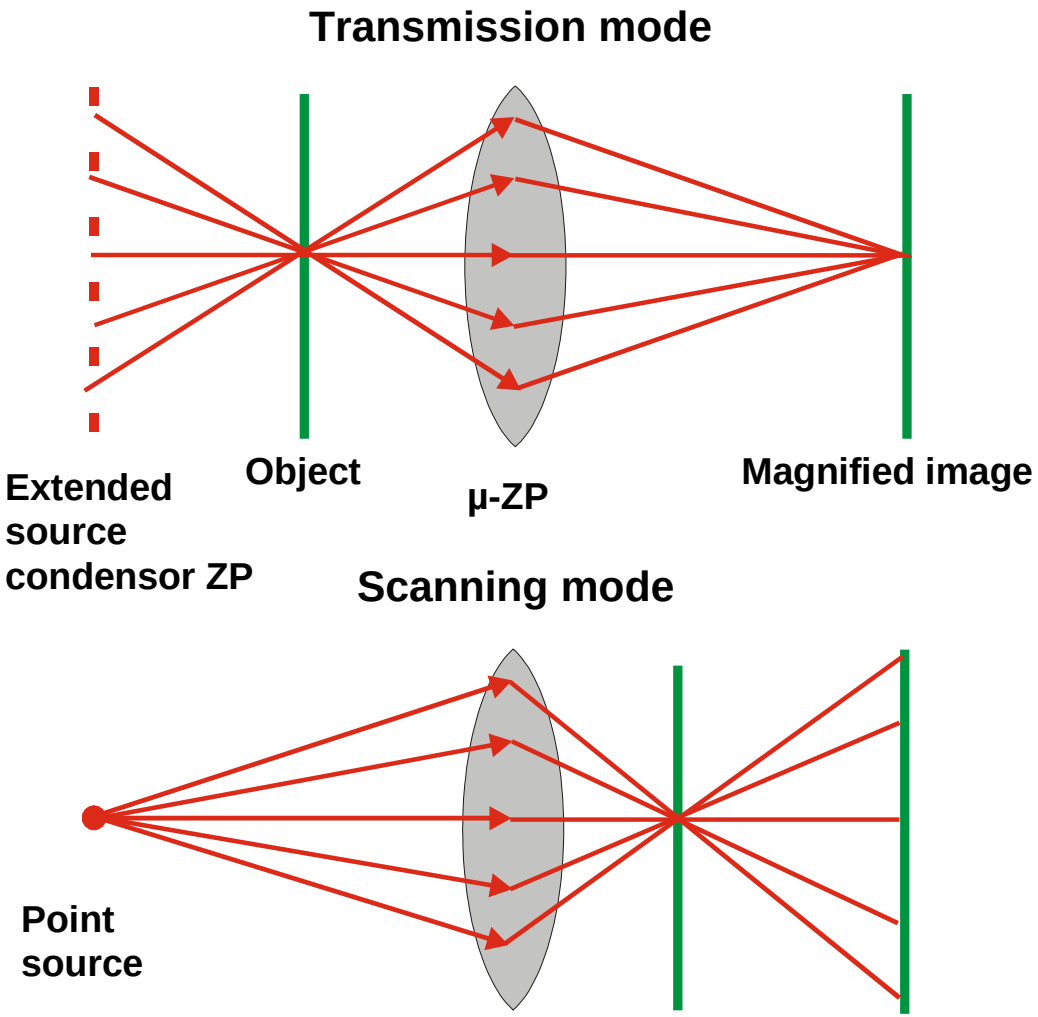
$\delta_c = D \bullet \Delta E / E$

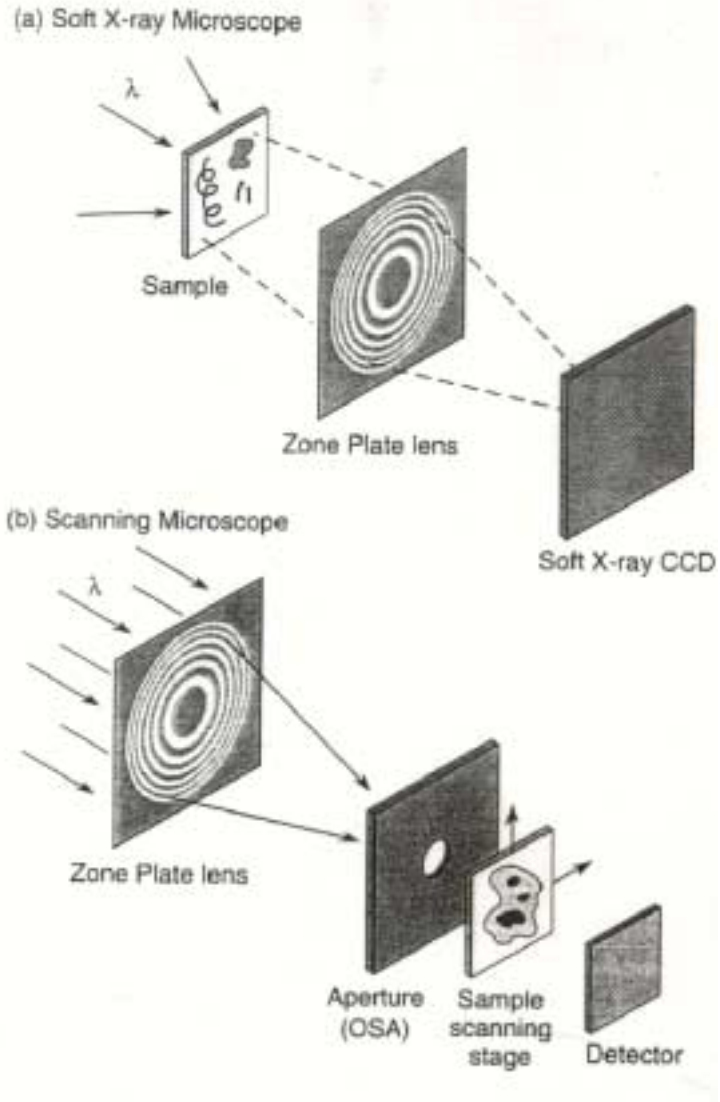
D: ZP diameter
$\Delta E / E$: resolving power of monochromator

monochromatic beam

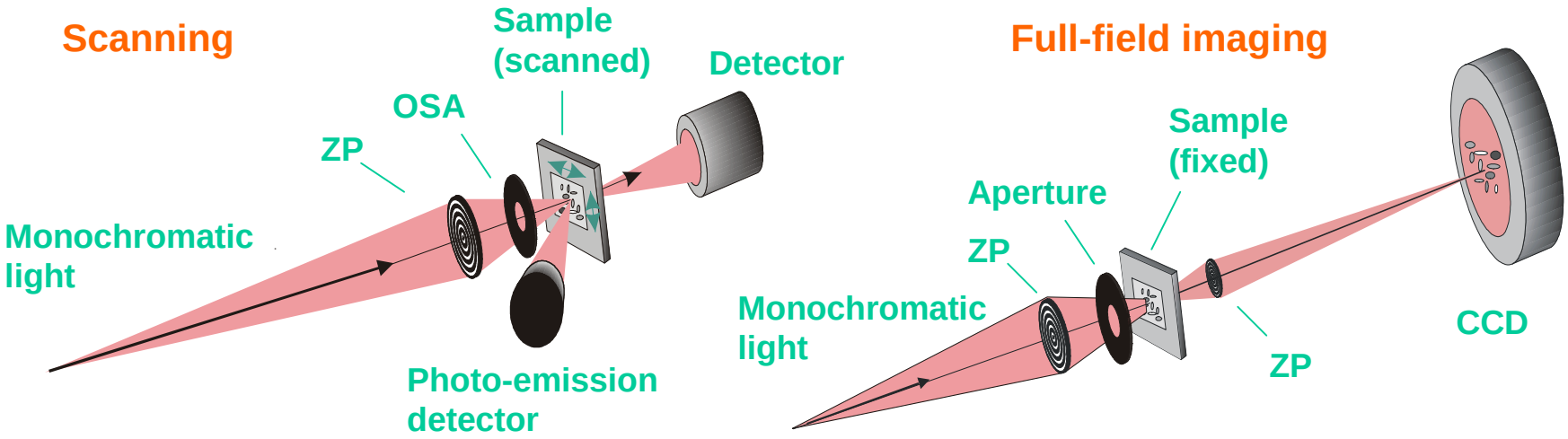
e.g. Δr=100 nm + typical values from synchrotron beamline
δ_r=122 nm, δ_i = 30 µm $\times$ 8 mm/3 m=80 nm, δ_c=100 µm $\times$ 0.2eV/500eV=40 nm =>δ=150 nm

Scanning versus full field imaging transmission microscopy





Scanning versus full field imaging transmission microscopy

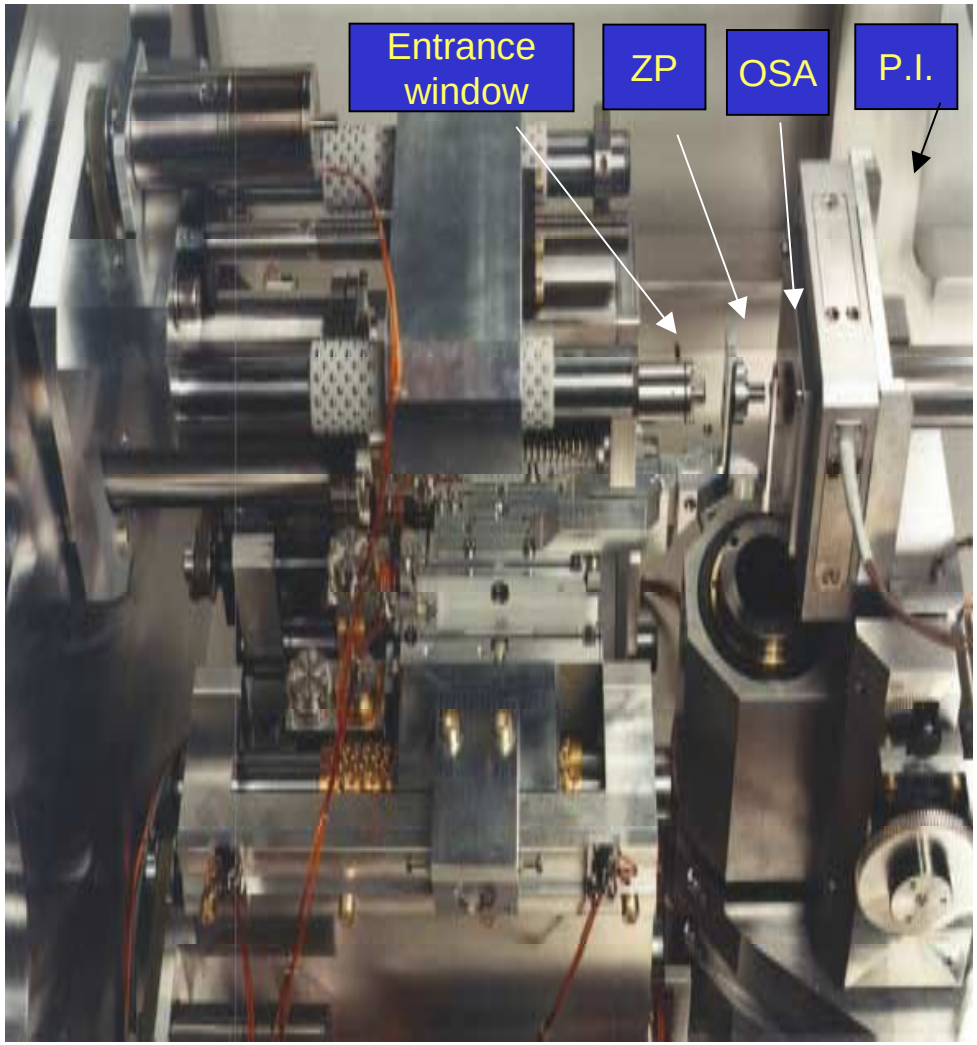


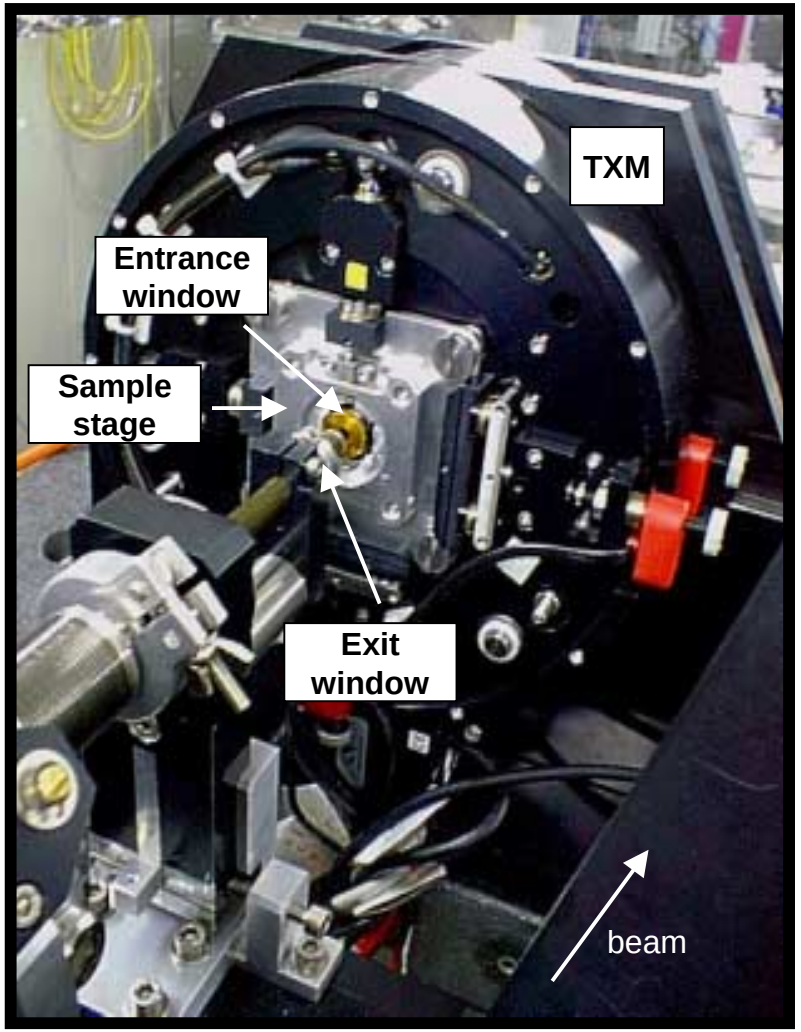
+ versatile detectors can be run simultaneously
+ low demands in the optics setup
+ SPEM possible
- long exposure times
- complex electronics

+ short exposure times
+ access to X-ray tomography in combination with spectro-microscopy
+ highest resolution due to static system
- complex optical alignment

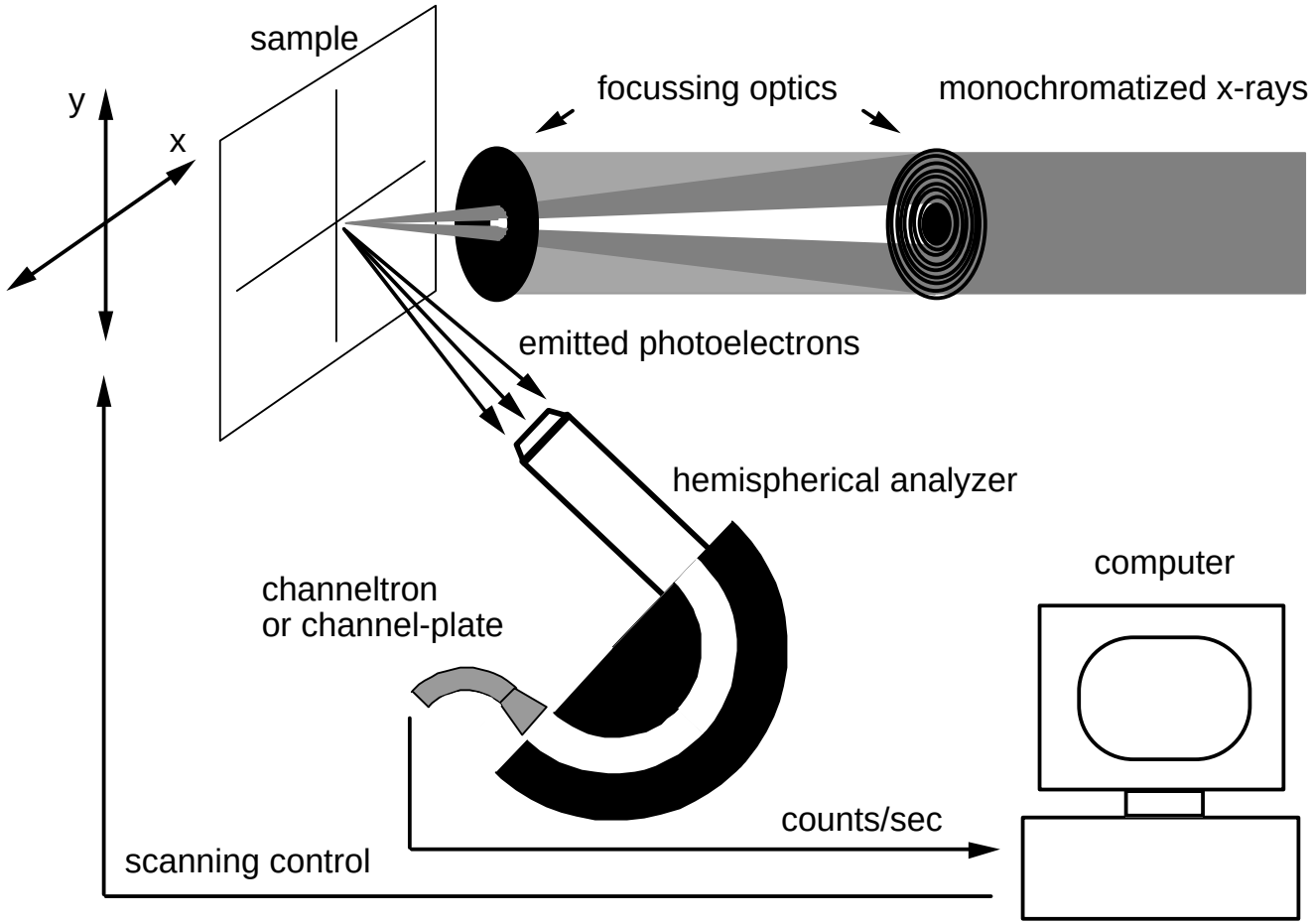
Scanning versus full field imaging transmission microscopy

e.g. at the ESRF

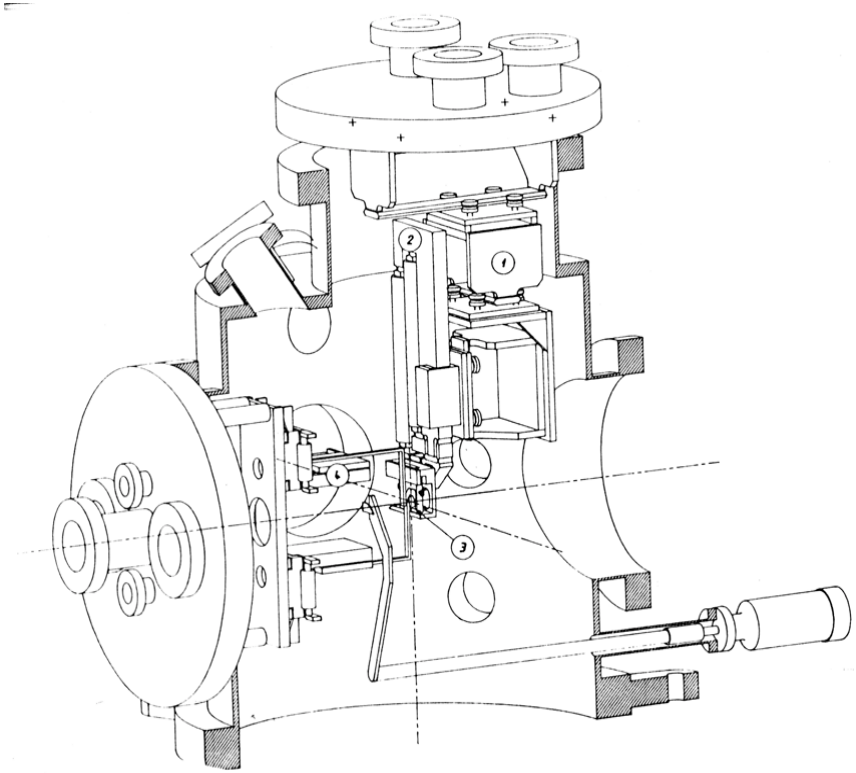


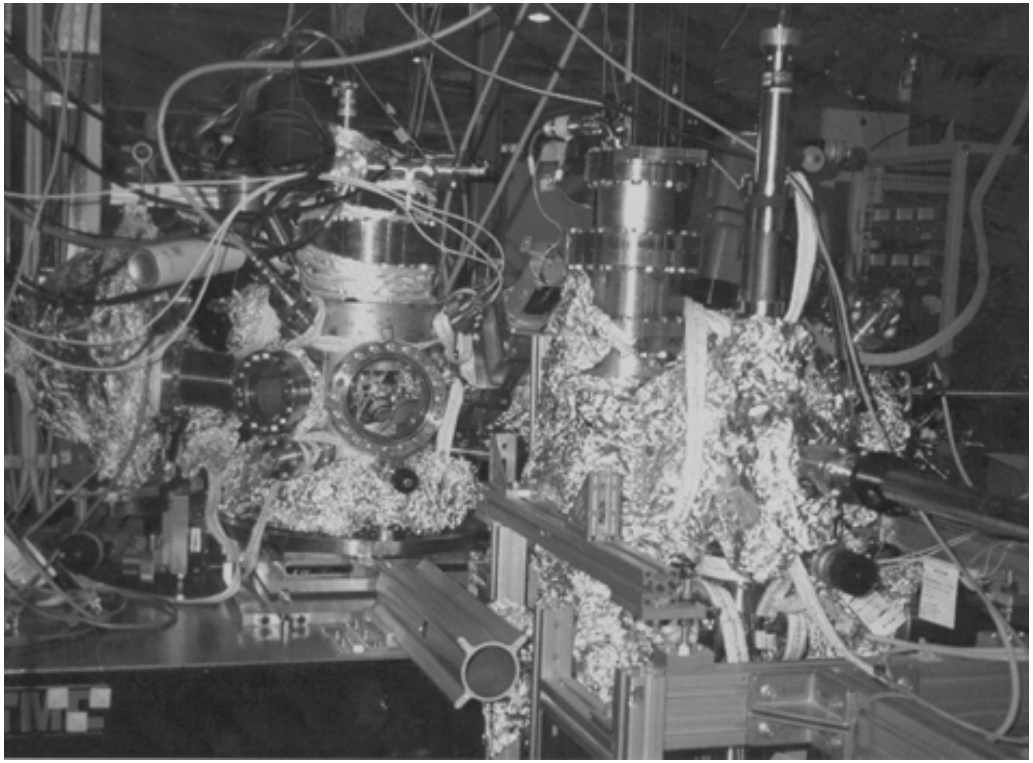


Scanning Photoemission Microscope

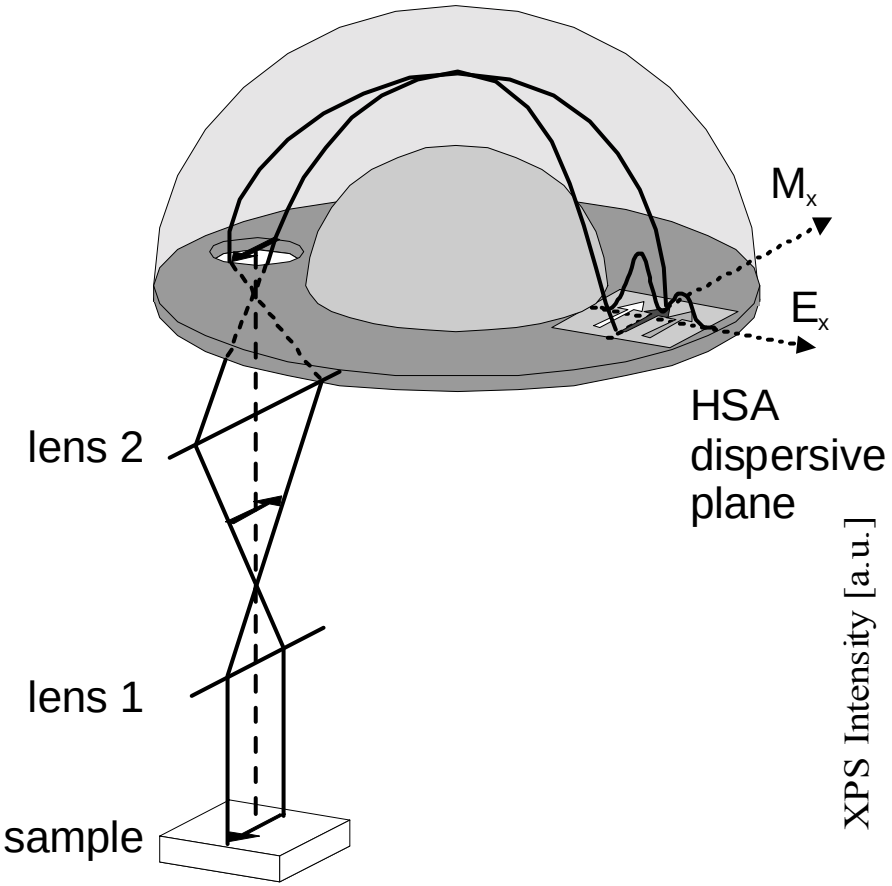


Scanning Photoemission Microscope at ELETTRA

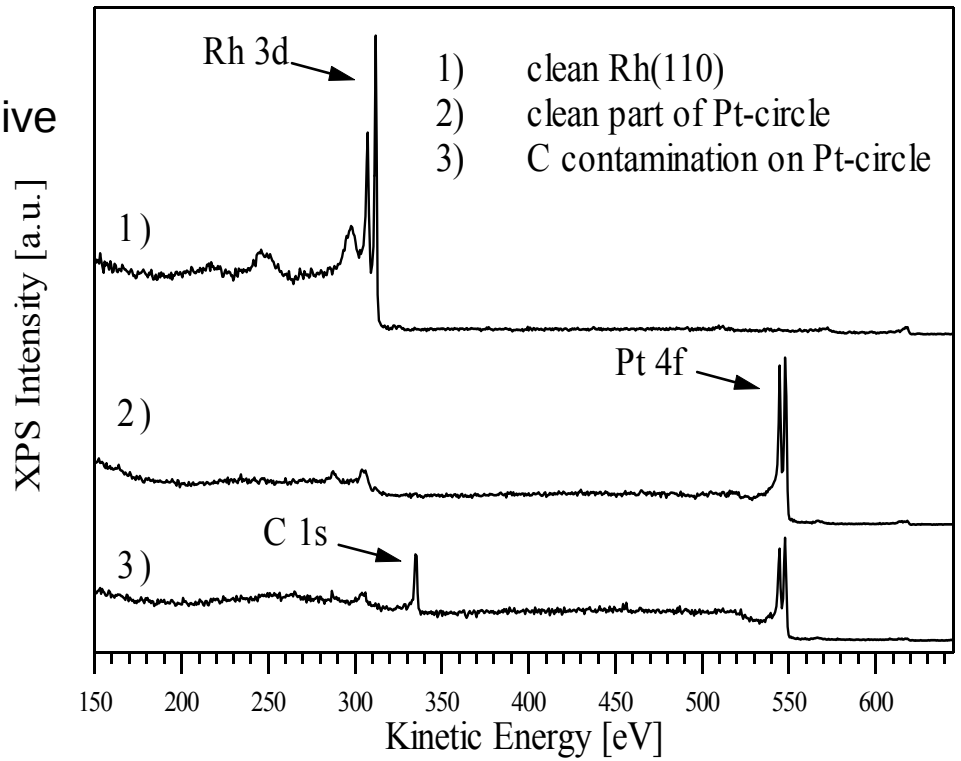




Scanning Photoemission Microscope at ELETTRA



Electron analyzer of the SPEM



SPEM: Multichannel-acquisition

Multi-channel acquisition

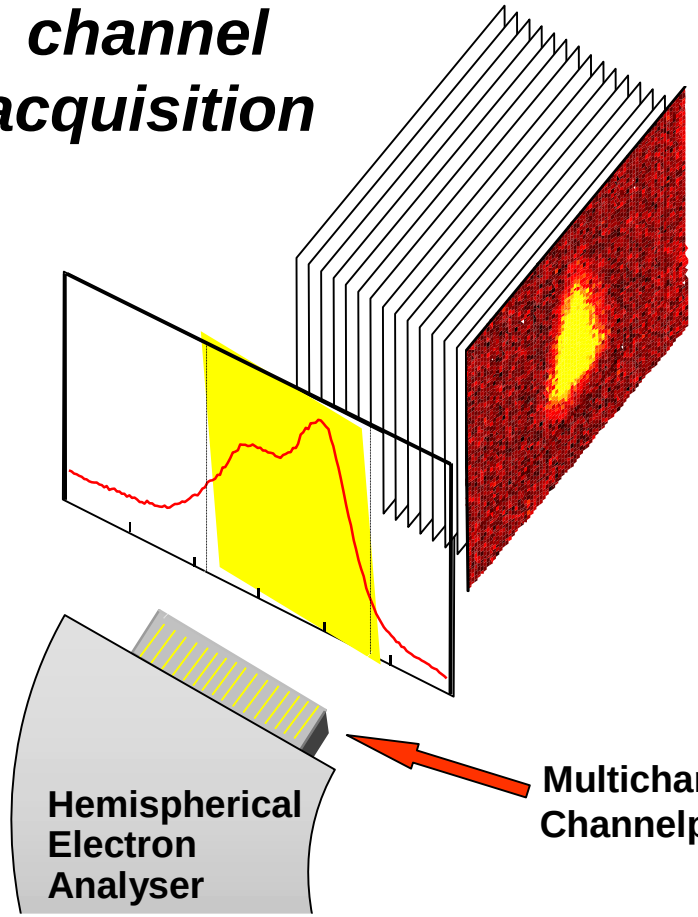
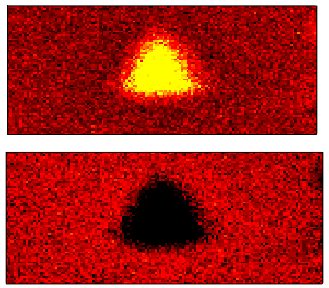
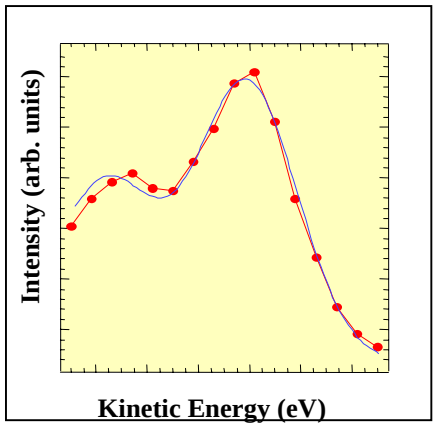


Image analysis

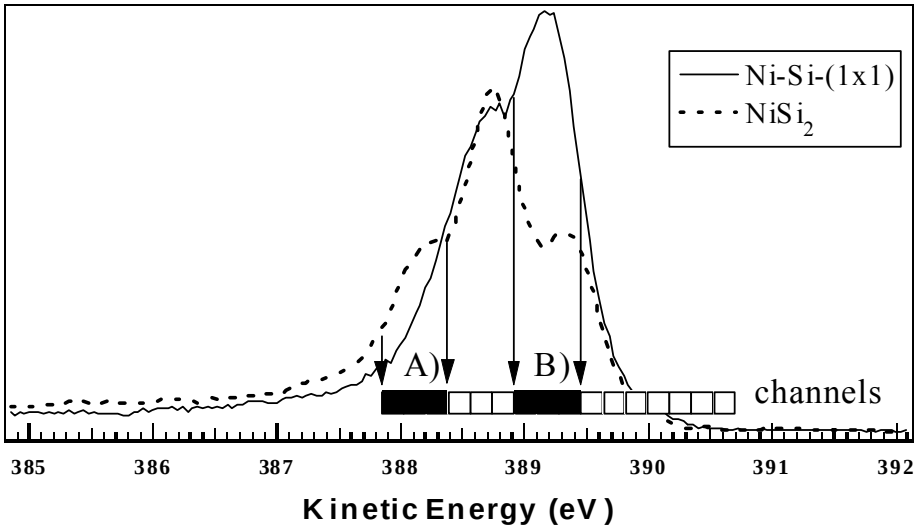


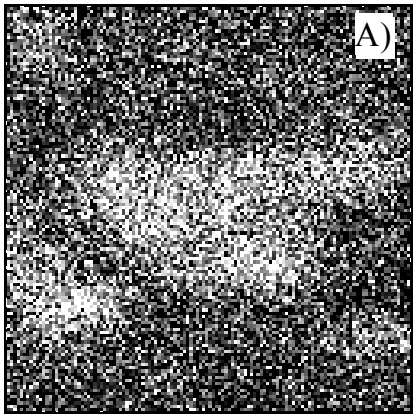
Single Channel Image

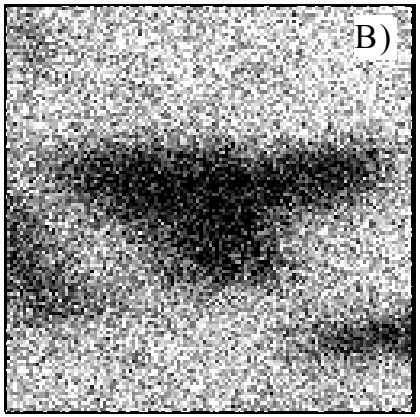


Spectra Imaging

SPEM: Multichannel-acquisition



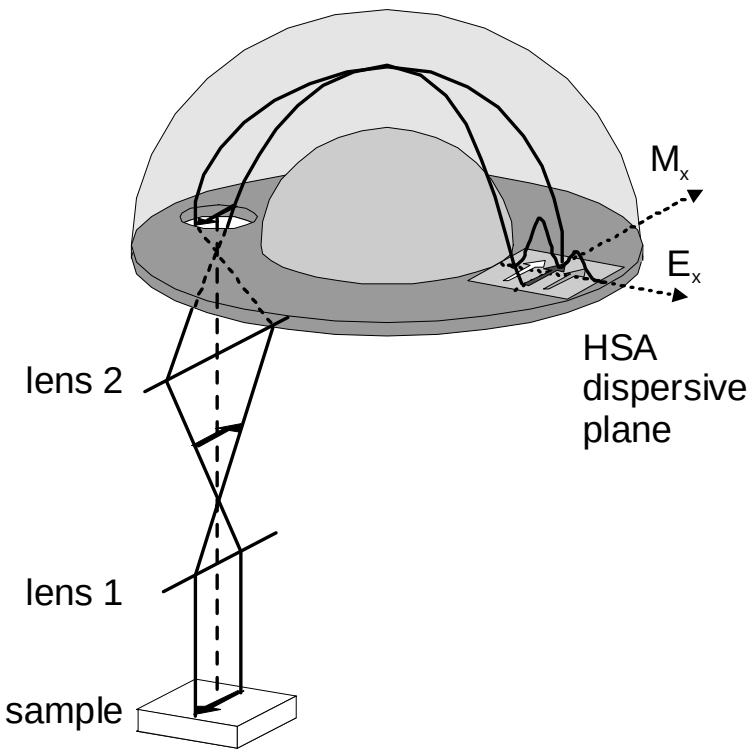




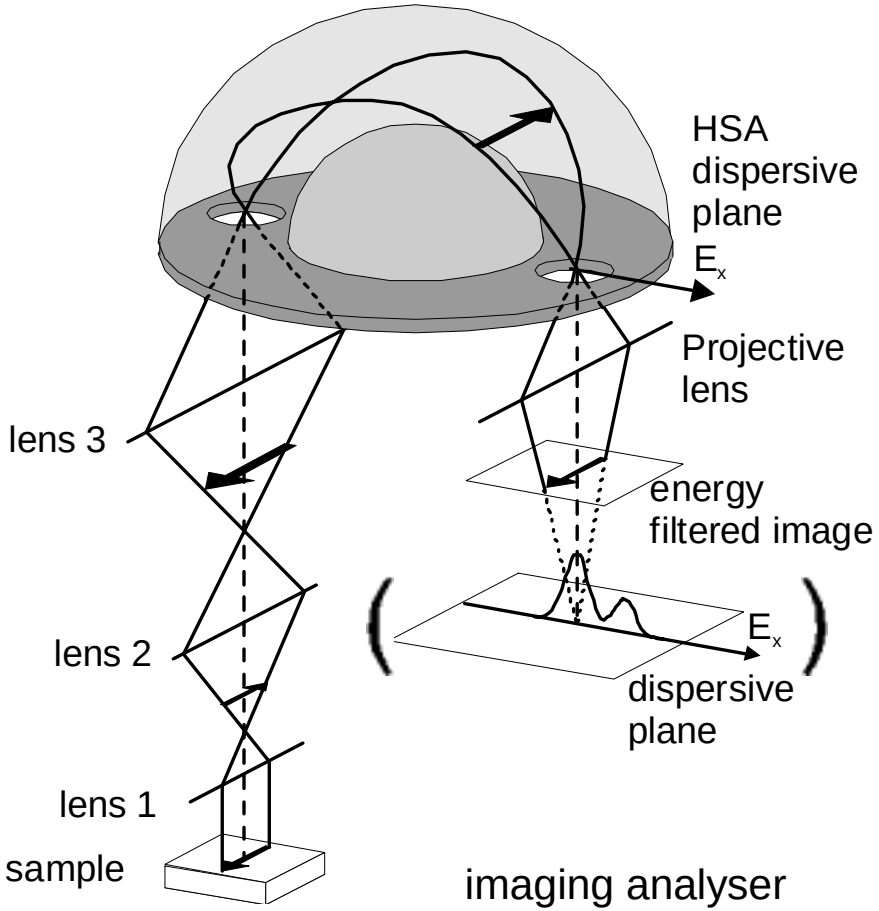
parallel recording of image set

imaging of chemical state

Electron imaging systems: Imaging XPS Analyser concept



non imaging analyser (as in SPEM)



imaging analyser

homogeneous illumination + scanning => imaging