Spectromicroscopy

An introduction to instrumentation and some experimental examples

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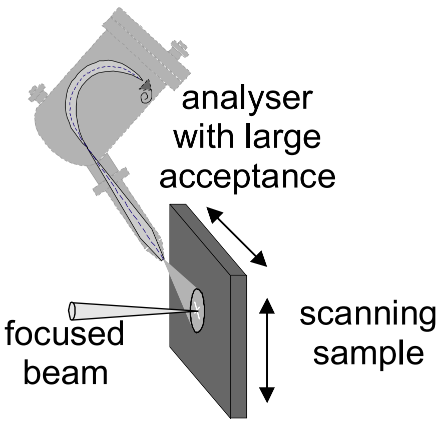
MAX-lab, Lund University

Spectromicroscopy = Spectral and spatial information

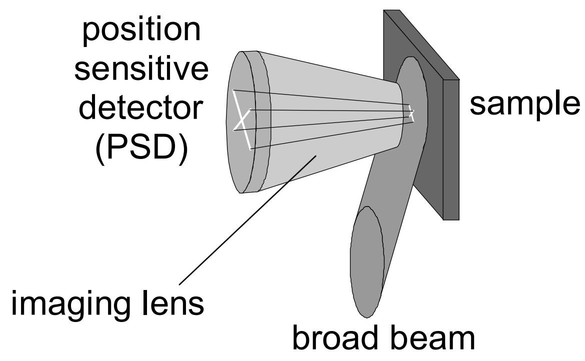
Spectral information from small areas of the sample
and
Image contrast obtained from different spectral features

Two ways to obtain both spectra and images:

Sequential or scanning
"Microspectroscopy"



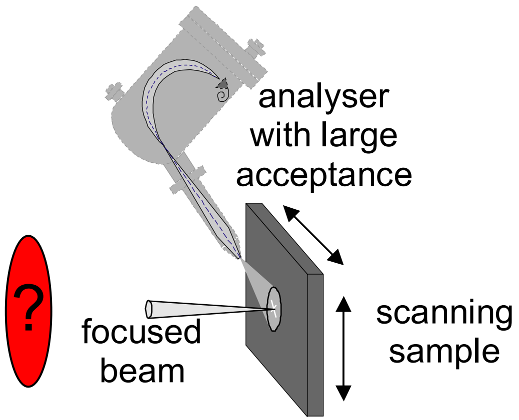
Parallel or imaging
"Spectromicroscopy"



Focusing optics

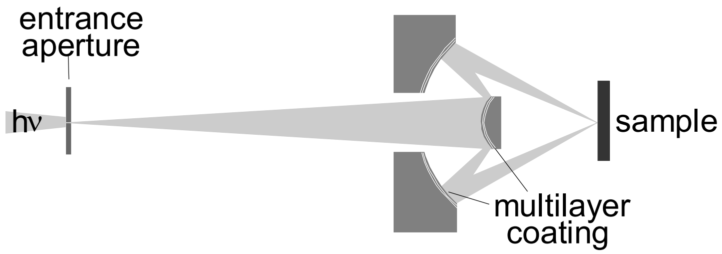
- Reflection with mirrors and diffraction with zone plates are most often used for focusing of VUV- and Soft X-ray radiation.
- Refraction in lenses can’t be used for VUV and Soft X-rays due to strong absorption and low refractive index.

Sequential or scanning
“Microspectroscopy”

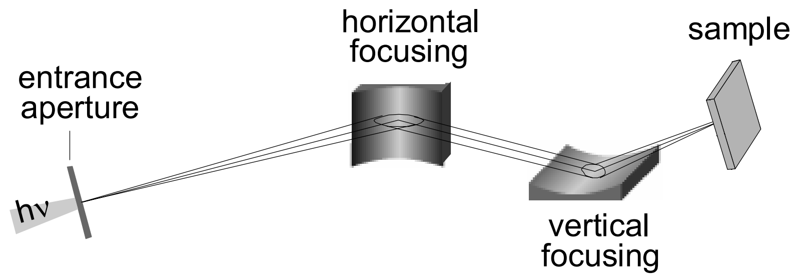


Imaging by focusing optics (mirrors)

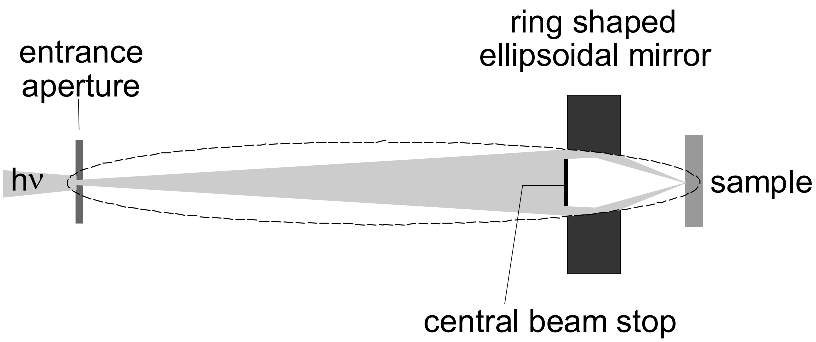
Schwarzschild



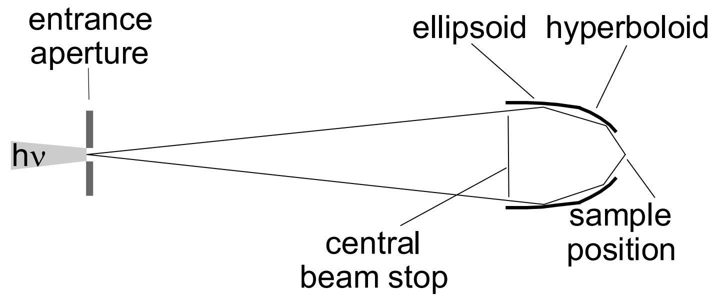
Kirkpatrick-Baez



Ellipsoid

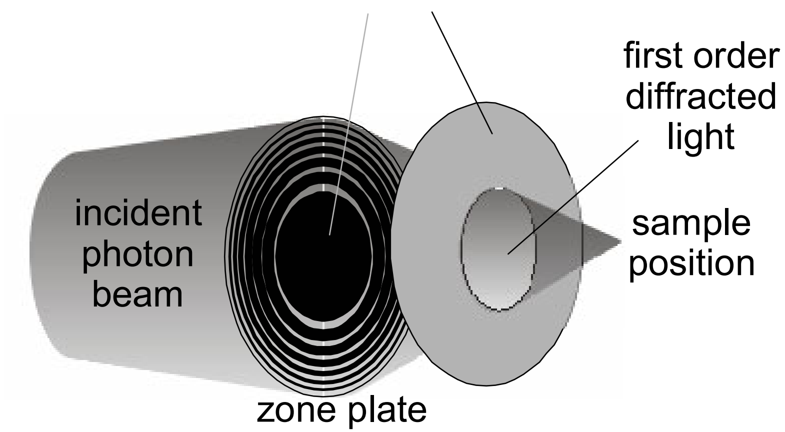


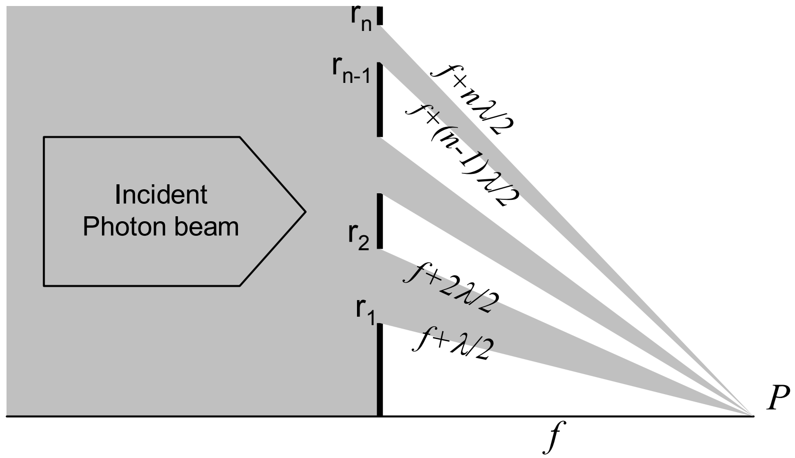
Wollter



MAX-lab

Imaging by focusing with a Zone Plate (ZP)





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

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

Due to a small numerical aperture

$$f >> \frac{n\lambda}{2} \Rightarrow r_n \approx \sqrt{n\lambda f}$$

See “Soft X-rays and Extreme Ultraviolet Radiation” David Attwood, Cambridge University Press, 1997

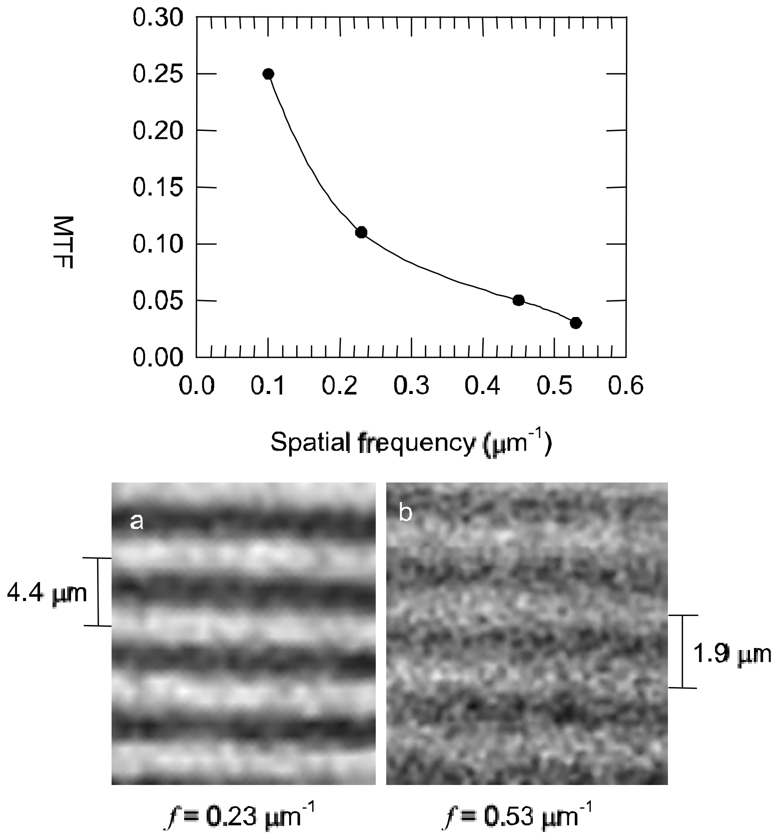
Properties

- Efficiency ~10 % in first order diffraction for soft X-rays
- Focal spot determined by the outermost zone width.
  - ~20 nm focal spot size has been achieved.
- Diameter ~30 µm to mm range, depending on application
- Focal distance varies with photon energy
  - Sample and zone plate must be moved during photon energy scan
- One or a few millimetres working distance

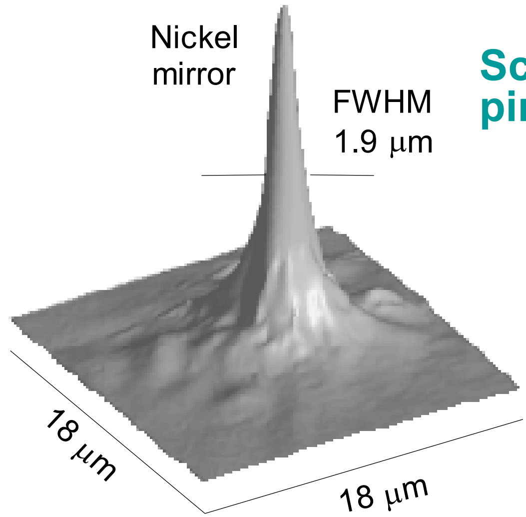
Spatial resolution measurements

Examples from the MAX-lab SPEM

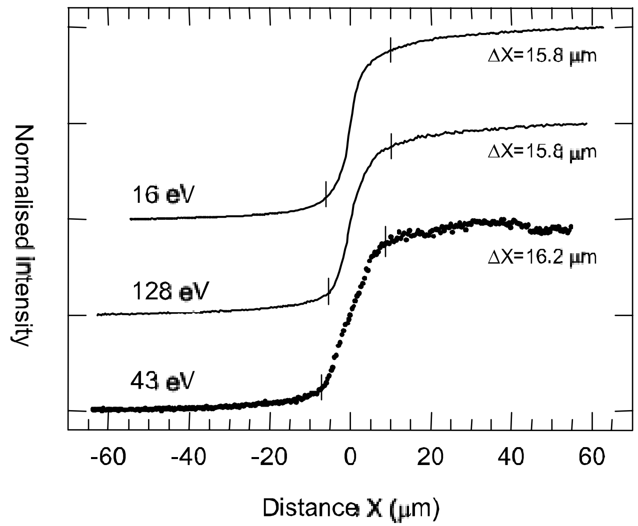
Scanning periodic structure



Scanning a pinhole

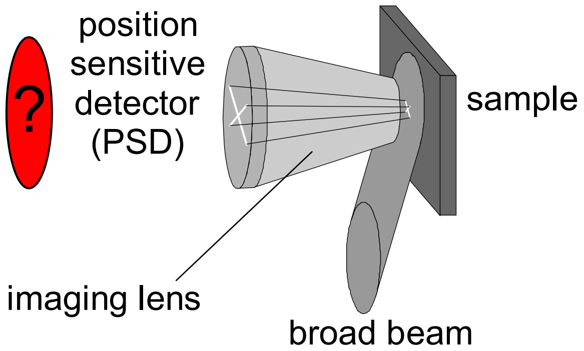


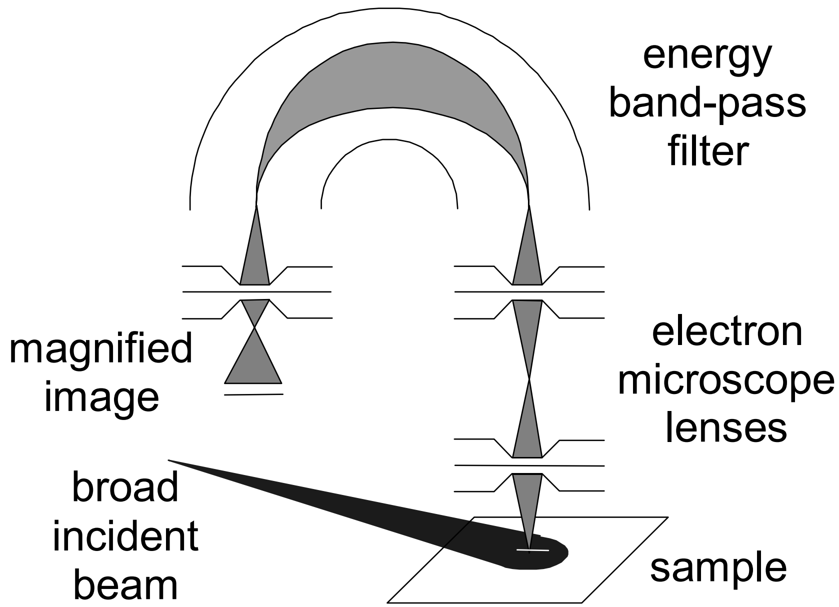
Scanning knife edge



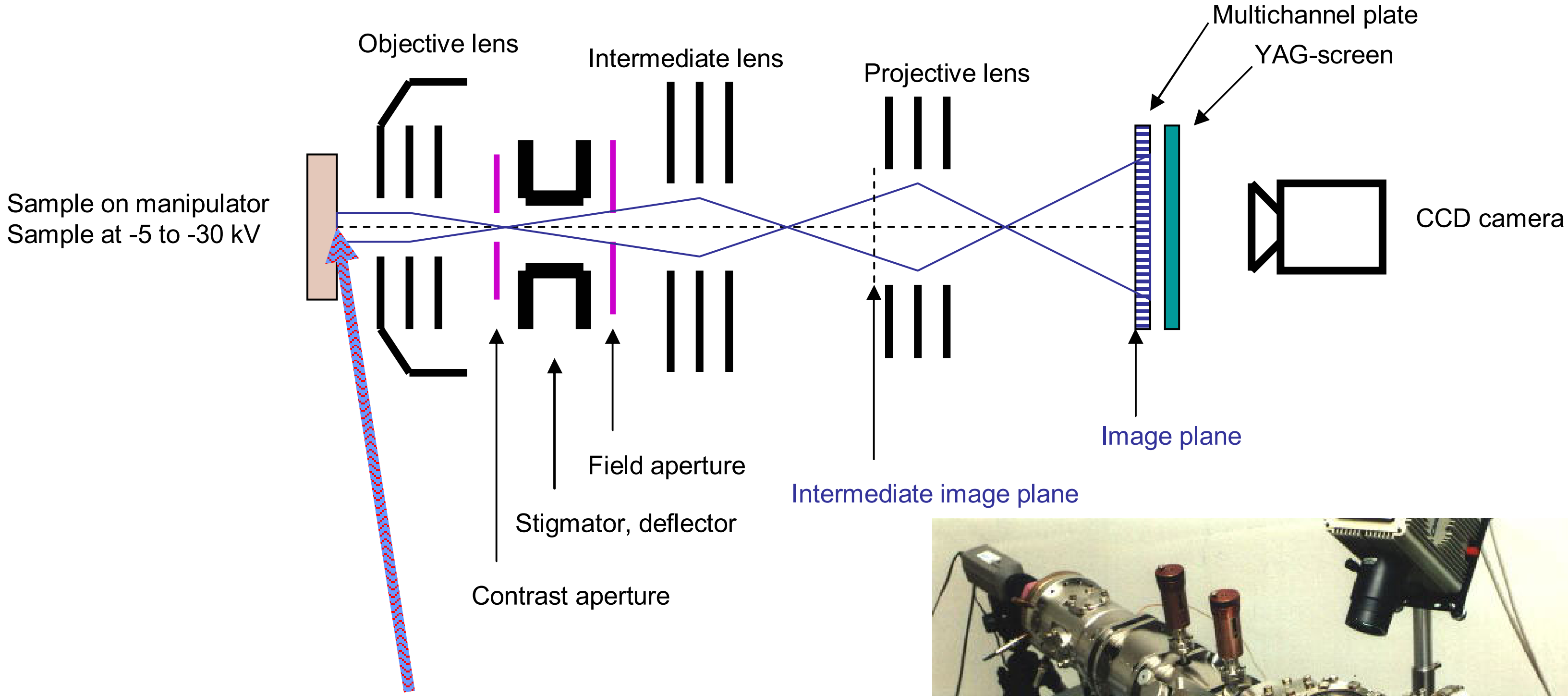
Imaging by projection

Parallel or imaging
"Spectromicroscopy"





Photoemission Electron Microscope (PEEM)

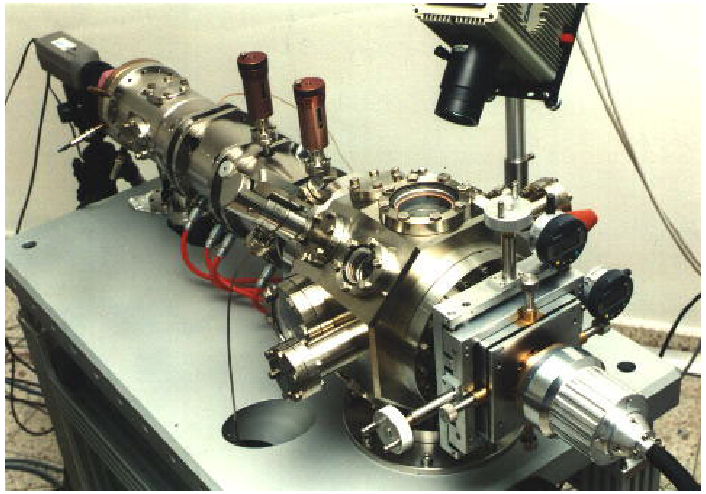


Photons

•Synchrotron radiation (XPEEM)

•UV lamp

•Mercury lamp, etc

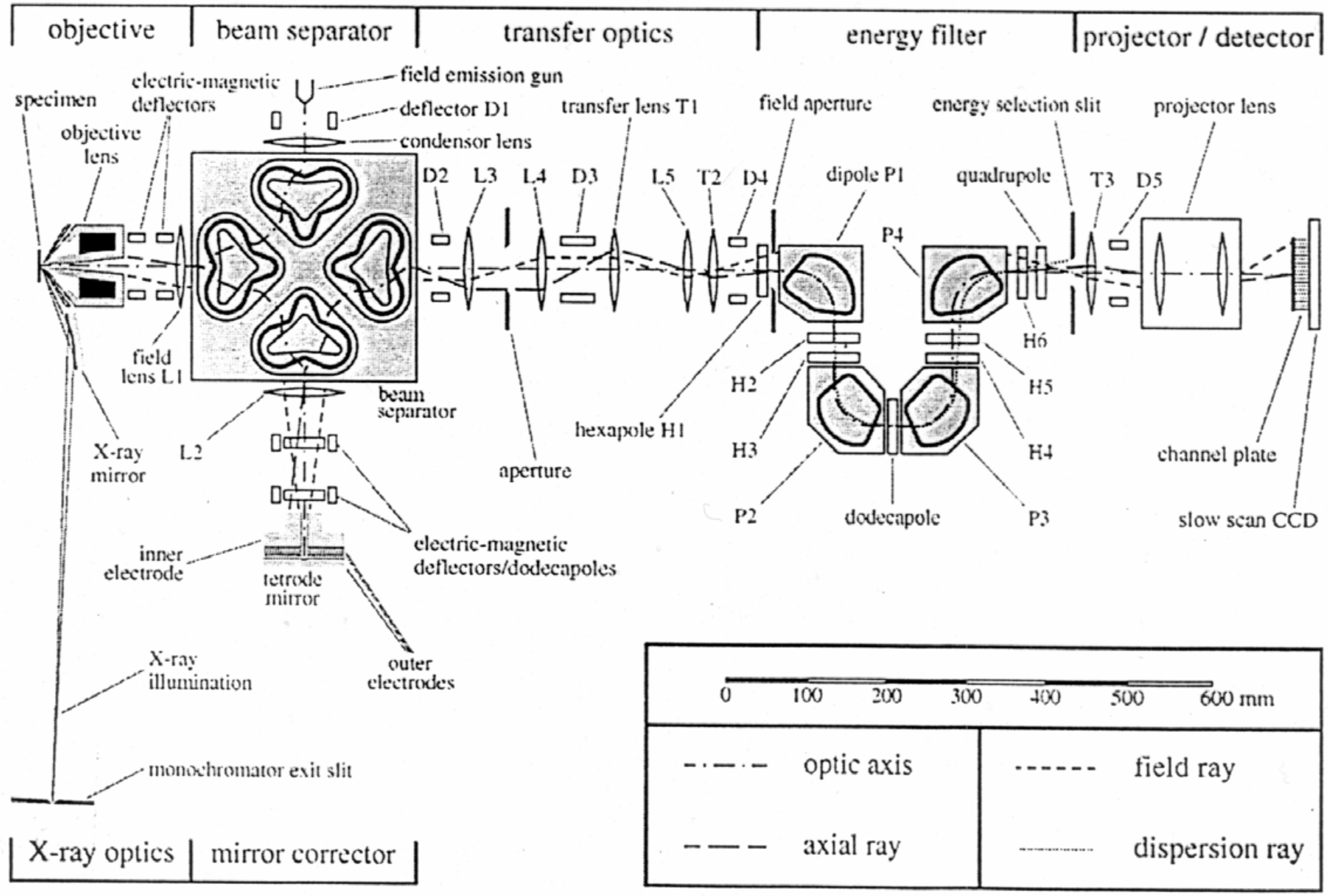


Elmitec PEEM. Ref:http://www.elmitec-gmbh.de

PEEM properties

- Very high spatial resolution
  - better than 20 nm with photoelectrons and secondary electrons. This depends strongly on the energy spread of the emitted photoelectrons.
- Easy change of magnification, field of view 2 - 150 μm
- Requires flat and conducting samples
- Gracing incidence photon beam can lead to shadow effects
- Needs homogeneous illumination
- Can be equipped with electron energy band-pass filter

Spectro Microscope for All Relevant Techniques – The SMART instrument at Bessy II



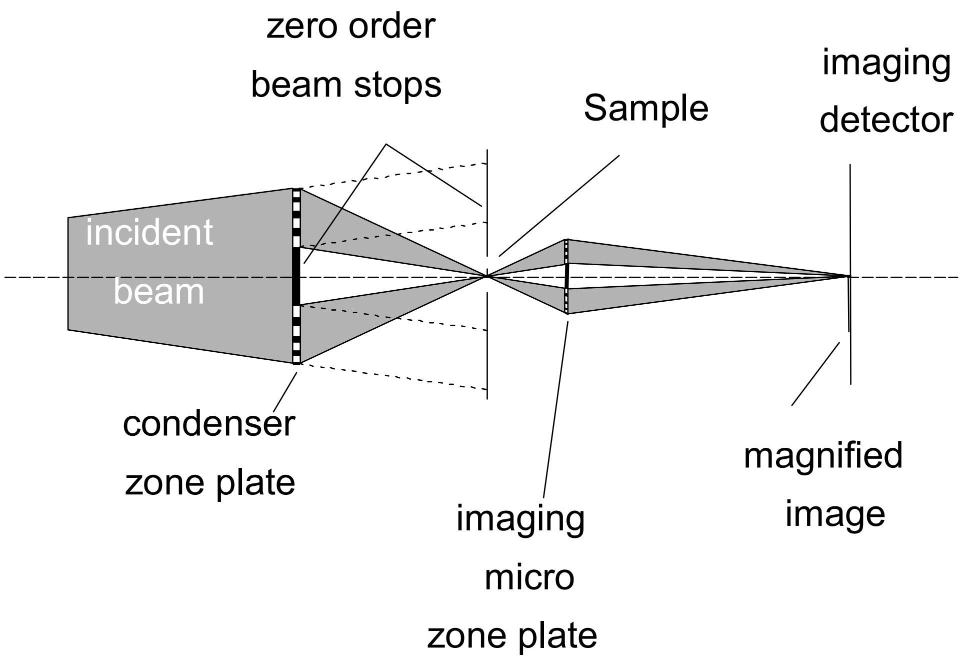
E. Bauer / Journal of Electron Spectroscopy and Related Phenomena 114–116 (2001) 975–987

Some examples from different microscopes

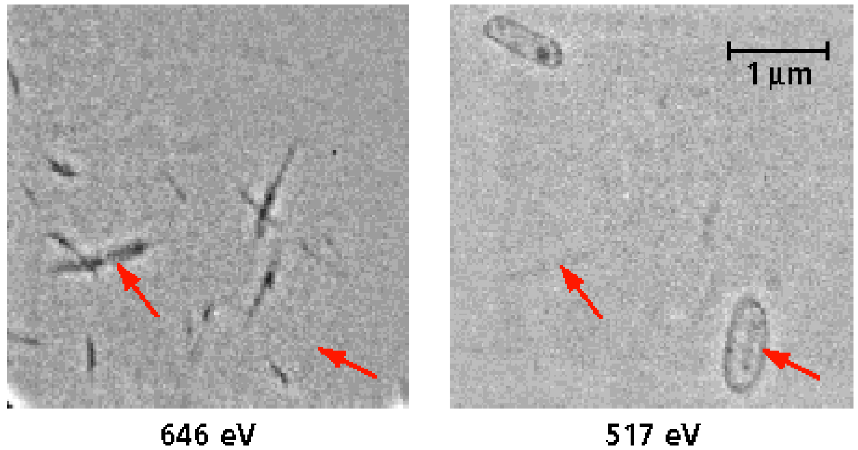
- **Imaging X-ray microscope using zone plates**
- **Scanning transmission X-ray microscope using zone plates**
- **Scanning photoelectron microscope using an ellipsoidal mirror**
- **X-ray fluorescence and μ-XANES microscope using Kirkpatrick-Baez objective**
- **Photoemission Electron Microscope (PEEM)**

X-ray imaging microscope

With λ between 40 Å (C K-edge) to 23 Å (O K-edge) wet samples can be investigated (The so called "Water window")



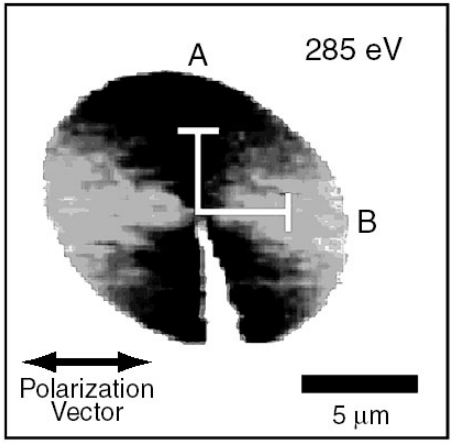
Manganese-Eating Microbes

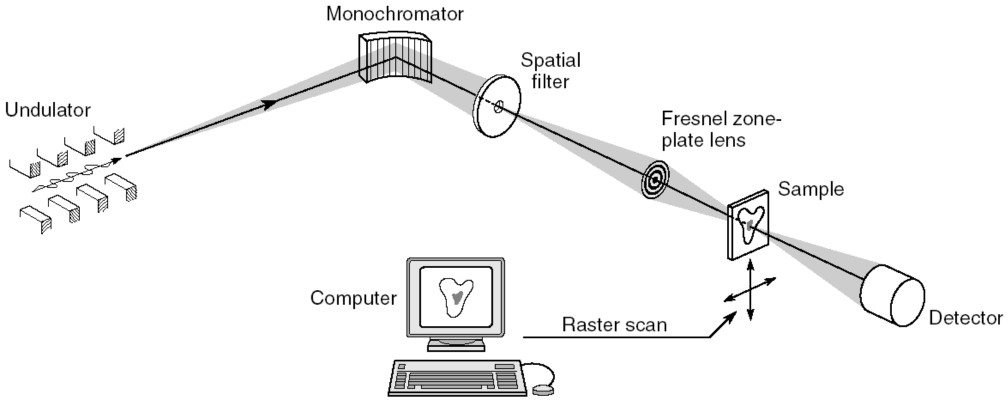


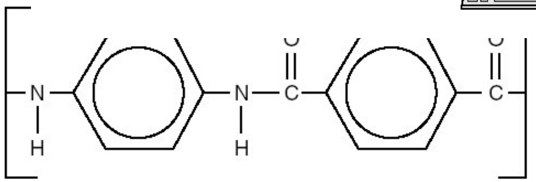
By B.P. Tonner (principal investigator) and K. Nealson (University of Wisconsin-Milwaukee), and W. Meyer-Ilse and J. Brown (Berkeley Lab's Center for X-Ray Optics), using the XM-1 microscope at Beamline 6.1.2. ALS.

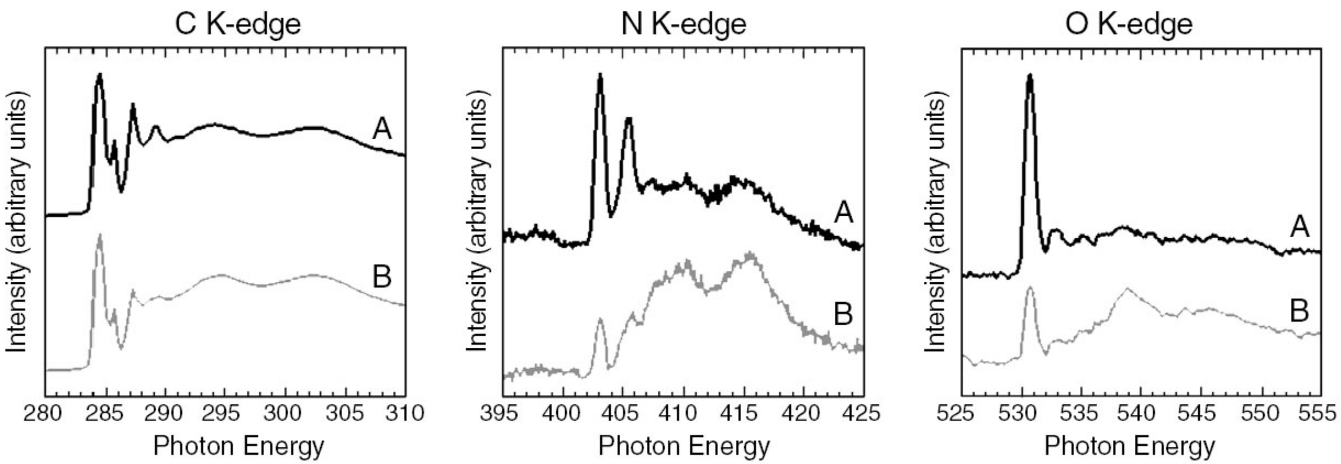
Scanning Transmission X-ray Microscopy

Cross-section of Kevlar fibre





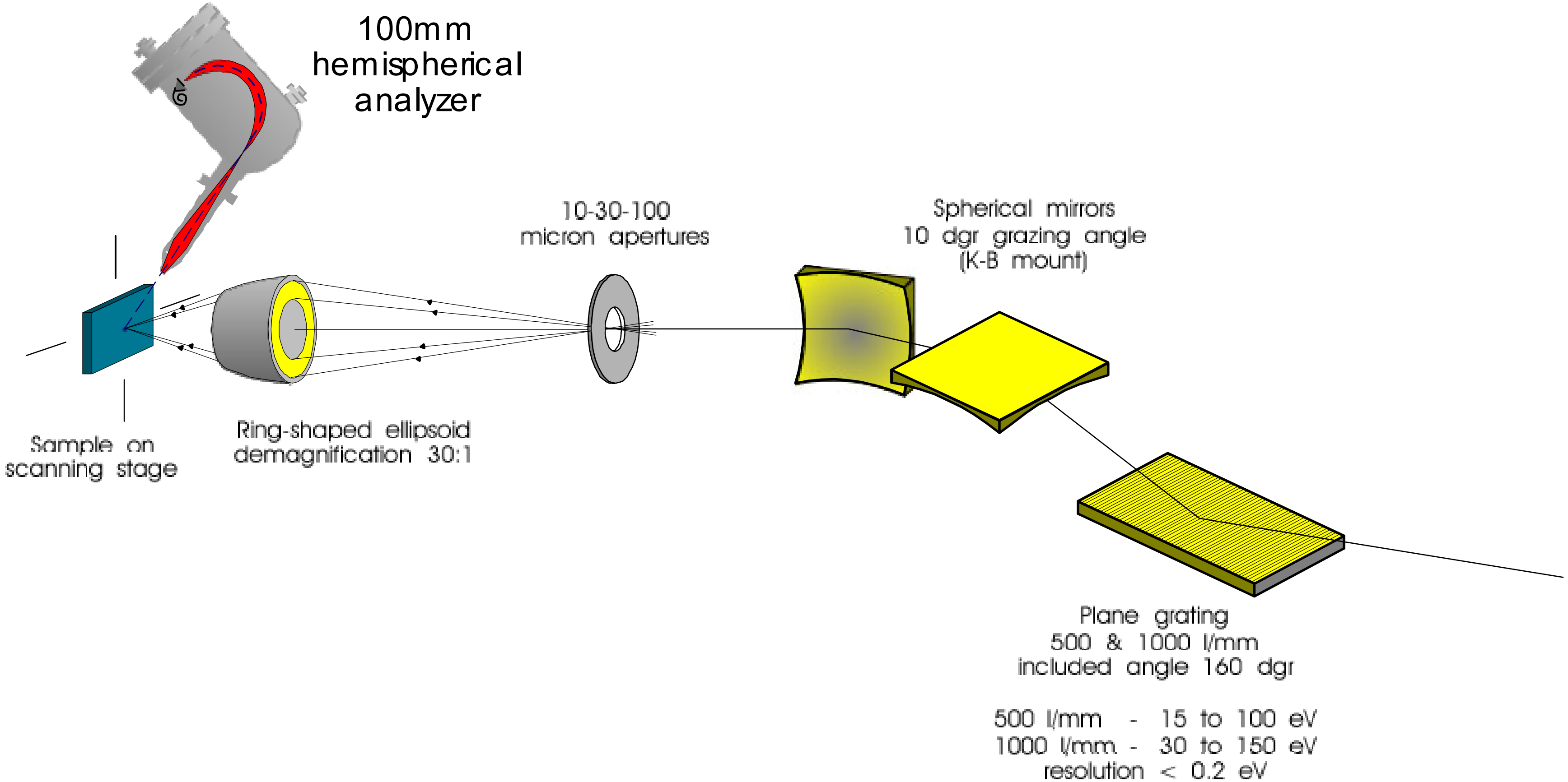




The polarization dependence (linear dichroism) shows the orientation of the polymer chains.

From:ALS, beamline 7.0.1, Data courtesy of H. Ade and A. Garcia (North Carolina State University).

The VUV Scanning Photoelectron Microscope (SPEM) at MAX-lab



MAX I

MAX II

MAX III

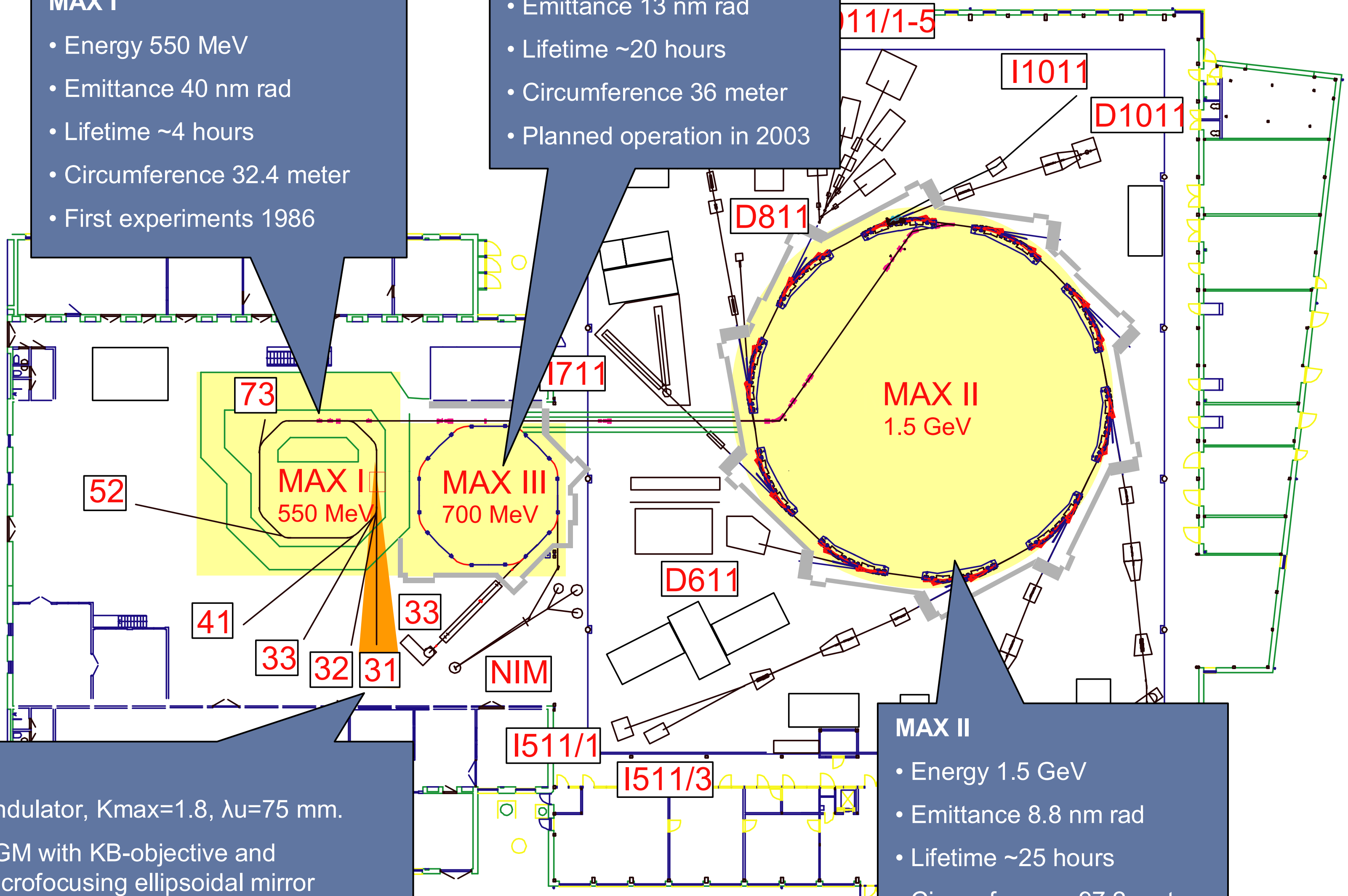
31

MAX I

- Energy 550 MeV
- Emittance 40 nm rad
- Lifetime ~4 hours
- Circumference 32.4 meter
- First experiments 1986

MAX III

- Energy 700 MeV
- Emittance 13 nm rad
- Lifetime ~20 hours
- Circumference 36 meter
- Planned operation in 2003



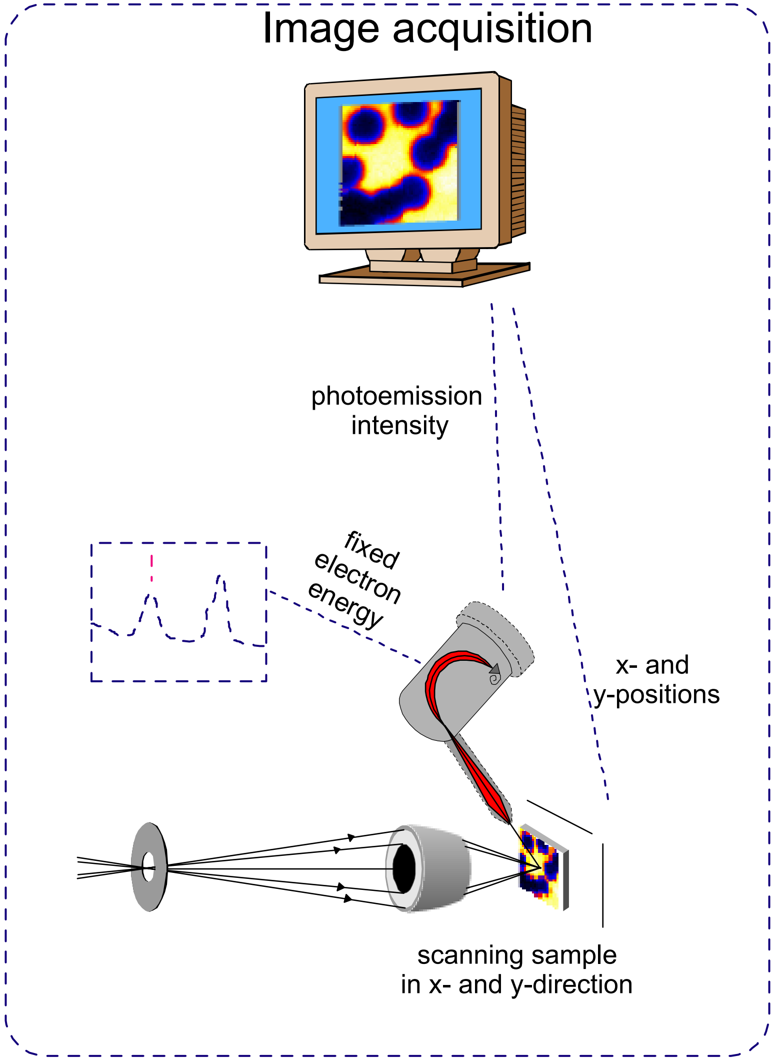
MAX II

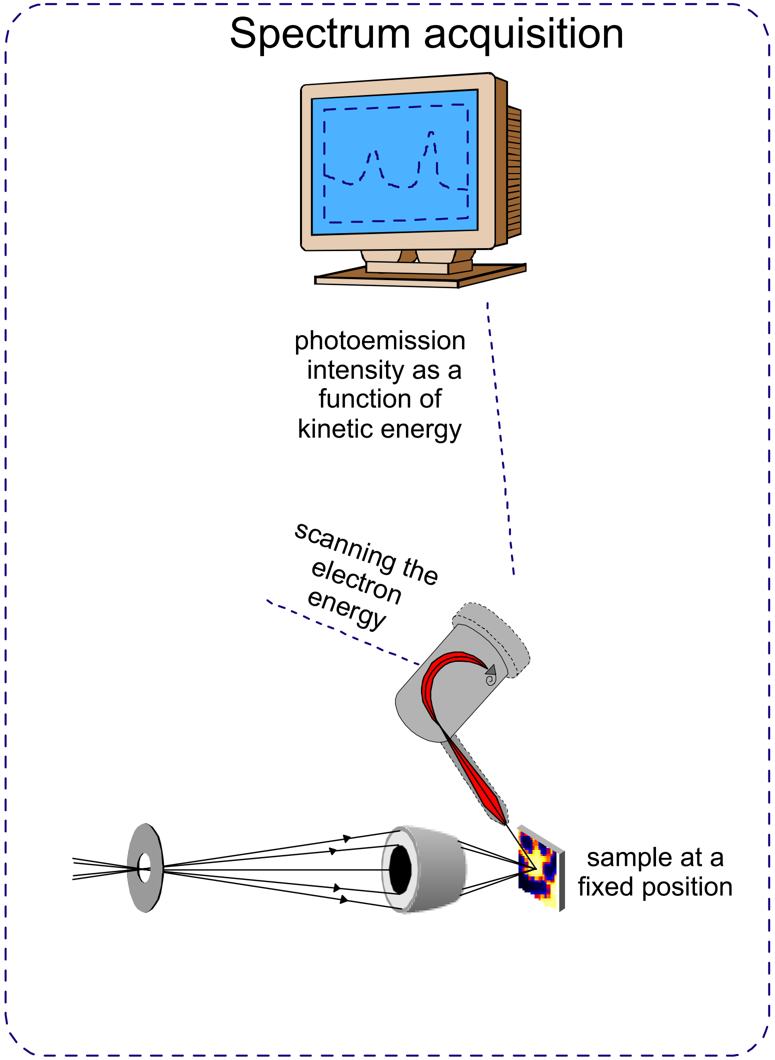
- Energy 1.5 GeV
- Emittance 8.8 nm rad
- Lifetime ~25 hours
- Circumference 97.2 meter
- First experiments 1997

31

- Undulator, Kmax=1.8, λu=75 mm.
- PGM with KB-objective and microfocusing ellipsoidal mirror
- hν = 15-150 eV
- Scanning photoelectron microscopy

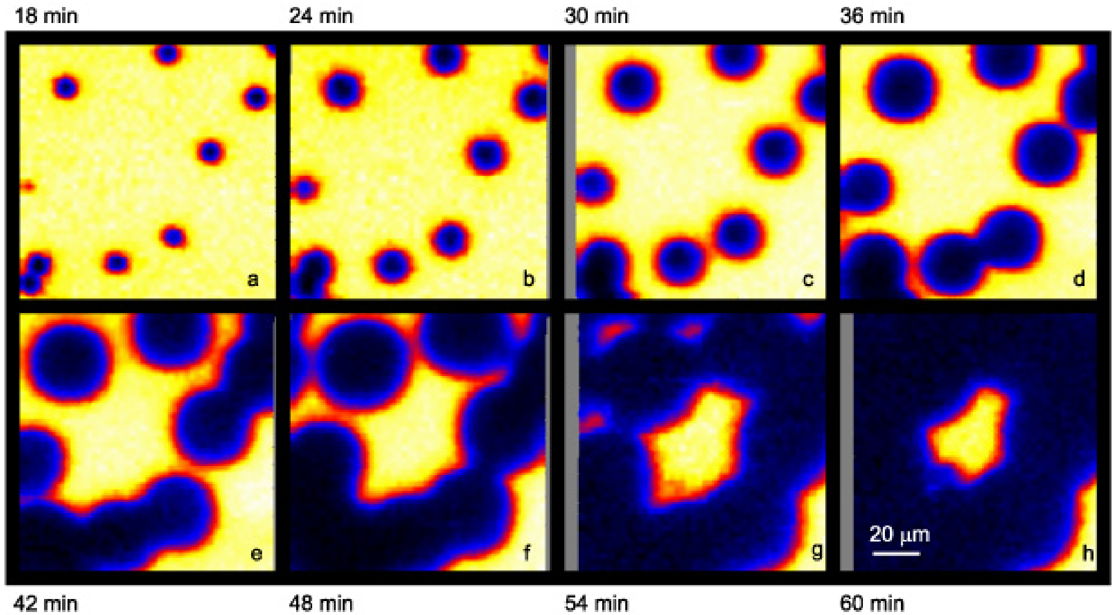
Data acquisition at the SPEM

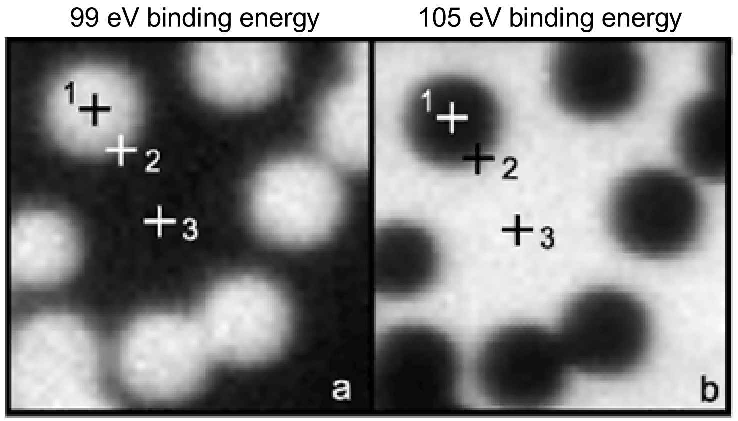


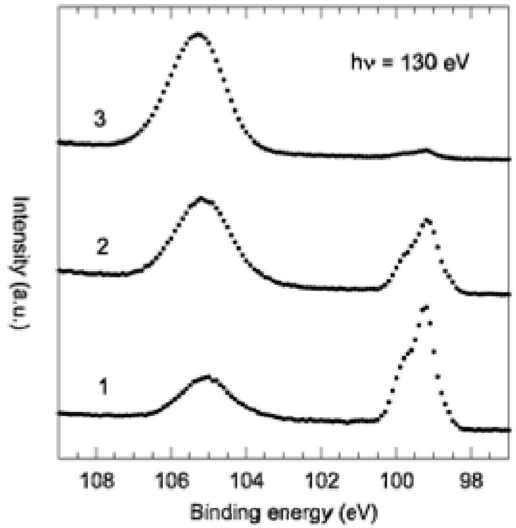


Photoelectron spectromicroscopy

Temperature induced void growth in SiO_2 overlayers on Si(100)



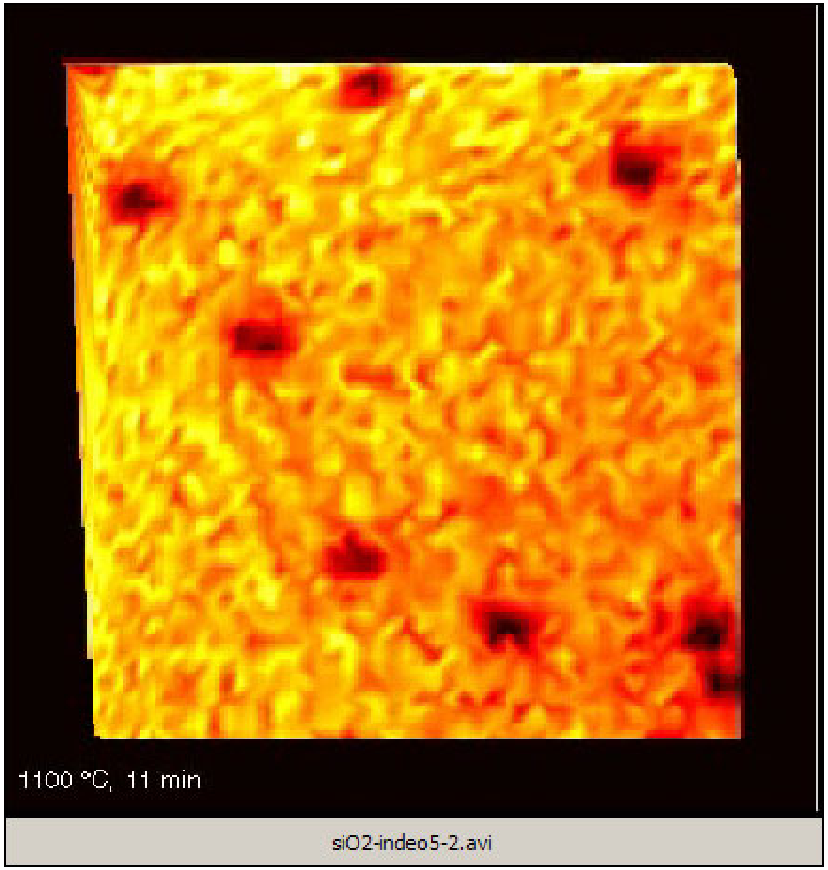




- Annealing temperature 1100°C
- Voids in the oxide layer grow with annealing time
- All voids are circular and of approximately the same size
- Yellow indicate SiO_2 rich areas, dark areas show Si from the substrate

U. Johansson, Thesis, Lund University, 1997

Void growth in SiO_2 overlayer on Si(100)



Video crated in MATLAB

Photon-induced phenomena

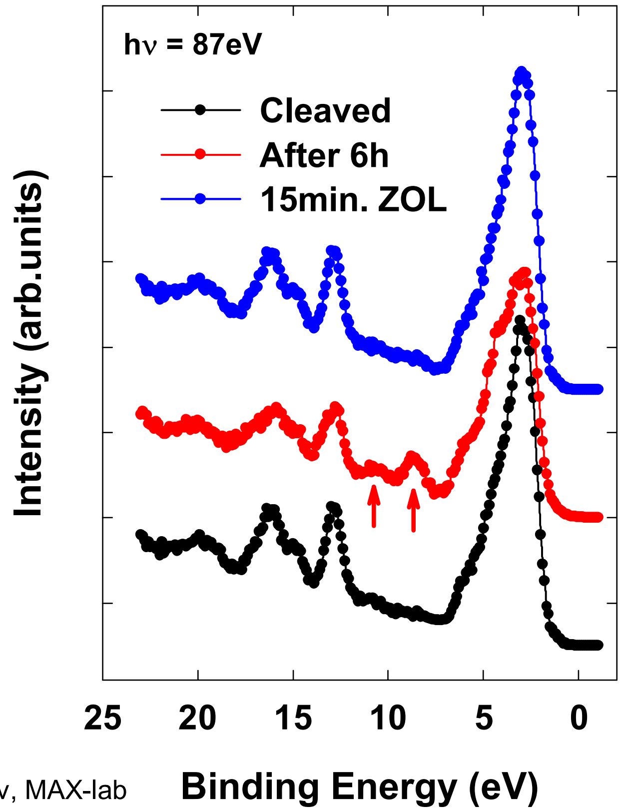
Beneficial

Deleterious

Flux: 10^9 - 10^{10} ph s^{-1} S = 1x1μm^2 Flux density: D = 10^{15} - 10^{16} ph $s^{-1}cm^{-2}$

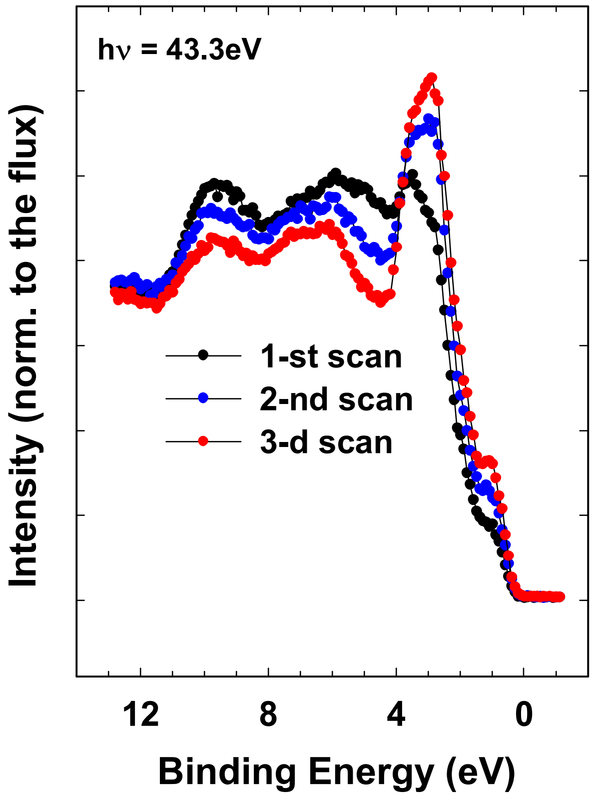
Surface cleaning

$Ba_{0.7}K_{0.3}BiO_3$



Beam damage

$Sr(Ca)CuO_2$

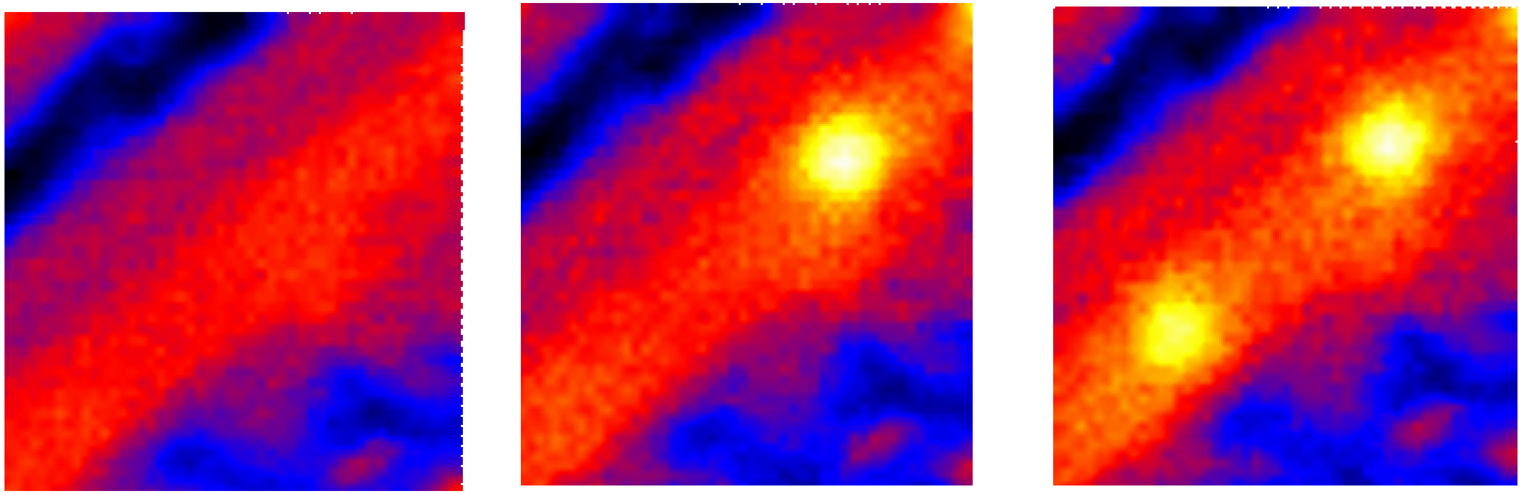


From A. Zakharov, MAX-lab

MAX-lab

Beam induced sample damage

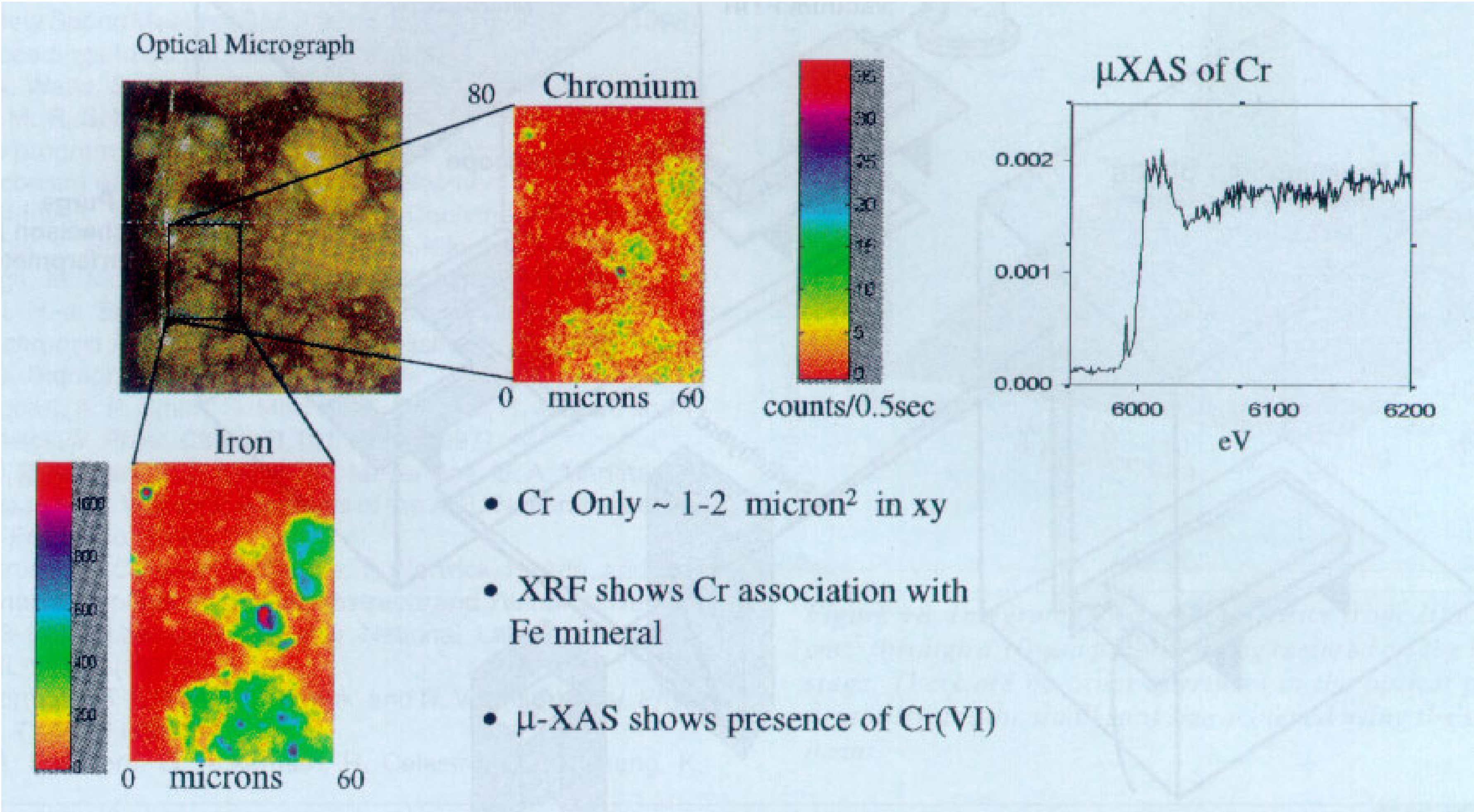
Ca(Sr)CuO_2 T_c=84K



120x120 μm^2 surface images of the same area, hν=43.18eV. The 20μm diameter bright spots are a result of beam-induced damage after just one scan (1min acquisition time) at each point.

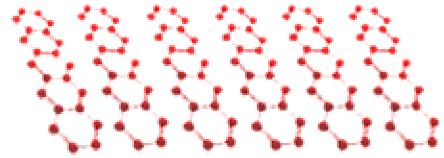
From A. Zakharov, MAX-lab

X-ray fluorescence and μ-XANES



From SRN, p. 19, Vol 14, N0. 4, 1998

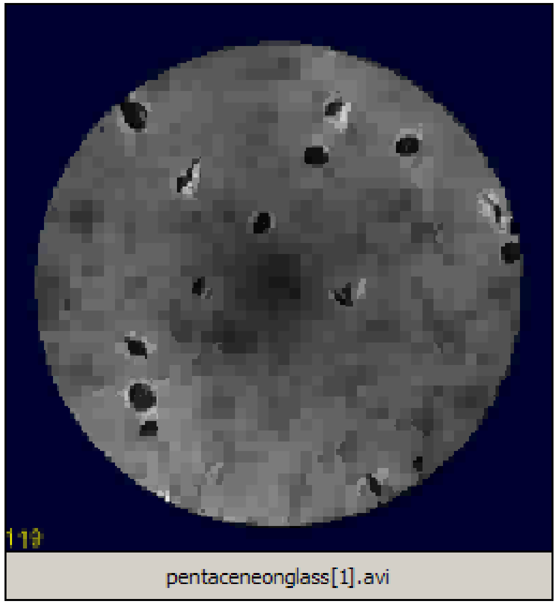
Pentacene growth on glas and Si(100)



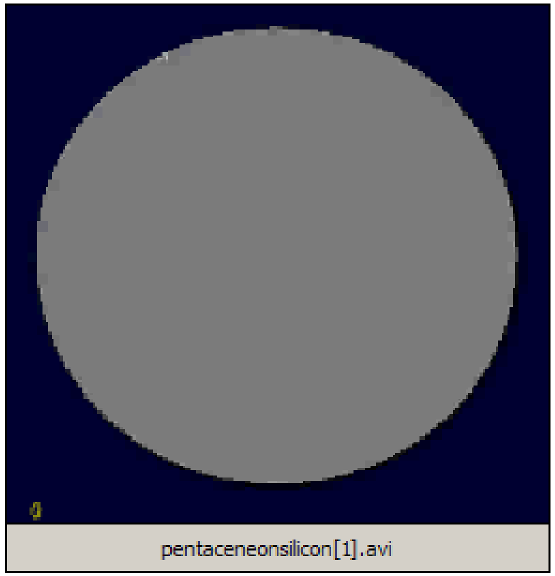
$C_{22}H_{14}$

- Experiments were done with a Merqury lamp
- Field of view 65 μm
- Images taken every minute

Pentacane growth on glass



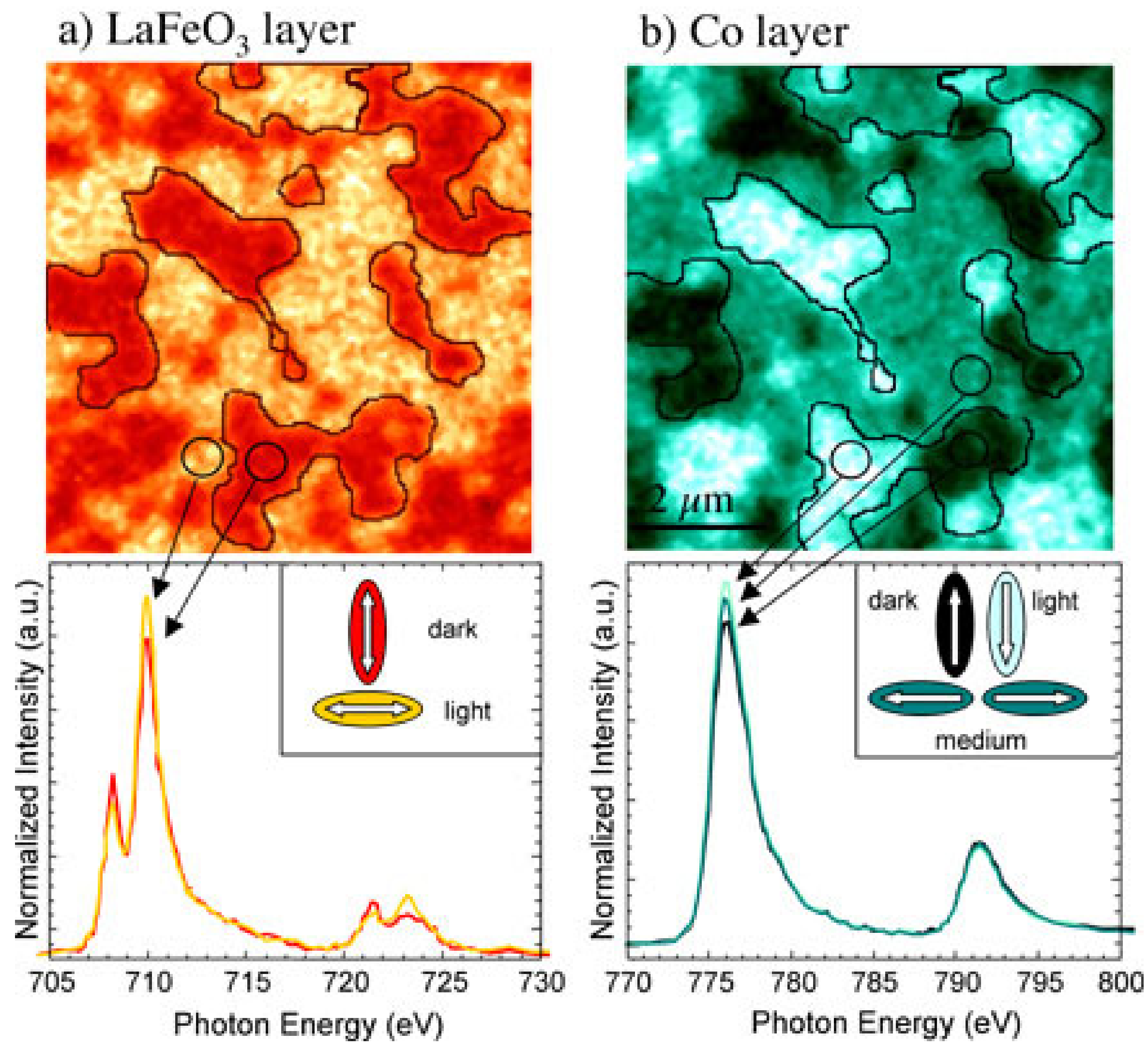
Pentacane growth on Si(100)



From: http://www.research –ibm.com

MAX-lab

Magnetic materials studied by Dichroism



Research conducted by F. Nolting, et al. ALS.

Fourier Transform Infrared Microscopy

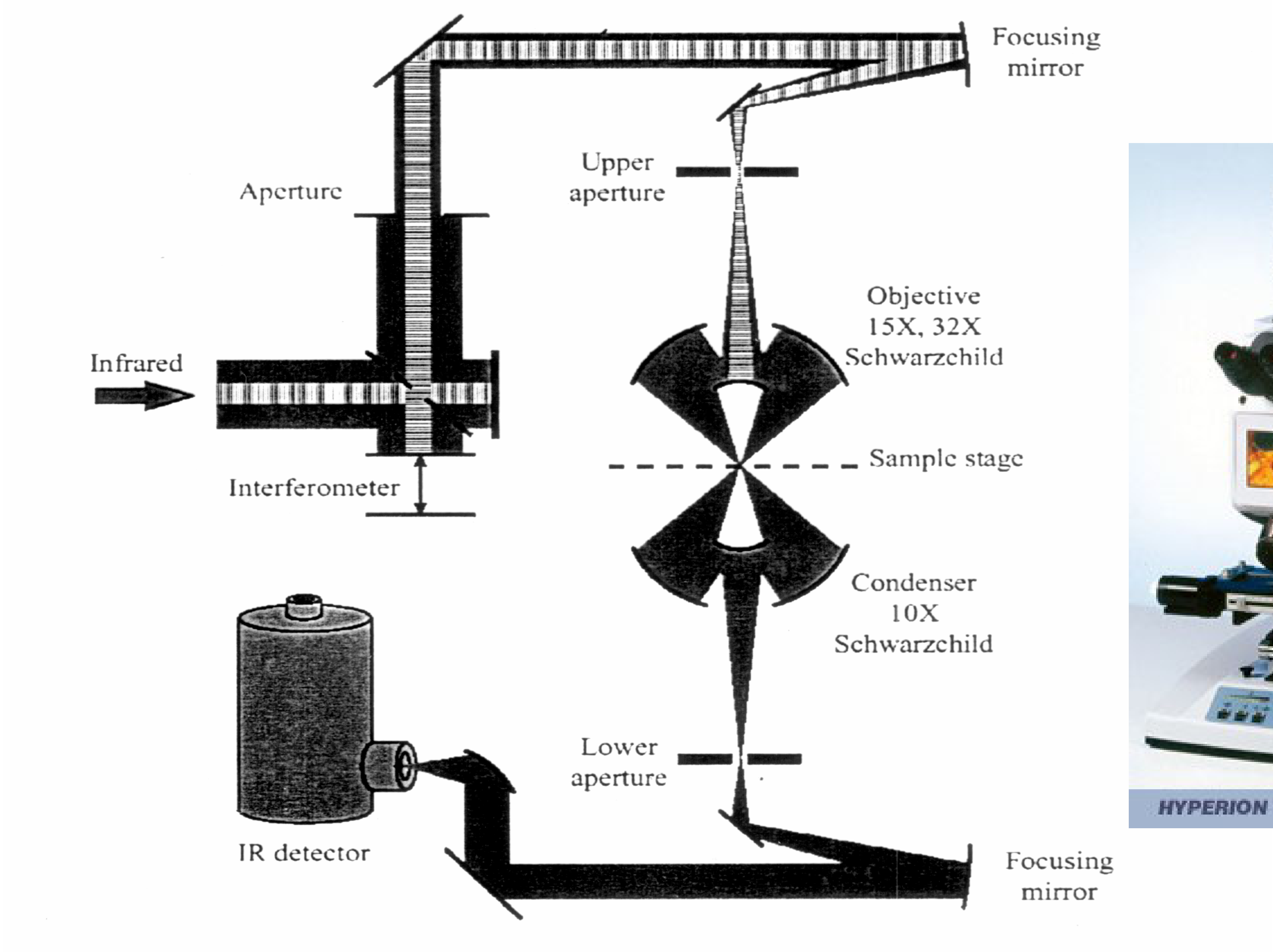
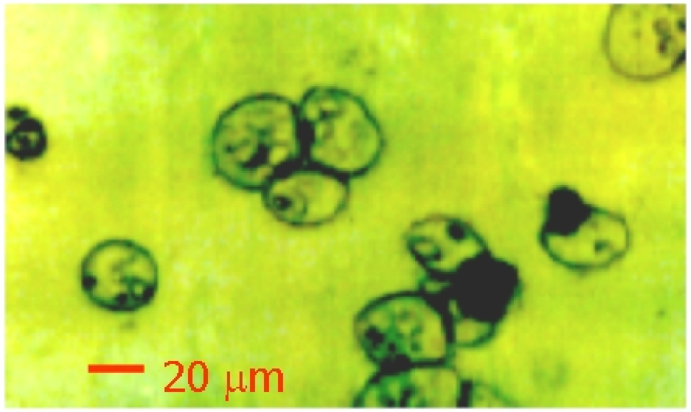
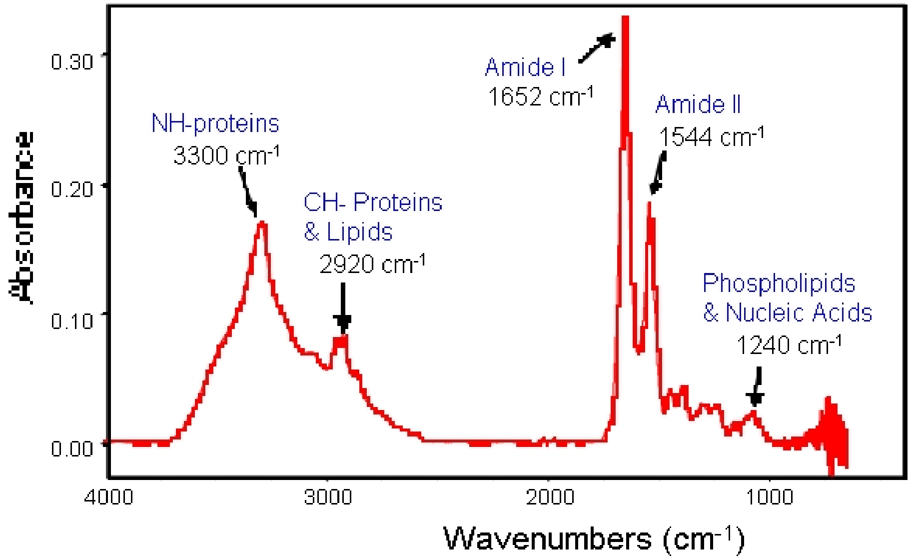
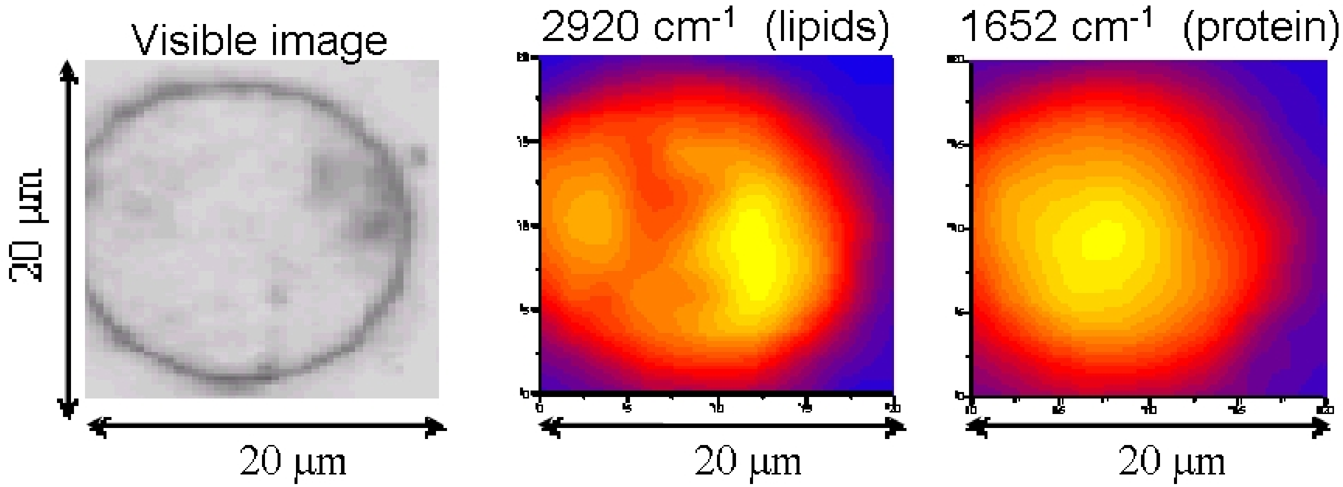


Image from G.L.Carr and G.P. Williams, in "Accelerator-Based Infrared Sources and Applications", SPIE Conf. Proc. Vol. 3135, p. 51, 1997

Chemical Imaging of Living Cells and Cellular Processes

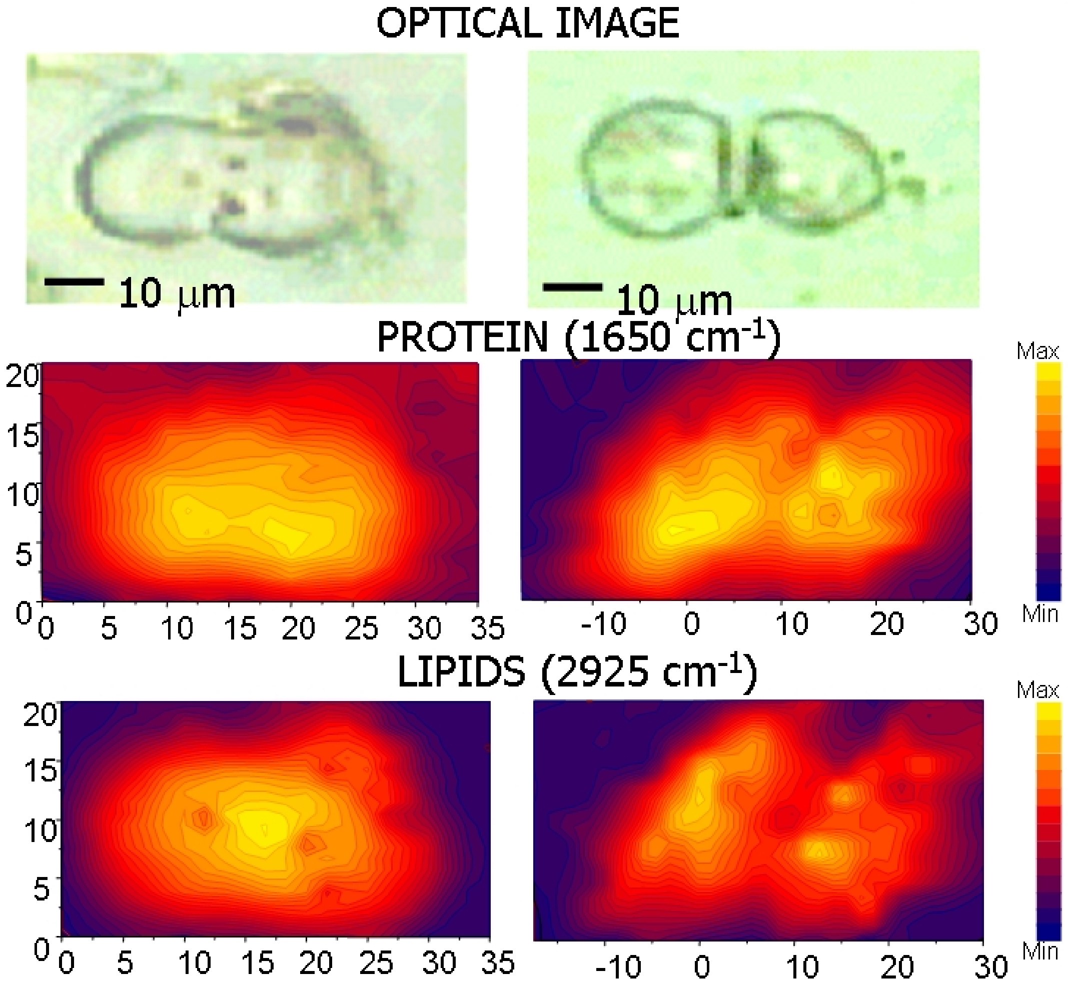






Chemical Imaging of Mitotic Cells

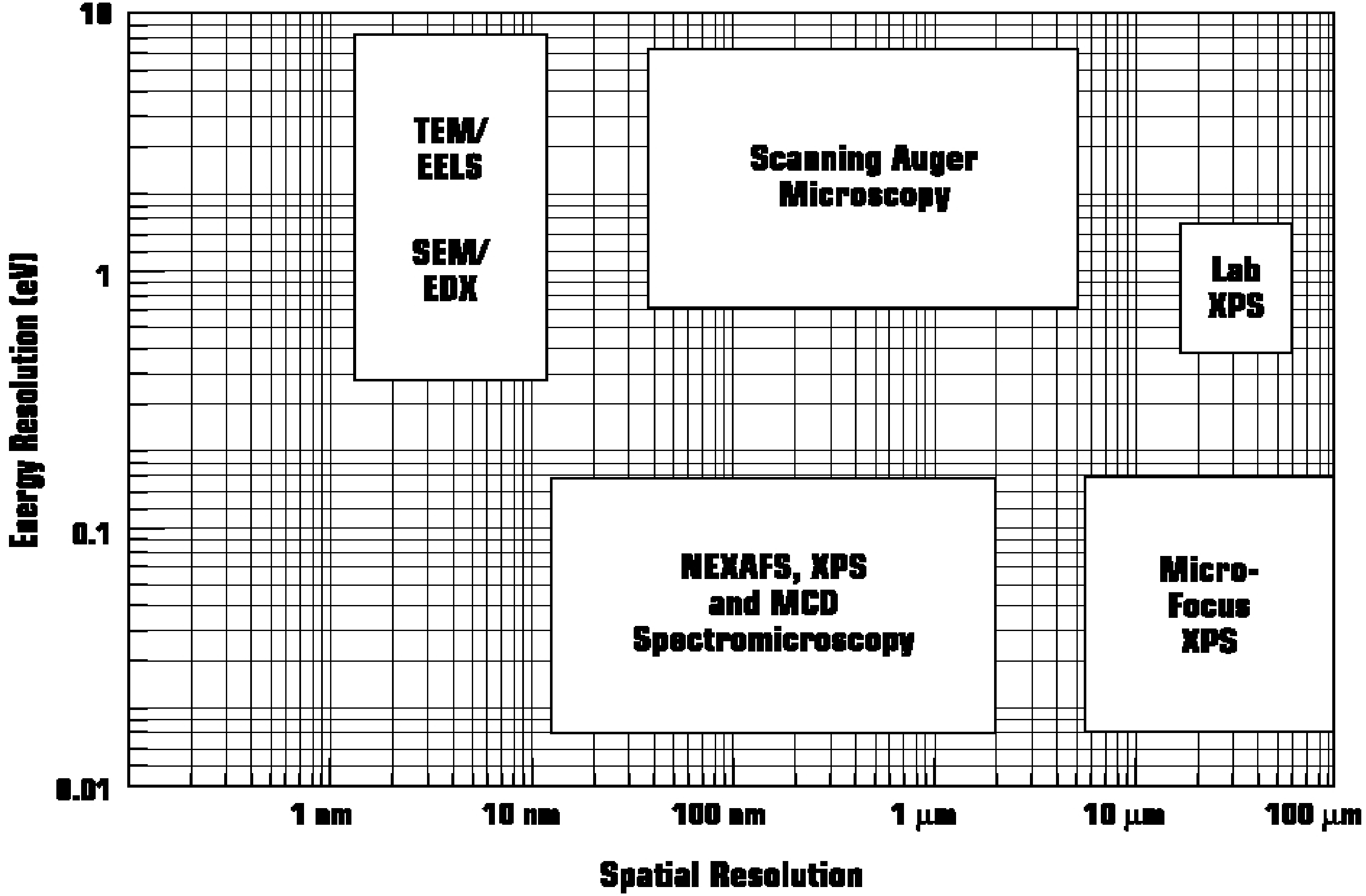
N. Jamin, P. Dumas (LURE), J.L. Teillaud (Institute Curie), G.L. Carr, G.P Williams (NSLS)



N. Jamin, P. Dumas, J. Moncuit, W.H. Fridman, J.-L. Teillaud, G.L. Carr and G.P. Williams, “Highly resolved Chemical Imaging of Living Cells by using Synchrotron Infrared Microspectrometry”, *Proceedings of the National Academy of Science*, **95** 4837 (1998).

Summary

- X-ray microscopy is a maturing field
  - Today a veriaty of spectroscopic methods are used
    - Photoemission spectroscopy
    - NEXAFS / XANES
    - Fluorescence
    - Linear and Circular Dichroism
  - Rapid development of focusing devices
    - Mirror systems
    - Zone plates
- IR-microscopy has found new applications
  - Diffraction limit reached due to the high brilliance of SR



Soft X-Ray Microscopy, PUB-786, ALS

Acknowledgments

Prof. Ralf Nyholm, MAX-lab
Dr. Alex Zakharov, MAX-lab