Detectors for Vacuum Ultra-Violet and Soft X-ray Science

Howard Padmore

Outline

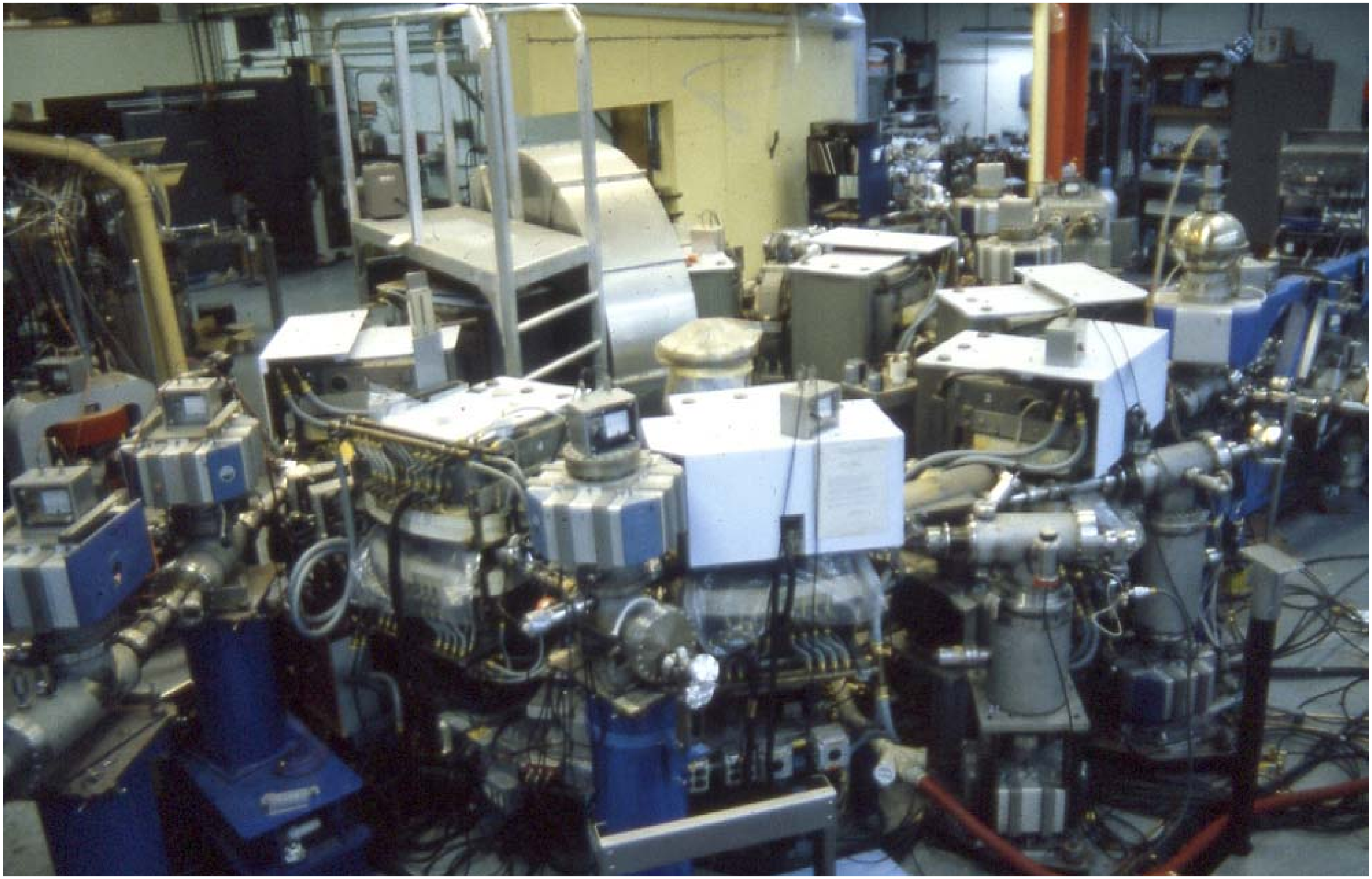
Spectroscopy

- angle resolved photoelectron spectroscopy (ARPES)
- core level photoelectron spectroscopy
- x-ray absorption spectroscopy with fluorescence detection

Microscopy

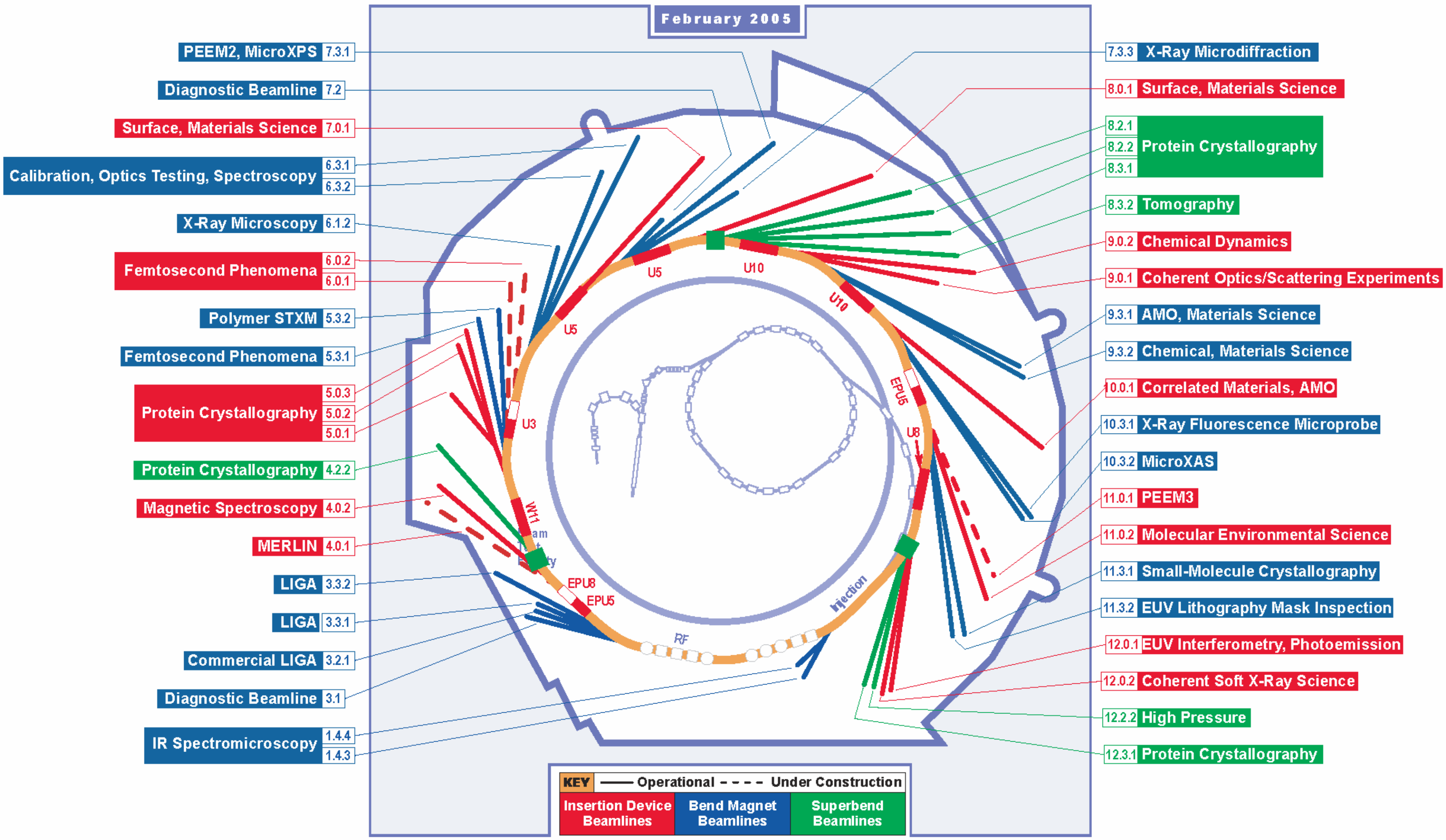
- x-ray nano tomography
- coherent x-ray diffraction microscopy

SR in the early days: Tantalus at Univ. Wisconsin



Beamlines at the ALS 2005

BERKELEY LAB



Why hasn't there been sufficient investment in detectors

Diversity of needs

- ALS has ~ 30 VUV – SXR beamlines
    - has > 30 different types of detector

Lots of 'easy' experiments to do, which give a big payoff with simple detectors

- photoemission
- x-ray absorption spectroscopy
- photoemission electron microscopy (PEEM)

Availability of some level of commercial detector systems

- eg. ARPES, SXR imaging / diffraction….

What is different now?

- scientific questions getting more difficult and demanding much more complex detection
- needs are tending to focus on a few high impact areas..
    - eg. photoemission applied to strongly correlated electronic systems
    - eg. diffraction microscopy applied to nanoscience
- relative maturity of microelectronic detector technology

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Photoelectron Spectroscopy

Angle Resolved Photoelectron Spectroscopy (ARPES)

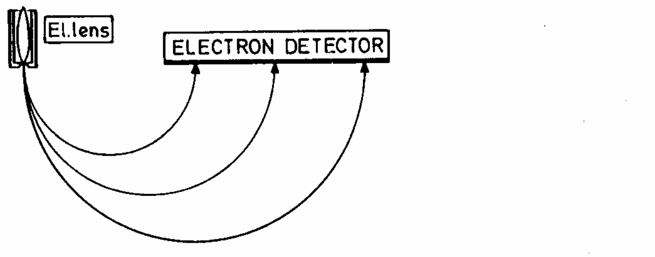
- Determination of electronic band structure of materials
    - correlated electronic systems, eg. high Tc, magnetic, manganites……
    - can be E, px, py, pz, x,y, T, δ coordinates, ie. 8 d data sets!
    - very high energy and momentum resolution

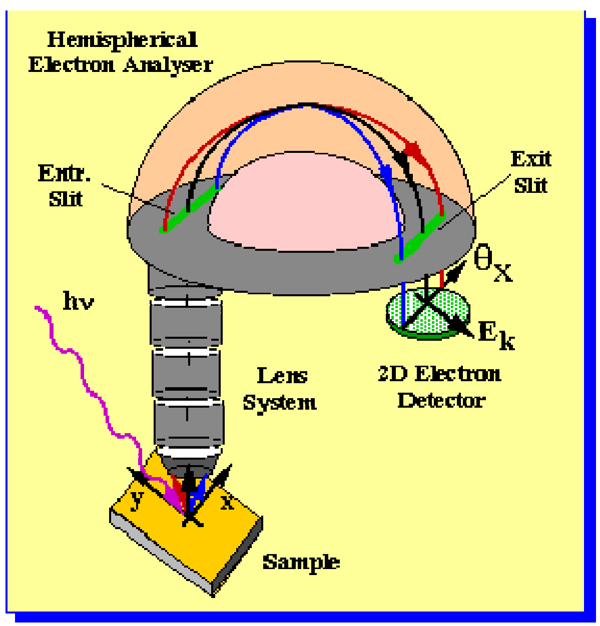
Core Level Photoelectron Spectroscopy

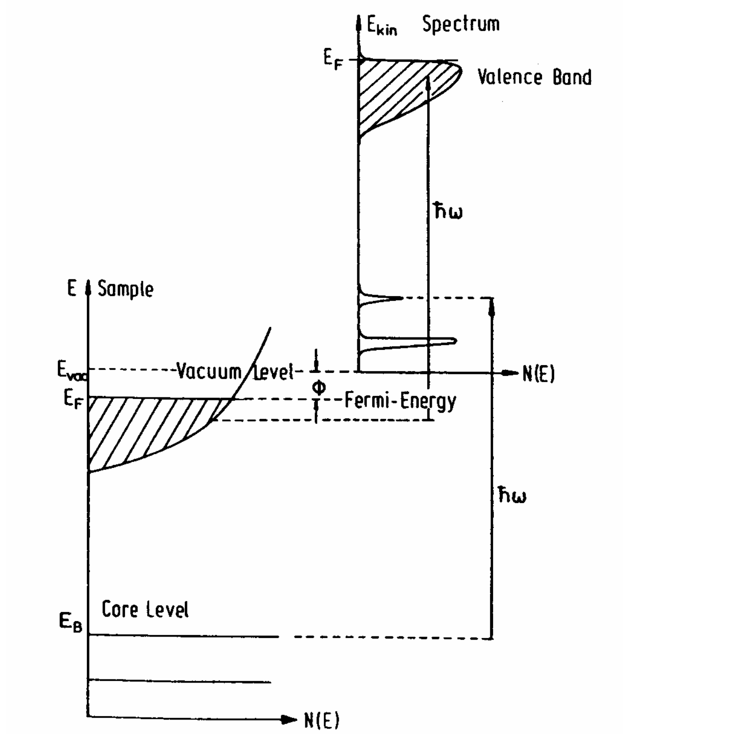
- Determination of chemical structure of a system from core level shifts
    - applied to surfaces, gases, and recently liquid surfaces / droplets,
- Determination of geometric structure of a system from photoelectron diffraction

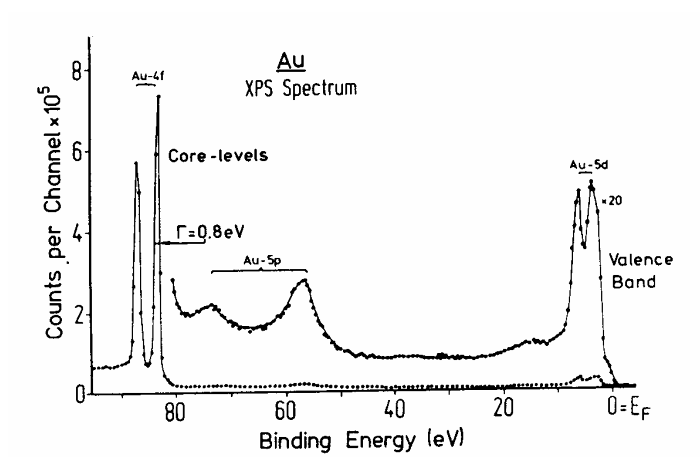
Photoelectron Spectroscopy

$$E_b = h\upsilon - E_{kin}$$



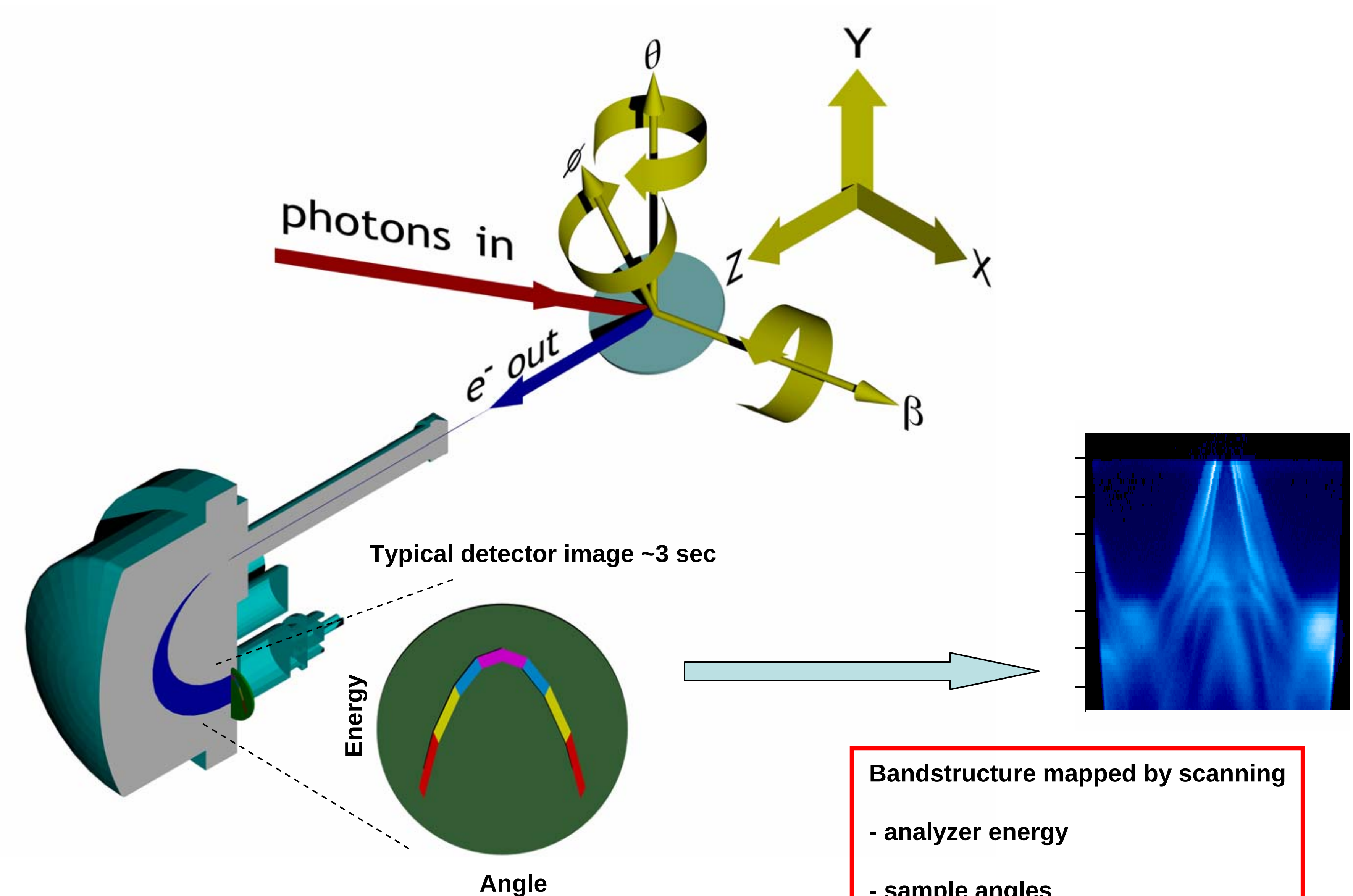






Hufner, Photelectron Spectroscopy

Valence Band Photoelectron Spectroscopy: Measuring Bandstructure

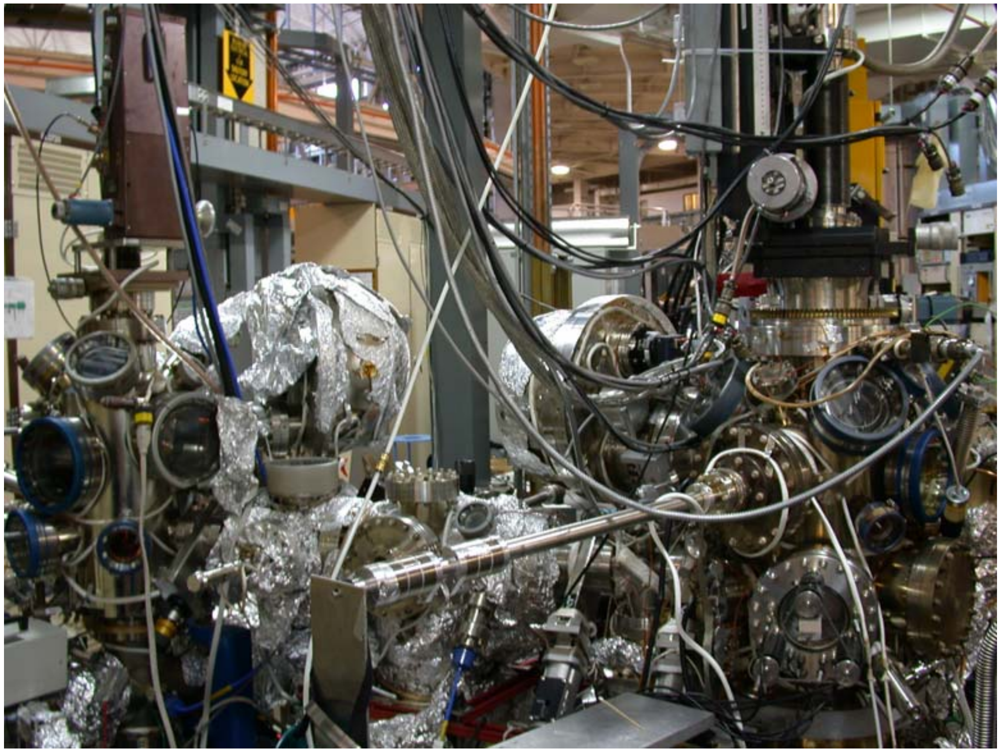


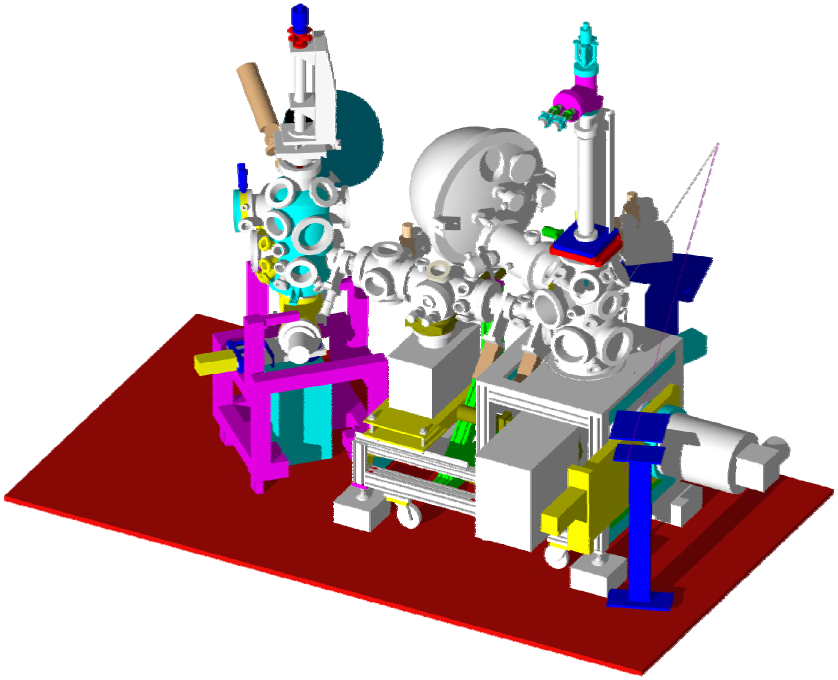
Bandstructure mapped by scanning

- analyzer energy

- sample angles

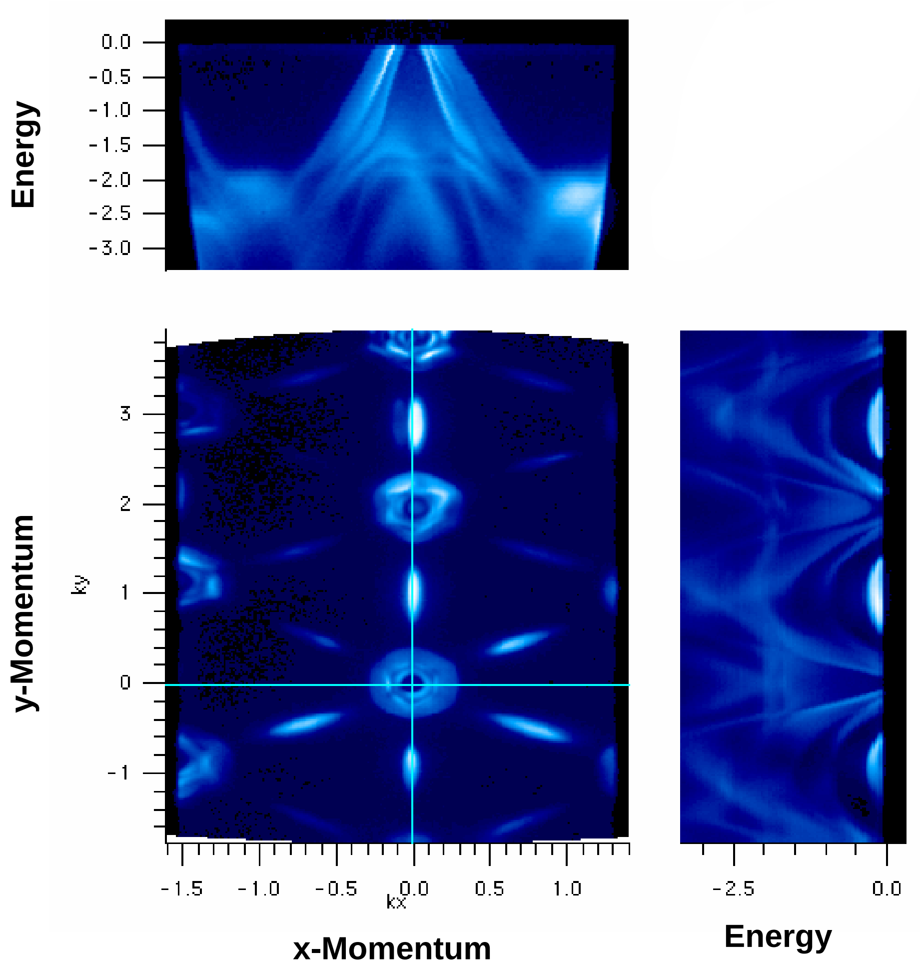
Angle Resolved Photo-Electron Spectroscopy (ARPES)





Electronic Structure Factory: Eli Rotenberg et al, ALS BL 7.0

Photoelectron Spectroscopy: I (E,px); I (E,py); I (px,py)



3 orthogonal slices
of a volume data set

Energy / x-Momentum / y-Momentum

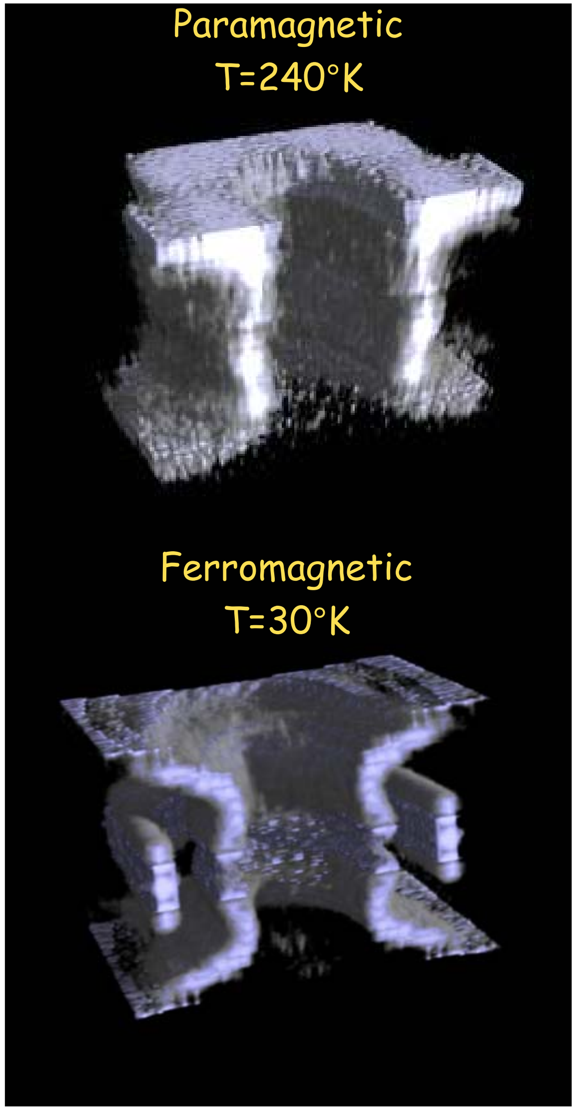
data courtesy K. Rossnagel, U. Kiel

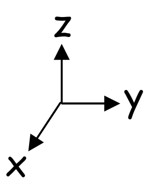
Photoelectron Spectroscopy: I (px, py, pz): const. E

Rare earth
metal Tb

px, py, pz at the
Fermi energy

- shows opening of
  a spin split gap





A third momentum direction can be scanned to build up a four dimensional data set.

Energy / x-Mom. / y-Mom. / z-Mom.

Also, Temperature and other parameters (sample composition, etc) may be scanned to yield higher dimensional data sets

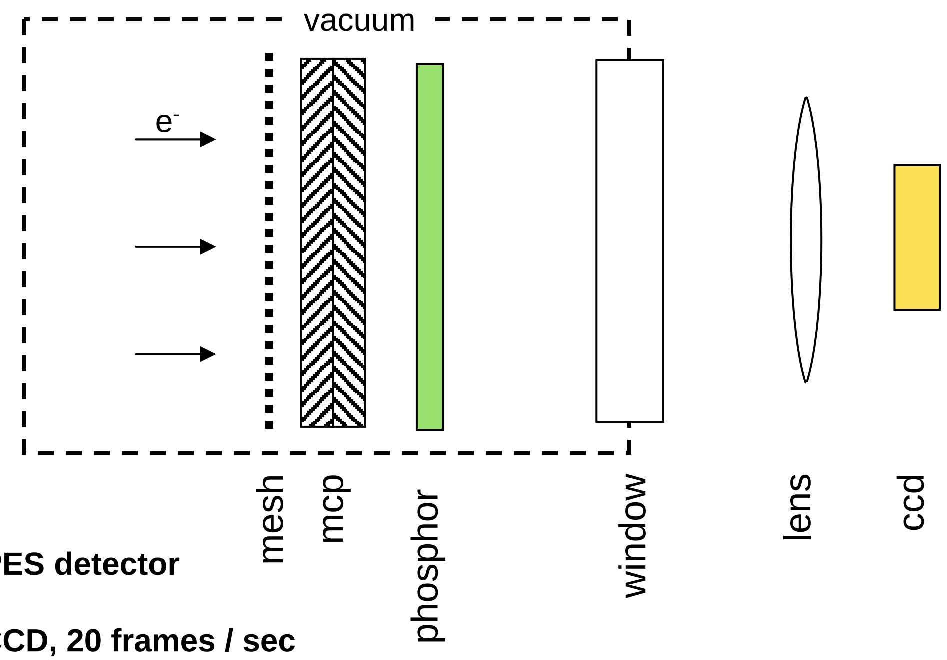
I (E,px,py,pz,x,y,T, δ)

- ie. up to 8 dimensional data sets

- need for high flux, high data rate

data courtesy K. Starke group, FU Berlin

Photoelectron Spectroscopy: 2d Detectors for ARPES



- standard ARPES detector

    - 10 bit CCD, 20 frames / sec
    - poor linearity, poor dynamic range, poor spatial resolution, too slow….

- improved ARPES detector

(a) - single MCP, fiber coupled, optimized phosphor, 30 x 30 mm active pixel sensor or CCD

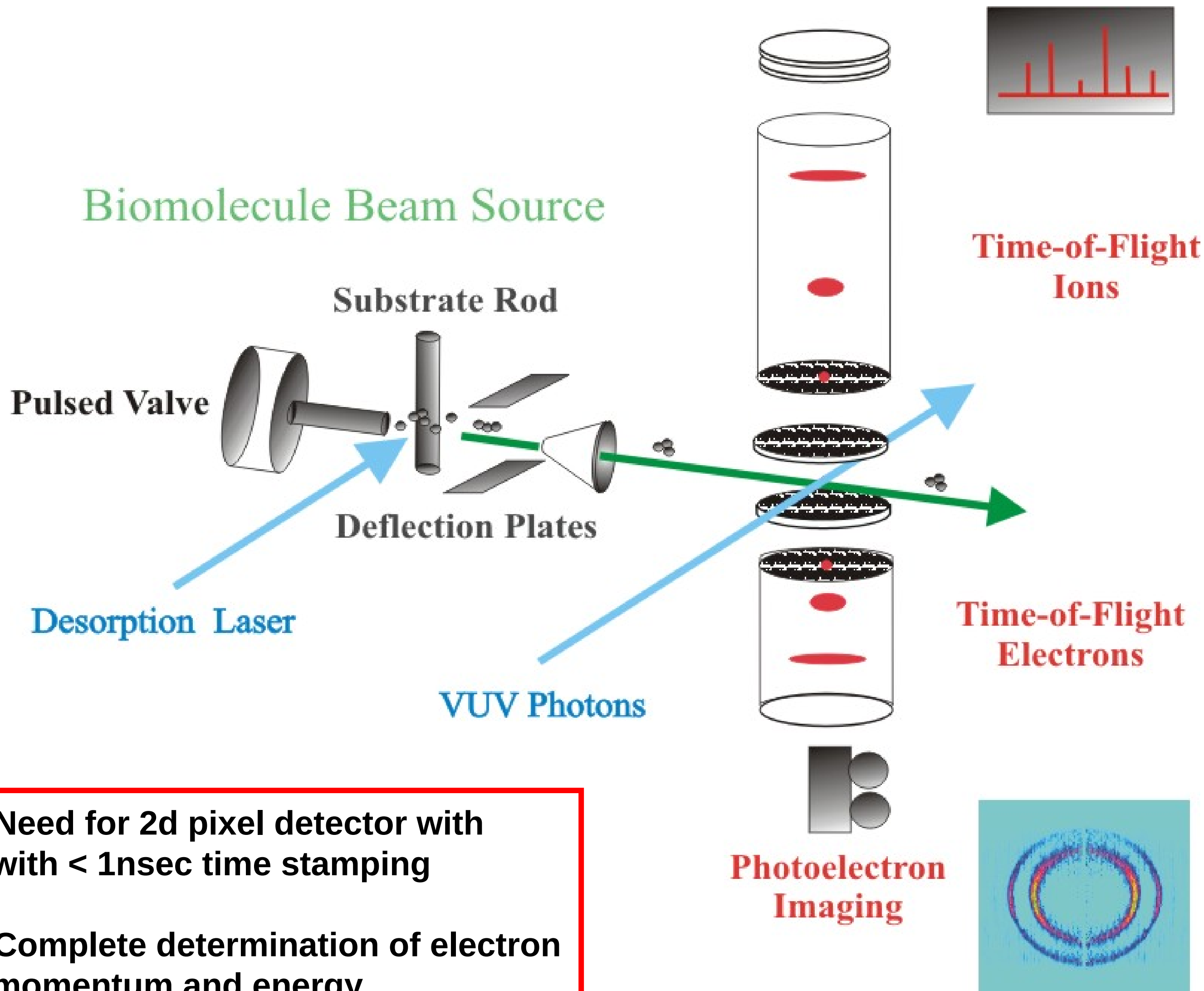
(b) - single MCP, large area electron optimized fast CCD (pixel limited psf)

(c) - double MCP, multichannel (CMOS ASIC) delay line readout (eg. Siegmund), NxN digital counter (eg. Denes)

10 MHz data rate over detector, 50 fps 'framing', UHV compatible, < 15 micron resolution (50% MTF)

- ideally time stamping to ~ nsec for TOF and pump – probe studies

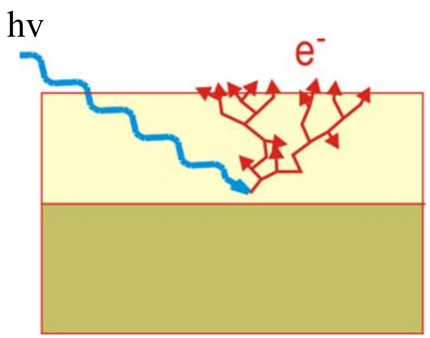
Photoelectron Spectroscopy with Time of Flight (TOF)



- Need for 2d pixel detector with with < 1nsec time stamping
- Complete determination of electron momentum and energy

Image from Musa Ahmed, LBNL

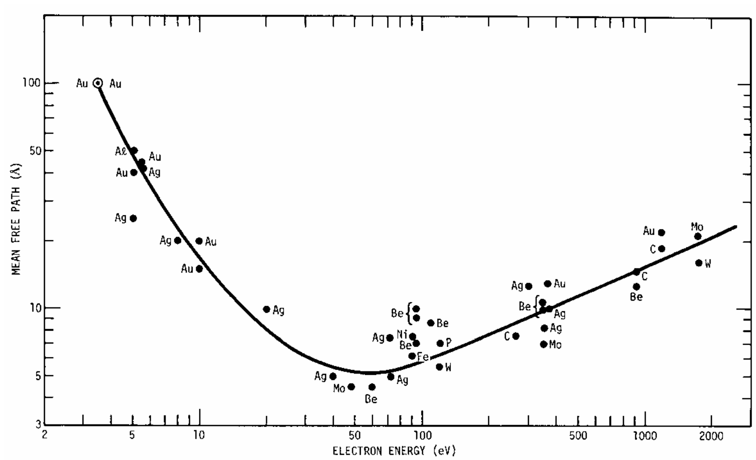
Core Level Photoelectron Spectroscopy



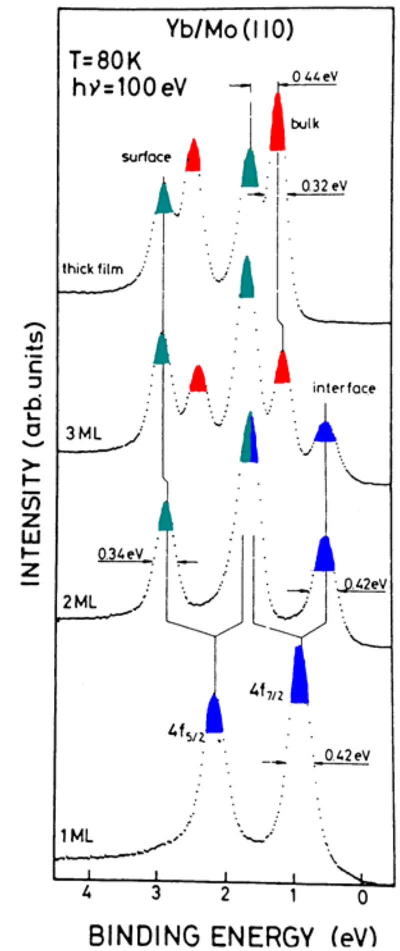
Electrons interact strongly

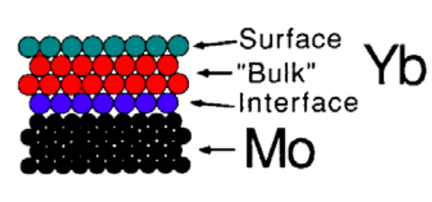
Surface Sensitivity

5-20 Å



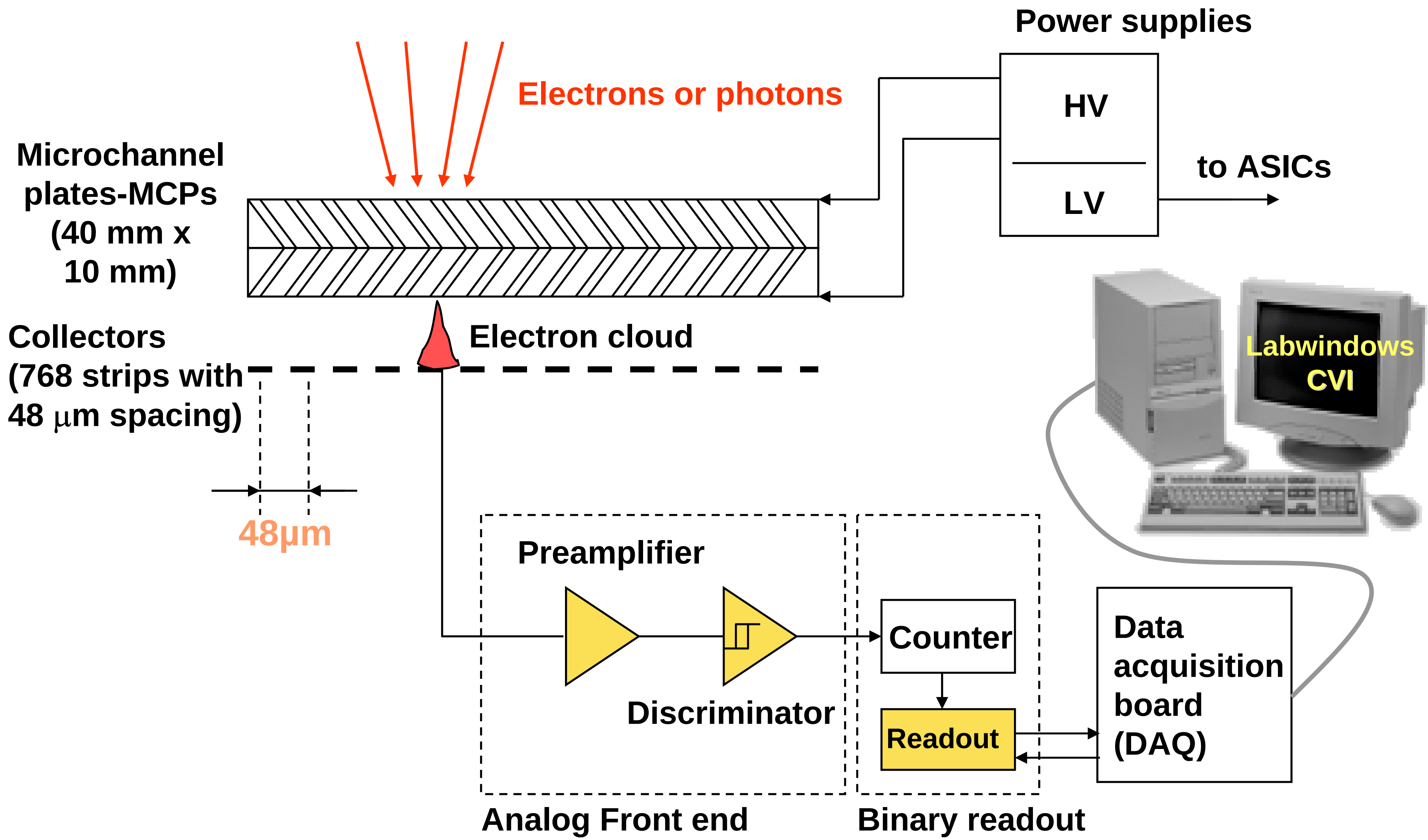
- high cross sections
- angle integration (20 x 20 degrees)
- very high data dates (GHz)



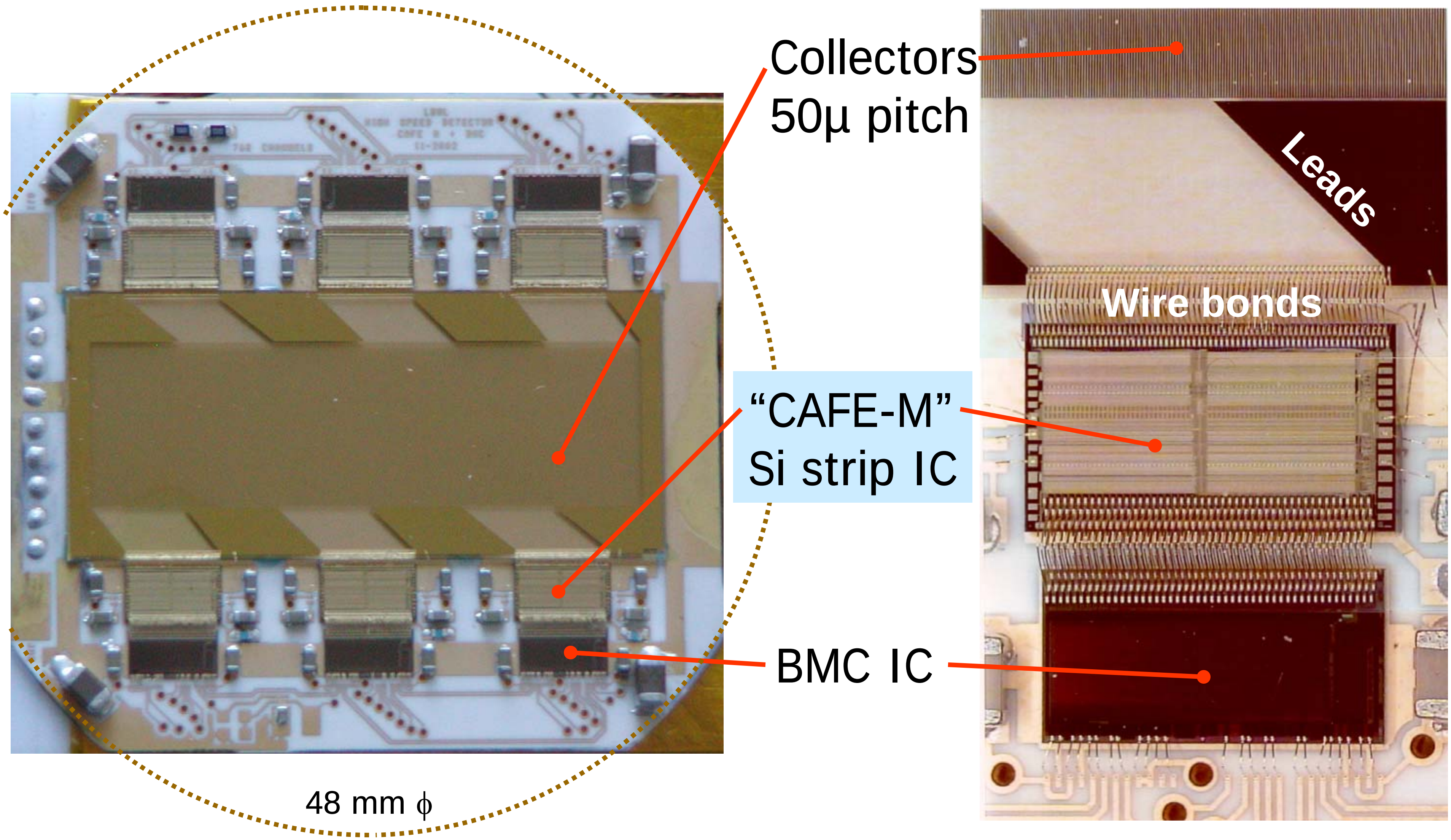


Mårtensson et. al. Phys. Rev. Lett. 60, 1731 (1988)

1D pulse counting detector for Core Level Photoemission

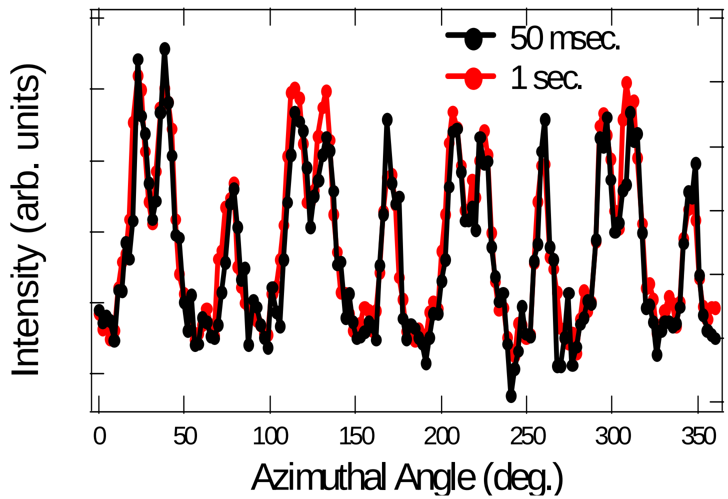


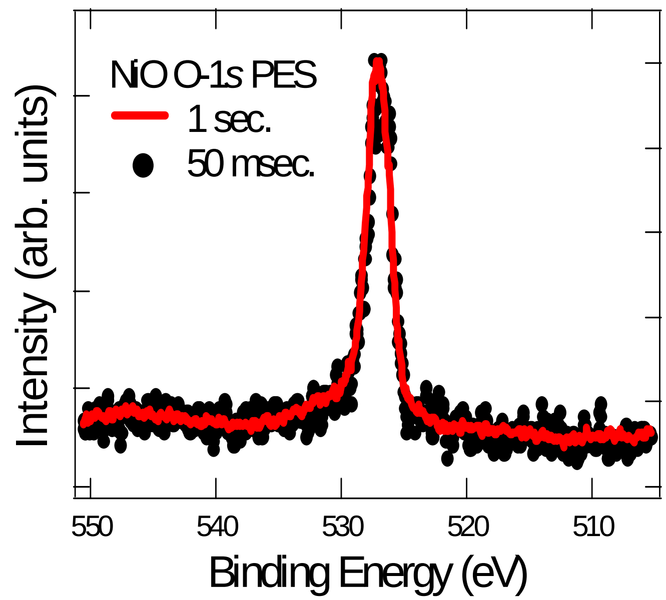
1D pulse counting detector for Core Level Photoemission



~ 1 GHz data rate over full 768 channels

1D pulse counting detector for Core Level PES:





A. Nambu, J.-M. Bussat, B.C. Sell, M. Watanabe, A.W. Kay, N. Mannella, B.A. Ludewigt, M. Press, B. Turko, M. West, G. Meddeler, G. Zizka, H. Spieler, T.Ohta, Z. Hussain, C.S. Fadley, Journal of Electron Spectroscopy and Related Phenomena, 137-140, 691 (2004).

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Microscopy

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- coherent x-ray diffraction microscopy

Soft x-ray Nano-Tomography

Tomographic transmission x-ray microscopy

- zone plate condensor, sample on rotation stage, zone plate objective, CCD detector

- record tilt series

- recover 3d image by filtered back projection

Tomographic coherent x-ray diffractive imaging (CXDI)

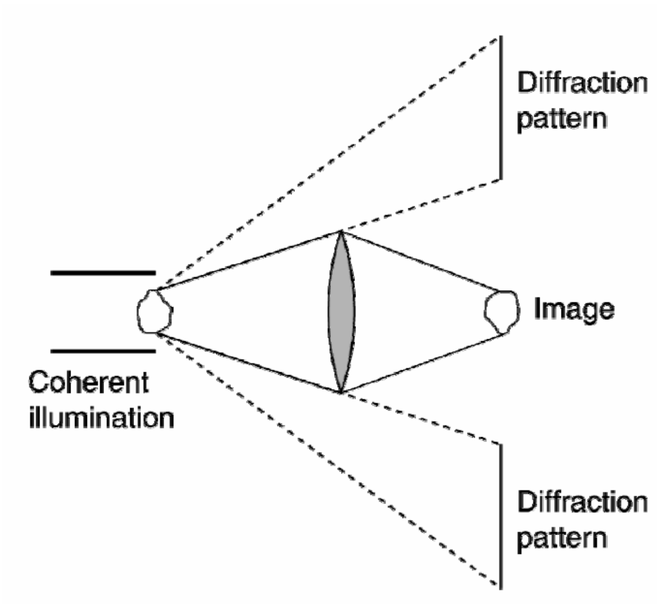
- lensless

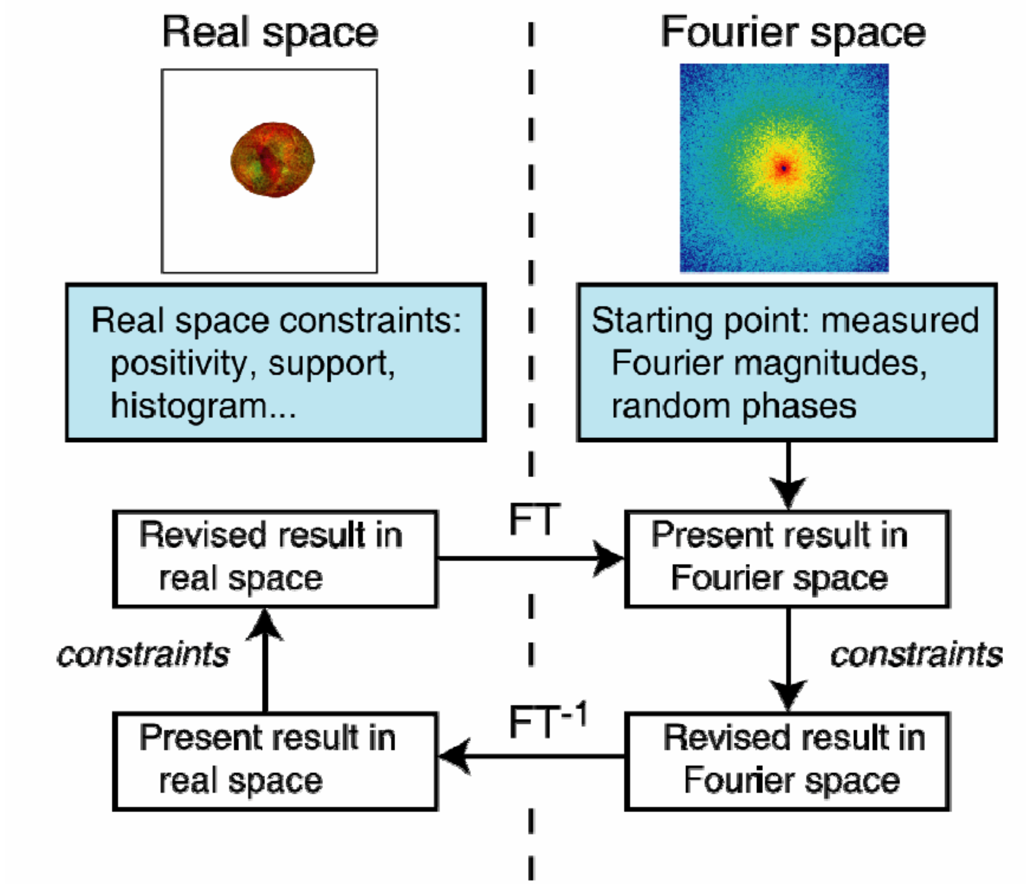
- illuminate sample with coherent light, record diffraction pattern on CCD

- record tilt series

- recover phase (and hence image) from iterative reconstruction using known supports

Coherent X-ray Diffraction Microscopy (CXDI)

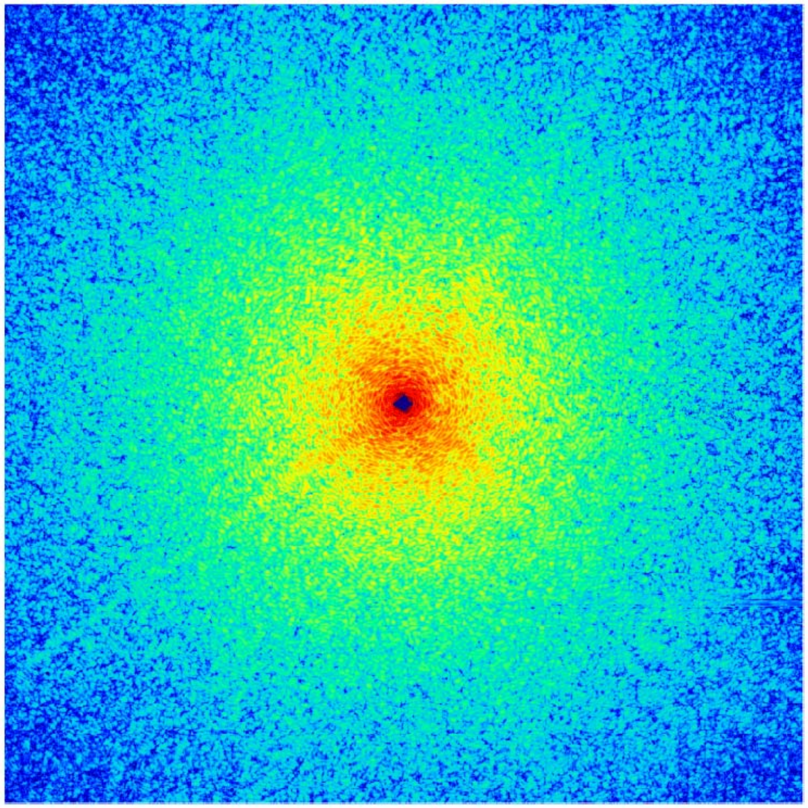


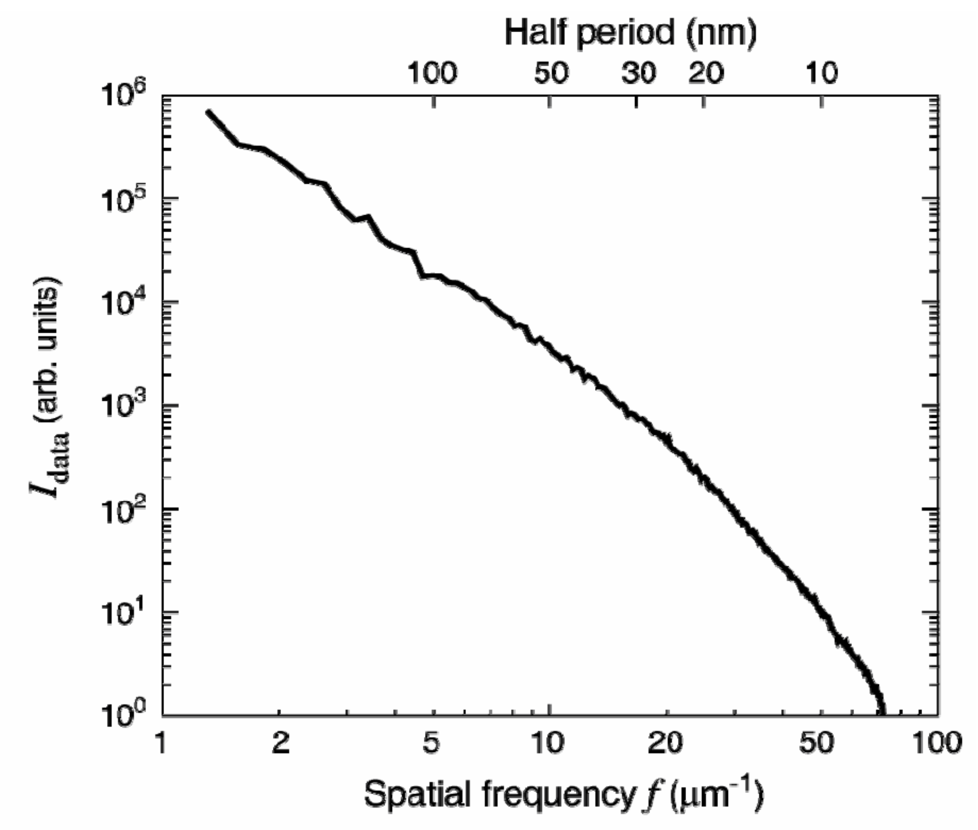


Sayre, 1980; Miao *et al.*, *Nature* **400**, 342 (1999)

Phasing algorithms: Feinup, *Opt. Lett.* **3**, 27 (1978); Elser, *JOSA A* **20**, 40 (2003); and others

Coherent X-ray Diffraction Microscopy (CXDI): Yeast Cell





D. Shapiro, C. Jacobsen, J. Kirz, D. Sayre et al

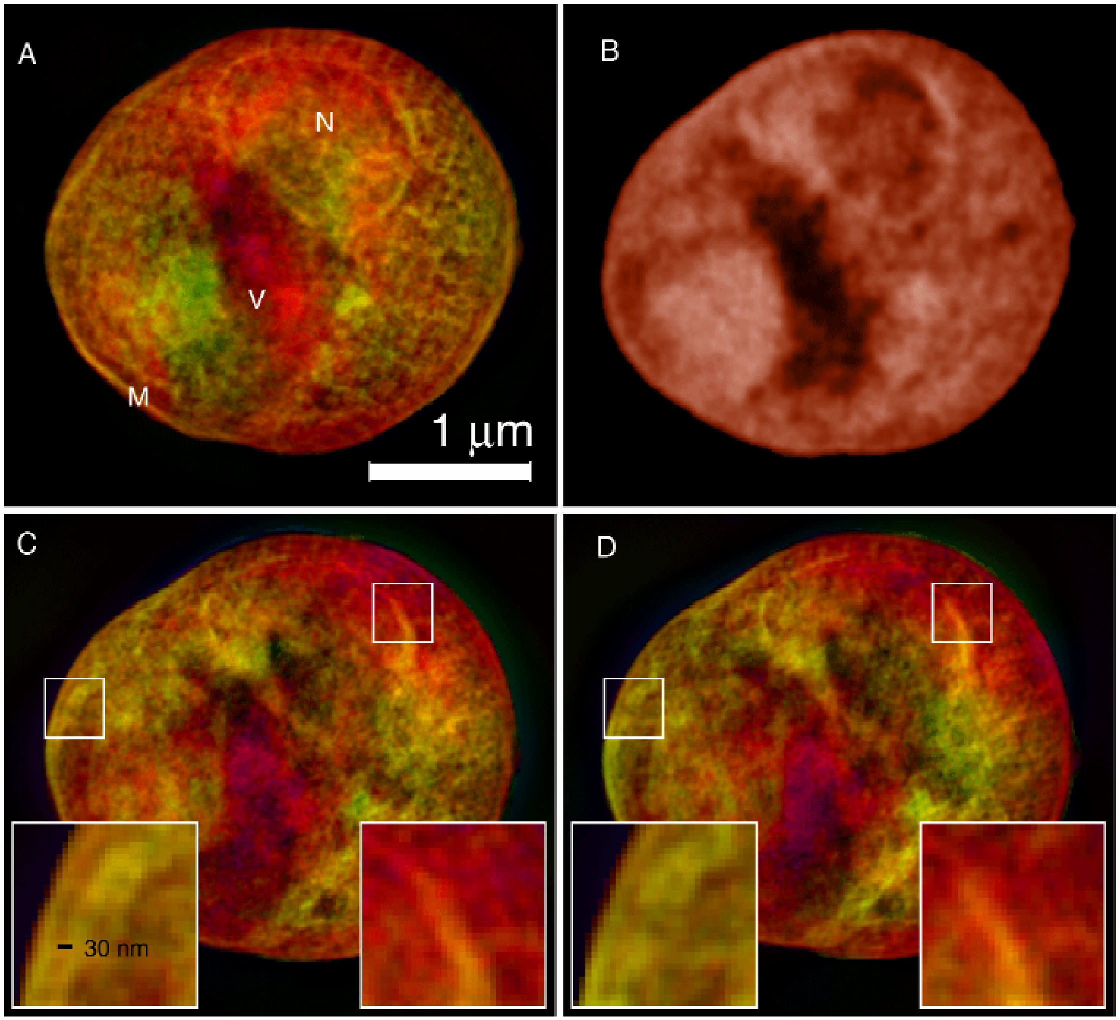
Coherent X-ray Diffraction Microscopy (CXDI): Yeast Cell

D. Shapiro *et al.*, Stony Brook (ALS, Berkeley)

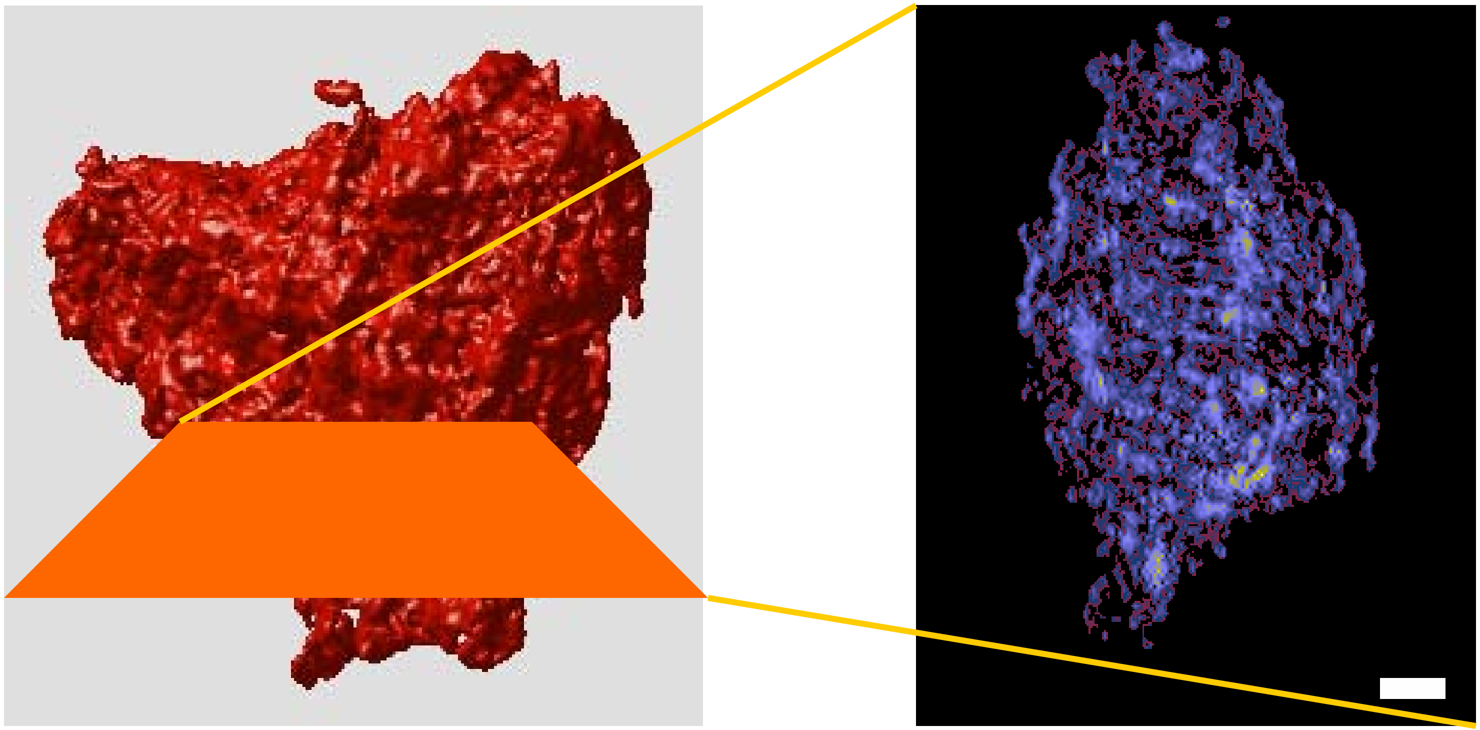
XDM image

STXM image

XDM images at two tilts, 1° apart



Coherent X-ray Diffraction Microscopy (CXDI): Ta_2O_5 foam particle

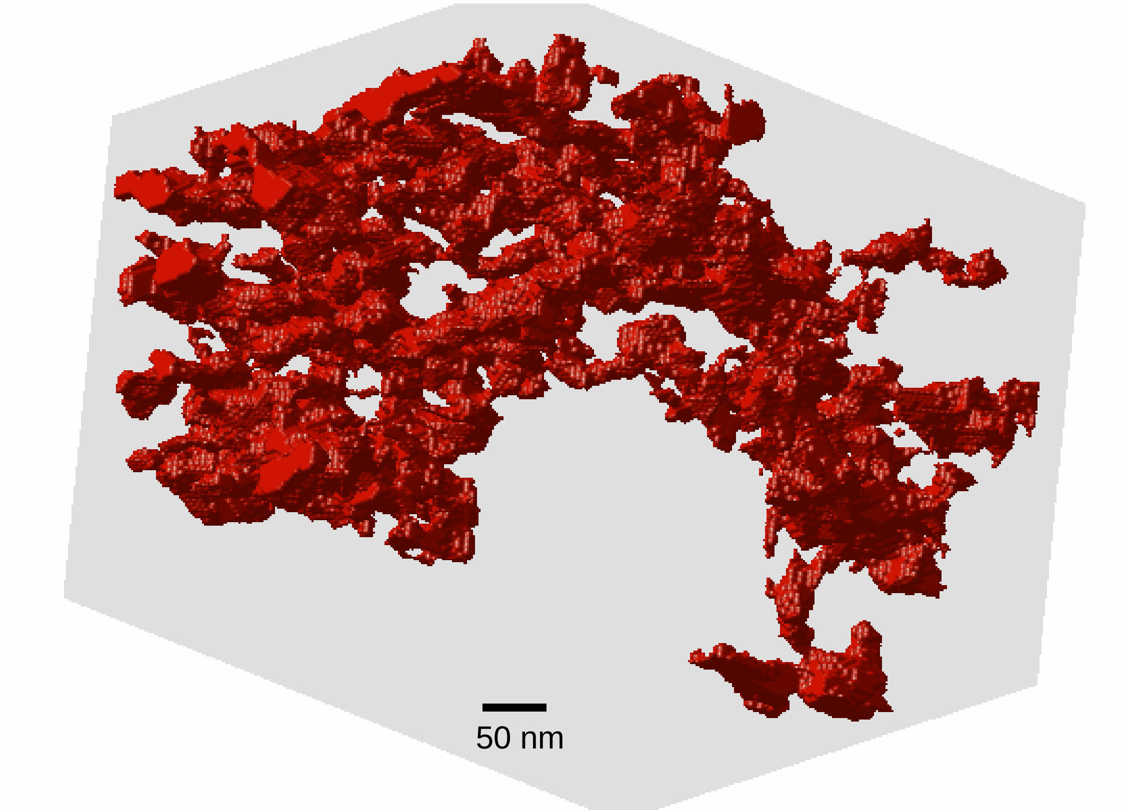


3D isosurface of lensless image of foam particle (size of particle ~2μm in max extent

Cross-section through particle (bar = 200nm)

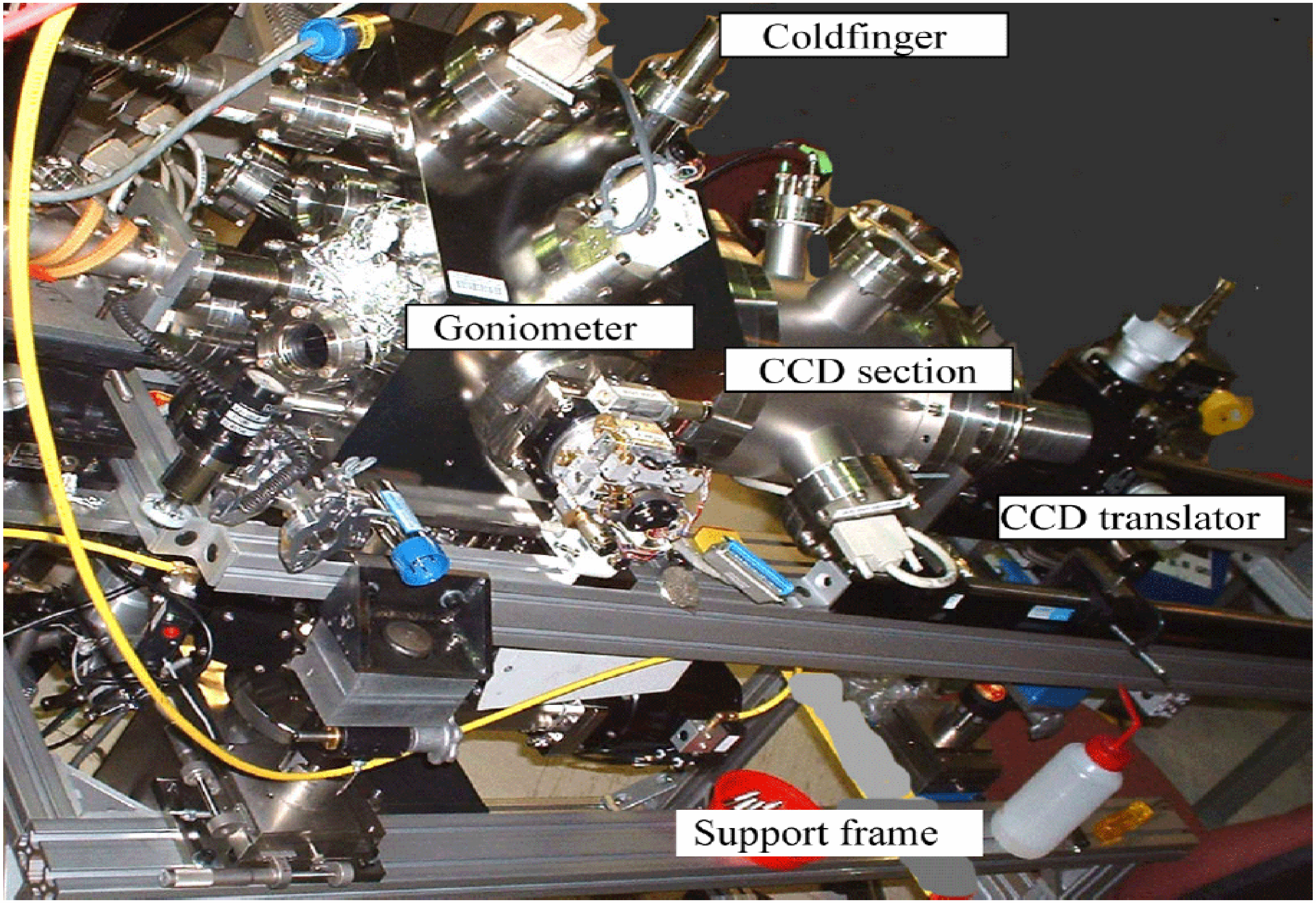
Reconstructed data courtesy of Anton Barty, Henry Chapman, Stefano Marchesini, et al.

Coherent X-ray Diffraction Microscopy (CXDI): Ta_2O_5 foam particle



Reconstructed data courtesy of Anton Barty, Henry Chapman, Stefano Marchesini, et al.

Coherent X-ray Diffraction Microscopy (CXDI)



Coherent X-ray Diffraction Microscopy (CXDI): Detectors

Dynamic range

- > 1e6 (higher resolution requires higher dynamic range, for same image size)

Number of pixels

- minimum of 2 x image size / resolution
  - eg. 10 micron cell, 10 nm resolution = 2k x 2k
  - no gaps allowed!

Pixel size

- < 50 microns to keep detector (in high vacuum) a reasonable size

Noise

- << 1 photon / read

Frame time

- 0.1-1 secs

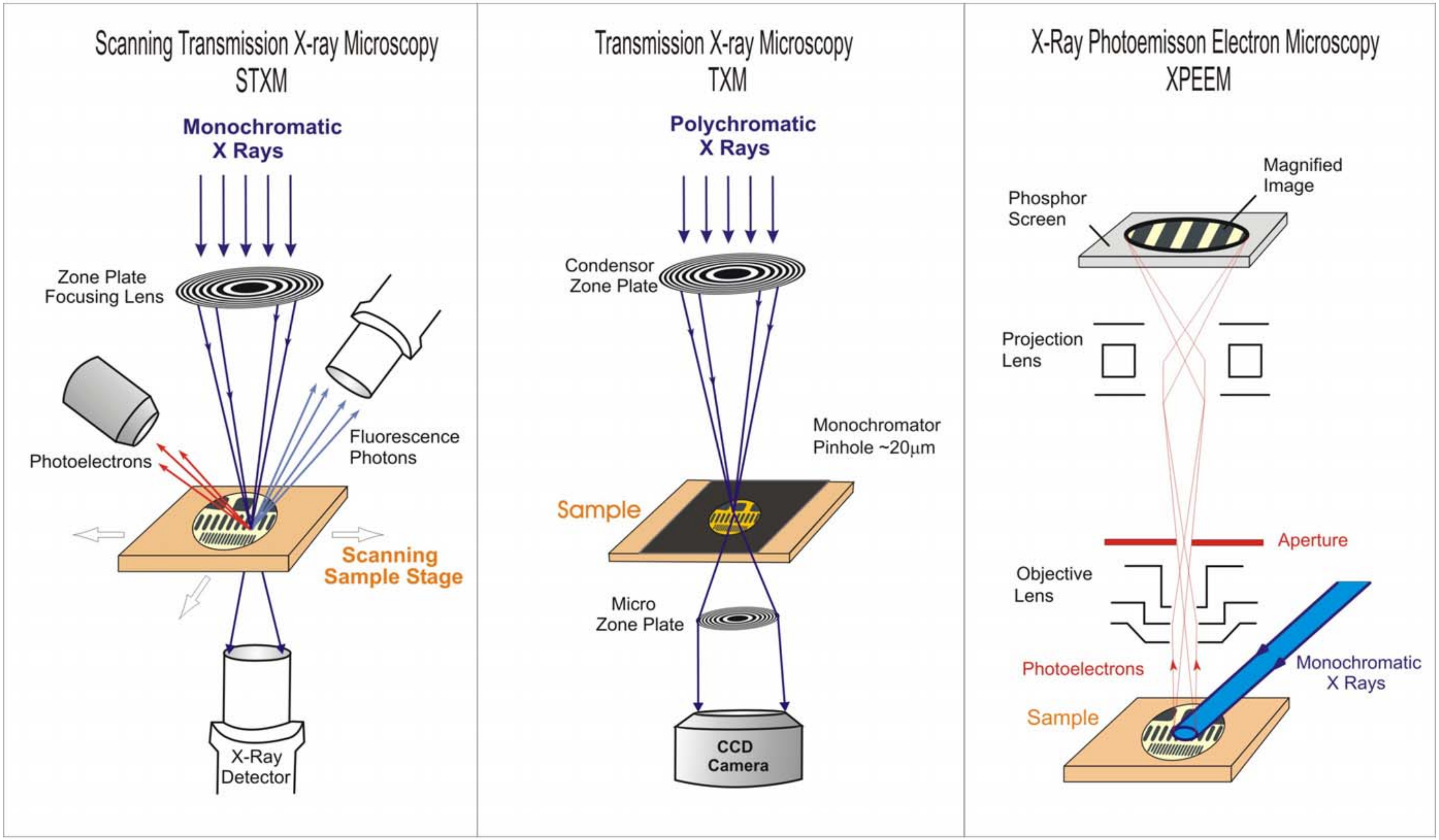
Coherent X-ray Diffraction Microscopy (CXDI): Detectors

Dynamic range issues

- pulse counting at > 1e6 ph/sec
    - pixel detectors…
        - gap issues (see Ed Westbrook's talk)
        - large pixels (may be small enough, eg. 50 micron Medipix 2)
        - low energy photons

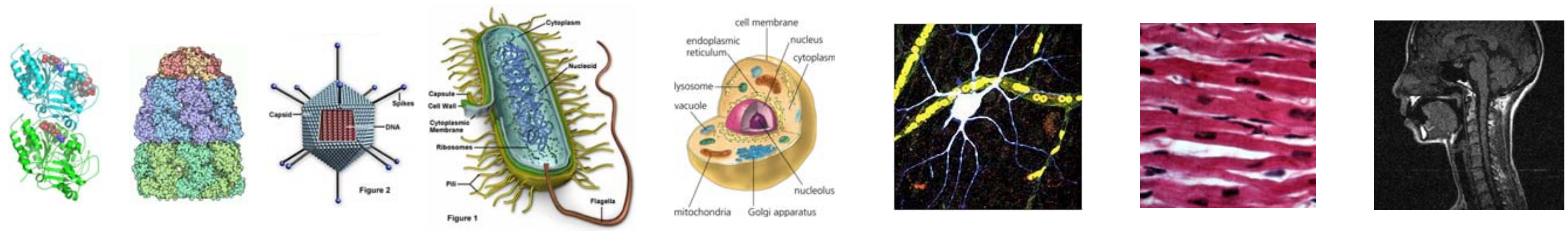
- direct detection in a ccd (presently used, but multiple frames with overlap)
    - 1 keV photons gives ~ 280 electrons / photon
    - non-MPP CCD well depth, 30 micron pixels, ~ 1e6 electron well depth
    - 3600 1 keV photons / read (max)
    - 12 bit resolution
    - need > 280 frames / sec (see Peter Denes's talk)
        - high frame rate + addition extends the dynamic range
            - possible because signal / noise ~ 280 / < 6 gives zero overlap

X-ray Microscopes



Imaging of Biological Systems

Size (m) 10^{-8} 10^{-7} 10^{-6} 10^{-5} 10^{-4} 10^{-3} 10^{-2} 10^{-1} 1



Molecules | Complexes | Viruses | Bacteria | Cellular compartments | Cells | Tissues | Whole organisms

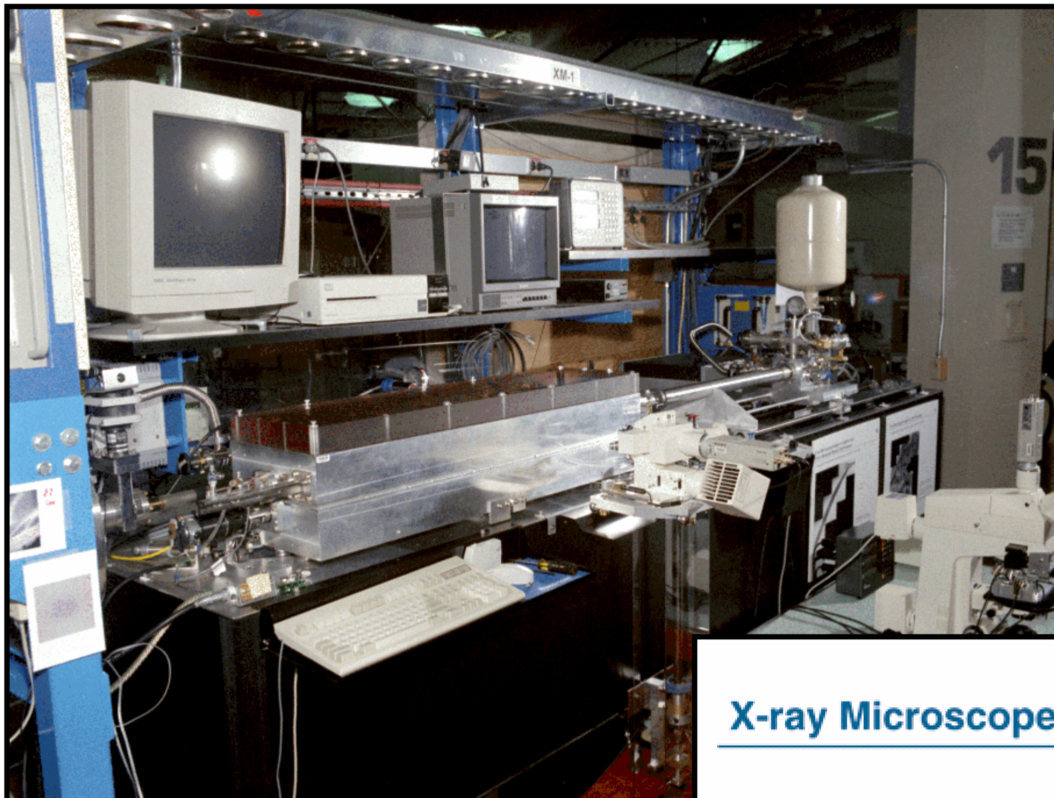
Cryo-electron microscopy

Light microscopy

X-ray crystallography
- atomic resolution
- needs protein crystals

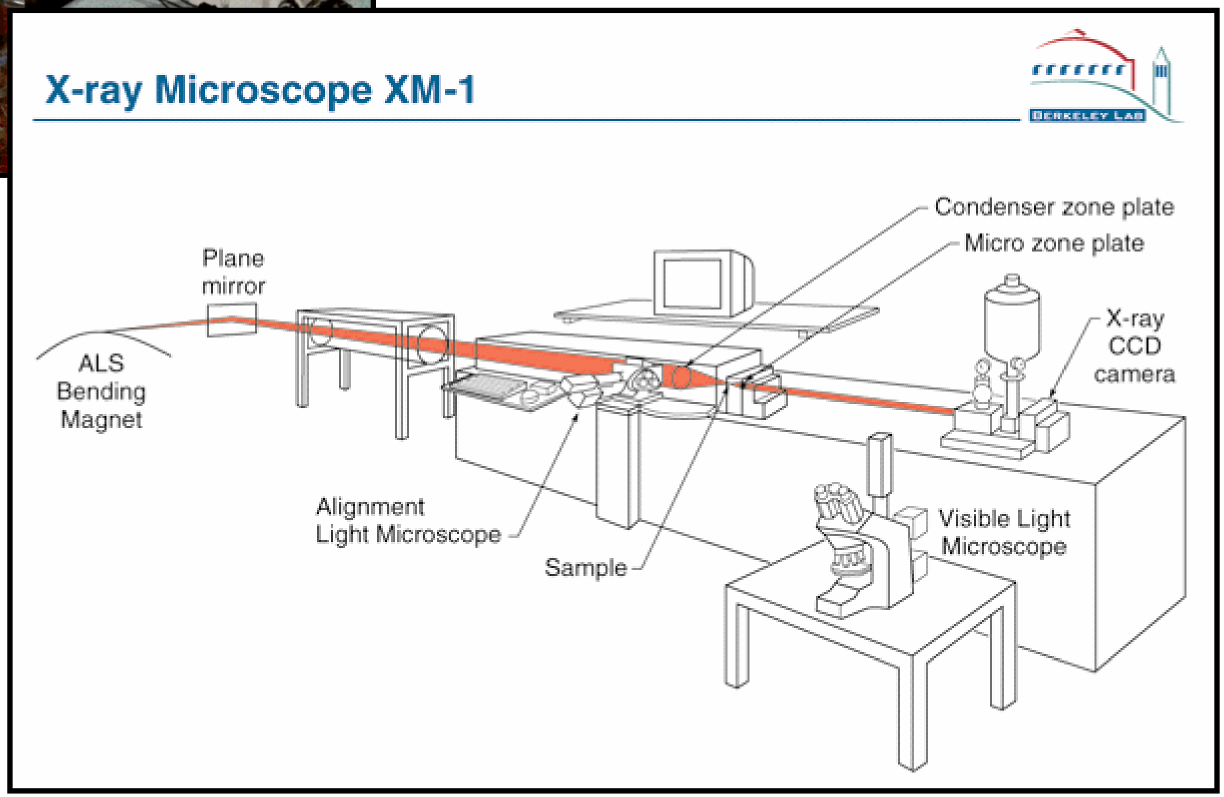
MRI

X-ray microscopy
- 15 nm resolution
- thick objects (compared to EM)



Beamline 6.1.2

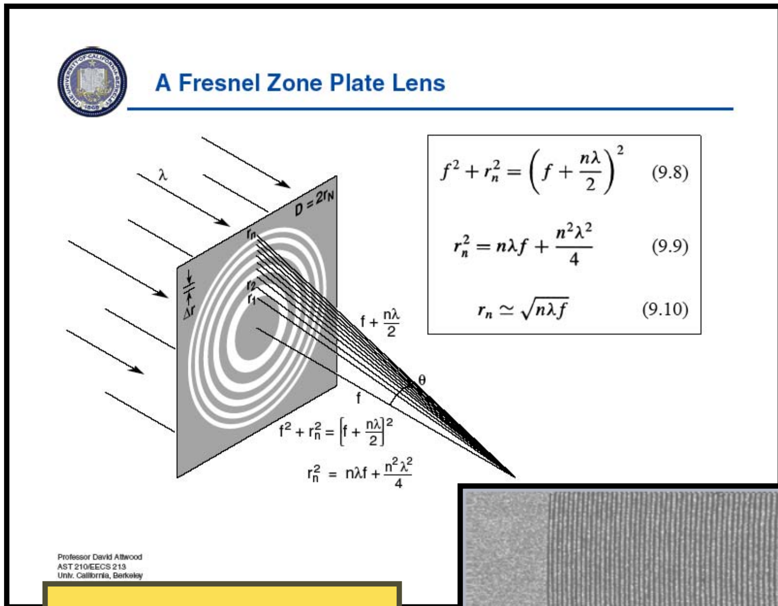
Full-field Transmission X-ray Microscope (XM-1)



Wavelength, 2.4 nm
Photon energy, 517 eV

Center for X-ray Optics

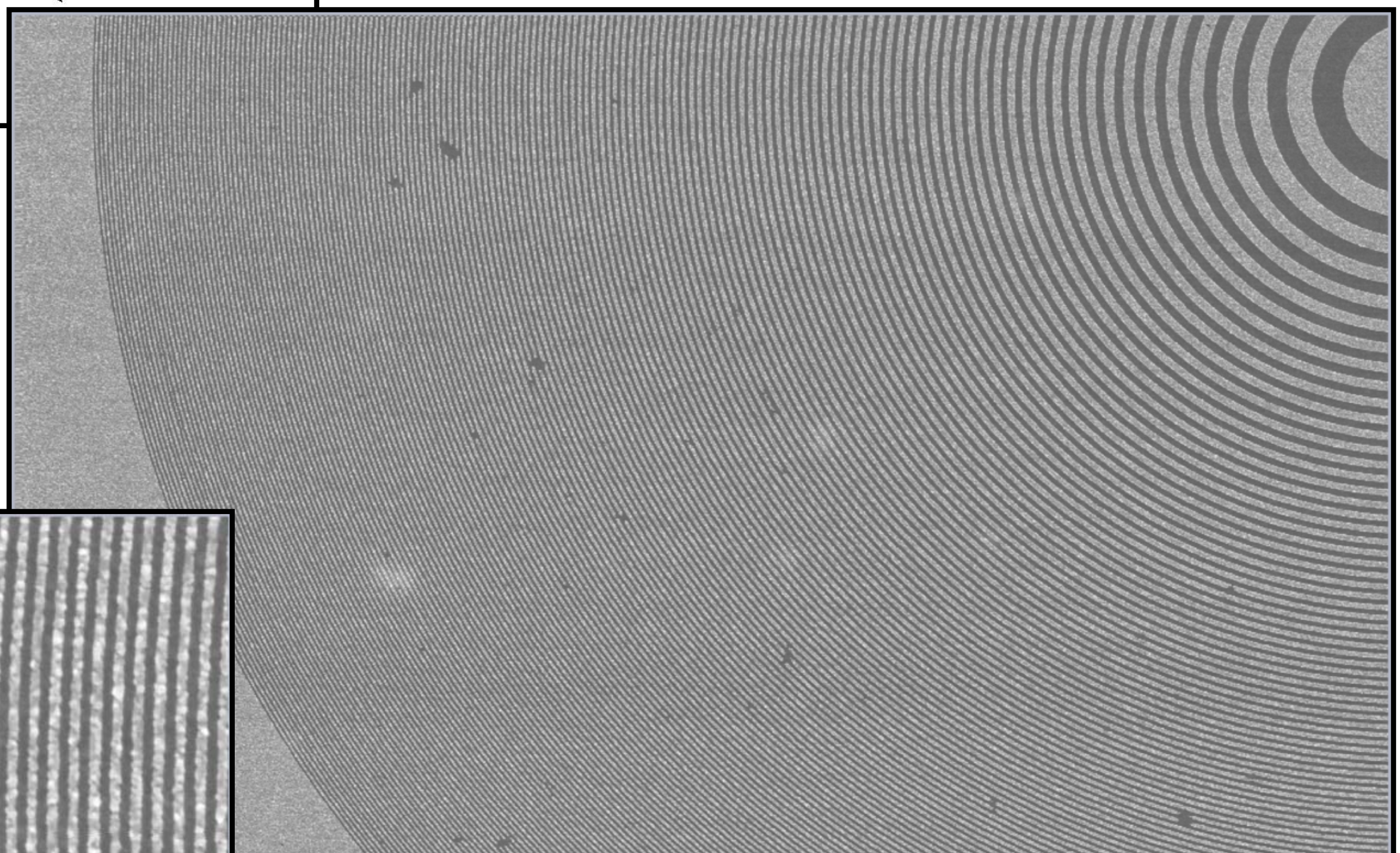
Erik Anderson
David Attwood

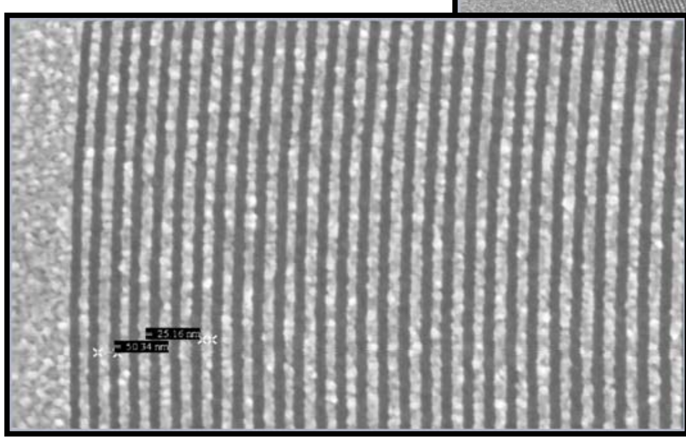


Zone Plates for 20 nm Resolution Microscopy (now 15 nm)

E. Anderson, D. Attwood, A. Liddle, D. Olynick, B. Harteneck, W. Chao; Center for X-ray Optics

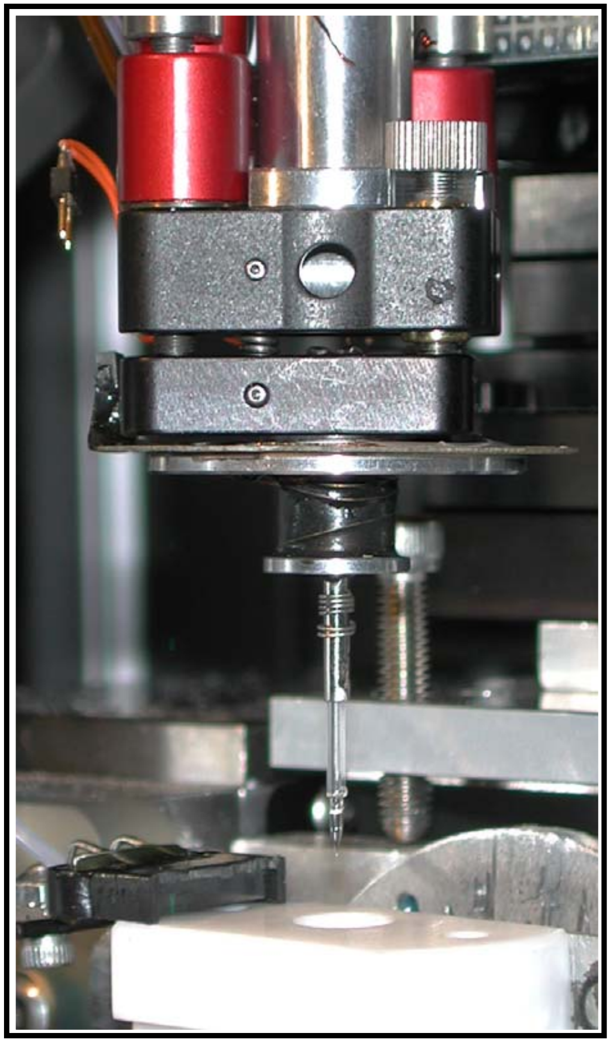
Dr = 25 nm
D = 63 um
N = 618 zones
f = 650 um
NA = 0.05
@ 2.4 nmλ

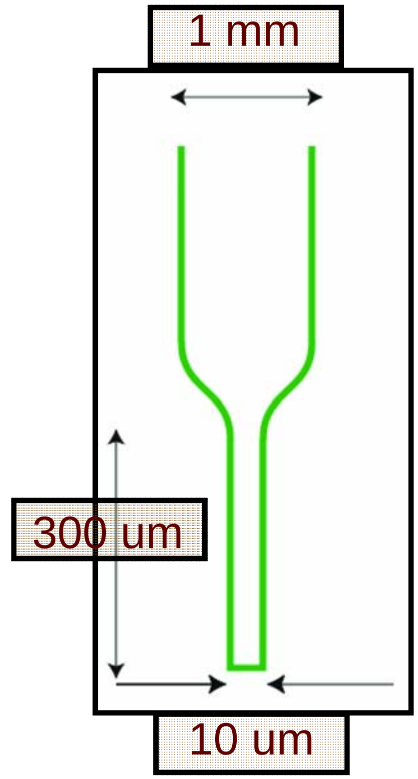


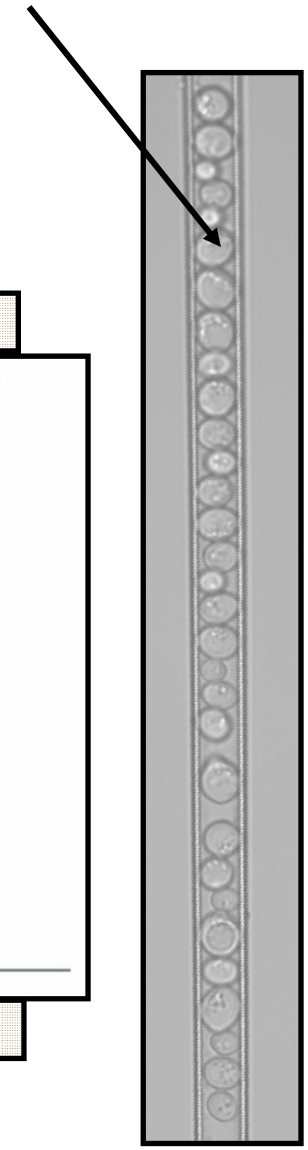


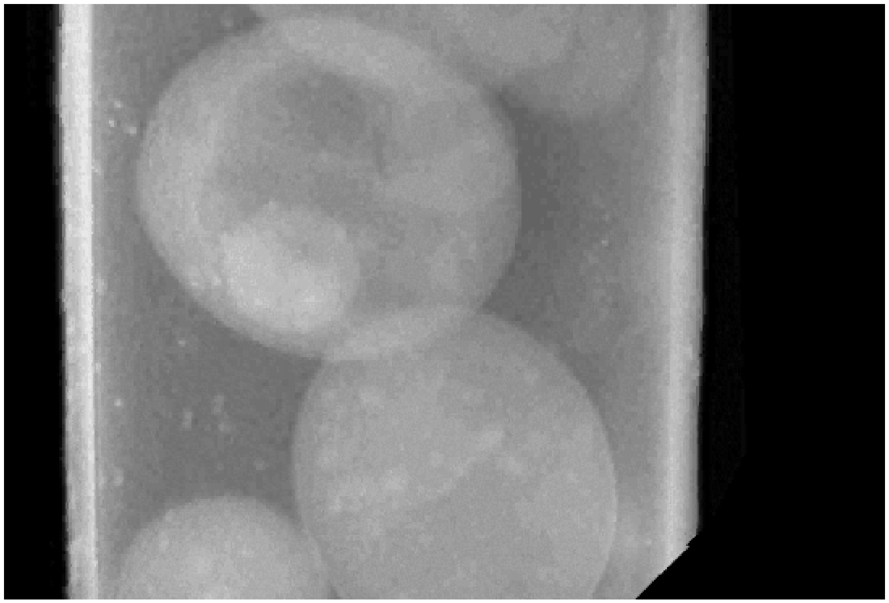
Cryo X-ray Tomography

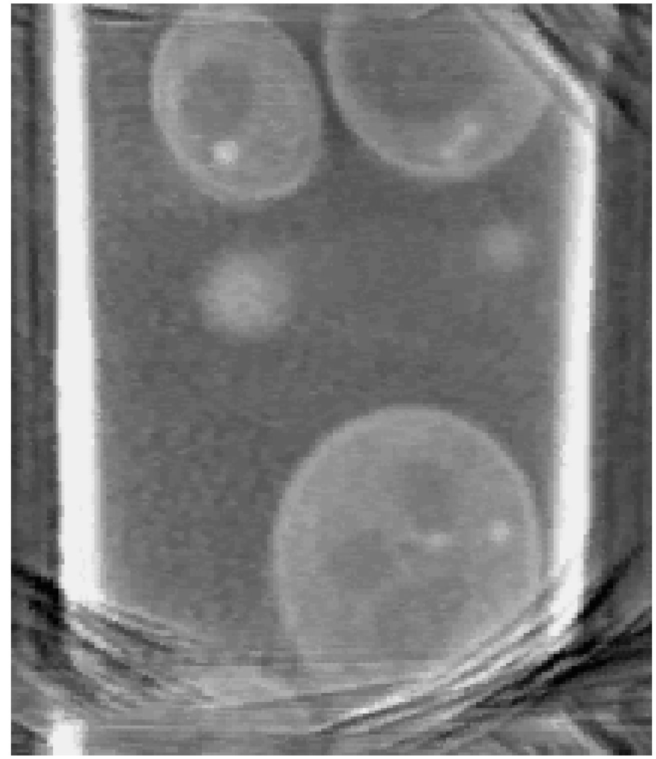
C.A. Larabell & M. A. Le Gros (2004).
Molecular Biology of the Cell, 15(3), 956-962











C.A. Larabell & M. A. Le Gros (2004).
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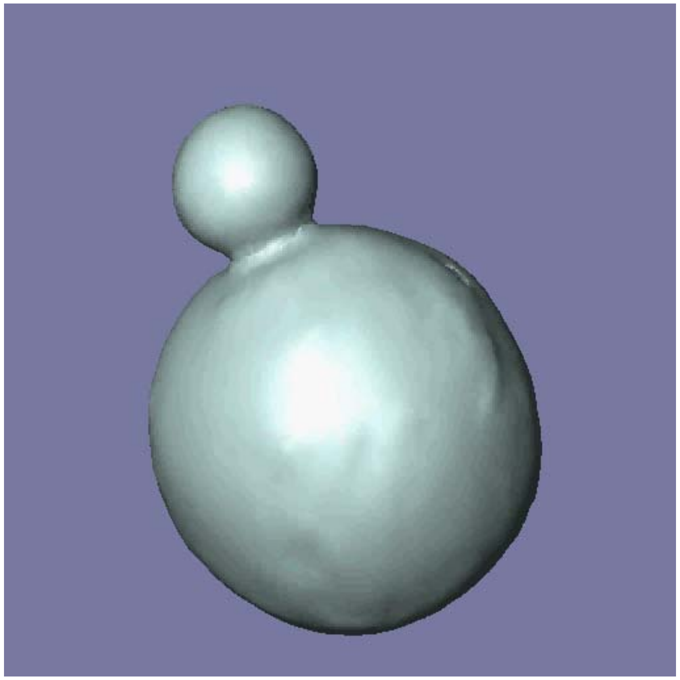
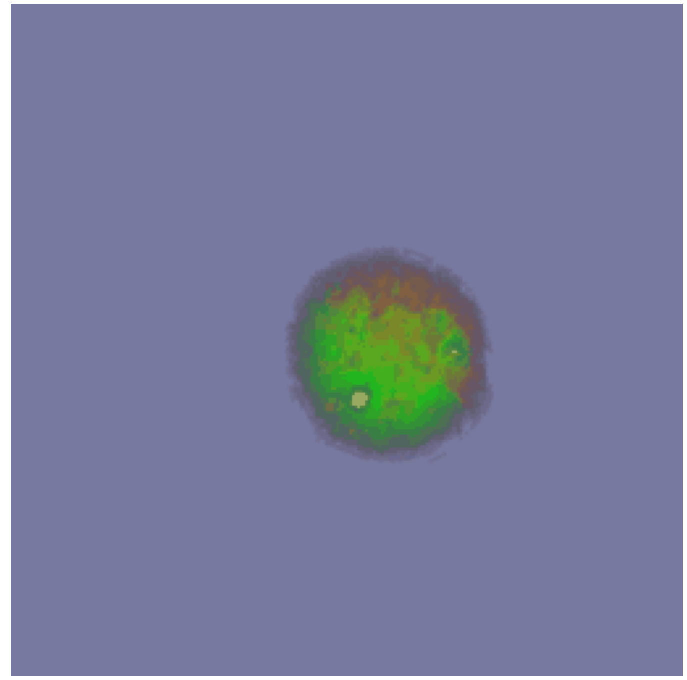
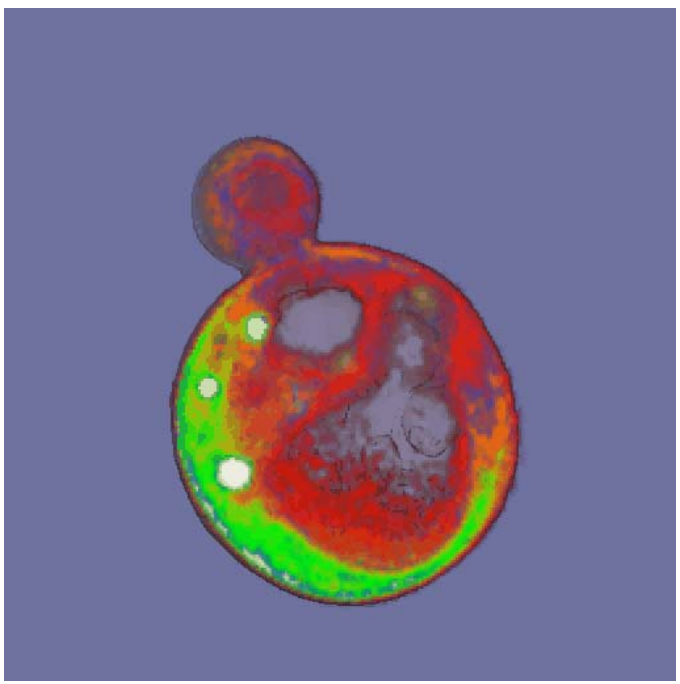
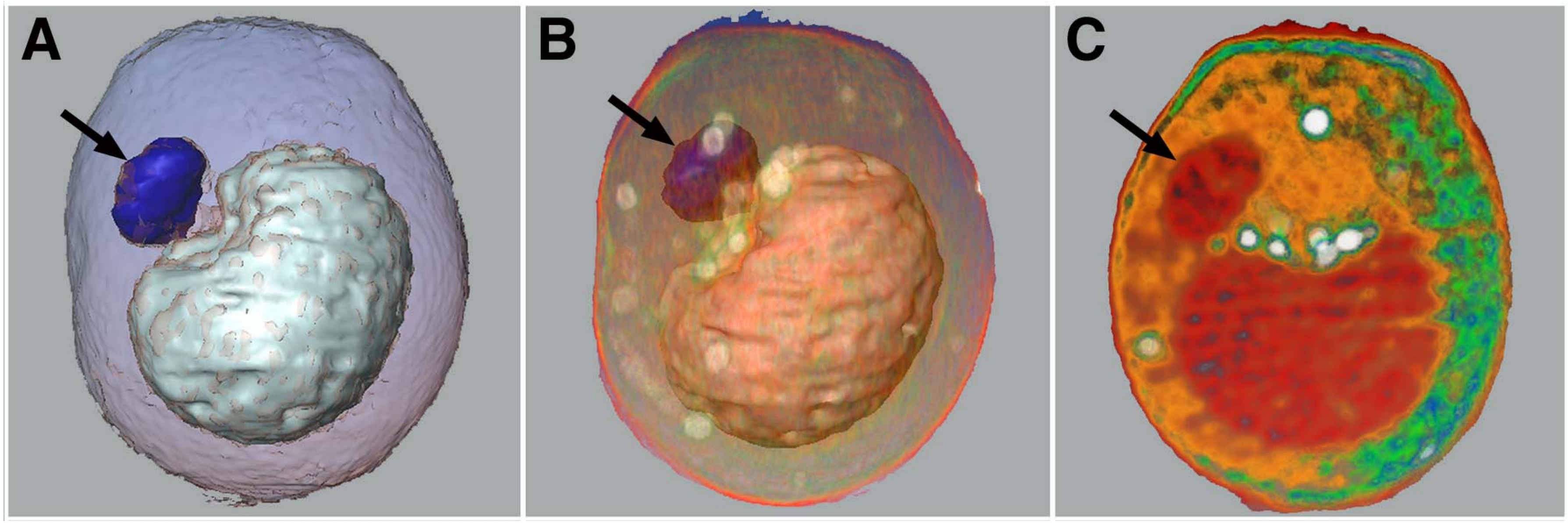






C.A. Larabell & M. A. Le Gros (2004). Molecular Biology of the Cell, 15(3), 956-962

Saccharomyces cerevisiae



Reconstructed data using different volume analysis algorithms.

A) Combination of translucent outer surface & opaque surfaces highlight internal organelles; arrow points to nucleus that has been color-coded blue

B) Surface views combined with volume rendering
- lipid droplets white
- surface of large vacuole pink

C) Computer section that was volume-rendered according to x-ray absorption
- lipid droplets white
- internal structures of vacuole and nucleus red
- other cytoplasmic structures appear green and orange.

C.A. Larabell & M. A. Le Gros (2004). Molecular Biology of the Cell, 15(3), 956-962

Tomographic X-ray transmission microscopy: Detectors

Dynamic range

- > 1e4

Number of pixels

- minimum of 2 x image size / resolution
  - eg. 20 micron cell, 10 nm resolution = 4k x 4k
  - no gaps allowed!

Pixel size

- related to focal length of zone plate (small) and reasonable object distance
  - < 20 microns

Noise

- << 1 photon / read

Frame time

- up to 100 frames / sec (ie. flux limited, to be able to take a tomogram in a few seconds)

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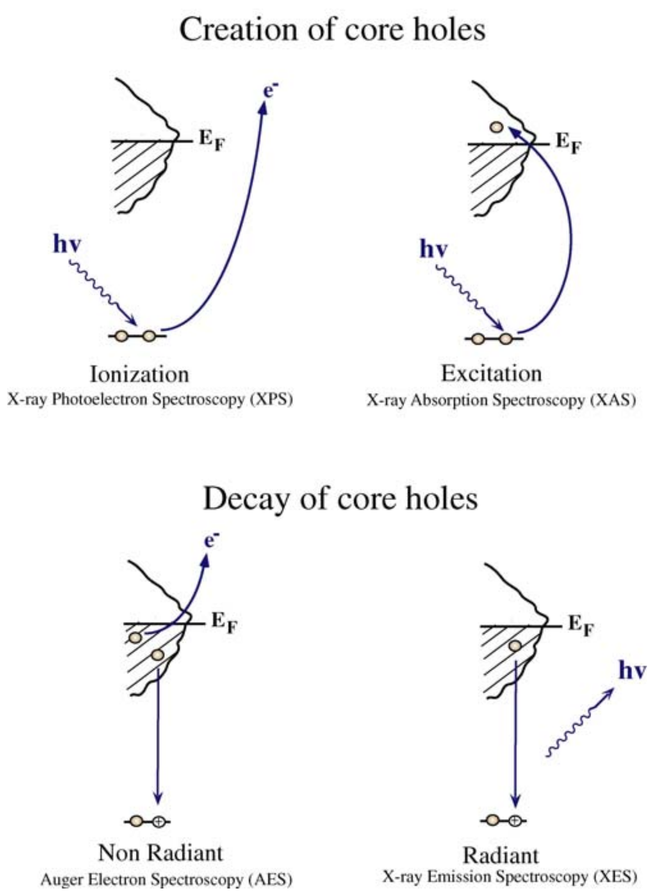
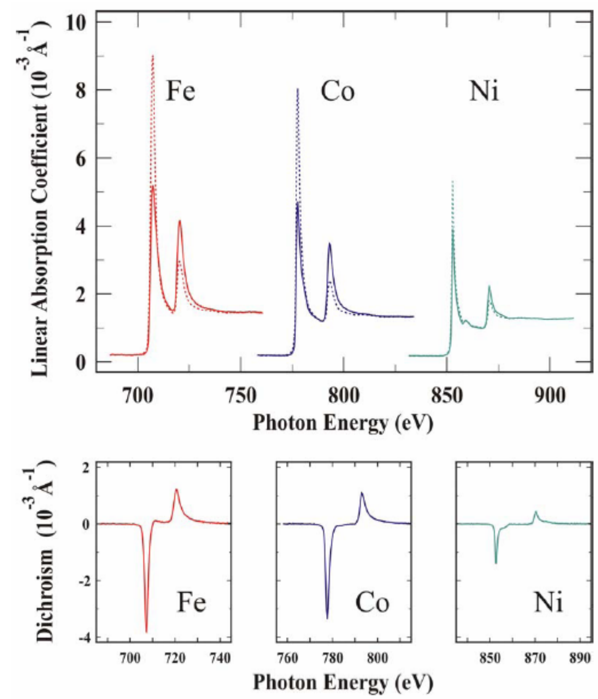
X-ray Spectroscopy



Absorption Spectroscopy



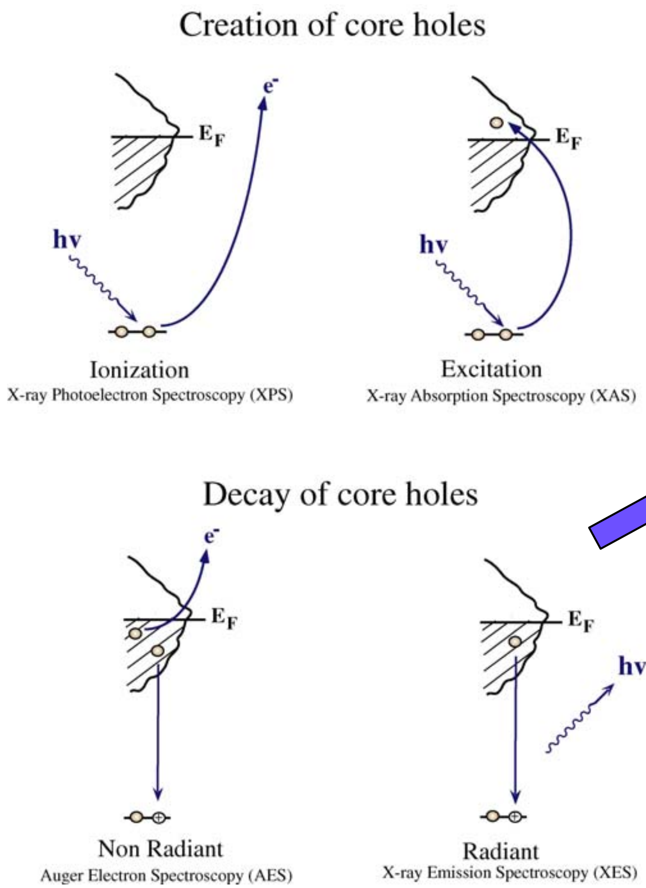
- **Excitation to continuum : EXAFS**
- **Excitation to unoccupied states : NEXAFS**

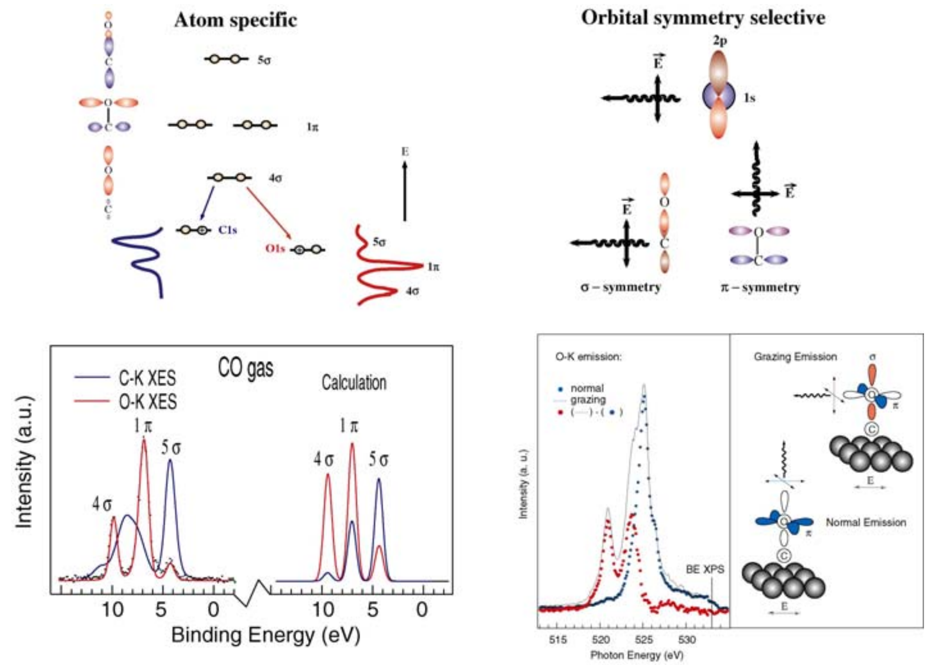
- **Absorption measured through**

  - **direct absorption**
  - **auger decay**
  - **electron yield**
  - **fluorescence decay**

X-ray Spectroscopy

X-ray Emission Spectroscopy



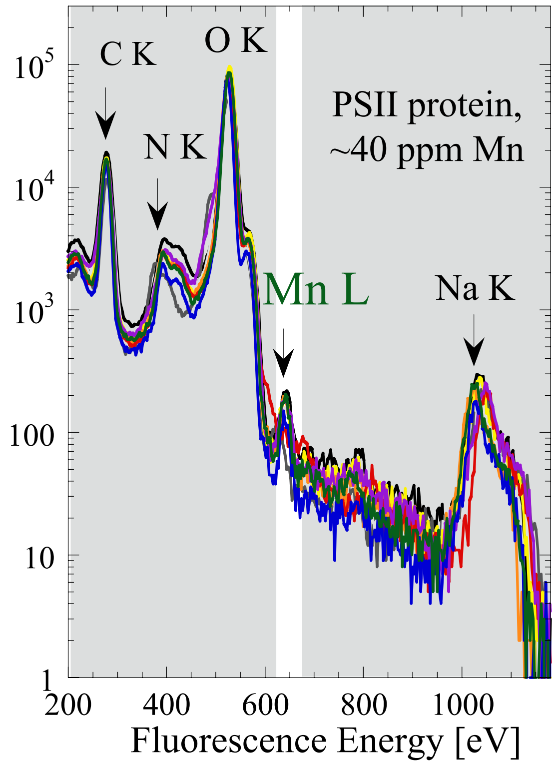


A. Nilsson et.al. J. Electron Spectr. 110-111, 12 (2000)

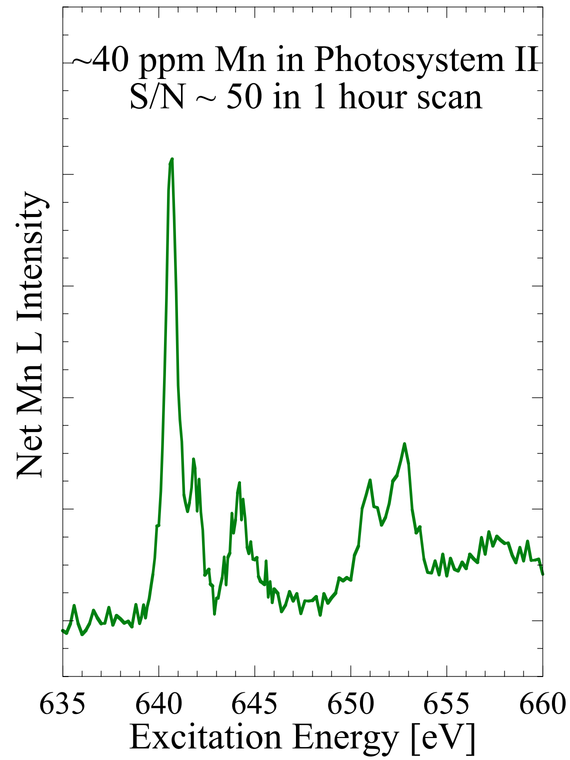
- **excitation of core electron**
- **de-excitation measured by**
  - **integration over fluorescence line, dE ~ 10 eV FY-XAS**
    - **chemical sensitivity from XAS, dE ~1 0eV**
  - **energy resolved fluorescence**
    - **chemical sensitivity from fluorescence spectrum, dE = 0.2 eV**
    - **requires use of a grating spectrograph**

X-ray Absorption with Fluorescence Detection: Required energy resolution

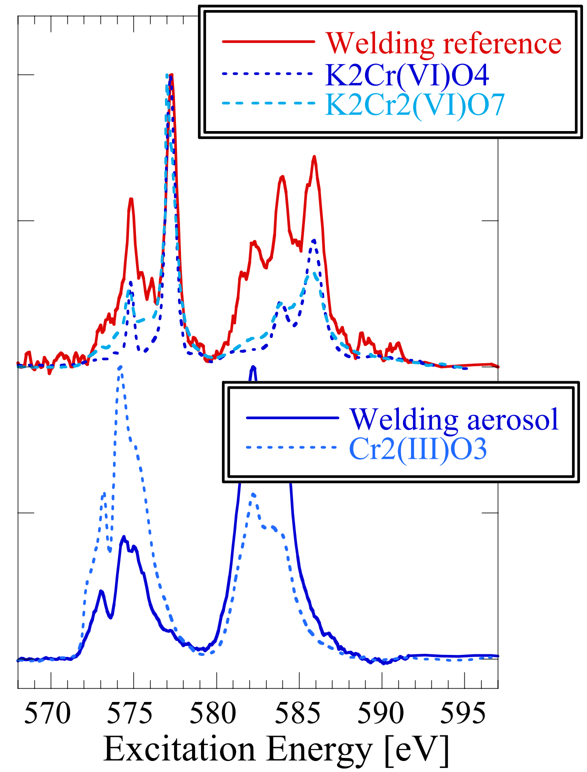
STJ detector response



e.g. Mn in Proteins



e.g. Cr in aerosols

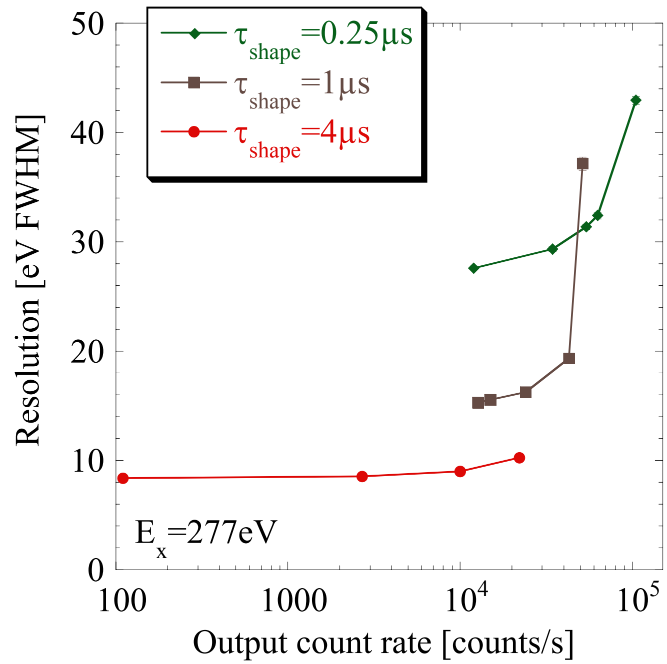


- FY-XAS allows one to look at dilute elements in a complex matrix, eg. metal center in a protein
- Optimal energy resolution is the width of the fluorescence emission spectrum: ~ 10 eV
    - minimises fluorescence background from adjacent elements
- High data rate crucial, in order to be able to handle fluorescence from concentrated matrix

X-ray Absorption with Fluorescence Detection

36 pixels $\Rightarrow$ 1.2 $\times$ 1.2 mm^2 total area
~8 mm from sample $\Rightarrow$ $\Omega/4\pi \approx 10^{-3}$

10 eV FWHM for E < 1 keV
>30,000 counts/s per pixel



-Need is for ~ 10 eV resolution to integrate over fluorescence spectrum (partial integration is a problem!)

- 1e12 ph/sec in, yields ~ 0.5% for low Z matrix, collect 10% of 4 pi = 5e8 ph/sec

- superconducting tunnel junction has right energy resolution, is robust and fast, but….

- need 30 x 30 array….see talk by Stephan Friedrich

Summary

Photoemission electron spectroscopy

- core level angle integrated PES….1d GHz array with CMOS readout….we are there!!
- angle resolved PES….upgrade to fast CMOS or CCD camera may be OK
    - TOF detector….2d, < nsec timing resolution, 2d pixel detector application??

Tomographic microscopy

- coherent x-ray diffractive microscopy CXDI
    - 1e6 dynamic range, small pixels, single photon sensitivity
    - KHz frame rate 10 bit encoded column parallel CCDs may be a solution
- Transmission x-ray tomography
    - 100 Hz frame rate, small pixels, single photon sensitivity
    - 16 bit encoded pseudo column parallel CCD may be a solution

X-ray Fluorescence Spectroscopy

- 10 eV resolution, very high rata rate, large solid angle
    - STJs may be the answer, but need 30 x 30 arrays