Scientific Highlights from 2-ID-B

David Paterson
X-ray Microscopy Group

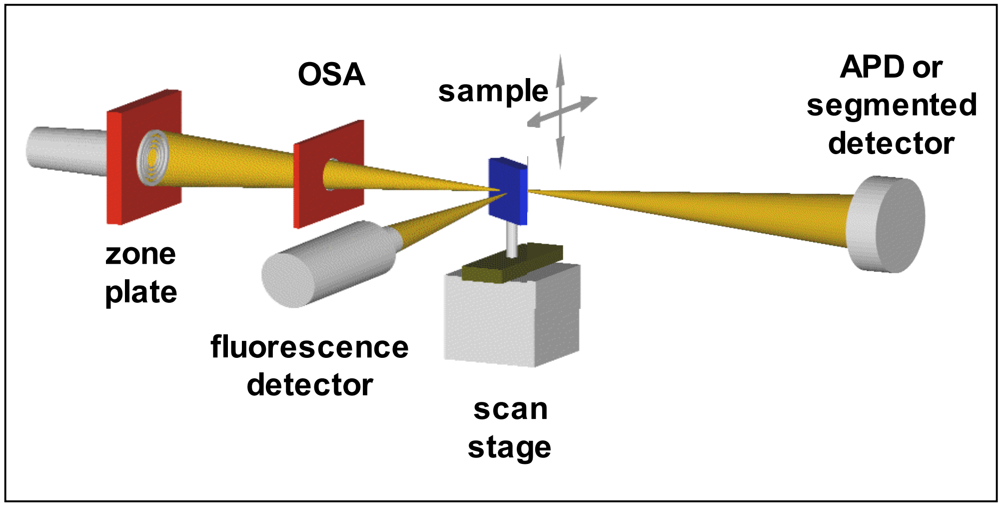
Highest resolution for solving questions on the nanoscale

Frontier science using soft x-rays

Introduction

- Beamline designed to maintain high coherent flux
  - 5.5 cm period undulator 1–4 keV
- Major instruments
  - Scanning x-ray microscope using zone plates
  - Coherent scattering and imaging station
  - Upgrade of monochromator 20X flux at key edges of P, S, Cl and soon Ar K and Ca
- Science highlights
  - Traditionally materials and coherence/phase studies
  - Recently venturing into environmental and biological studies, particularly microspectroscopy or µXANES

Intermediate Energy Scanning X-Ray Microscope



Characteristics

- 2,000 hour/yr
- 1 – 4 keV
- δ = 60 nm
- 10^8 ph/s

Contrast modes

- Transmission
- Fluorescence
- Micro-XANES

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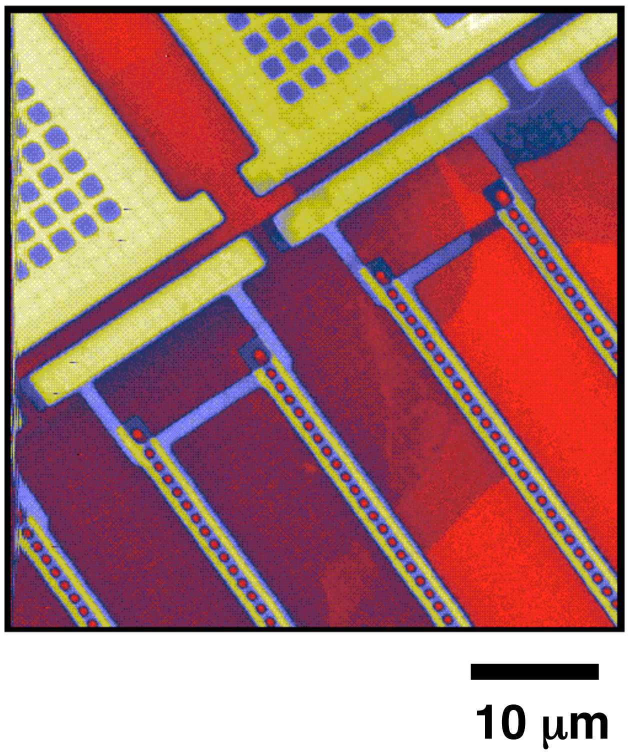
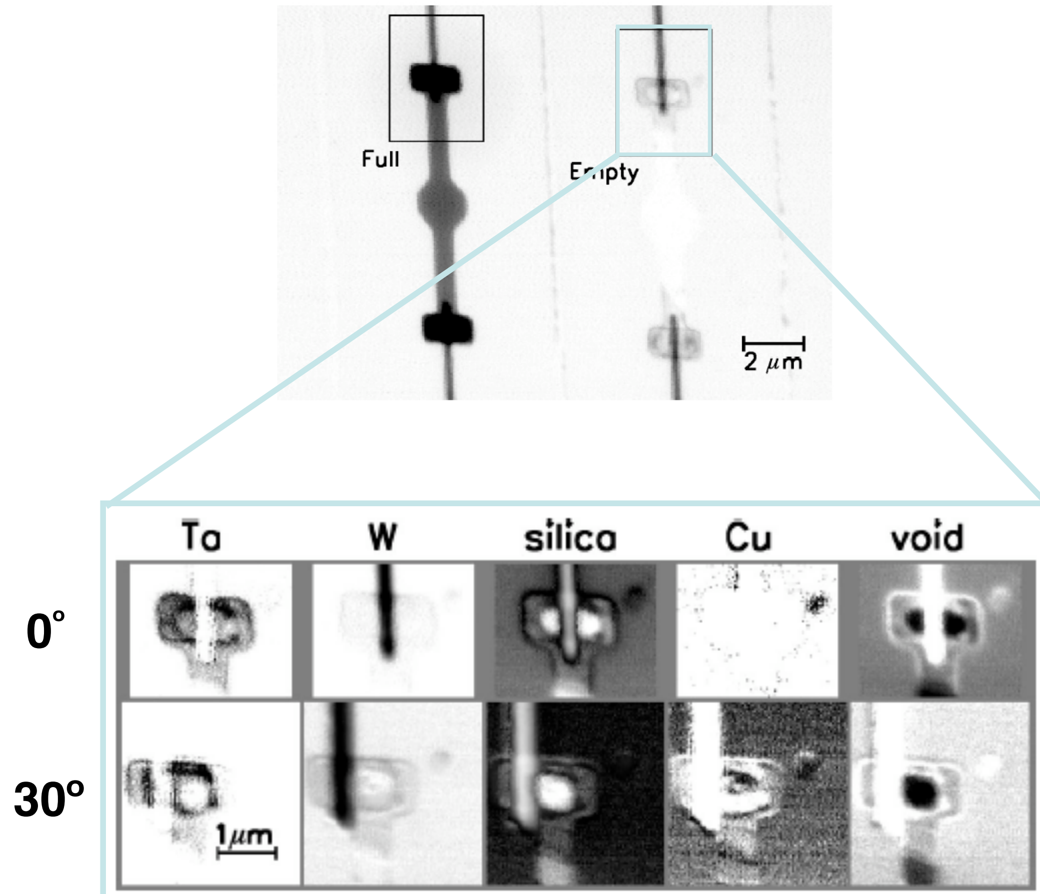
Materials science: defects in buried interconnects



Example: Ta-lined Cu interconnect, W vias



Z. Levine, *et al.*, *J. Appl. Phys.* 95, 405 (2004)

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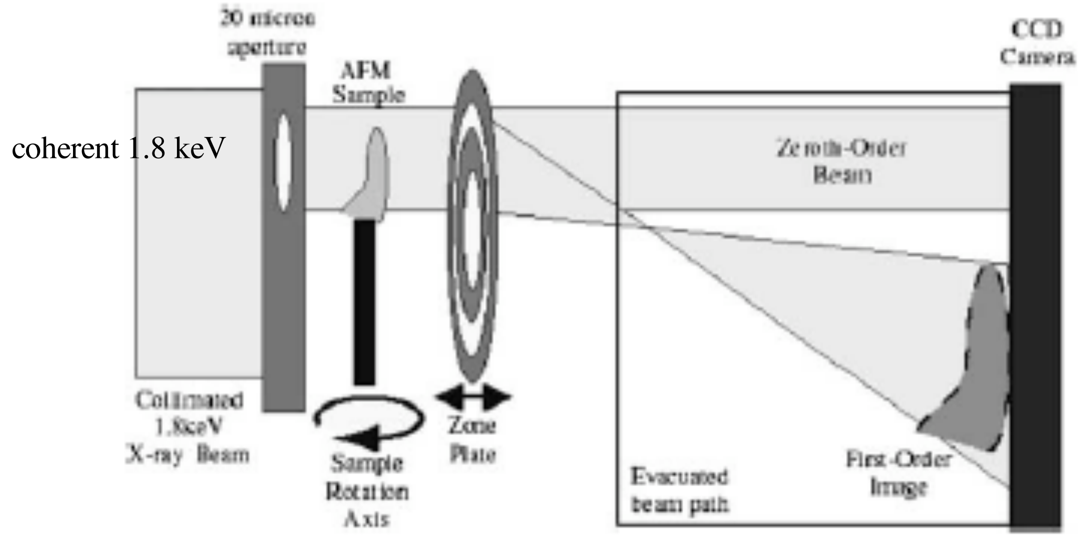
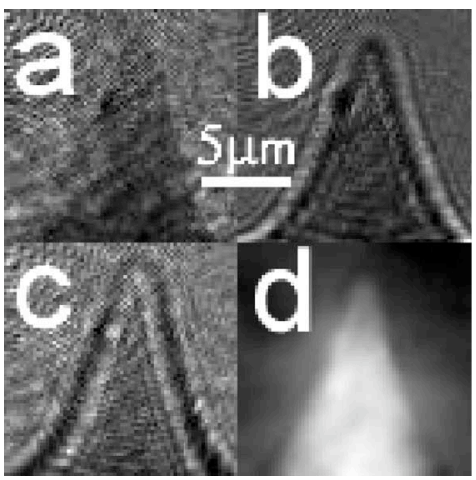
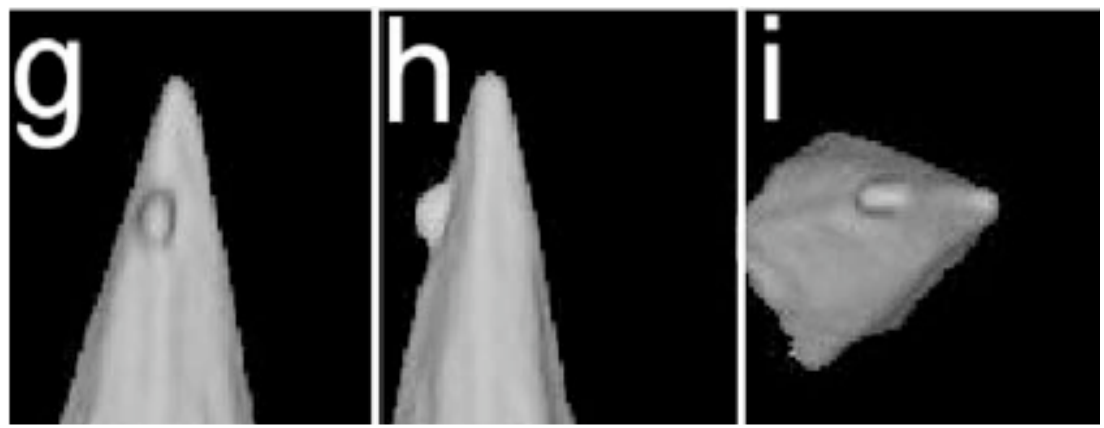
Quantitative Phase Tomography

- Use zone plate for sub-micron resolution
- Quantitatively reconstruct of the real part of the refractive index of an AFM tip
- Use 3 images at different focal positions (a, b, c)







P. J. McMahon *et al.,* Optics Comm. **217**, 15 (2003)

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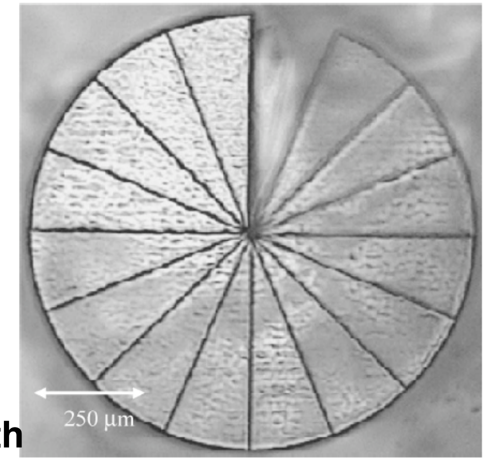
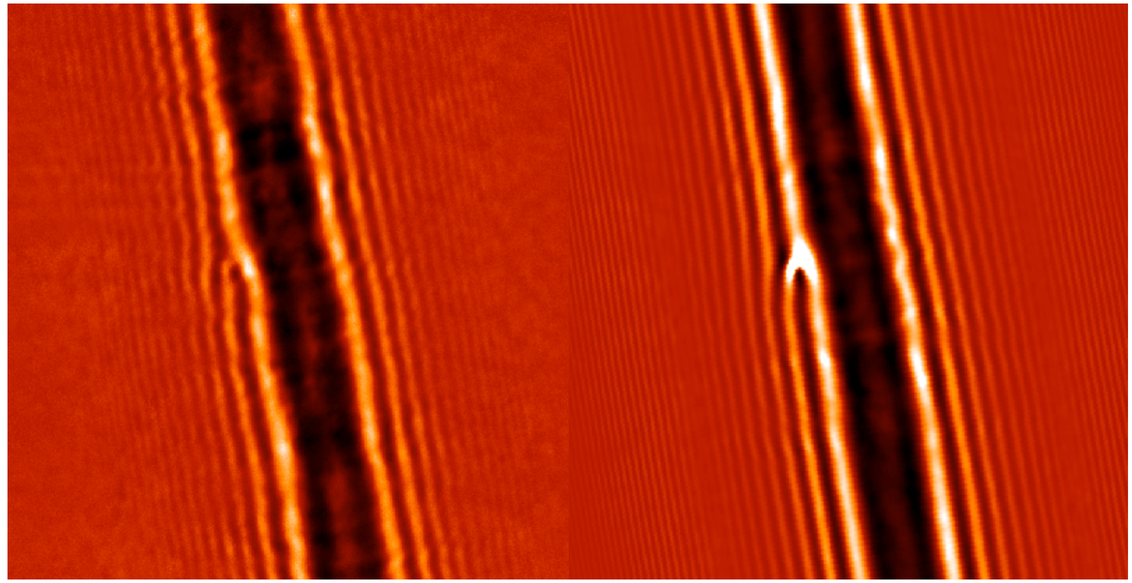
X-ray vortex: observation and manipulation

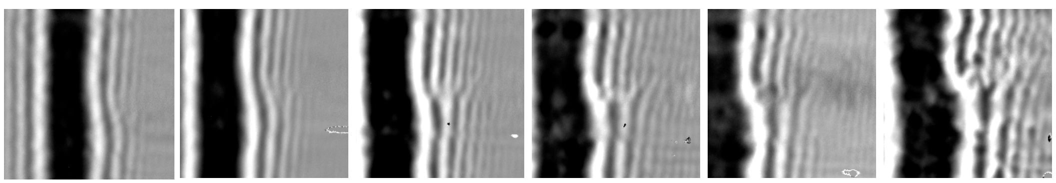


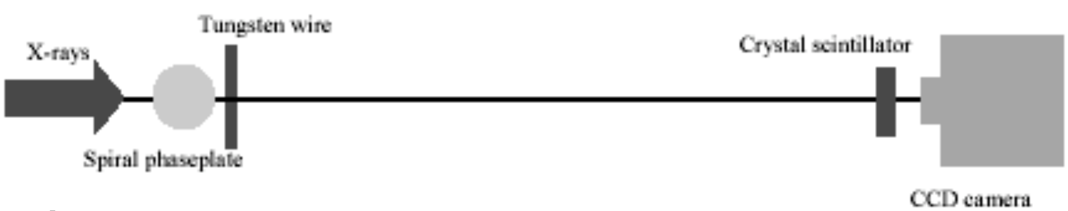
Polyimide
34 μm depth
~2π phase
shift at 9
keV



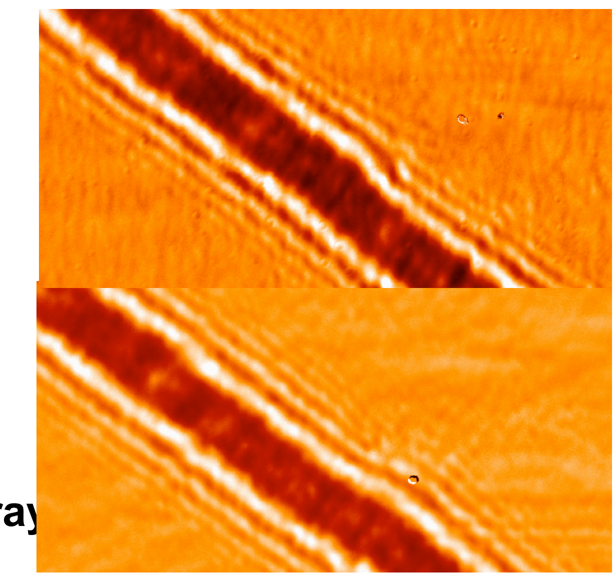
Experiment

Simulation





Experiment geometry



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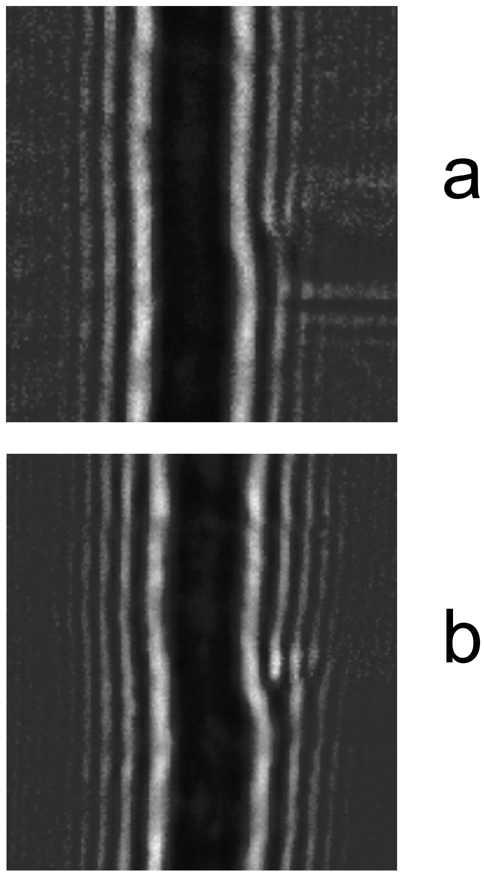
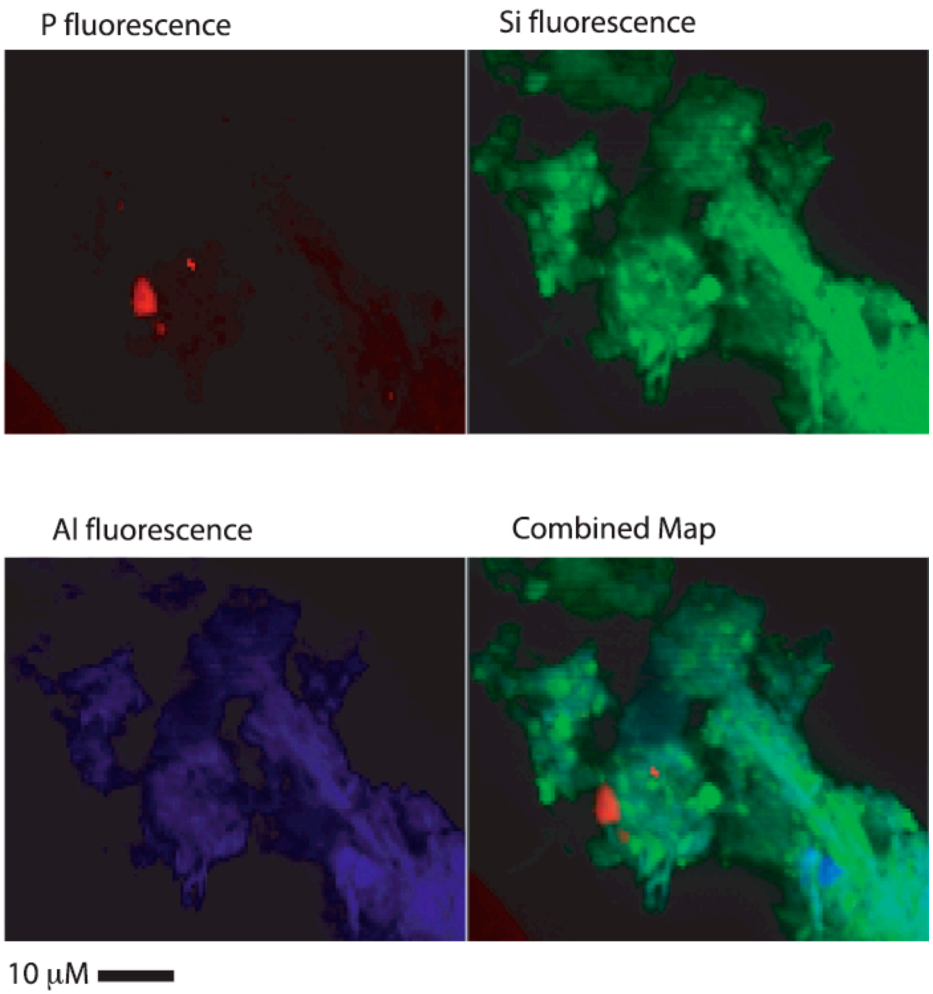
Intermediate energy vortex

- Demonstration of the presence of vortex states of charge 1 and 2
- X-ray energies of 2.13 (0.58 nm) and 1.63 keV (0.76 nm) were used to produce the images shown in (a) and (b)

Peele, *et al.*, Optics Letters 27, 752 (2002)
A. Peele, *et al.*, J. Opt. Soc. A (in press)



Phosphorus fluorescence mapping of particulates



- Absorption mapping of marine organic phosphorus particulates high density regions are not necessarily the P rich
- 2190 eV incident energy
- Phosphorus x-ray fluorescence map to find P hotspots

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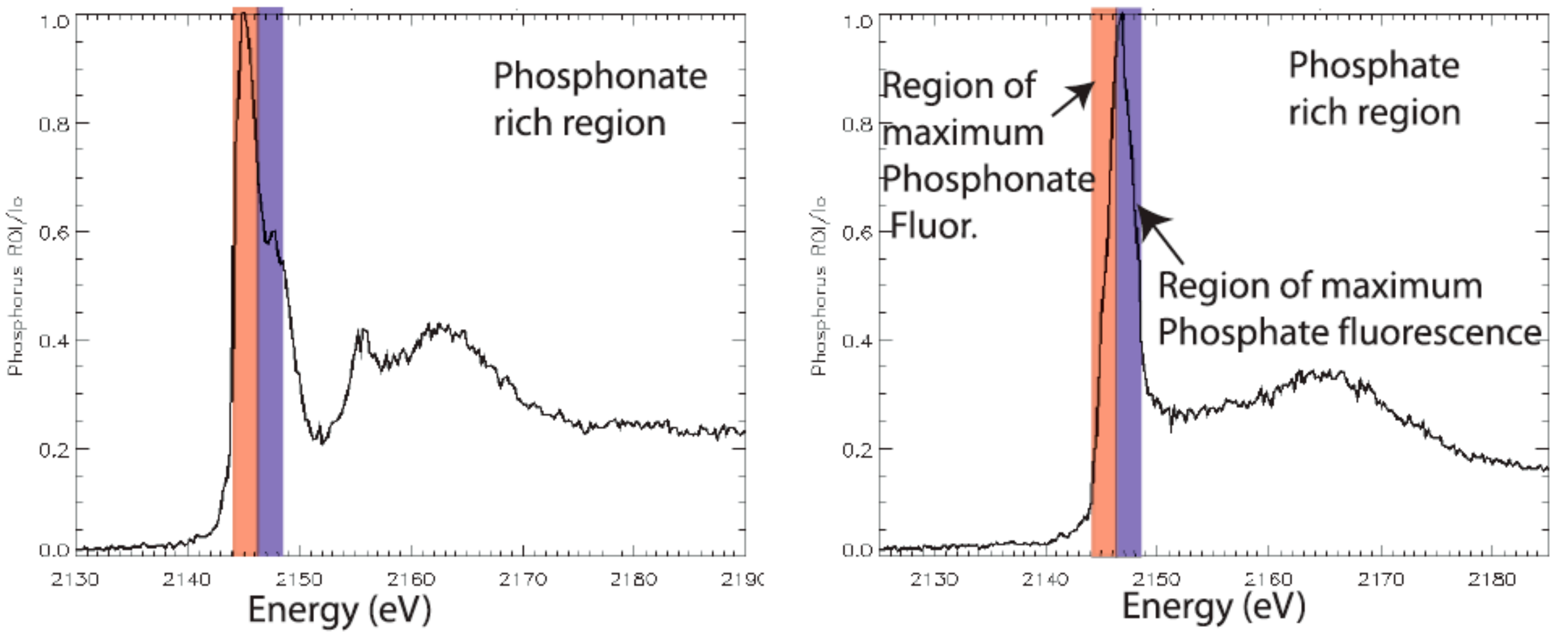
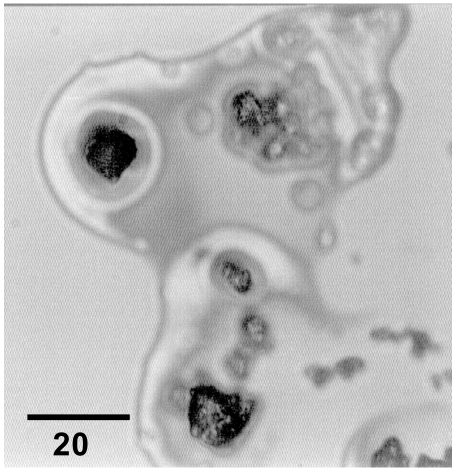
Spectra from submicron regions

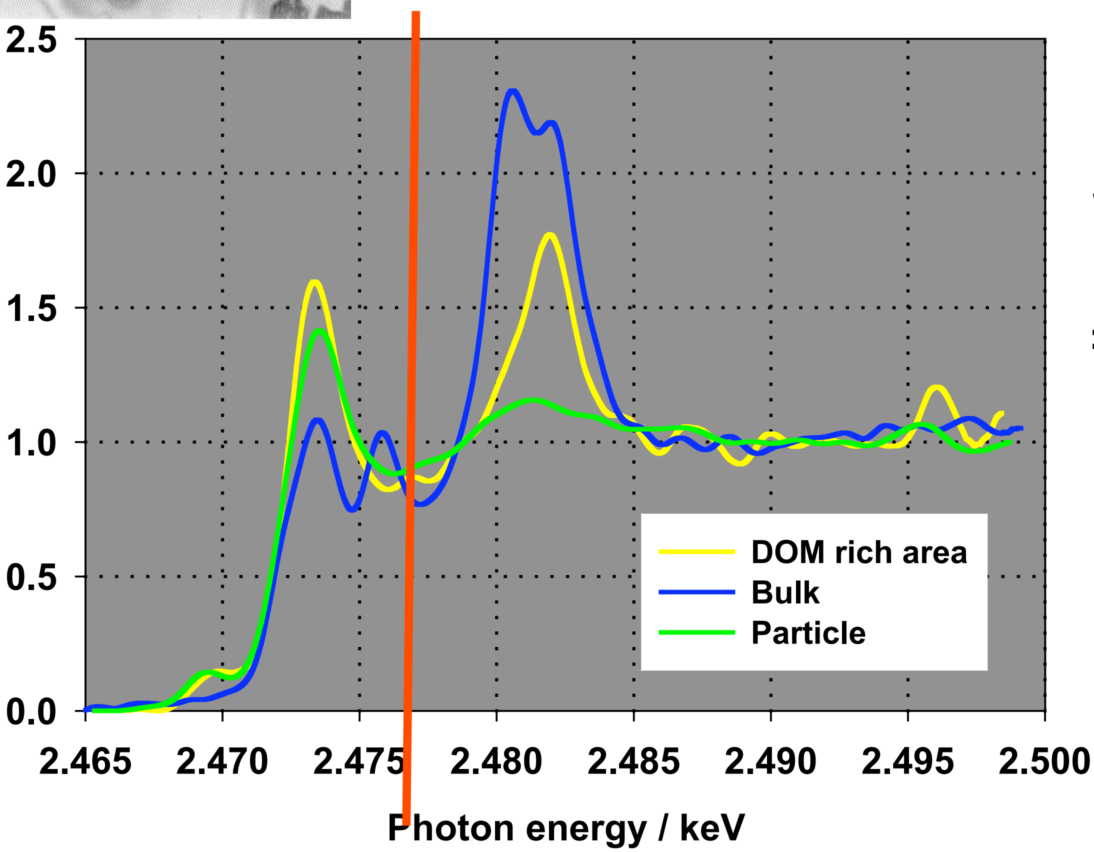


Soil spectromicroscopy

- X-ray spectromicroscopy provides a unique combination of
  - high spectral resolution and
  - high spatial resolution
- It allows for characterization of associations of soil colloids and soil micro habitats regarding
  - structure
  - sulfur speciation
  - differences within and between associations
- Opens up totally new perspectives in soil science



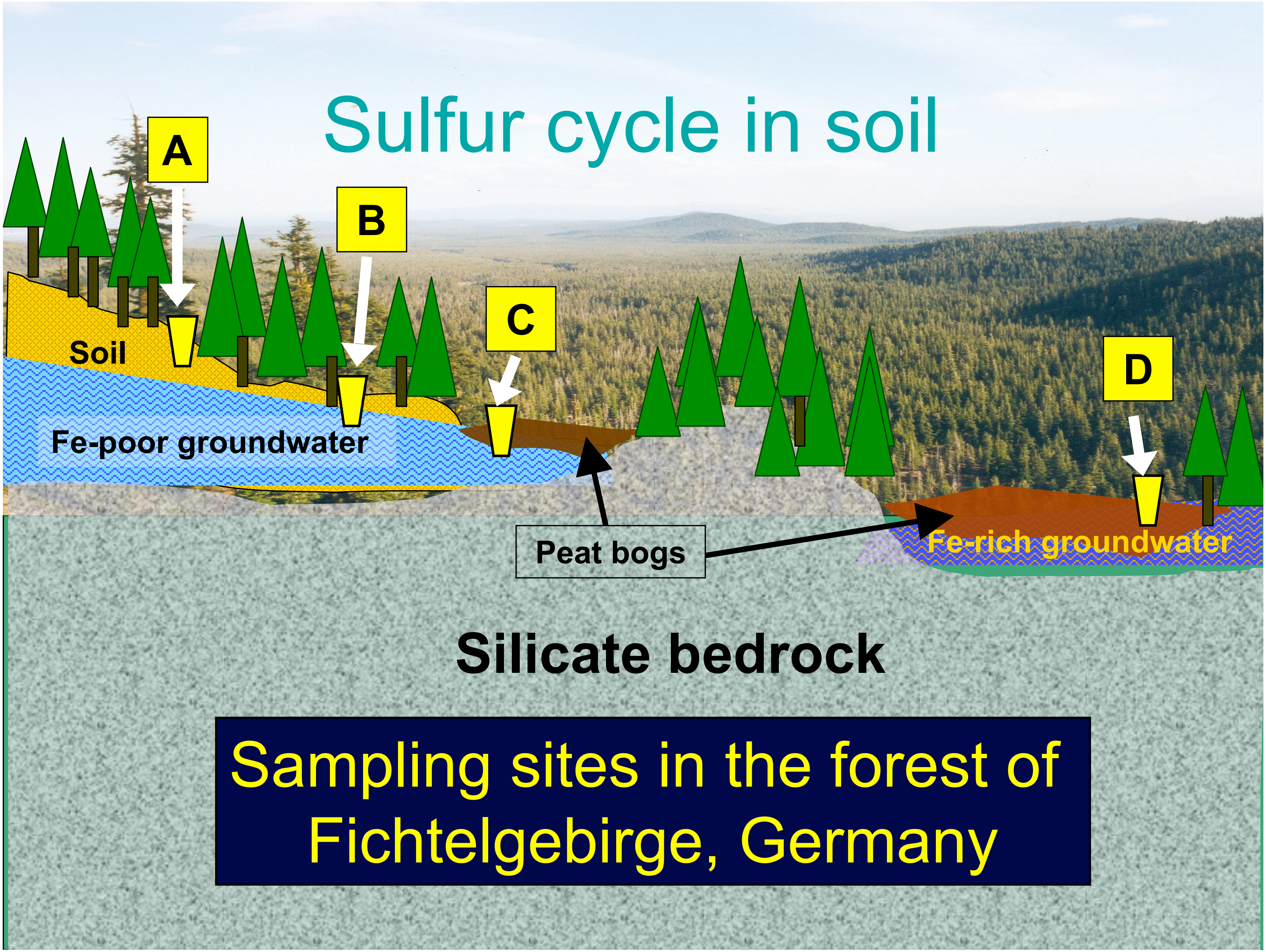
Sulfur distribution in soils



Black Forest soils
Dissolved Organic Matter
Spectrum:
Bulk ≠ Particle
≠ DOM rich area

Chemical analysis:
Integrated
Reduced ⇔ Oxidized

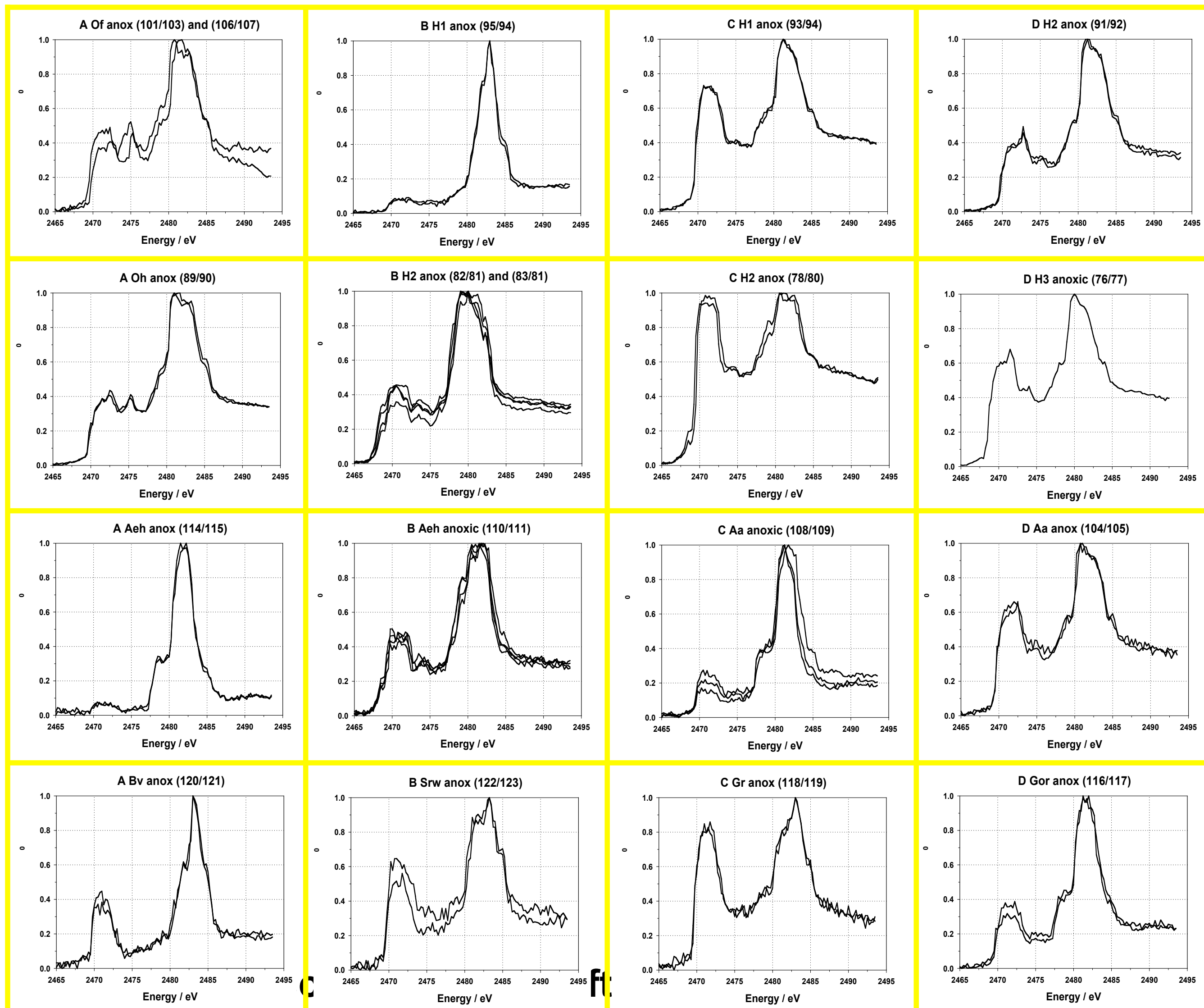
J. Prietzel, J. Thieme, U. Neuhäusler, J. Susin
I. Kögel-Knabner, EJSS 54(2003) 1-11


Sulfur cycle in soil
A
B
C
D
Soil
Fe-poor groundwater
Peat bogs
Fe-rich groundwater
Silicate bedrock
Sampling sites in the forest of Fichtelgebirge, Germany

Bulk spectra variations

Sampling site A B C D

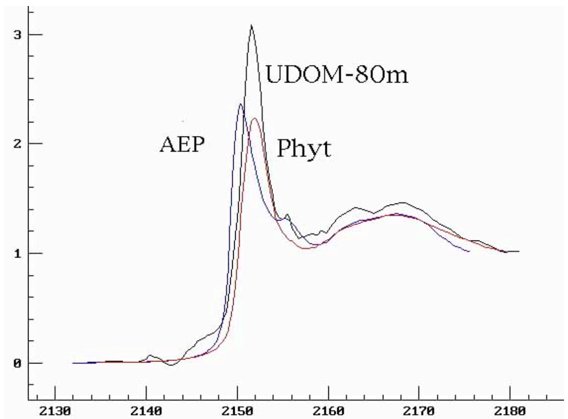
Depth



Summary and future directions

- Highest resolution microscopy studies
  - Transmission
  - Fluorescence
- Microspectroscopy of key elements
- Coherent scattering and imaging
  - Dedicated station

Acknowledgements

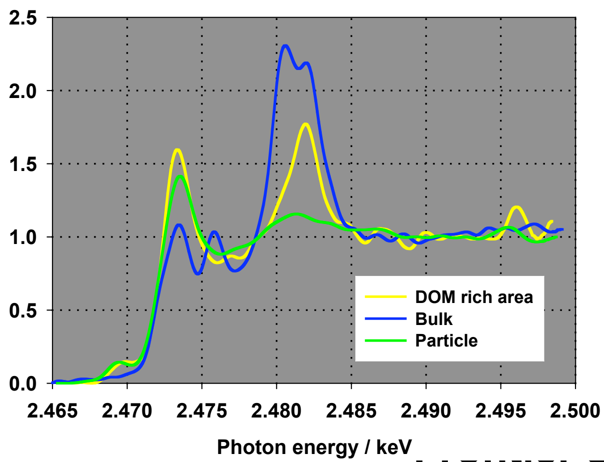


Jay Brandes[1] & Ellery Ingall[2]

[1]University of Texas at Austin

[2]Georgia Institute of Technology

- Examining marine particulate organic matter at sub-micron scales using scanning transmission x-ray microscopy (STXM) and carbon X-ray adsorption near edge spectroscopy (C-XANES) (in Press) Marine Chemistry



Jürgen Thieme[3] & Jörg Prietzel[4]

[3]Institut für Röntgenphysik,Universität Göttingen

[4]Lehrstuhl für Bodenkunde, TU München

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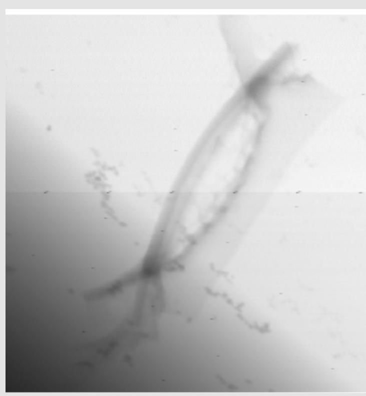
Future plans: scanning microscopy

- **Improved detection schemes**

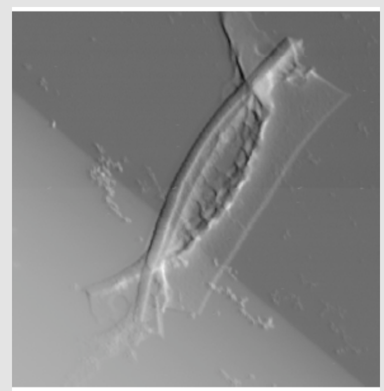
  – Increased acceptance: approach 2π sr
    - Reduce radiation damage
    - Increase throughput
  – Routine acquisition of differential phase contrast
    - Fast overview scan
    - Correlation between elemental maps and cellular structure
    - Quantification of biological mass

Absorption contrast



Differential Phase contrast

10 μm



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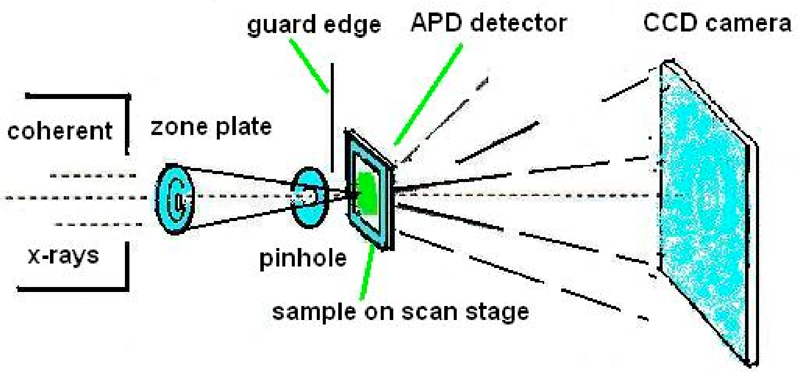
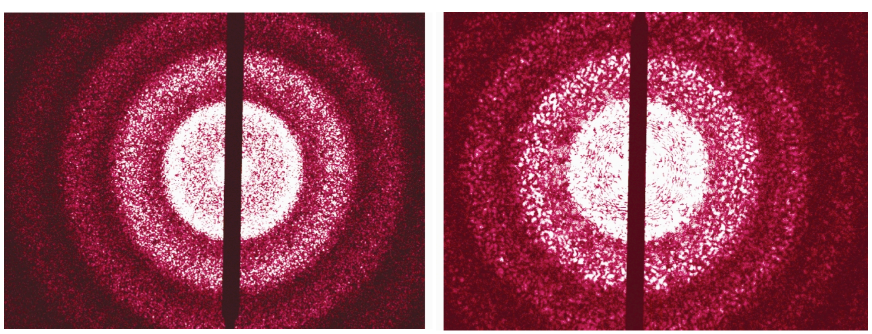
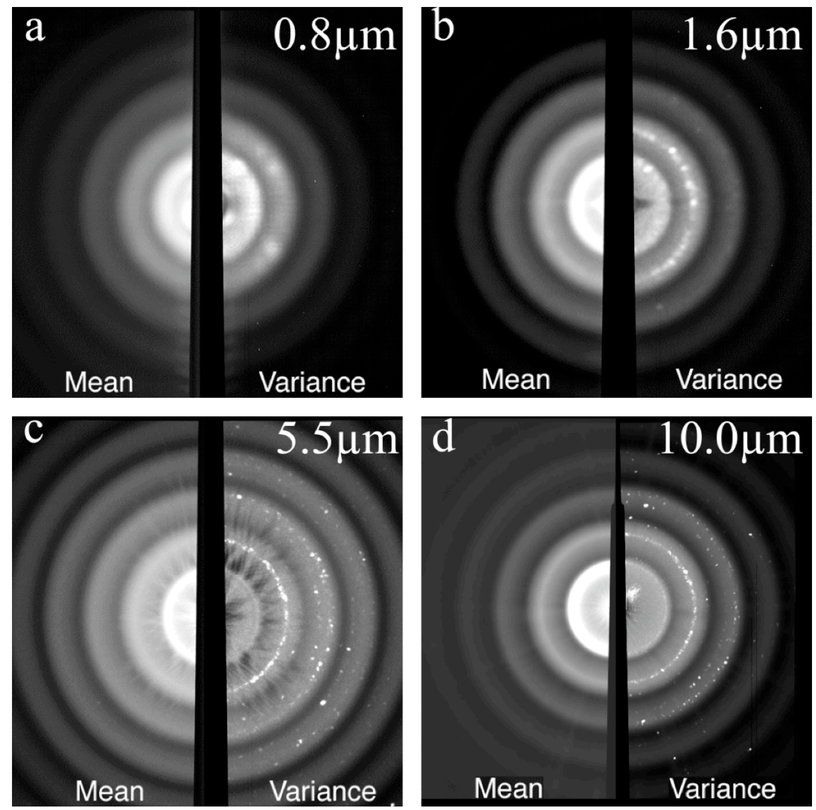
X-ray fluctuation microscopy





Speckle patterns produced by polystyrene latex spheres with 1.83 keV X-rays defined by 5.5-μm (left) and 10-μm (right) pinholes



Mean and variance from 3600 speckle patterns of a film of latex spheres 277 nm diameter with various coherent illumination sizes

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Scanning Microscopy Modalities

- Transmission
  - Fast (~msec/pixel)
  - Elemental contrast around absorption edge
  - Differential phase contrast for light elements

- Fluorescence
  - High trace element sensitivity (10^{-18} gram for Zn)
  - Quantitative
  - Spectroscopy → Chemical state

- Diffraction
  - High throughput/μm-resolution structural analysis
  - High resolution: min. sampling volume ~ 10^{-5} μm^3

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2-ID-B SXM specifications

Zone Plates

Material		Au	Au	Au	Ni	Ni	
Radius		38.5	40	80	45	49	μm
Central stop radius		(none)	(none)	(none)	(none)	20	μm
Zone thickness		420	450	450	110	130	nm
Finest zone width		100	45	50	45	40	nm
Transverse resolution		122	55	61	55	49	nm
Focal length	(1.83 keV)	11.4	5.3	13.3	6.0	5.7	mm
Depth of field	(1.83 keV)	72	11	25	15	12	μm
Efficiency	(1.83 keV)	20	18	18	2.5	3.0	%

Sample Stage (XYZθ)

	Coarse	Fine	
Linear range	±25	±0.1	mm
Linear resolution	500	0.8	nm
Linear velocity	2	20	mm/s
Angular range	360	360	degrees
Angular resolution	0.001	0.14	degrees
Max scan speed	25	1	ms/pixel

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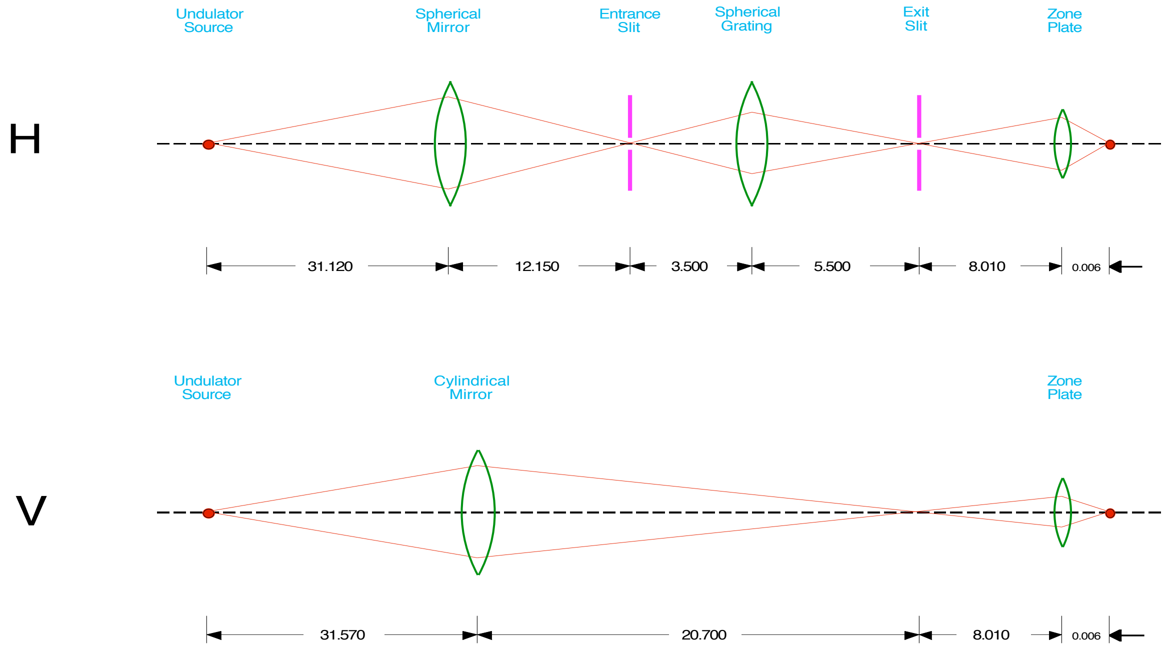
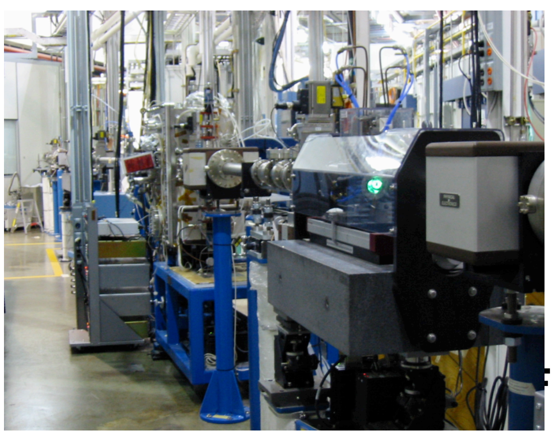
2-ID-B beamline performance

Monochromaticity	**~1000 typical, > 3000 peak**	
Coherent area	**50 μm × 50 μm**	
Coherent flux	**2×10^5 ph/μm^2/s/0.1% BW**	
Focused flux	**4×10^7 ph/s/0.1% BW**	**50 nm spot (ZP40)**
	2×10^8 ph/s/0.1% BW	**60 nm spot (ZP50)**
	2×10^8 ph/s/0.1% BW	**150 nm spot (ZP100)**

2-ID-B intermediate-energy beamline





Environmental Science techniques

- Scanning fluorescence x-ray microscopy and microspectroscopy (μ-XANES) will be the workhorse probes
- Microtomography, including fluorescence methods, will provide complimentary information in the third dimension.
- Intermediate energy x-ray probes with access to the K edges of the light elements, combined with hard x-ray probes accessing the heavy metals, will become increasingly important.
- Improvements in the throughput and spatial resolution will be crucial to capitalize on the opportunities ahead.

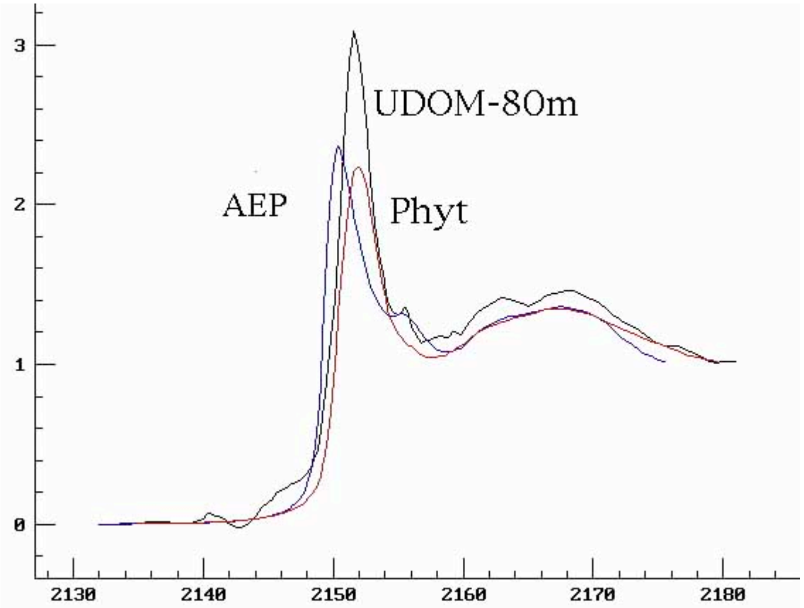
Phosphorus speciation in marine environment

- Phosphorus is a vital nutrient sustaining primary productivity in the world's oceans but little is known about the speciation in particulate and dissolved organic matter
- Presence or absence of oxygen has a marked effect upon phosphorus release from particulate phases and NMR studies have indicated that marine organic phosphorus consists of a mixture of phosphates (C-O-P bonding) and phosphonates (C-P bonding)
- Distribution of each phosphorus type is highly variable
- Use P-XANES with transmission and fluorescence x-ray microscopy of phosphates in a suite of natural samples
- Mapping is essential to separate the variety of phases heterogeneously
- New model of marine phosphorus cycling that takes into account micro-scale heterogeneity and speciation

Phosphorus speciation in marine organisms and particulates

Phosphorus speciation spectra:

AEP = 2-aminoethylphosphonate
Phyt = Phytic Acid
UDOM = Ultrafiltered Dissolved marine Organic Matter



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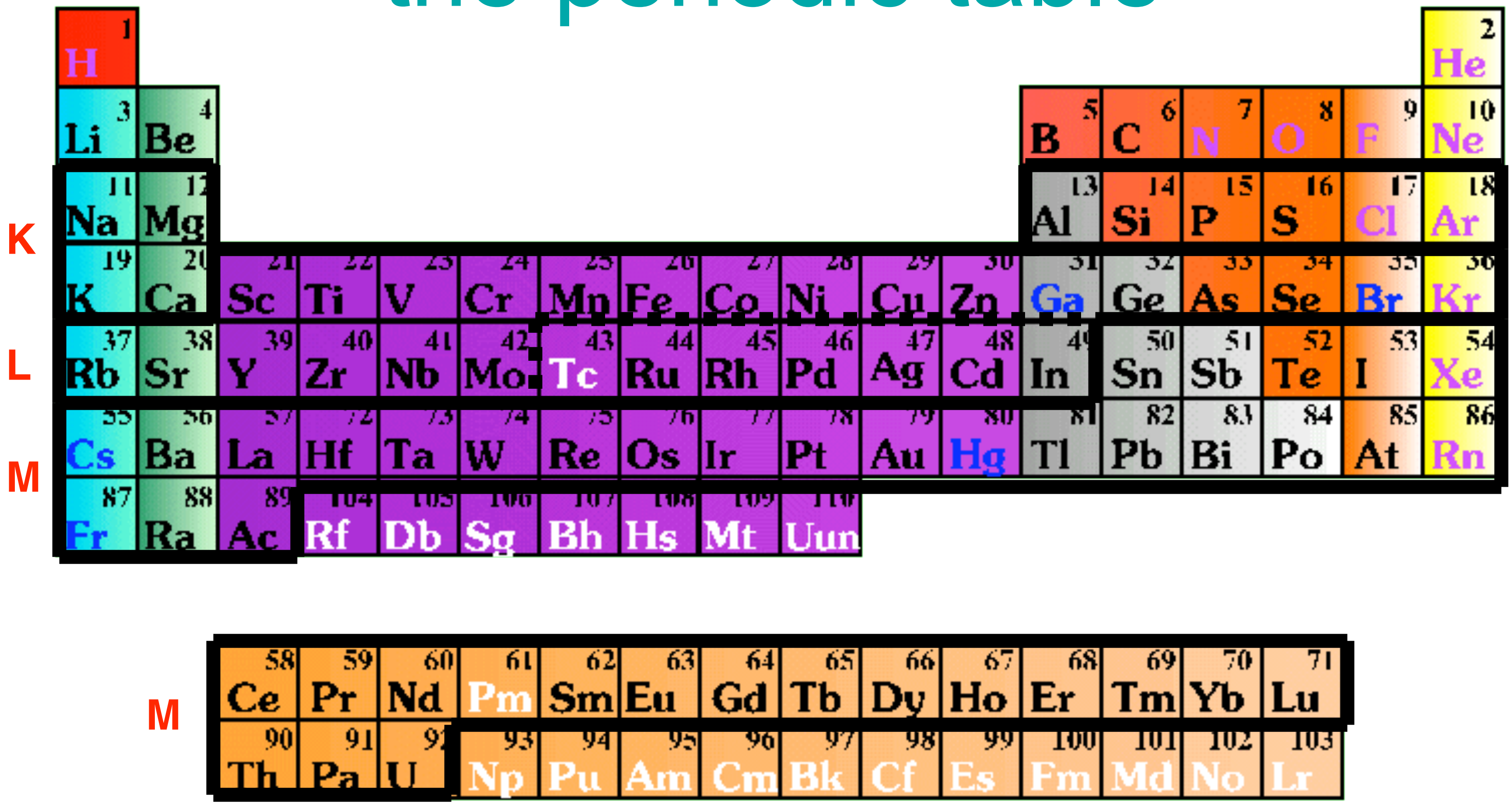
1–4 keV: access to most of the periodic table



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