Imaging in Earth, Planetary and Environmental Science

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Outline of Talk

- Overview of imaging and microanalysis needs in earth and planetary science
- Example applications:
  - Absorption microtomography
  - Fluorescence microtomography
  - Microspectroscopy mapping
- Future Goals and Needs

Earth and Planetary Materials

- Heterogeneity ranges from km to nm scale.
  - There can be a scale below which material is homogeneous (e.g. 10 micron mineral crystal), but not always.
  - Somewhere between material science and biology in complexity.
- Natural systems have large variability
  - Analyzing 1 or 2 samples is rarely sufficient
- Geochemically or environmentally important elements may be present at very low concentrations
- Oxidation state and coordination environment are very important for geochemical and environmental applications

Earth and Planetary Materials

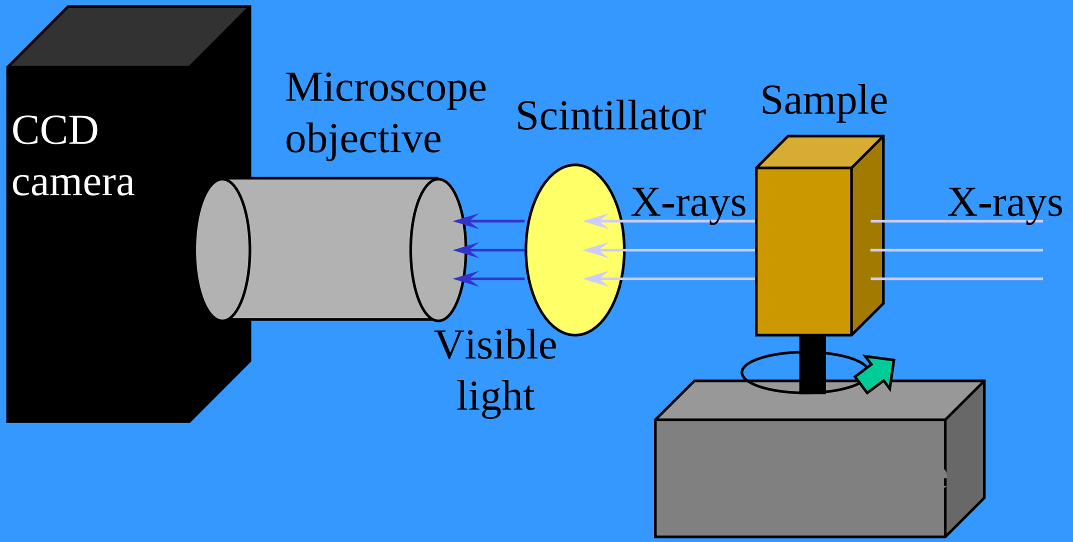
- Extreme environments
  - High temperatures of deep earth, volcanic melts, etc.
  - High pressure of deep earth
- Real-time imaging
  - Studies of dynamic processes
- Technique requirements
  - Rapid: analyze many samples; dynamic processes
  - Sensitive: measure low concentrations
  - Chemically specific: measure valence, coordination state
  - High-energy: penetrate high-pressure, high-temperature cells

Techniques for Obtaining Chemical Composition Using Microtomography

- Simple absorption tomography, image of linear attenuation coefficient, μ
  - Requires a priori knowledge of the sample, e.g. high-Z minerals are magnetite, etc.
- Differential absorption, imaging above and below an absorption edge
- X-ray fluorescence microtomography

Absorption Tomography Setup

- X-ray Source
  - Parallel monochromatic x-rays, 8-80 keV
  - APS bending magnet source, 20 keV critical energy
  - 1-50mm field of view in horizontal, up to 6 mm in vertical
- Imaging System
  - YAG single crystal scintillator
  - 5X to 20X microscope objectives, zoom lens
  - 1300x1100 pixel CCD camera

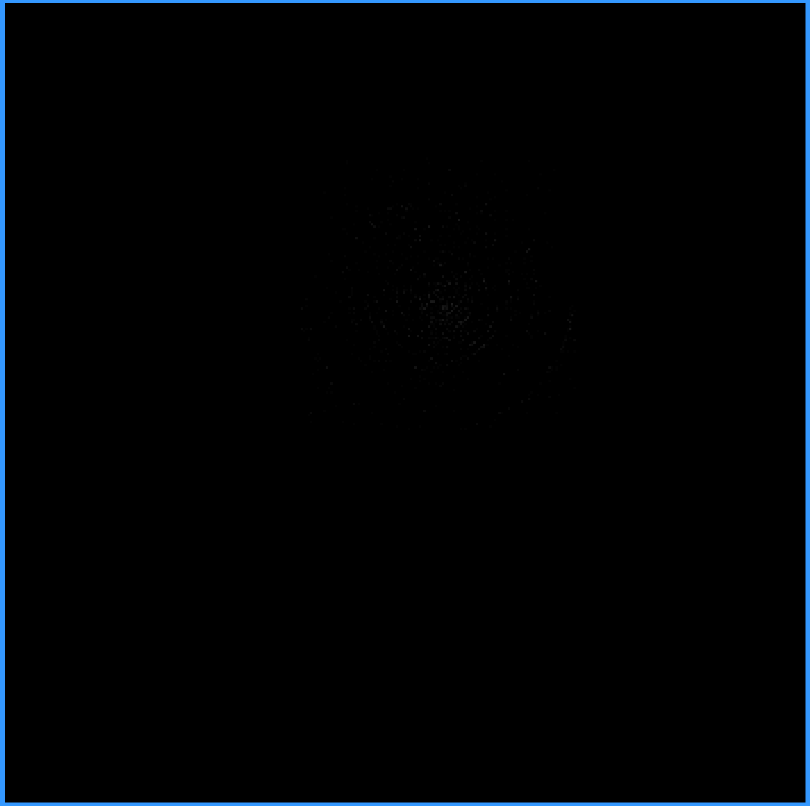


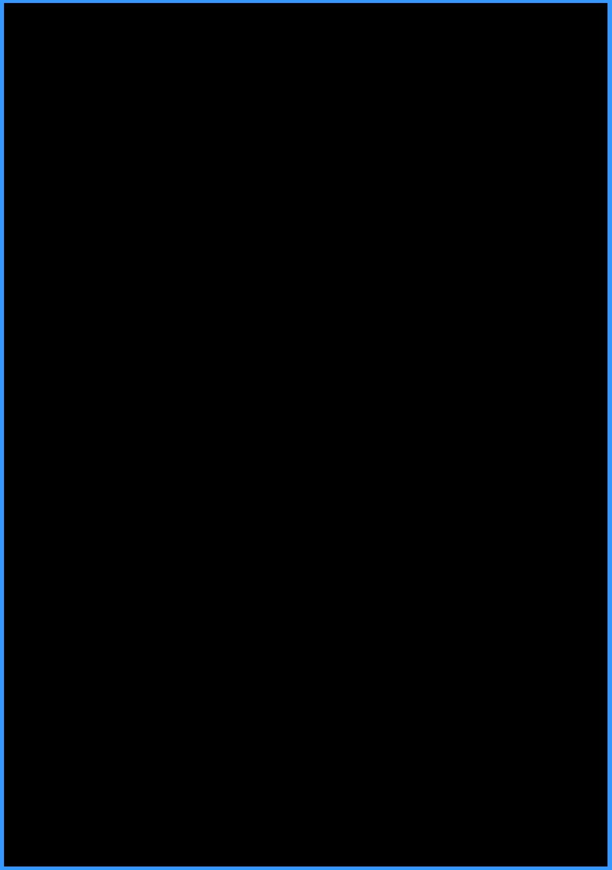
Simple Absorption Tomography

- Fast
  - Typically 360 or 720 projections to create a 650x650x520 voxel image
  - 10-60 minutes
- Data intensive
  - 650x650x520 volume is about 500MB
  - Users collect 50GB of data in 2 days
- Challenge is to extract quantitative numbers from the data
  - Porosity, permeability, etc.

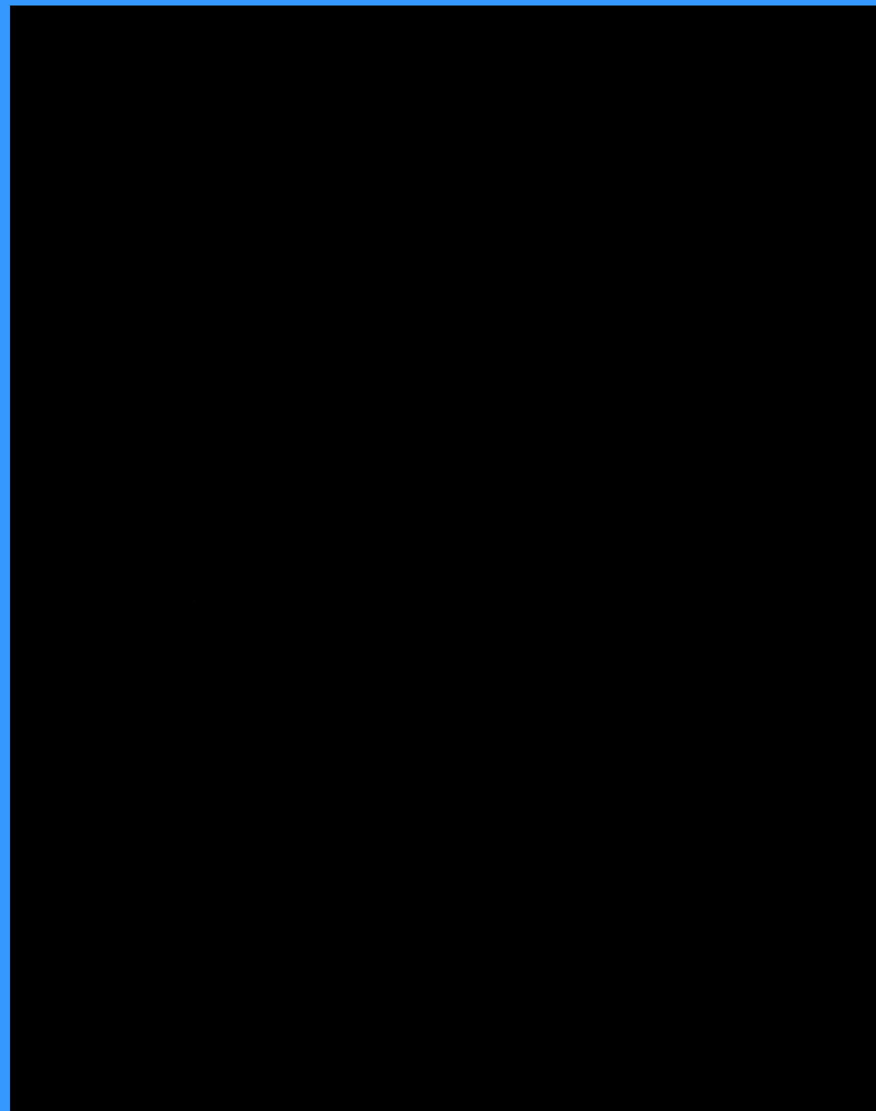
Eocene snail fossil, 20 mm tall

X direction



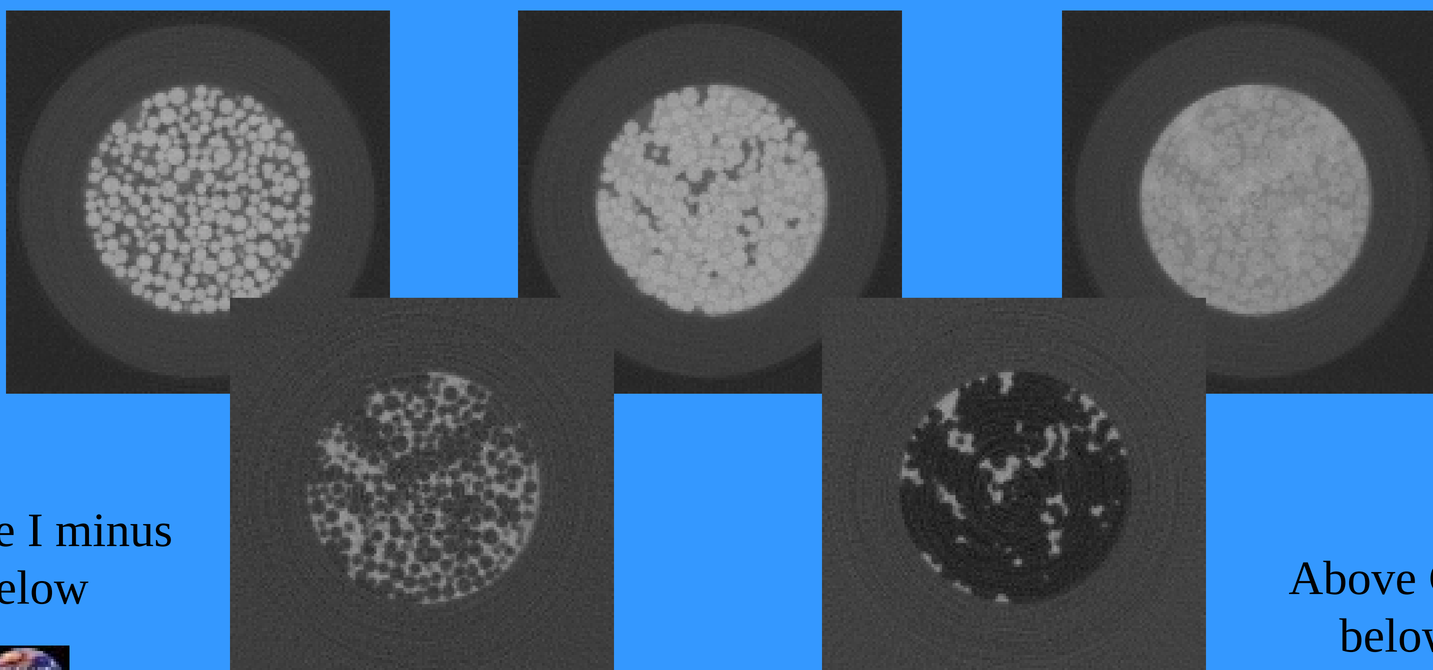


Metereorite with shock features



Differential Absorption Tomography

- Collect 2 absorption data sets, above and below the absorption edge of the element of interest – also fast
- Requires a substantial change in linear attenuation coefficient due to element of interest
  - Major elements, not trace elements
- Example: Sand with I bearing oil, Cs bearing water

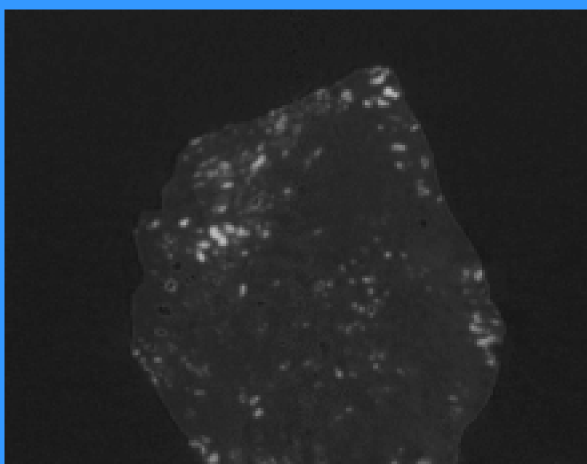


Above I minus below

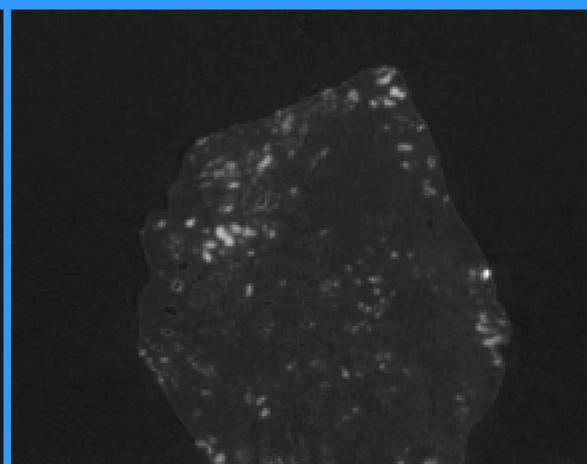
Above Cs - below

Carbonado diamonds – looking for zircons above and below Zr K edge

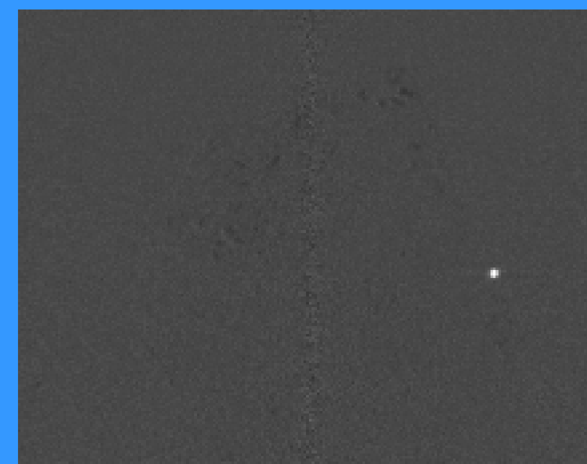
Zircon is used for dating – need to find crystals in a very hard, opaque matrix



100 eV below Zr K absorption edge



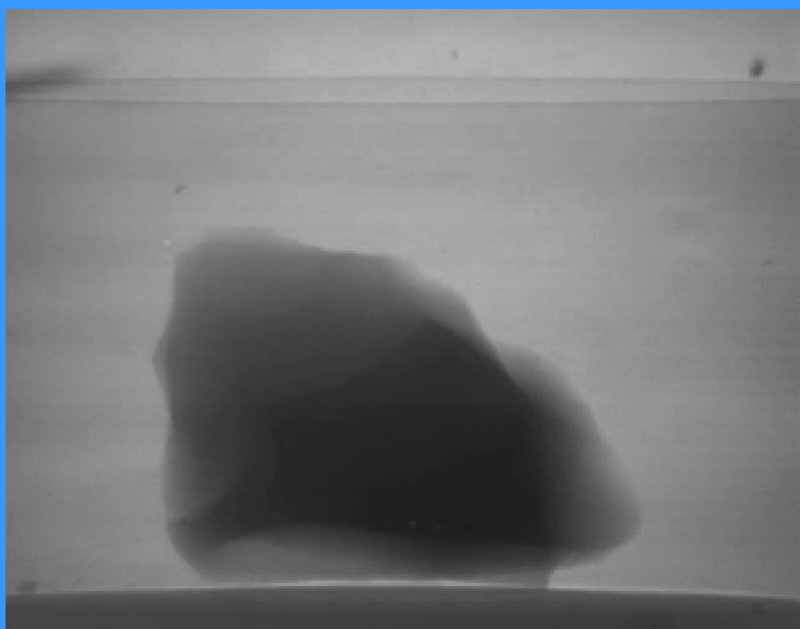
100 eV above Zr K absorption edge

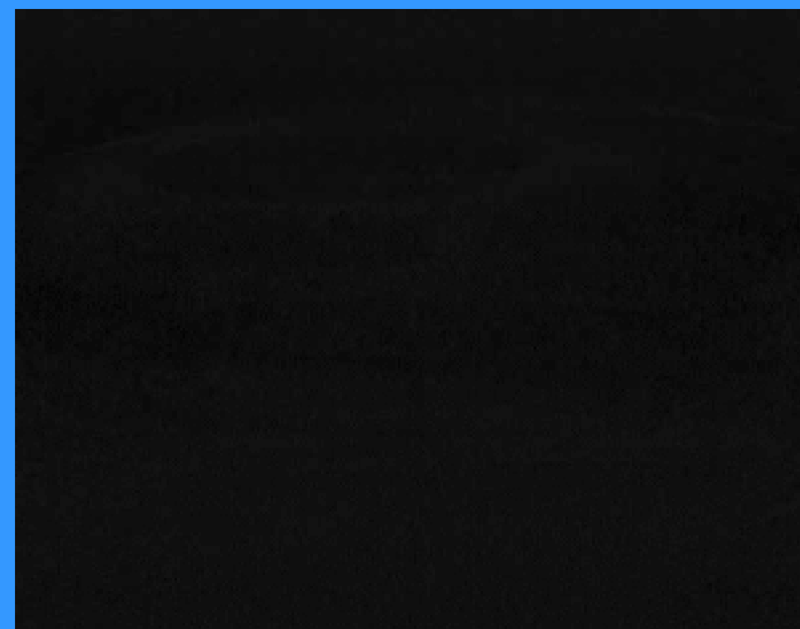


Difference of above and below edge images

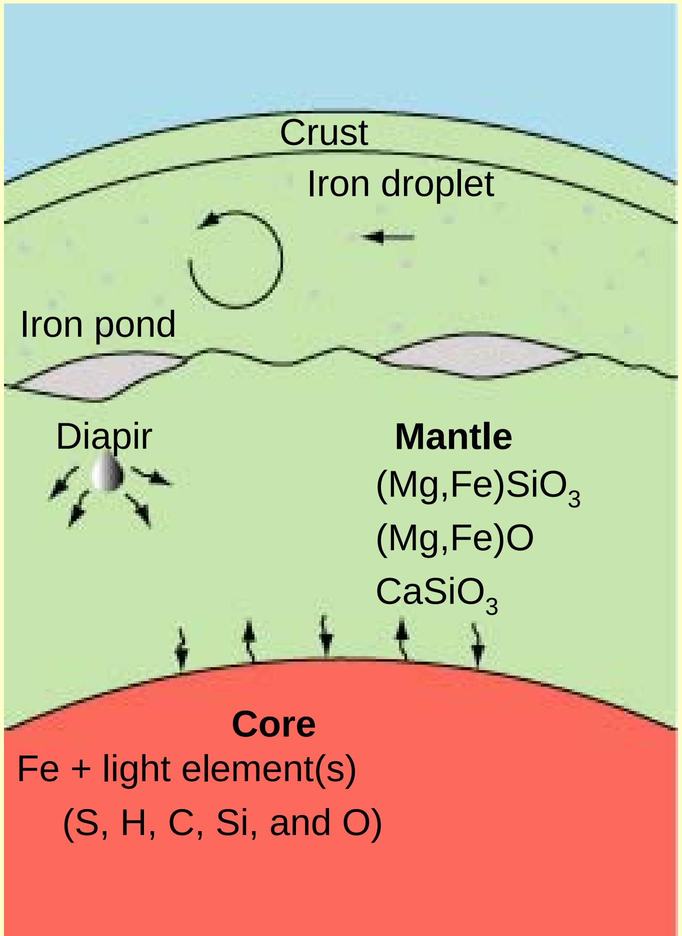
Bubble formation in granitic melts

Bubble formation (foaming) is what drives explosive volcanic eruptions. Want to understand kinetics and composition dependence.





Tomography at High-Pressure

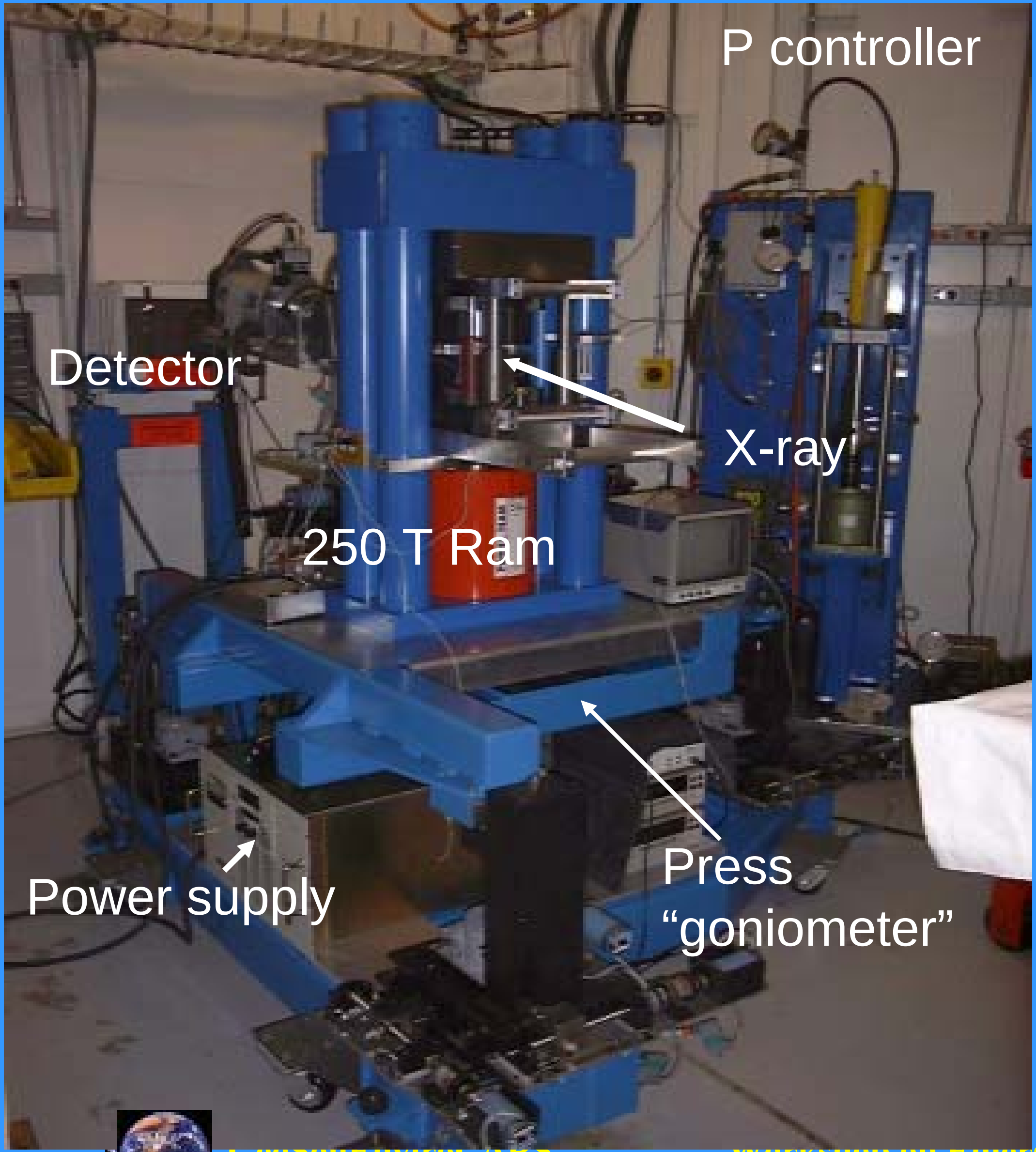


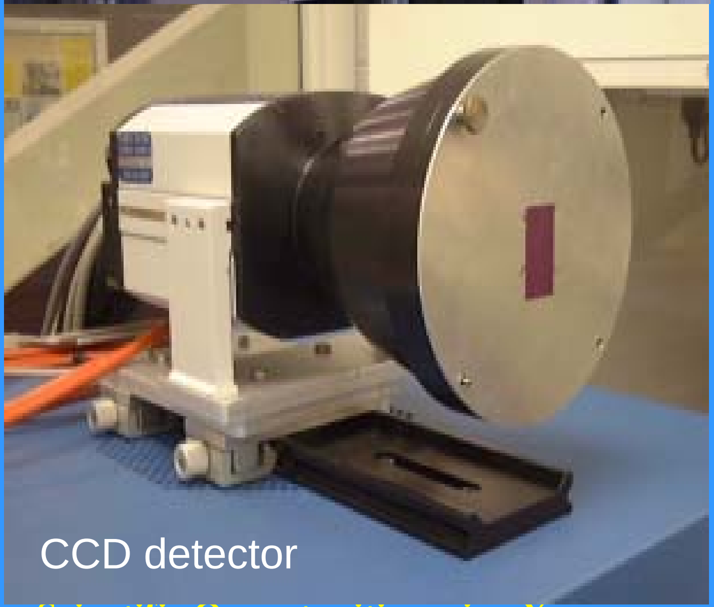
Modified from Stevenson [1990]

Issues concerned with the Earth's Core:

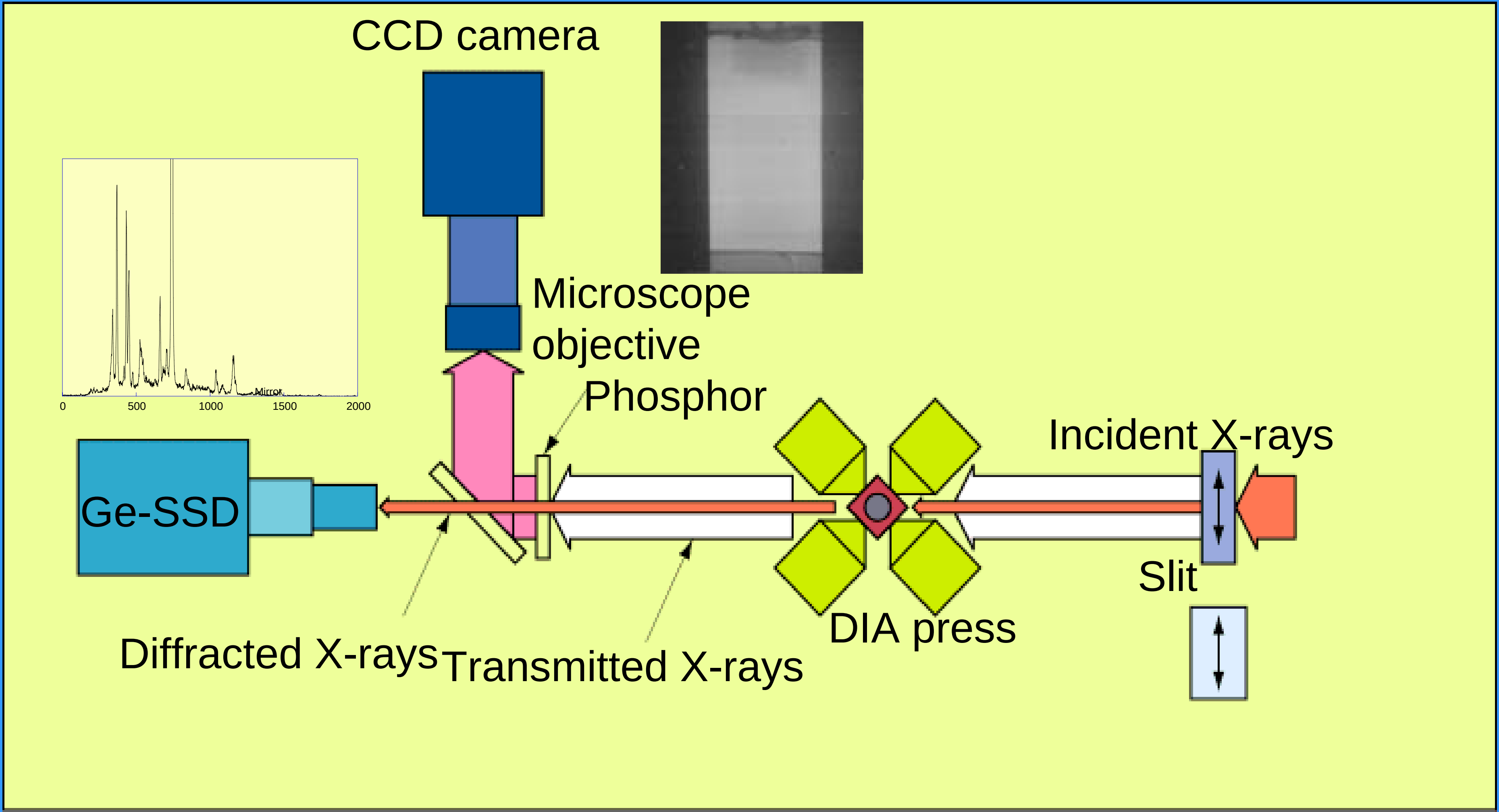
- Composition
  - Density of Fe alloy
  - Potential light elements
- Formation of the core
  - Fe segregation from silicates
  - Interaction during core formation

High Pressure Instrumentation

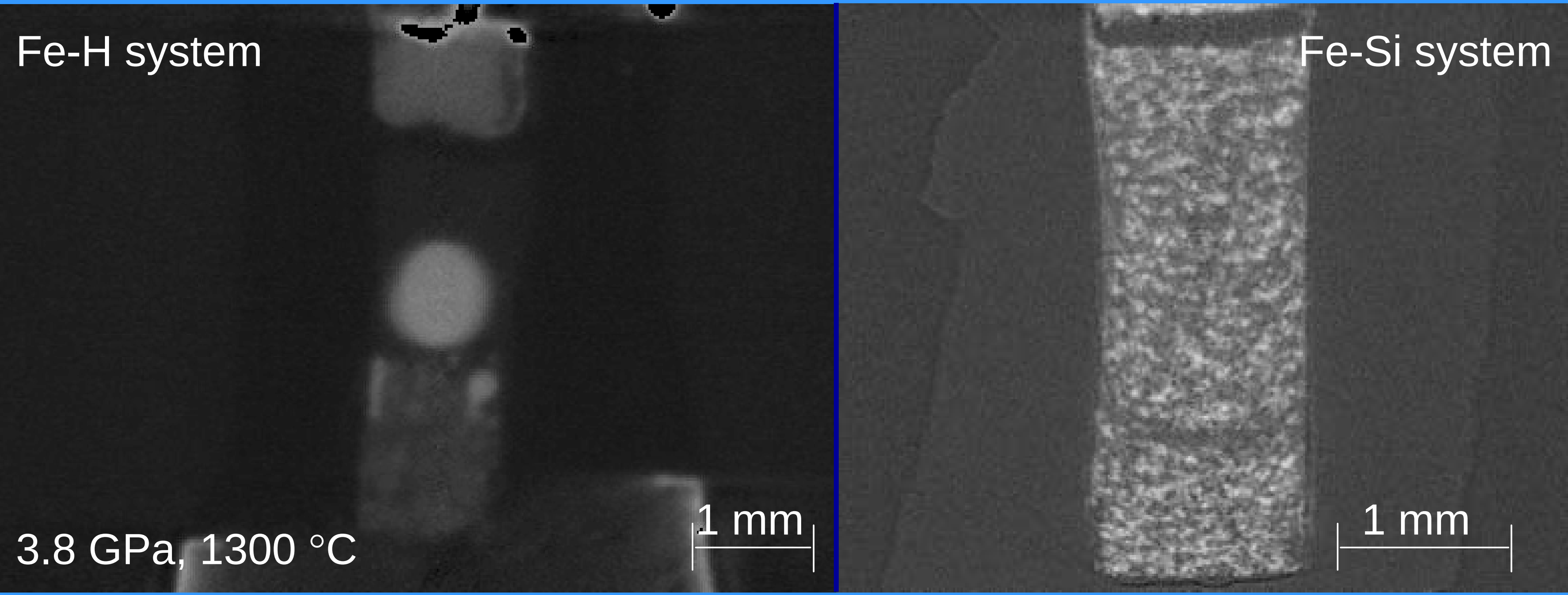




Diffraction and Imaging Setup

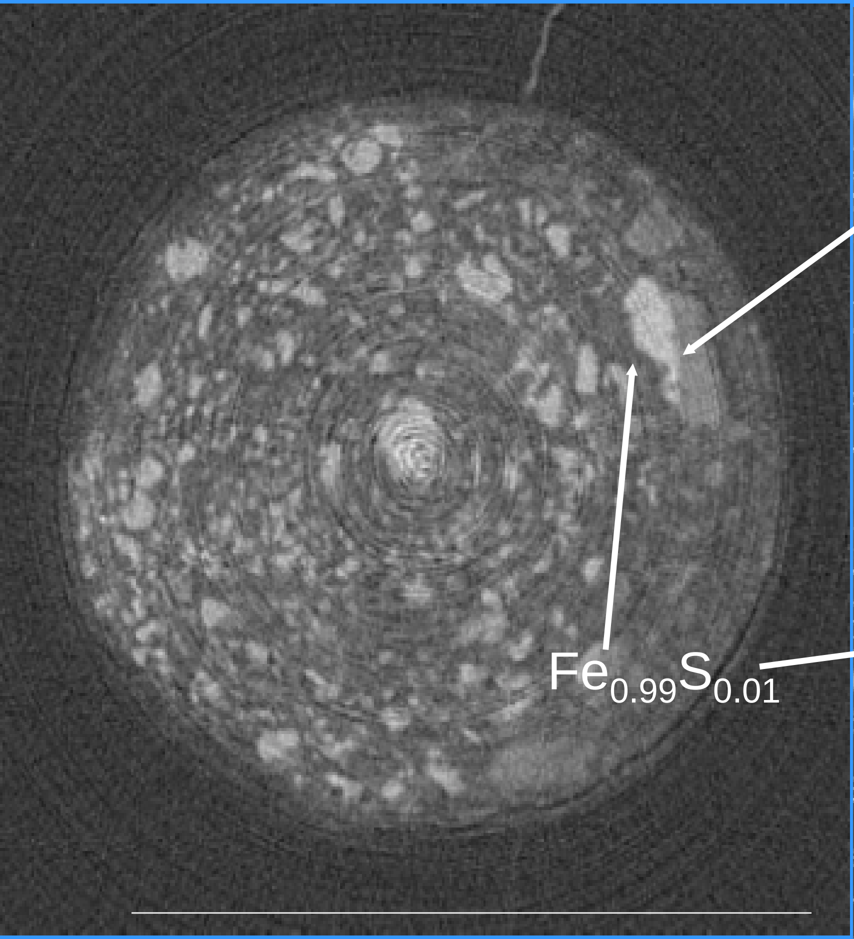


Tomography on Recovered Samples

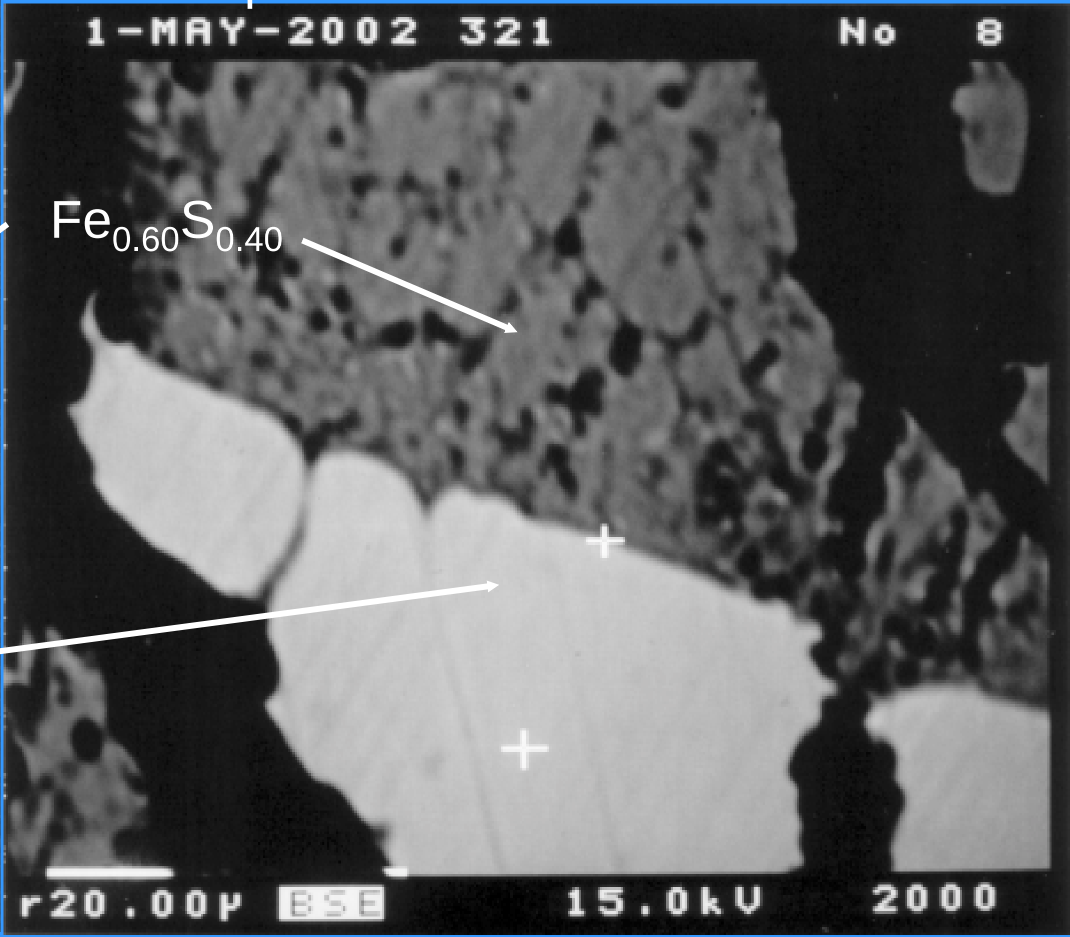


Tomography Vs. Microprobe

Tomography



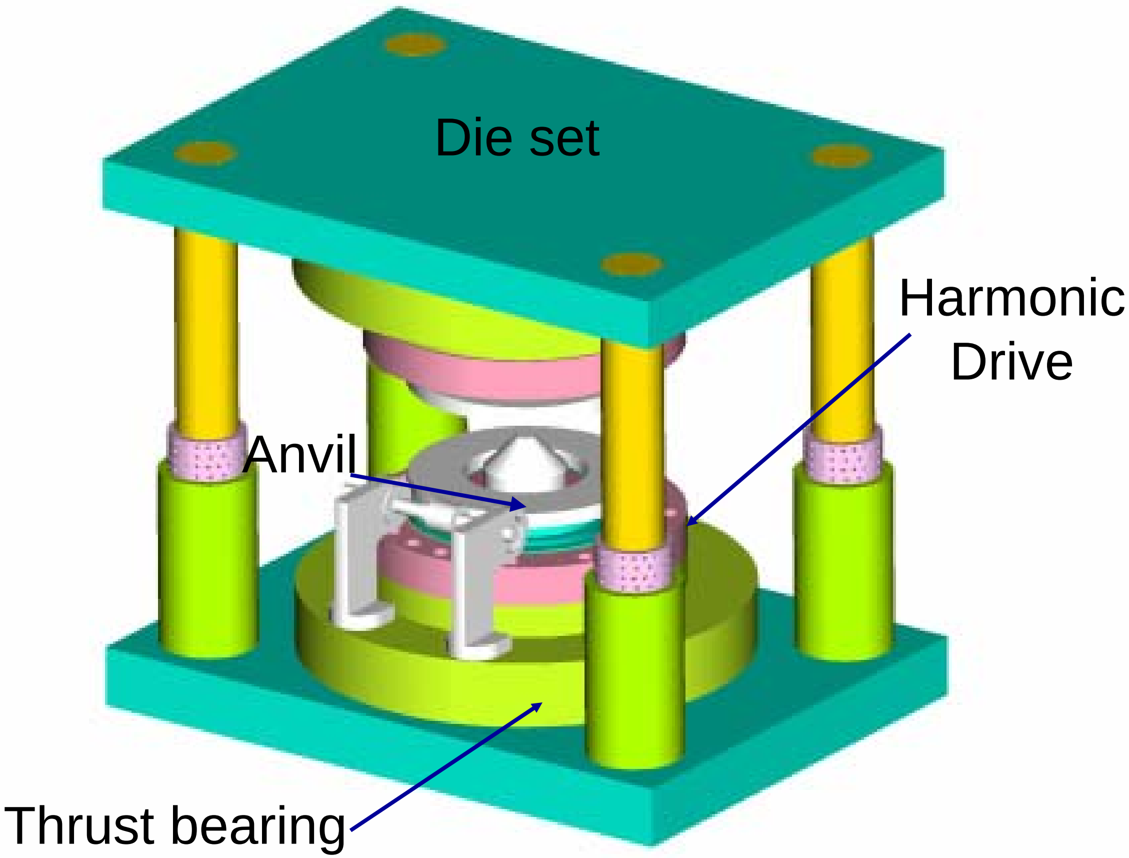
Electron probe

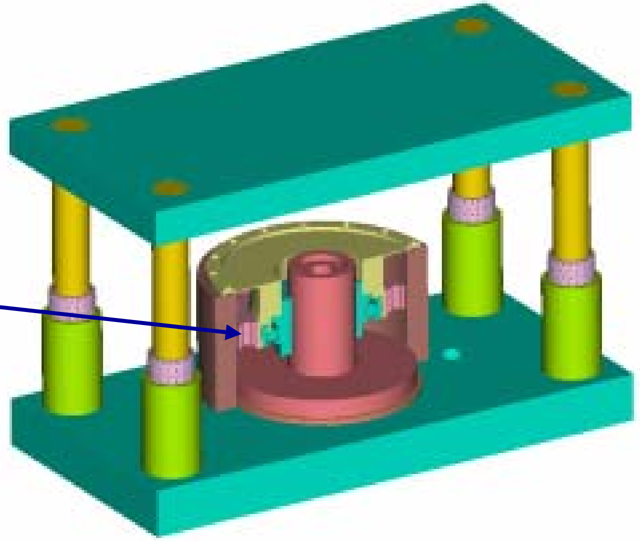


Design Parameters

- Max load capacity: 50 T
- Rotation range: full 360° with 0.02° repeatability
- Lateral translation of apparatus by ~ 50 mm, repeatable within 2 μm
- Modes of operation:
  - Anvils rotating in-phase for high-P tomography
  - Anvils rotating out-of-phase for shear deformation
- P-T range: 10 GPa, 1500K
- Sample size: 1 mm diameter, 0.5 mm long
- Monochromatic diffraction and imaging
- Spatial imaging resolution: ~2 μm

Rotation Mechanism





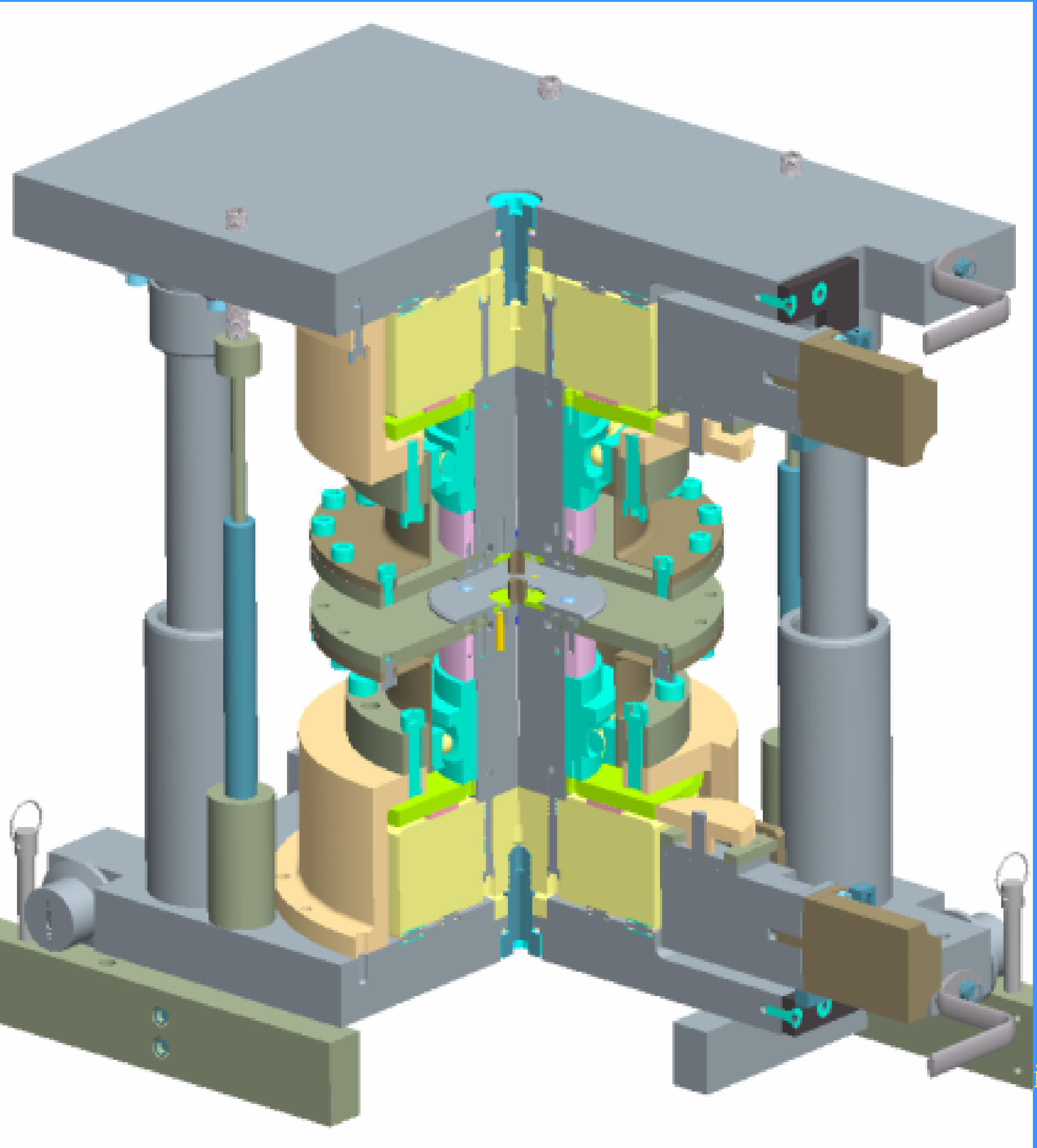
Concept based on rotational deformation apparatus
(Yamazaki and Karato, RSI, 2001)

Potential Applications

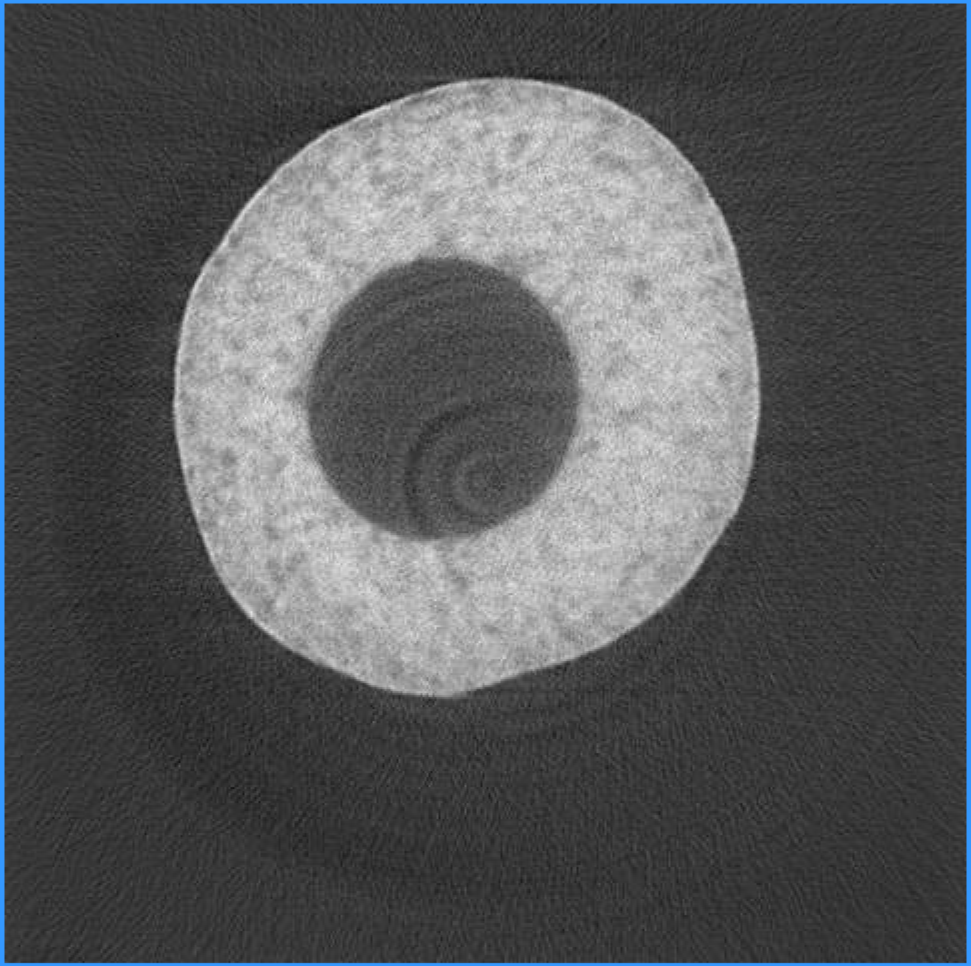
1. High-P-T tomography
   - -In situ observations of phase (incl. melt) separation
   - -Effects of deformation on melt texture evolution
   - -Density of melts/liquids (absorption)
2. Volume measurements of liquids/melts
3. Monochomatic diffraction
   - -Structure of melts/liquids
   - -Preferred orientation in deformed solids

Rotation Mechanism

Concept based on rotational deformation apparatus (Yamazaki & Karato, RSI, 2001)



First High-Pressure Tomography



45 keV

6 micron pixels

12 tons load

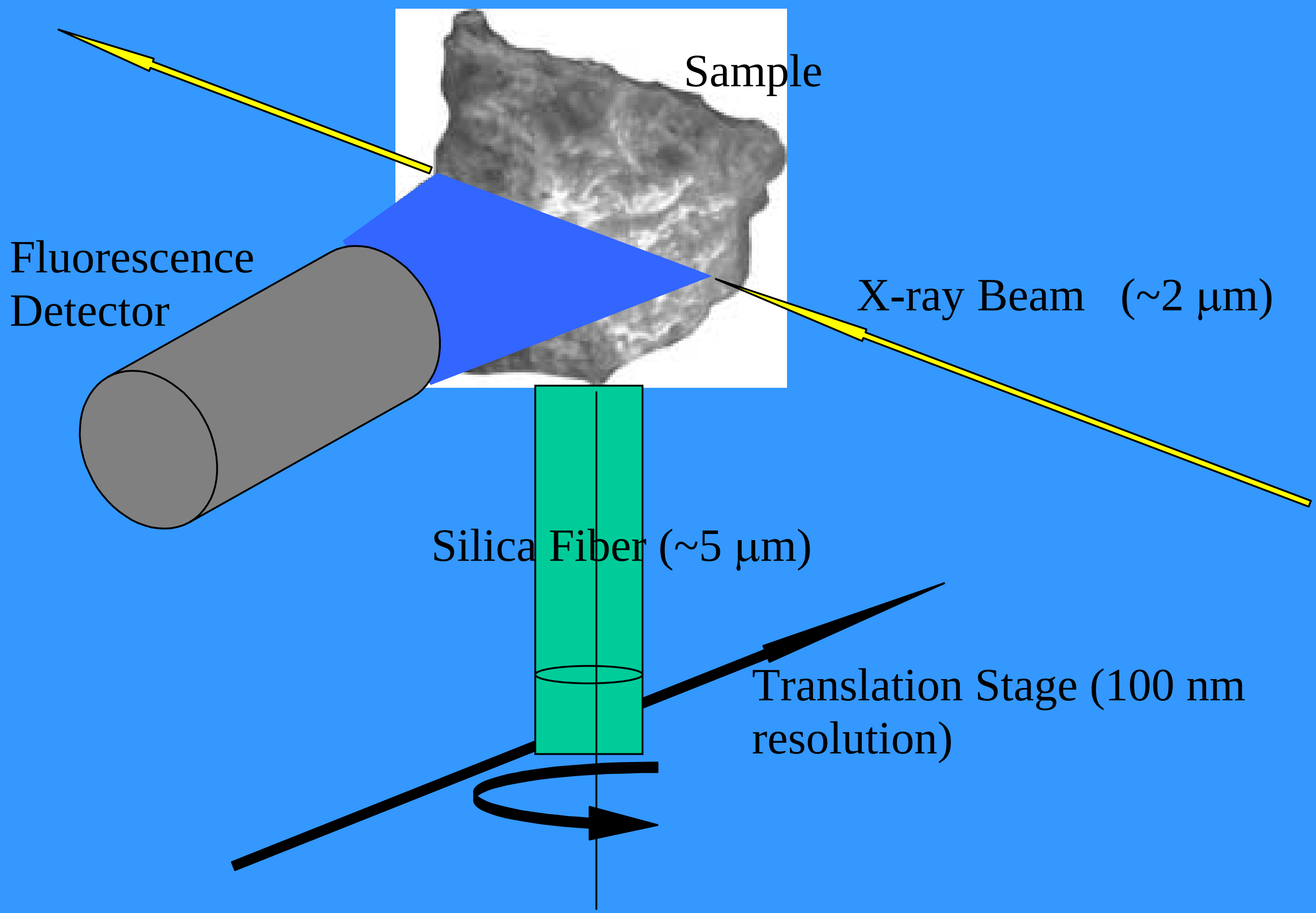
40 kbar (4 GPa)

Sapphire sphere in Fe-9%S powder.

Fluorescence Microtomography

- Images of the internal distribution of specific elements
- Synchrotron XRF is very sensitive, sub-ppm
- Valuable when object cannot be sectioned at required resolution
- Absorption must not be too large
- First generation (pencil beam)
  - Requires NX translations and NR rotations for each slice.
  - Slow – but similar to conventional 2-D map
- APS Undulator Source
  - Less than 1mm vertical by 3mm horizontal
  - 1000 times more intense monochromatic beam than bending magnet

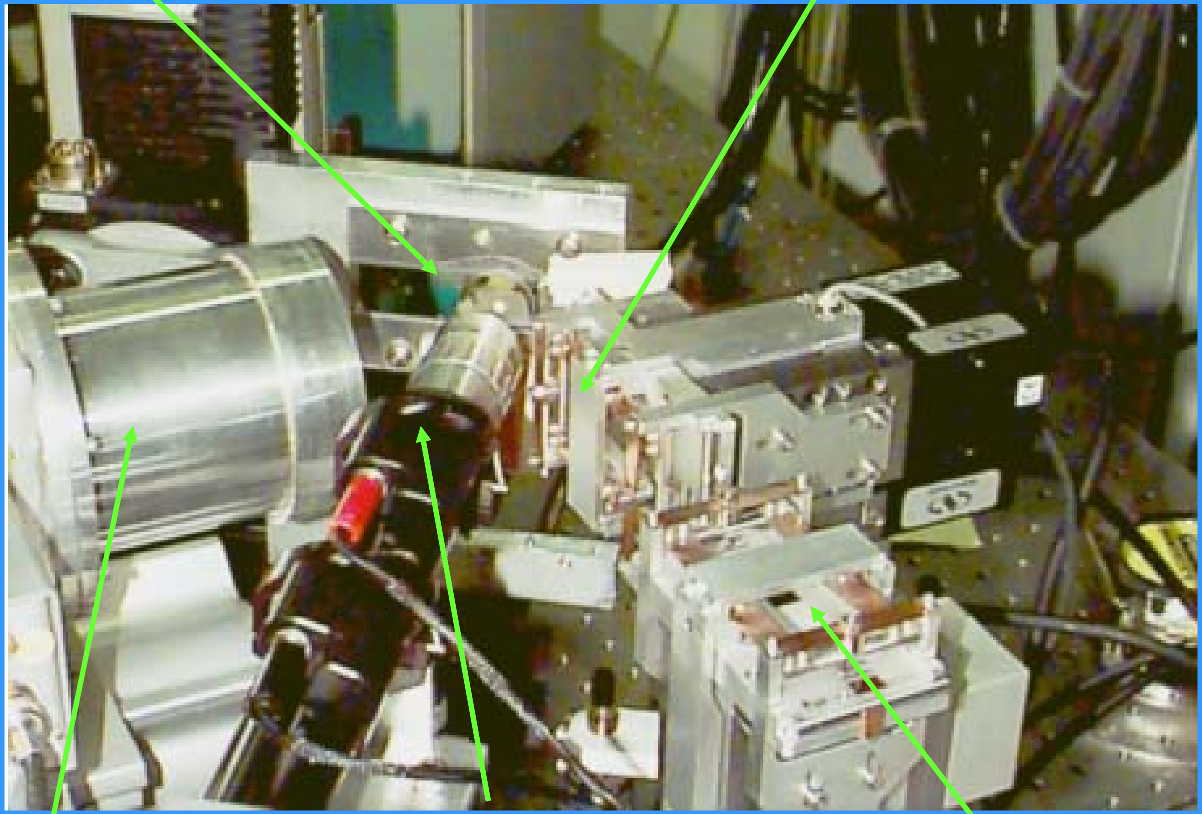
Fluorescence Microtomography Apparatus



X-ray Microprobe at GeoSoilEnviroCARS (APS)

X-Y-Z-θ Sample Stage

Horizontal Focusing Mirror



Fluorescence Detector

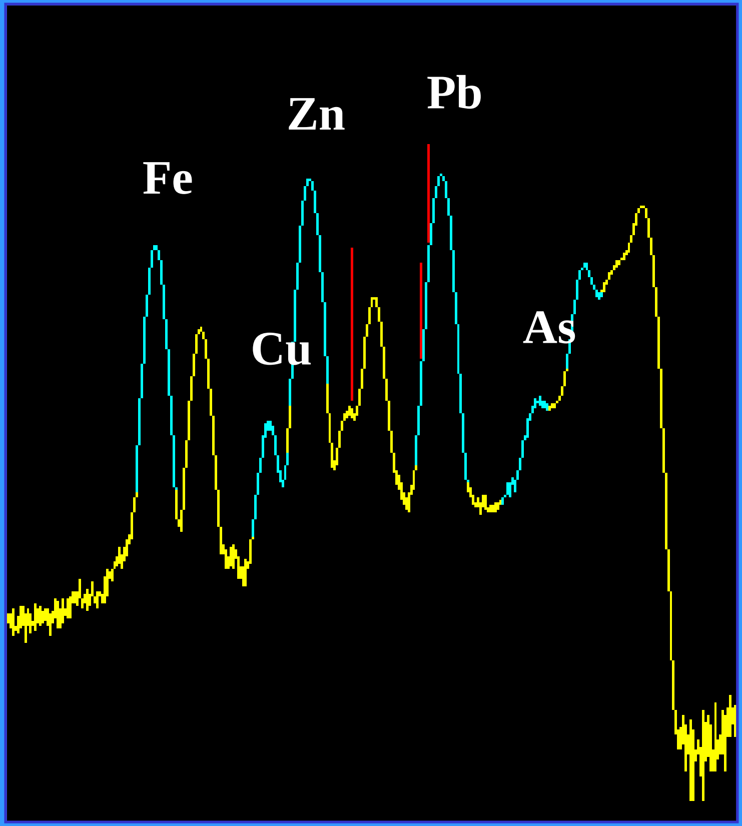
Optical Microscope with CCD Camera

Vertical Focusing Mirror

Data Collection Setup: Fluorescence Detector

- Regions of Interest (ROIs) are selected for each element of interest
- Fluorescence intensity in each peak is collected simultaneously at each pixel.

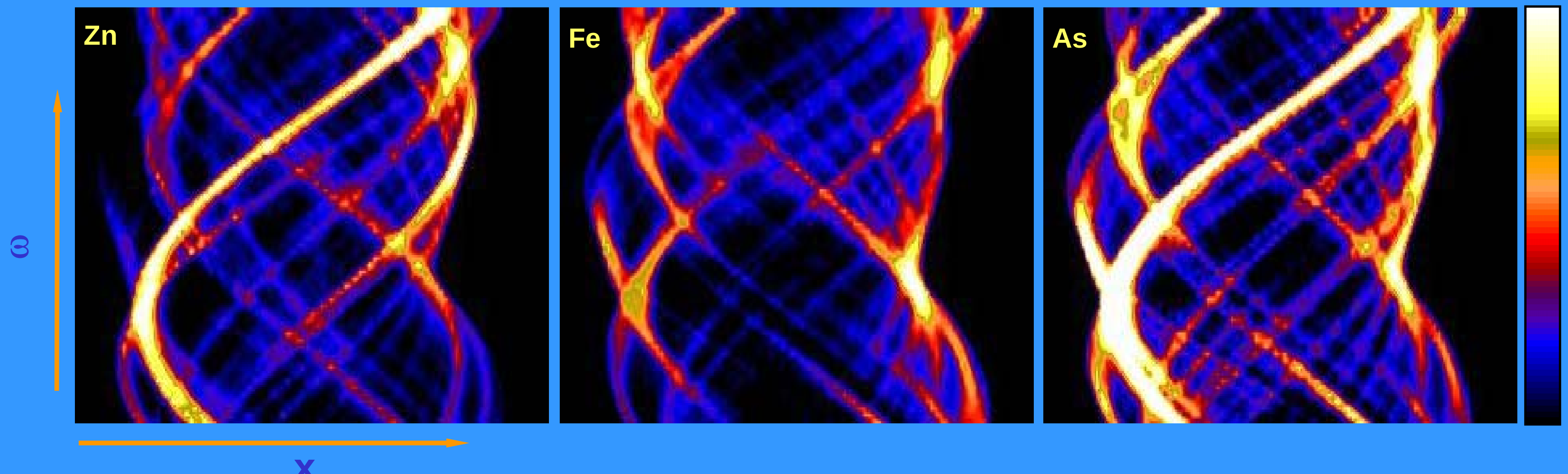
X-ray Fluorescence Spectrum (logarithmic display)



Fluorescence Tomography: Sinograms

The raw fluorescence tomography data consists of elemental fluorescence (uncorrected for self-absorption) as a function of position and angle: a ***sinogram***. This data is reconstructed as a virtual ***slice*** through the sample by a coordinate transformation of $(x,\omega) \rightarrow (x, y)$. The process can be repeated at different z positions to give three-dimensional information.

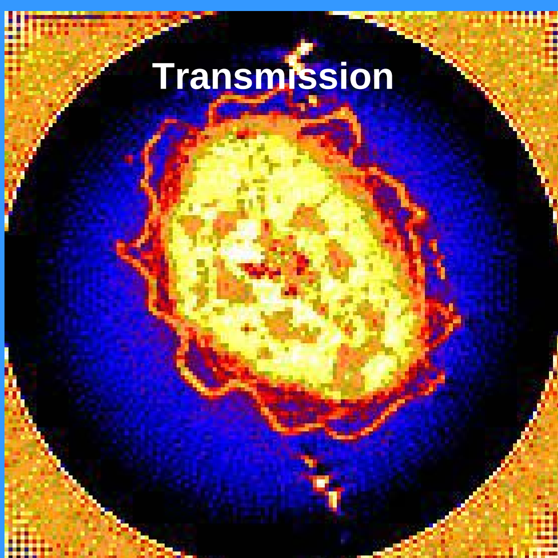
Fluorescence sinograms collected simultaneously for Zn, Fe, and As for a cross-section of As-contaminated cattail root (photo, right): **x**: 1100μm in 10μm steps **ω**: 180° in 3° steps

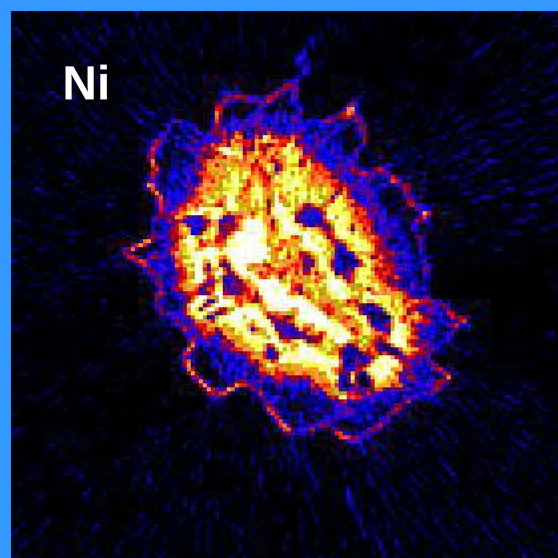


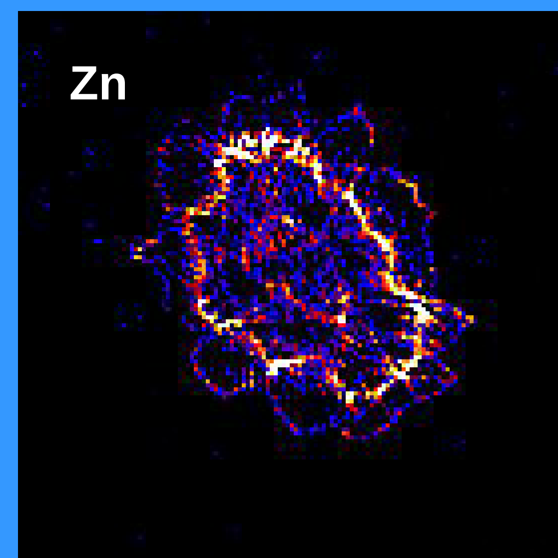
Nickel Distribution in Hyperaccumulating Alyssum Plants

(D. Sparks et al. U. Delaware)

• Fluorescence and transmission tomograms of Ni and Zn microdistributions in an Alyssum stem (~ 800 μm dia) from plant grown in Ni-enriched soil (8 μm x 2 degree x 1 second)
• Transmission tomogram shows ~density distribution
• Allows one to study the uptake mechanisms of specific elements.







Future Directions

- 3-D imaging with trace element sensitivity
  - Better detectors!
- Real-time imaging at extreme conditions
  - Better detectors!!!
- Technique development (higher pressure, in-situ high temperature, better resolution)
  - More beam time!!!