3D Energy Dispersive Spectroscopy – Elemental Tomography in the Scanning Transmission Electron Microscope

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Topics

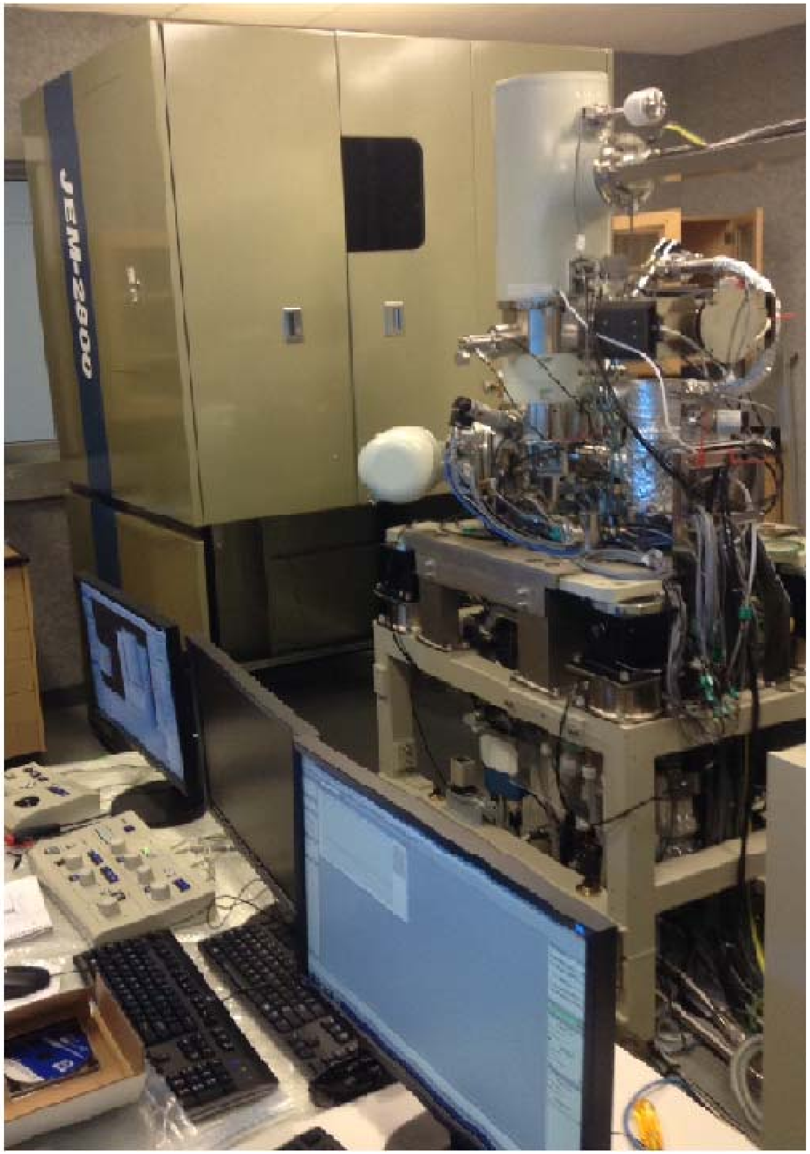
1. Introduction to EDS in the STEM

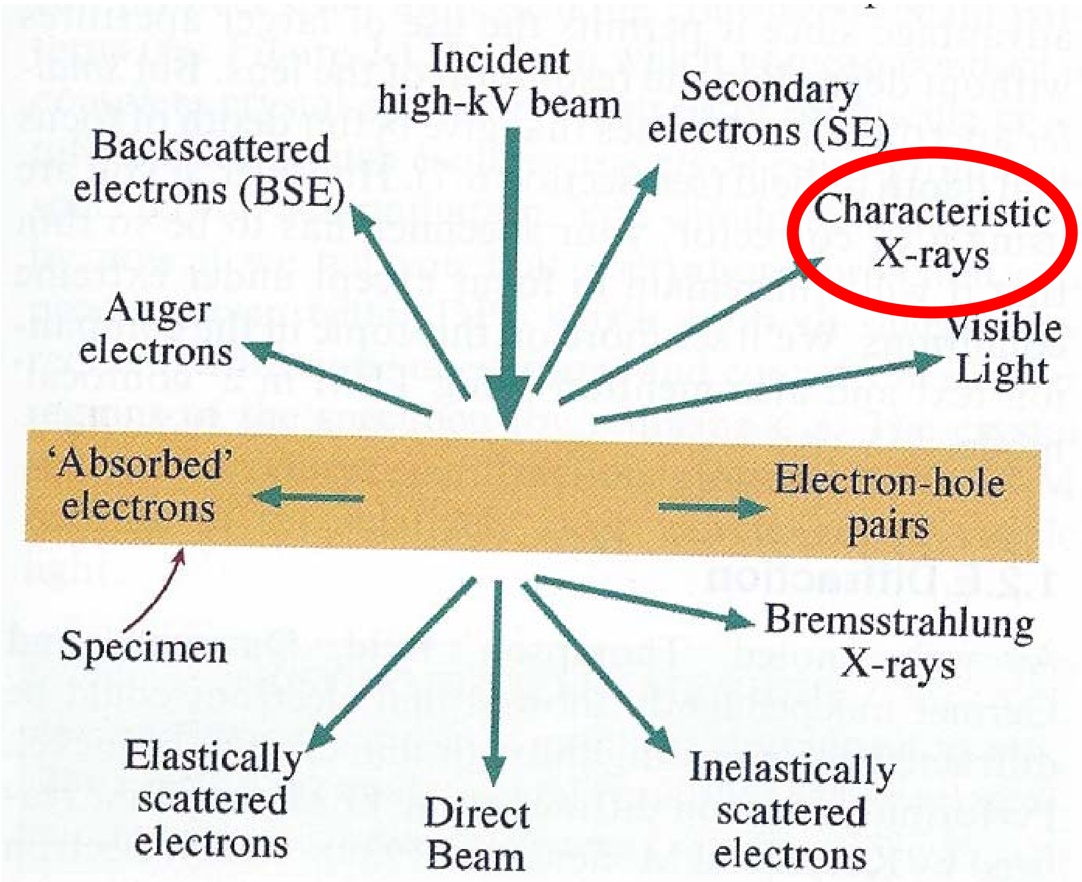
2. Extending EDS into three dimensions

3. Considerations and challenges for EDS tomography in the STEM

4. Examples of 3D EDS datasets

STEM Imaging: Possible Information Acquired



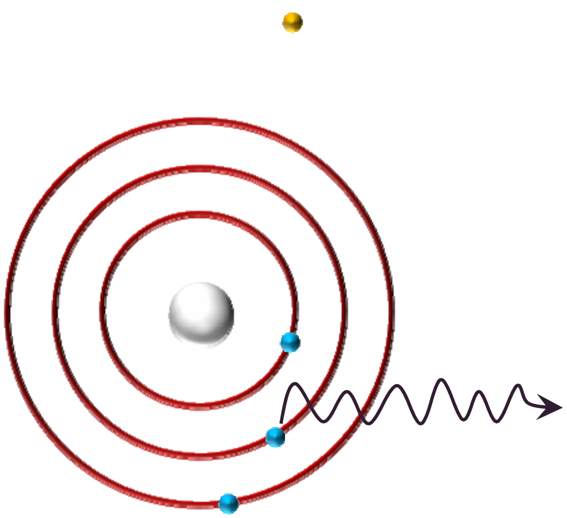


Bright Field
STEM

Dark Field
STEM

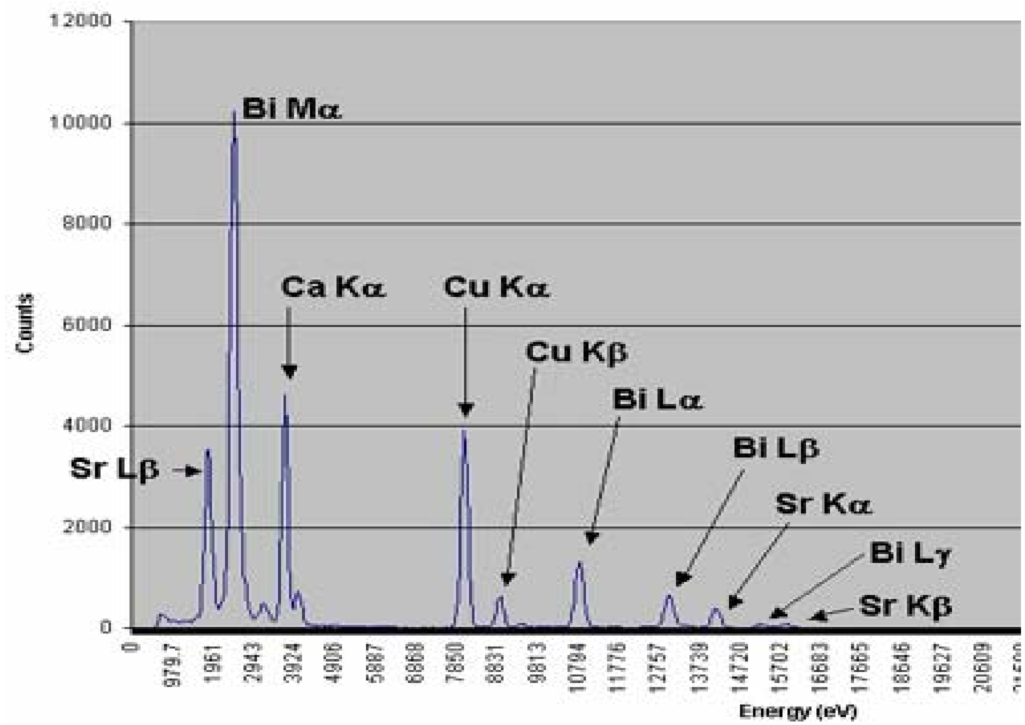
Figure from Williams & Carter:
"Transmission Electron Microscopy"

Characteristic X-Ray Generation



X-rays generated will have energies that are characteristic of atoms where they come from. Allowing identification and quantification of elemental components in a sample

i.e. $E\,K\alpha = BE_{L\ Shell} - BE_{K\ Shell}$



Beam-Sample Interaction Volume for X-Ray Generation

Sample surface

Simple approximation of beam spreading:

$$b = 8 \times 10^{-12} \frac{Z}{E_0} (N_v)^{1/2} t^{3/2}$$

Z = atomic number
E_0 = beam energy
N_v = atoms/m^3
t = sample thickness

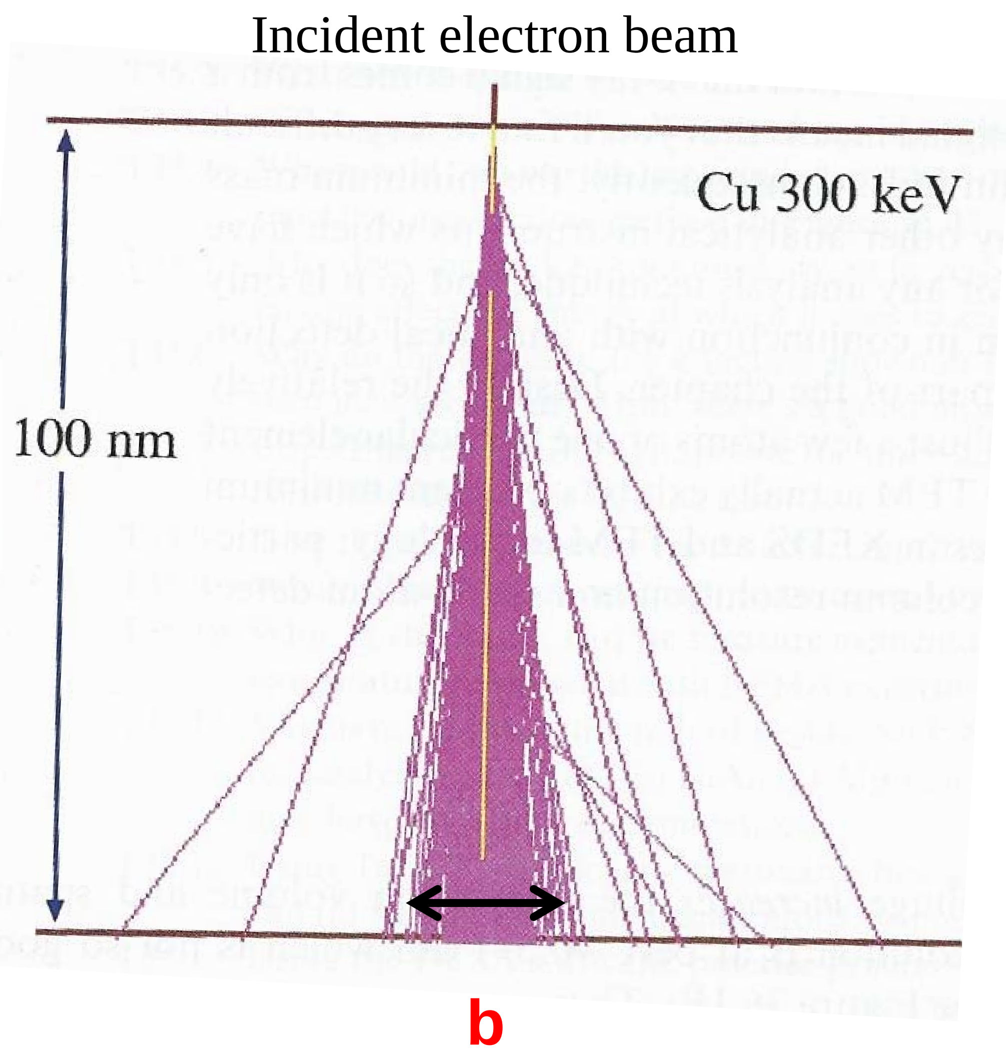
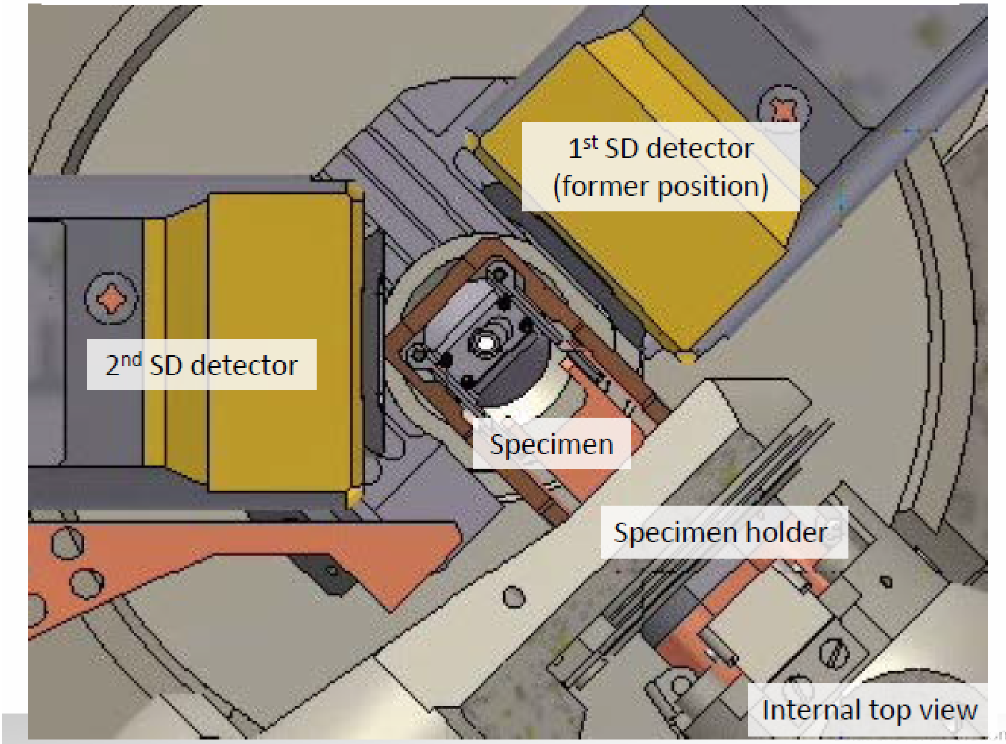


Figure from Williams & Carter: "*Transmission Electron Microscopy*"

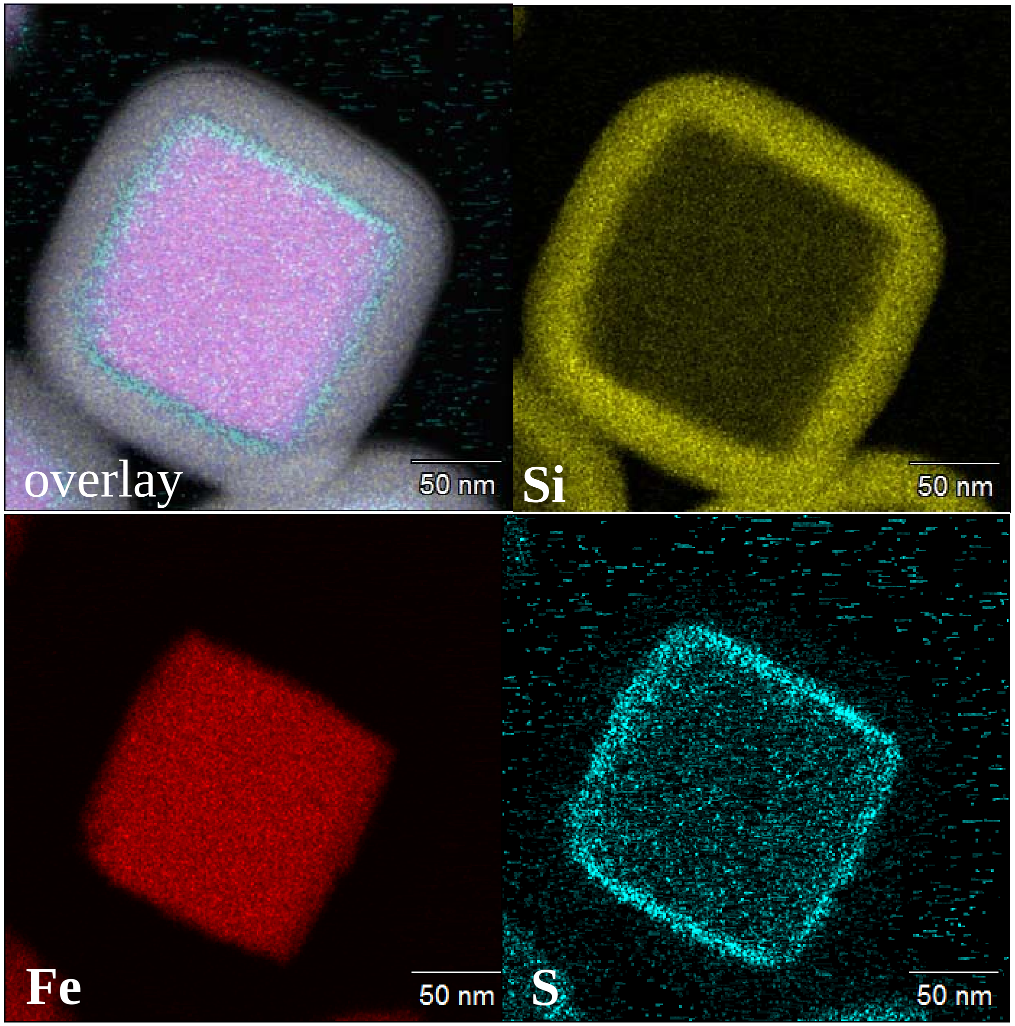
Dual EDS Detector System on JEOL 2800 at U of U

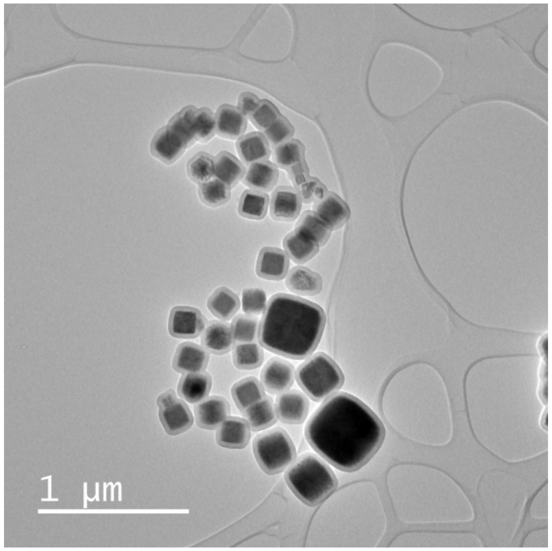
- Dual 100 mm^2 detectors
- Combined solid angle collection efficiency of 1.9 sr (best in class)
- This is still only about 13% of all signal (4 □ sr in a complete spherical volume of excitation)

1st SDD and 2nd SDD make the angle of 135 degrees



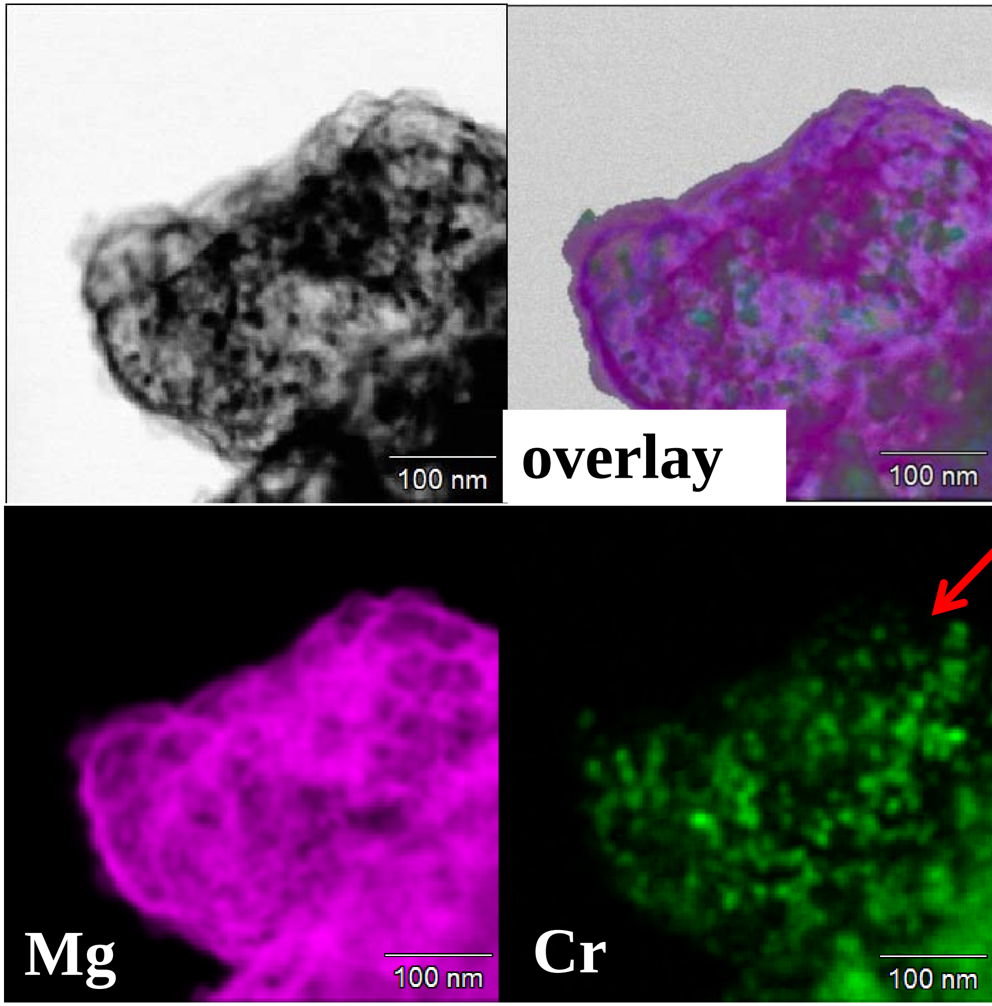
Two Dimensional Elemental Mapping





Core-Shell iron/silica nanoparticles Marc Porter group, University of Utah

Two Dimensional Elemental Mapping



What about depth distribution? Cr near surface or deeper within structure?

MgH_2 nanocomposites with Cr

Samples from Zak Fang group, University of Utah

Tomographic EDS

- **Tilt specimen around single axis at regular intervals (usually 1 or 2°)**
- **Acquire spectral image (“projection”) data at each tilt angle**
- **Resolution of 3D spectral image is a function of**
  - **Maximum tilt angle (a) (+/- 80° with JEOL HTR holder)**
  - **Number of projections**
  - **S/N of projections**

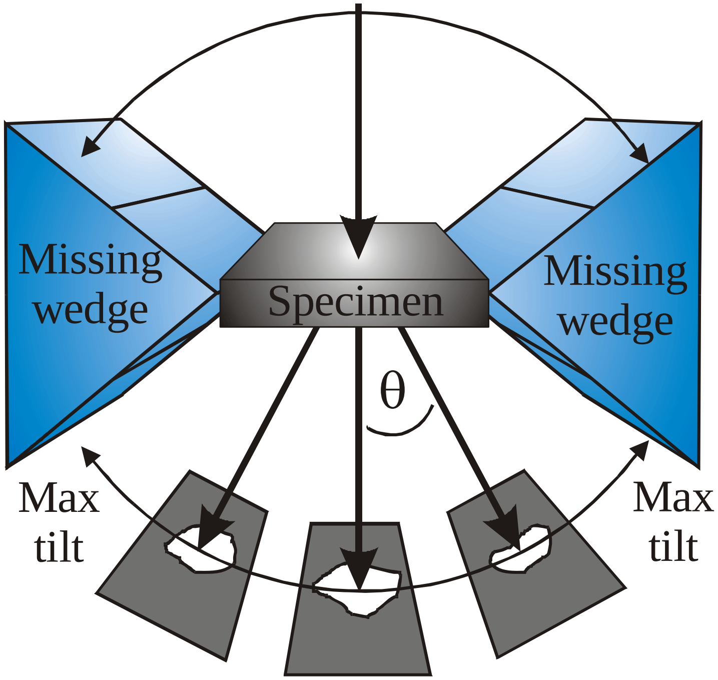
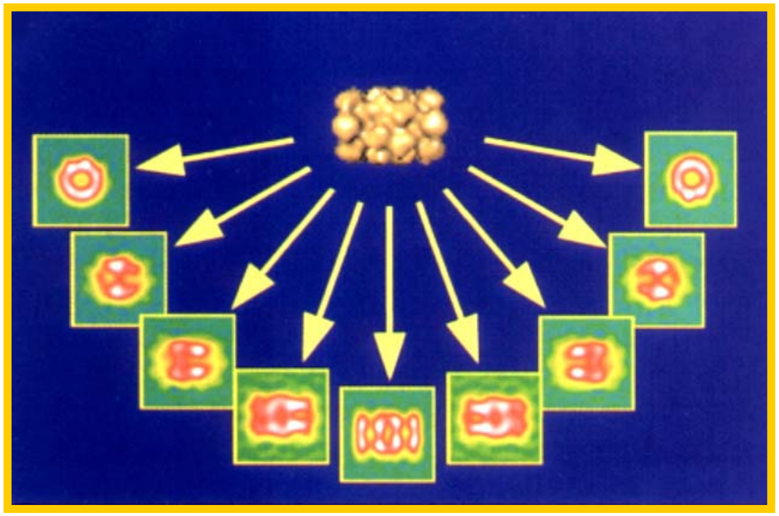


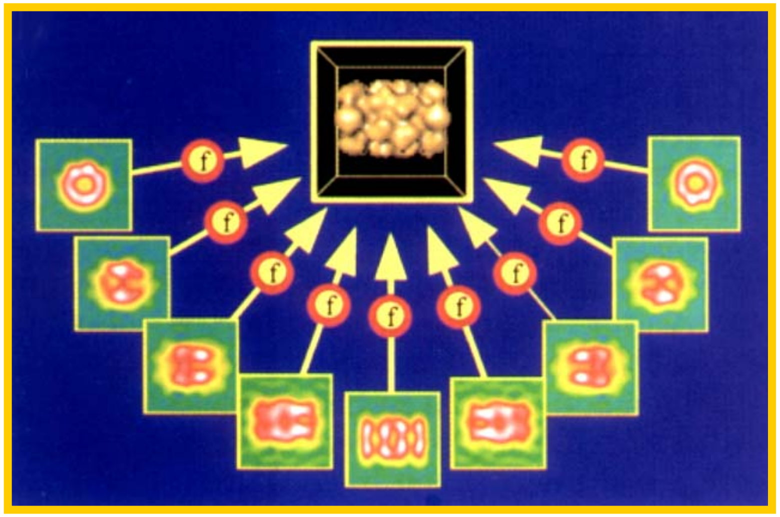
Figure courtesy of :
K. McIlwrath JEOL USA, Inc.
M. Weyland and P.A. Midgley
Department of Materials Science and Metallurgy
University of Cambridge

Tomographic EDS

Projection



Back-projection

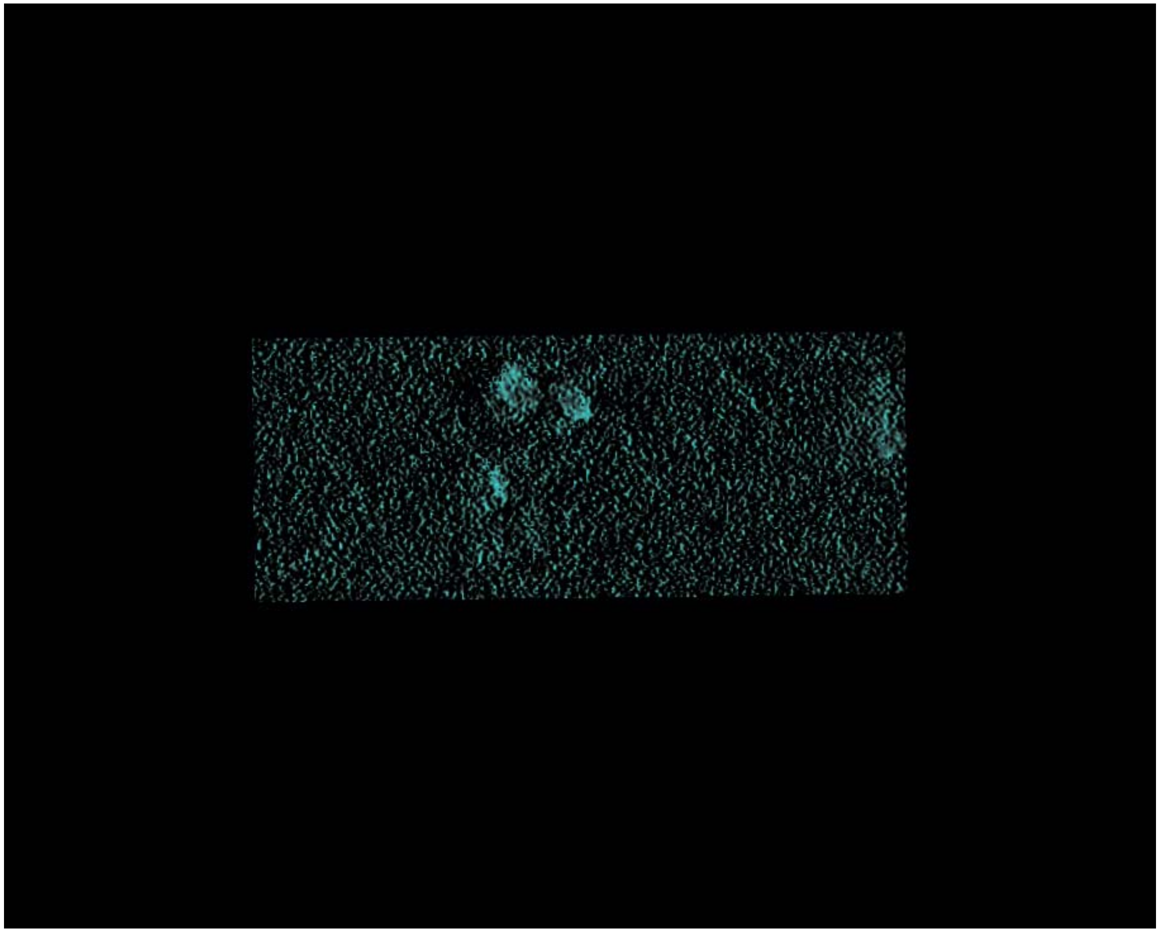


- Projections are combined to reconstruct 3D image
- Algorithms used for reconstruction
  - *Weighted back projection with mathematical filtering of data*
  - *Iterative reconstruction*

Slide courtesy of K. McIlwrath – JEOL USA, Inc.

Example 1: Tomographic EDS

New upgrade (installation completed 08/21/15)

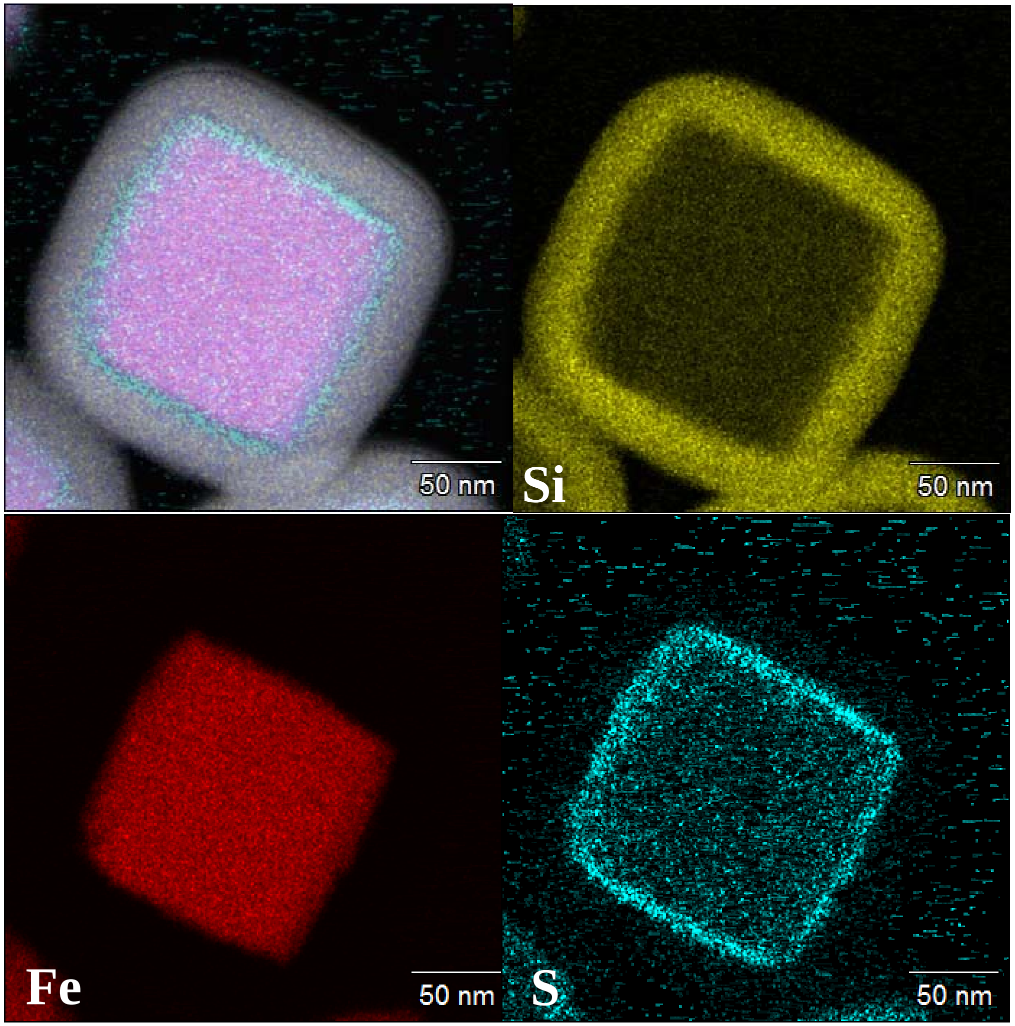


- Spheres are 262 nm in diameter
- Blue color, C
- Gold color, Au

3D tomogram of latex spheres on Au grating

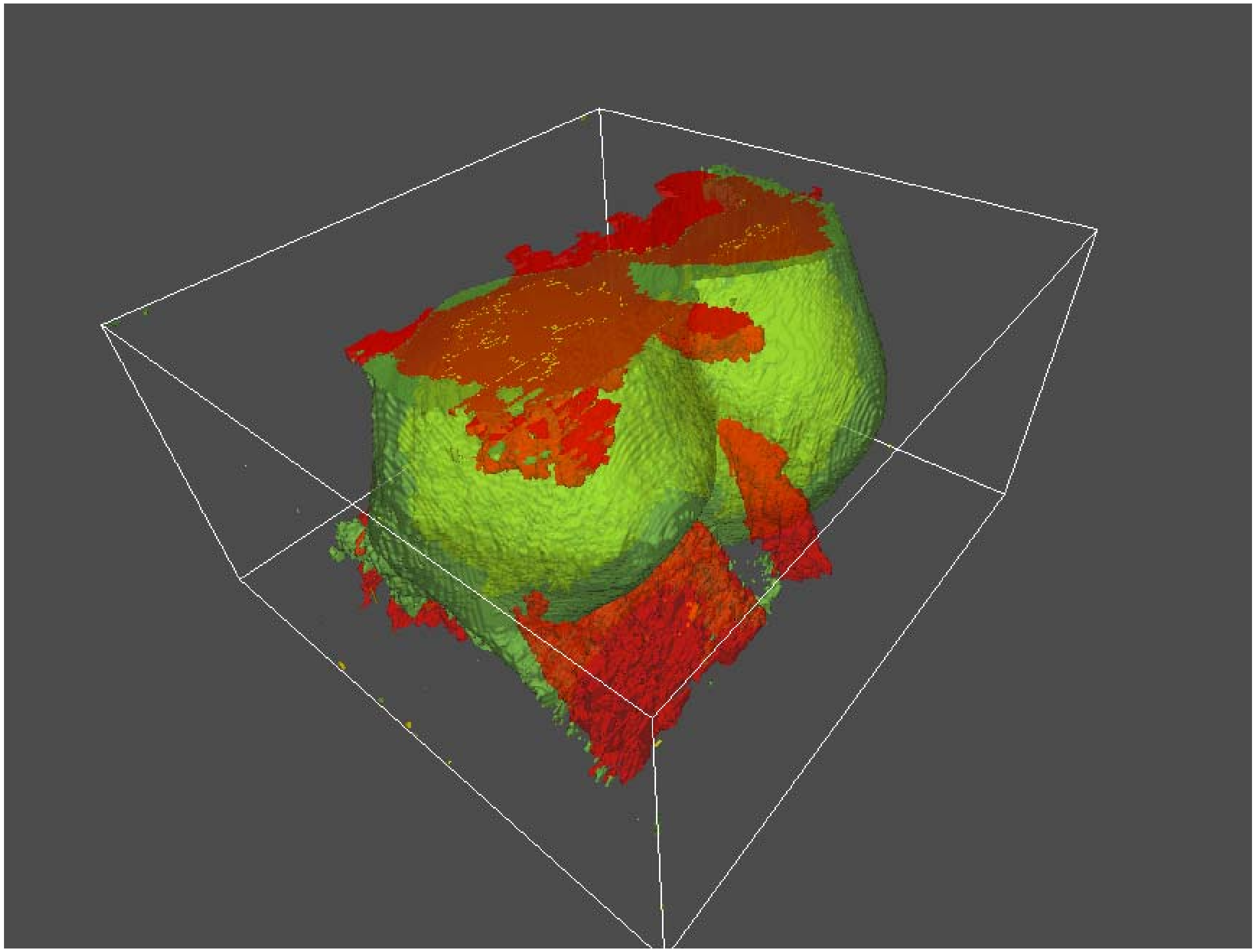
Example 2: Tomographic EDS on Core/Shell Nanoparticles

2 D EDS maps



- Tilt series performed from -64º to +59º in 3º steps
- Total of 41 tilts at 5 minute per tilt acquisition time
- Complete acquisition time of 205 minutes (3 hrs. 25min)

Example 2: Tomographic EDS on Core/Shell Nanoparticles



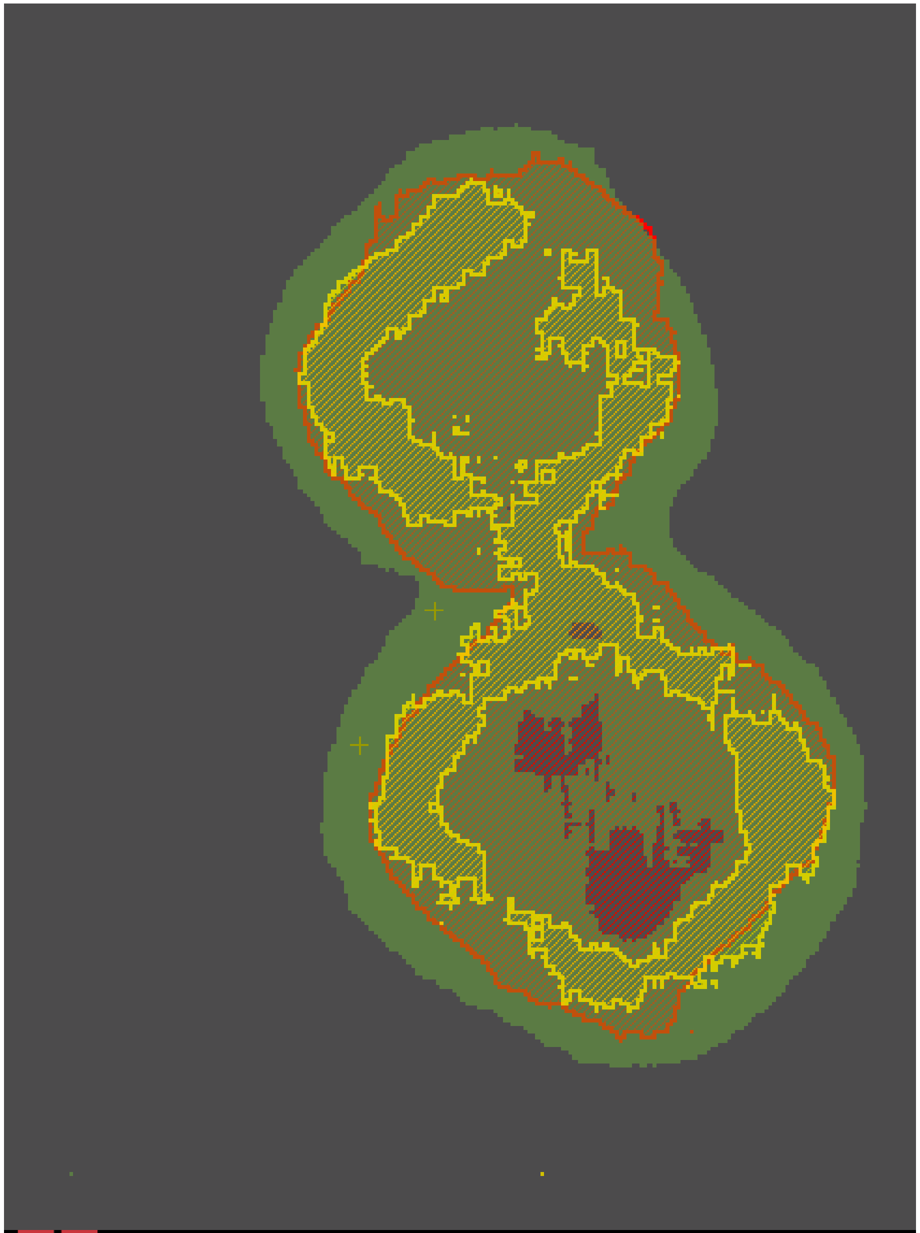
Green – Si

Red – Fe

Yellow - S

3D rendering of EDS data after tilt series

Example 2: Cross Sectional Representation



- Fe "bleed through"
- Long collection times at high magnification presents challenges
- Other challenges:

  Currently no quantitative models to deal with 3D tilt series data for EDS

  Inherently low S/N of EDS data in STEM

Special Thanks

Randy Polson – U of U Surface Analysis Lab

Kevin McIlwrath – JEOL USA, Inc.

Steven Kim – AppFive