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

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. \( E_K \alpha = BE_{L \text{ Shell}} - BE_{K \text{ Shell}} \)
Beam-Sample Interaction Volume for X-Ray Generation

Sample surface

Simple approximation of beam spreading:

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

- \( Z \) = 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 mm² 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)
Two Dimensional Elemental Mapping

Core-Shell iron/silica nanoparticles Marc Porter group, University of Utah
Two Dimensional Elemental Mapping

MgH$_2$ nanocomposites with Cr

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

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