Electron **tomography**

**CHARACTERIZATION OF MATERIALS IN 3D**

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3D techniques for materials science

FIB sectioning


Atom probe tomography


X-ray tomography

3D techniques for materials science

1 = atom-probe tomography;  
2 = scanning transmission electron microscopy (STEM) focal sectioning;  
S/TEM = electron tomography;  
AFM = atomic force microscopy sectioning;  
SIMS = secondary ion mass spectroscopy;  
FIB/SEM = focused ion beam/scanning electron microscopy.

History of tomography


Development of electron tomography

- **1960’s**: first applications of tomography related technique in electron microscopy in biological sciences (1952 Nobel Prize for Klig)
- **1990’s**: routine application of TEM tomography in biological sciences
- **2000**: first application of TEM tomography in catalysis by Geis/Jansen de Jong/Koster [3,4]
- **2001**: routine application of TEM tomography in catalysis by Janssen/de Jong/Koster [6,7].
- **2001**: first applications of electron tomography to HAADF-STEM and spectroscopic (EFTEM, EDX) images by Midgley/Weyland [14] and Moll [15]

Timeline:

- **1917**: formulation of mathematical base for tomographic techniques by Radon (Radon Transform)
- **1960’s**: development of X-ray computerized tomography (1979 Nobel Prize for Cormack and Hounsfield)
- **1990’s**: development of automated TEM tomography by Agard [18] and Baumeister [19]
- **1990**: first commercial systems enable data acquisition in ~4 h
- **2001**: development of pre-calibration electron tomography by Koster/Ziese [20,21]
- **2001**: commercial systems making use of pre-calibration enable improved accuracy and data acquisition in ~30–60 min

Nearly 50 years later to the work of Radon, tomographic X-ray scanning for 3D medical imaging was proposed and EMI, built the first X-ray computed tomography scanner in 1971.
Tomography in microscopy

**Input**: 2D projections acquired at different orientations.

**Output**: A mathematical algorithm based on the *Radon transform* is used to reconstruct the 3D Volume.
Tomographic reconstruction: *Radon Transform*

Tomography originates in a 1917 paper of Radon. It is the basis for all the tomographic techniques no matter if light, X-rays electrons, neutrons... are used.

2D slices obtained from 1D projections. Stacking the 2D slices a reconstruction in 3D is obtained.
Central Slice Theorem: ‘a projection of an object at a given angle is a two-dimensional central section through the three-dimensional Fourier transform of that object’.

In practice, most reconstruction algorithms are based on real space processing.
Tomographic acquisition: limitations

TEM sample holders for tomography

Artefacts can arise in the 3D reconstruction:

- Missing wedge
- Irradiation damage
- Contamination of the sample
Tomographic reconstruction: artefacts

In practice, limited experimental data usually means that some Fourier space information is missing, and back-transform of the object is then degraded.

For a single tilt axis, the resolution of the reconstruction is different in each spatial direction:

\[ d_y = \frac{\pi D}{N} \]

\[ d_z = d_y \cdot e \]

# Electron Tomography

<table>
<thead>
<tr>
<th>Types of signal/techniques used for tomography in electron microscopy</th>
<th>TEM</th>
<th>S(T)EM</th>
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</thead>
<tbody>
<tr>
<td><strong>Coherent</strong></td>
<td><strong>Incoherent</strong></td>
<td><strong>Coherent</strong></td>
</tr>
<tr>
<td>Elastic scattering</td>
<td>Bright-field/dark-field (BF/DF) (only for amorphous and biological samples)</td>
<td>Annular dark-field (ADF)</td>
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<td>Diffraction</td>
<td>Exitwave restoration (EWR)</td>
<td>Convergent-beam electron diffraction (CBED)</td>
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<td>Lorentz</td>
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<td>Off-axis holography</td>
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<td></td>
<td>Transport of intensity (TIE)</td>
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<tr>
<td>Inelastic scattering</td>
<td>EFTEM (Low-loss)</td>
<td>EFTEM (Core-loss)</td>
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Two-dimensional projections, or *tomograms*, used for 3D reconstruction must meet the ‘projection requirement’.

The signal of a HAADF image has several advantages:

- It is proportional to the atomic number.
- It is mainly incoherent.
- It meets the “projection requirement” for tomography.
Tomography workflow

**Acquisition**
- choose maximum tilt range
- choose tilt increments
- limit applied dose to prevent beam induced changes

**Alignment**
- sequential cross-correlation (apply filter to images)
- fiducial marker model (refine model to minimize errors)

**Reconstruction**
- weighted backprojection (most common)
- iterative reconstruction techniques (common)
- Fourier Transform based reconstruction (less common)

**Visualization**
- sequential numerical cross-sections (e.g. IMOD, Inspect3D)
- isosurface and volume rendering (e.g. Amira, Chimera)
- Image denoising and segmentation (e.g. TOM, IMOD, Amira)
HAADF STEM tomography applications: Particle structure

HAADF STEM tomography applications: PSD

HAADF STEM tomography applications: PSD

Tomographic reconstruction of a magnetotactic bacterium.


Left, typical high-resolution TEM image of a crystalline nanoparticle and 3D reconstruction.


The size and location of Au particles inside the material can be seen unambiguously and subjected to statistical evaluation.
HAADF STEM tomography applications: Supported catalysts

2D projection TEM

3D volume. HAADF STEM Tomo
The nanoparticles (red) appear to prefer to anchor themselves at the saddle points.

“In the heterogeneous catalysts we have analyzed herein, after being activated under reducing or oxidizing environments, the dispersed gold nanoparticles show a strong preferential location for nanocrystal boundaries and stepped sites of the oxide support”

HAADF STEM tomography applications: Pores

The accumulation of Iron in cells of the brain is a symptom present in Alzheimer patients.

HAADF STEM tomography applications: irradiation damage

Localised irradiation of a thin sample (SiO₂/Ge) using STEM

2D

3D

EFTEM tomography: chemistry in 3D

Using inelastic scattered electrons for chemical mapping in 3D. EFTEM tomography in low-loss and core-loss.

Other 3D techniques in EM: Towards atomic resolution I

Electron tomography fullerene-like particles toward atomicscale resolution.

Other 3D techniques in EM: Towards atomic resolution II

HRSTEM + Discrete tomography

Other 3D techniques in EM: Towards atomic resolution III

New aberration-corrected scanning transmission electron microscopes permit very short depth of focus, opening the door to focus sectioning and confocal methods.

Summary

• Many techniques are available in materials science for recovering quantitative 3D information. Some techniques are destructive and others not. For example, atom probe provides 3D chemical information with atomic resolution. Not all the samples can be analysed and it is destructive (the sample is lost).

• The most suitable technique depends on your sample and the problem you want to solve.

• Tomographic techniques are based on acquiring tilted series of images (projections) and reconstructing the original volume using computer algorithms.

• Missing information means that reconstruction have artefacts which need to be taken into consideration for.

• Different groups are studying different ways of performing atomic resolution tomography with chemical sensitivity in TEM.

