## **Analytical Electron Tomography: Methods and Applications**

Georg Haberfehlner<sup>1</sup>, Angelina Orthacker<sup>1</sup>, Franz P. Schmidt<sup>2</sup>, Anton Hörl<sup>3</sup>, Daniel Knez<sup>2</sup>, Andreas Trügler<sup>3</sup>, Ulrich Hohenester<sup>3</sup> and Gerald Kothleitner<sup>1,2</sup>

Electron tomography is a powerful technique for 3D characterization at the nanoscale. Recent developments focus on extracting a wide range of information about a sample in 3D [1]. Of special interest is the combination of electron tomography with spectroscopic techniques - EFTEM, EELS and EDS - to recover the information present in spectroscopic signals in three dimensions. Analytical electron tomography allows mapping of chemical variations and gradients, approaching the goal of full 3D elemental quantification [2]. Additionally, EELS tomography can be used to extract information about materials properties or chemical bonding [3,4]. In this presentation we will discuss the steps necessary to successfully combine spectroscopy and tomography and show respective applications.

For analytical electron tomography a tilt series of spectrum images is recorded, over the widest tilt range possible for the actual sample geometry. The key to a successful reconstruction then lies within data processing, in particular in the extraction of the desired information from the spectroscopic signal, and the alignment and reconstruction of the tomographic tilt series. We will discuss novel, reliable and automated procedures for tomographic alignment together with advanced reconstruction algorithms, which incorporate additional information in the reconstruction process available from the sample. Particular emphasis is laid on compressed sensing algorithms, including total-variation (TV) minimization [5].

To give an example for a simultaneous EELS and EDS tomography and 3D mapping experiment, we demonstrate the reconstruction of phases in Al-alloys via TV minimization algorithms [2,6]. In this case the technique was applicable, since sharp interfaces between different materials in the sample were present, even though the quality and number of projections was limited. In addition we recovered spectral EDS and EELS data, by reconstructing each spectral channel, providing four-dimensional datasets with spectra available separately for each voxel (see Fig. 1).

We will also discuss the power of EELS tomography for the extraction of optical material properties in the form of 3D surface plasmon fields for metallic nanostructures (see Fig. 2). Correct reconstructions need meaningful physical models as basis for special algorithms needed in the 3D reconstruction of the low energy-loss signal [7].

The paper attempts to give insight into the current status of analytical electron tomography, its potentials and current limitations. [8]

<sup>&</sup>lt;sup>1.</sup> Graz Centre for Electron Microscopy and Nanoanalysis, Graz, Austria

<sup>&</sup>lt;sup>2</sup> Institute for Electron Microscopy and Nanoanalysis, Graz University of Technology, Graz, Austria

<sup>&</sup>lt;sup>3</sup> Institute of Physics, University of Graz, Graz, Austria.

- [1] PA Midgley and JM Thomas, Angew. Chem. Int. Ed. **53** (2014), p. 8614.
- [2] G Haberfehlner *et al*, Nanoscale **6** (2014), p. 14563.
- [3] O Nicoletti et al, Nature **502** (2013), p. 80.
- [4] B Goris et al, ACS Nano 8 (2014), p. 10878.
- [5] R Leary et al, Ultramicroscopy **131** (2013), p. 70.
- [6] G Kothleitner et al, Microsc. Microanal. 112 (2014), p. 678.
- [7] A Hörl, A Trügler and U Hohenester, Phys Rev Lett 111 (2013), p. 076801.
- [8] This research has received funding from the European Union within the 7<sup>th</sup> Framework Program [FP7/2007–2013] under grant agreement no. 312483 (ESTEEM2). We thank Jiehua Li and Peter Schumacher from University of Leoben and Harald Ditlbacher and Joachim Krenn from University of Graz for samples and discussions.

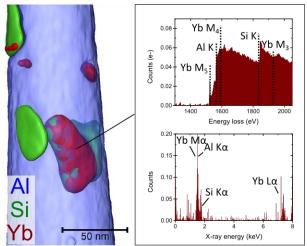
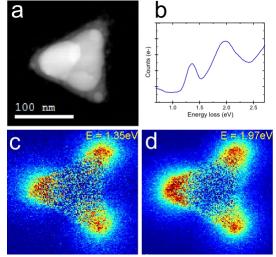


Figure 1. 3D elemental maps of an AlSi-alloy and single voxel EELS and EDS spectra [2].



**Figure 2.** Dipole modes in a triangular metal-insulator-metal structure: (a) HAADF STEM image (b) energy-loss spectrum, (c) & (d) EELS maps of coupled dipolar modes visible in (b).