Multi-mode electron tomography for the 3D morphological and structural characterization of nanomaterials.

Z. Saghi, X. Xu and G. Möbus

Dept Engineering Materials, University of Sheffield, Sheffield S1 3JD, UK.

z.saghi@shef.ac.uk
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In the last decade, electron tomography has enabled the 3D characterization of a wide range of nanomaterials, using various TEM imaging modes such as BF-TEM, ADF-STEM, and EFTEM [1]. In this work, we explore the combination of lattice-resolved HREM tomography and binarised shape reconstruction [2] for the retrieval of 3D crystallographic information and faceting of nanocrystals. HREM tomography is a promising technique which would push electron tomography resolution to the atomic scale [3]. In extension to our earlier work on weak-phase tomograms from full tilt series, here we explore the reconstruction from three zone-axes only. Figure 1(a) shows the 0º exit wave projection of a simulated cone-shaped CeO_2 supercell with a maximum thickness of 2.5nm and a microscope voltage of 1250kV. The reconstructed tomogram (figure 1(b)) from three zone-axis projections shows the 3D atomic distribution of Ce and oxygen atoms, but we notice from the slices through the tomogram (figure 1(c,d)) that the star artefacts prevent a reliable estimation of the outer facets of the crystal. To retrieve the 3D shape of the crystal, a complete tilt series is acquired and binarised (figure 1(e)). The intersection of the backprojected shadows provides an estimation of the 3D shape and faceting of the crystal (figure 1(f)) [4]. This second tomogram can then be multiplied to the lattice-resolved tomogram (figure 1(b)) so that both the crystallographic and the faceting reconstructions are superimposed (figure 1(g,h)).

To illustrate the technique experimentally, we selected a CeO_2 nanoparticle which was aligned to the goniometer rotation axis, such that a {200} fringe system is visible throughout a tilt series from -60º to +70º with 10º increment. Figure 2(a-d) show four members of the tilt series. The HREM images in figure 2(a) and figure 2(b) show zone-axis patterns of <110> type and near to <100> respectively, and were backprojected in full contrast, after noise filtering to select the major Bragg spots. In parallel, all 14 images were binarised and the outer shape of the particle was reconstructed using the shape-from-silhouette algorithm. Figure 3(e) shows the isosurface of the tomogram obtained from the binarised tilt series, while figure 3(f) shows the reconstruction by superposition of only two zone-axis projections. The combination of the two is essential, as the shared volume of the two backprojected HREM contrast patterns (both roughly cylindrical) does not reveal the particle shape but rather a geometric artifact (common volume of two cylinders) which depends on the tilt difference of the two zone axes. The geometric tomography however reveals the right particle shape, as also shown in [5].

Figure 1 illustrates the concept of this hybrid method, but experimental challenges prevent the results in Figure 2 from providing a true atomic resolution. Conditions for a successful atomic reconstruction would include: (i) all HREM images should be “Structure Images” in the sense of [6], which means that either all atoms, or at least the sublattice of heavy atoms, generate spots at the correct atomic positions; (ii) the surface contour images should allow alignment to the precision required to define the lateral positions of the atomic resolution images as well; (iii) all HREM images must be precisely aligned not only to the rotation axis, but also with respect to the crystallographic zone axes [7].

1. G. Möbus and B.J. Inkson, Mat. Today **10** (2007) p18.
2. K.C. Tam, *J nondestructive evaluation* **6** (1987) p189.
3. G. Möbus et al., *MRS Symp.Proc.* **738** (2003) G1.2, 15.
4. Z. Saghi et al., *J. Microsc.* **232**(1) (2008) p186.
5. X. Xu et al., *MRS Symp.Proc.* **1026E** (2007) 1026-C08-04.
6. J. Spence *Experimental High-Resolution Electron Microscopy* (1988) (New York: Oxford University Press).

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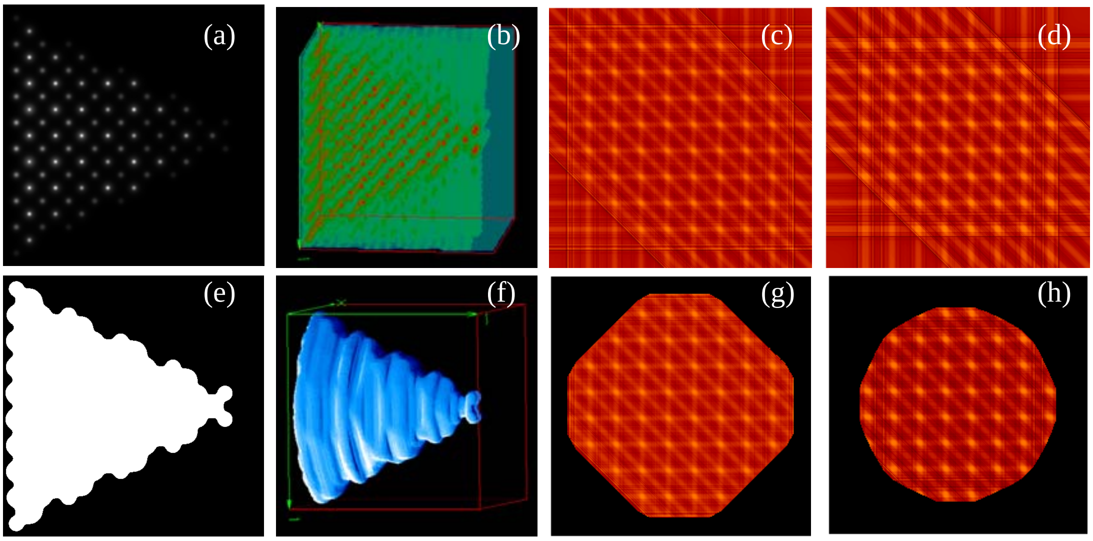

Figure 1. 0° imaginary part of the exit wave function of a CeO_2 conical supercell. (b) Reconstruction from 3 zone-axis projections (-45°, 0° and +45°), and slices through the volume (c,d). (e) Shadow projections after binarisation of (a); (f) the reconstructed shape from binary projections is used as a mask to superimpose the 3D shape information to the lattice-resolved tomogram (g,h).

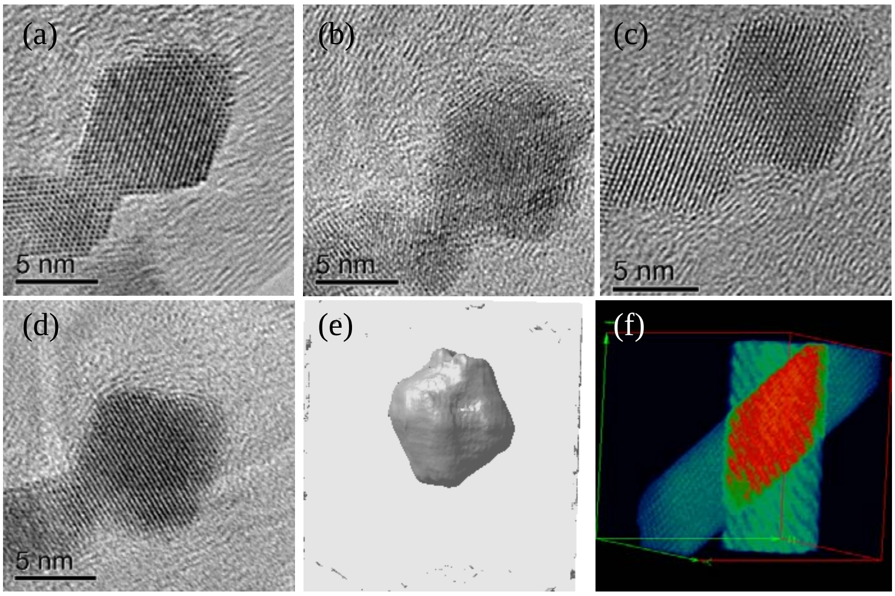

Figure 2. HREM projections of an isolated CeO_2 nanoparticle, at -50° (a), -10° (b), +10° (c) and +40° (d). (e) Shape-from-silhouette reconstruction from the full binarised tilt series. (f) Voxel projection view of the HREM reconstruction from the two zone-axis projections (a) and (b).

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