Electron tomography for nanoscale materials science

P.A. Midgley[1], M. Weyland[1], T. Yates[1], J. Tong[1], R. Dunin-Borkowski[1], J.M. Thomas[1], I. Arslan[1,2] and N. Browning[2]

1. Department of Materials Science and Metallurgy, University of Cambridge, Pembroke Street, Cambridge, CB2 3QZ, UK
2. Department of Chemical Engineering and Materials Science, University of California-Davis, One Shields Ave, Davis, CA 95616, USA

pam33@cam.ac.uk
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Electron tomography is being used with increasing regularity within the materials science community [1], implementing techniques developed originally for the life sciences. In particular, the tomographic 'projection requirement' necessitates the use of incoherent imaging modes for crystalline and strongly scattering objects, prevalent in materials science. This has led to the development of STEM-based tomography using a HAADF detector [2] and EFTEM tomography [3, 4], using core-loss edges to yield 3D compositional information.

Tomography is a two-stage process based on acquisition of a tilt series followed by a 3D reconstruction. The acquisition step is now largely an automated process for STEM and TEM-based tomography and it is quite common to record a series of images every 1 or 2 degrees from +70 to –70 degrees. Such angular sampling and high tilt range leads to high fidelity 3D reconstructions, computed using either standard weighted back-projection or iterative techniques incorporating constraints at each iteration.

The inherently high spatial resolution of STEM tomography lends itself to the imaging of nanoscale 3D objects. Here we show two examples of this. Firstly we show results, in Figure 1, from a Pt-Ru heterogeneous catalyst. This catalyst has been developed for the hydrogenation of *trans-trans* muconic acid (derived from sugar), converting it into adipic acid, a necessary precursor for the production of nylon [5]. The reactivity and selectivity depends critically on the distribution of the nanoparticles within its high surface area mesoporous silica support. MCM-41 has been used with 3nm diameter pores arranged in a hexagonal array. The reconstruction in (a) is shown in perspective. The spatial resolution is ~1nm in all three dimensions, proven by the clarity with which the pore structure is revealed and the reconstruction of the 1nm diameter nanoparticles of $Pt_{10}Ru_2$. In (b) a single pore is shown that corresponds to the circled pore seen in (a). This pore volume contains two nanoparticles. The ability to investigate the particles within each pore can yield unique information about the local catalyst loading and distribution.

Figure 2(a) shows four representative images from a tilt series recorded from a CdSe tetrapod supported on a holey carbon film. The film has curled to give a crescent-shaped support as seen in 2(b). The tetrapod is clearly reconstructed showing 4 distorted 'arms' each about 4nm in width. One of the arms has folded back to be parallel with another and there is evidence for secondary growth on the arms in the form of pyramids and similar shapes [6].

1. P.A.Midgley and M.Weyland, Ultramicroscopy **96** (2003)413.
2. P.A.Midgley, M.Weyland, J.M.Thomas and B.F.G.Johnson, Chem. Comm. **18** (2001) 907.
3. G. Mobus and B.J. Inkson, Applied Physics Letters **79** (9) (2001) 1369.
4. M. Weyland and P.A. Midgley, Microsc. Microanal **9** (2003) 542.
5. J.M. Thomas, B.F.G. Johnson, R. Raja, G. Sankar, and P.A. Midgley, Accounts of Chemical Research **36** (2003) 20.
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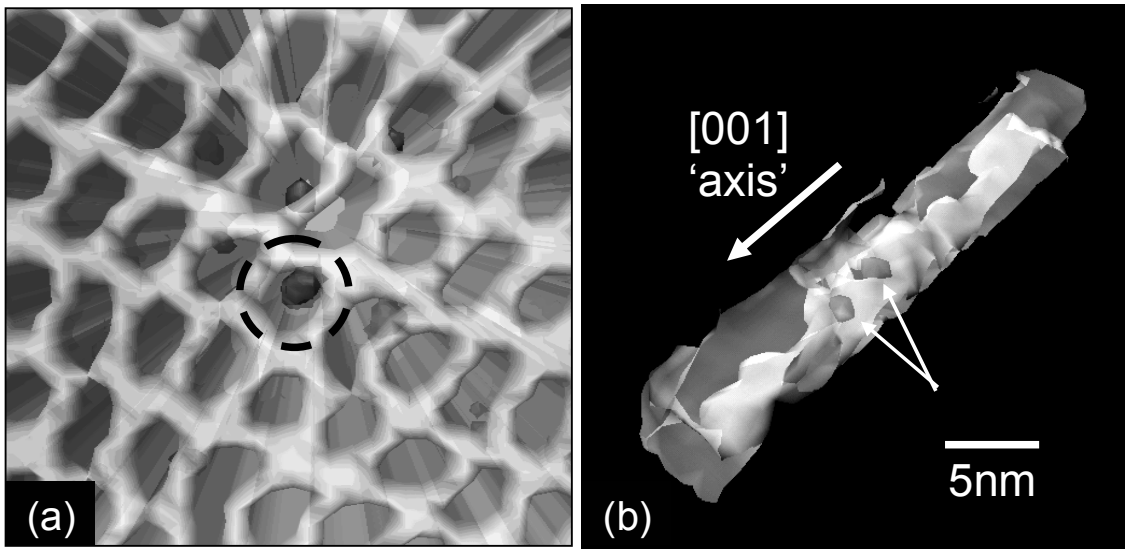

Figure 1. (a) Perspective view of MCM-41 mesoporous silica acting as support for catalytically active Pt-Ru nanoparticles. The pores are 3nm in diameter and each particle about 1nm in diameter. (b) A single pore (circled in (a)) is extracted from the 3D reconstruction to show the presence of two particles with approximately 4nm separation within this region of the pore.

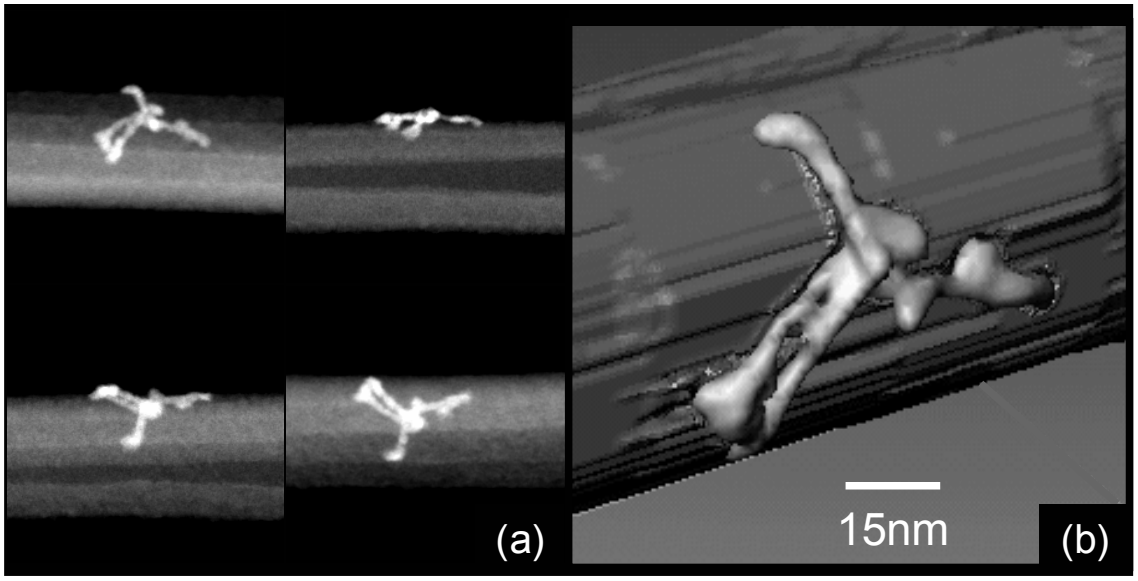

Figure 2. (a) 4 individual STEM HAADF images from the original tilt series of a CdSe tetrapod nanoparticle. The tetrapods are supported on a crescent-shaped carbon support film. (b) Surface render of the tetrapod reconstruction.