EFTEM Tomography on Nanomaterials

N.Y. Jin-Phillipp, C. T. Koch and P. A. van Aken

Stuttgart Center for Electron Microscopy, Max Planck Institute for Metal Research
Heisenbergstr. 3, 70569 Stuttgart, Germany

nyjin@mf.mpg.de
Keywords: tomography, EFTEM, carbon nanotube, copper whisker

TEM images are two-dimensional projections of a three-dimensional (3D) structure. In many cases, particularly in nano-sciences, having a complete picture of the structure in all three dimensions is very important. BF image contrast from most specimens in materials science which are of crystalline nature depend almost entirely upon the diffraction condition of the crystal, and therefore cannot be used for tomography. Energy-filtered TEM (EFTEM) is considered to be an alternative imaging mode for tomography [1, 2], provided that multiple scattering can be neglected and that the diffraction contrast due to orientation is low. Being element specific and electronic-state sensitive, EFTEM tomography may provide 3D information not only of the shape but also of the composition or electronic state of the nano-objects. Here, we report on our 3D EFTEM tomography study of multi-wall carbon nanotubes (CNTs) filled with iron and of copper nano-whiskers.

Tilt-series of EFTEM images were acquired with a Zeiss EM912 microscope operated at 120kV using the Gatan EFTEM tomography plug-in for Digital Micrograph. The core-loss elemental maps were obtained by the three-window method (acquisition of 2 pre-edge images for extrapolating the background which was then subtracted from the post-edge image). The alignment of the elemental map tilt series by cross-correlation and the reconstructions using weighted back-projection (WBP) and simultaneous iterative reconstruction technique (SIRT) were performed with the software package Inspect3D of FEI.

For the CNT filled with Fe tilt-series of both C-K and Fe-L_{23} EFTEM images were acquired at angles ranging from +60° to -60° in 2° steps. Figure 1(a) shows a 2D image of C-K and Fe-L_{23} elemental mappings at 0° tilt. The two tilt-series of the elemental maps were aligned simultaneously, and the volume marked by the white rectangle in Figure 1(a) was reconstructed. The three orthogonal views of the 3D reconstruction by SIRT with 10 iterations are shown in Figure 1(b), (c), and (d), where the *x*-*y* plane is the image plane at 0° tilt. Assuming a round cross-section of the CNT the elongation factor along *z* from the SIRT reconstruction is ~ 1.15, much lower than that from the WBP reconstruction (not shown here) and the expected value of 1.55 for the tilt range of ±60°. Interestingly, the CNT is revealed to be incomplete at its top so that the Fe catalyst particle seems not to be enclosed. An extended EELS study shows that the crown of the catalyst particle contains oxygen and the iron there is oxidized. These results may be useful for further understanding of the growth of the CNTs, as well as their applications.

Tilt-series of both C-K and Cu-L_{23} EFTEM images of a copper whisker were acquired at angles ranging from +60° to -50° in 2° steps. Figure 2(a) shows a 2D image of the C-K and Cu-L_{23} elemental mappings at 0° tilt. The two tilt-series of the elemental maps were aligned simultaneously and the tip part of the whisker inside the white rectangle was reconstructed using SIRT. The 3D reconstruction viewed along *z* is shown in Figure 2(b). It may be seen that, though being obscured by the elongation along *z* and artifacts of reconstruction, the whisker is faceted and that C distributed irregularly on side-facets.

In addition to the many factors influencing the resolution of 3D reconstruction in tomography, which have been widely discussed in the literatures, e.g. tilt range, alignment of

G. Kothleitner, M. Leisch (Eds.): MC2009, Vol. 1: Instrumentation and Methodology,
DOI: 10.3217/978-3-85125-062-6-035, © Verlag der TU Graz 2009

projections, and reconstruction techniques, we find that the accuracy of tomographic reconstruction needs to be extensively discussed, e.g. the effects of the mechanical instability of the tilt-axis, tilt increment and specimen drift, beam damage, contamination, and the diffraction contrast. Our preliminary results of EFTEM tomography with conical illumination demonstrate that the contrast of the EFTEM images due to the effect of crystal orientation has been substantially eliminated.

1. P.A. Midgley and M. Weyland, Ultramicroscopy **96** (2003) p413.
2. C. Kübel, et al., Microsc. Microanal. **11** (2005) p.378.
3. The authors acknowledge financial support from the European Union under the Framework 6 program under the contract for an Integrated Infrastructure Initiative Reference 026019 ESTEEM. They thank Kersten Hahn for his assistance and Nicole Grobert (Oxford University) and Gunther Richter for providing the CNTs and copper whisker sample respectively.

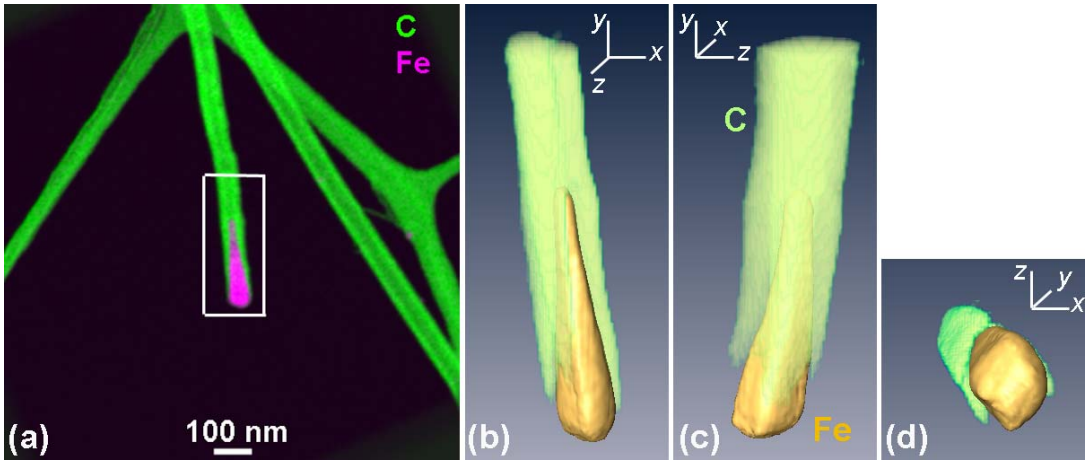

Figure 1. (a) Combined C-K (green) and Fe-L_{23} (purple) elemental maps of an Fe-filled CNT at 0° tilt. The reconstruction volume is marked by the white rectangle. (b)-(d) Three orthogonal views of the Fe filled nanotube reconstructed by SIRT, where *x*-*y* is the image plane at 0° tilt. Note the slight elongation along *z*, parallel to the direction of the incident electron beam.

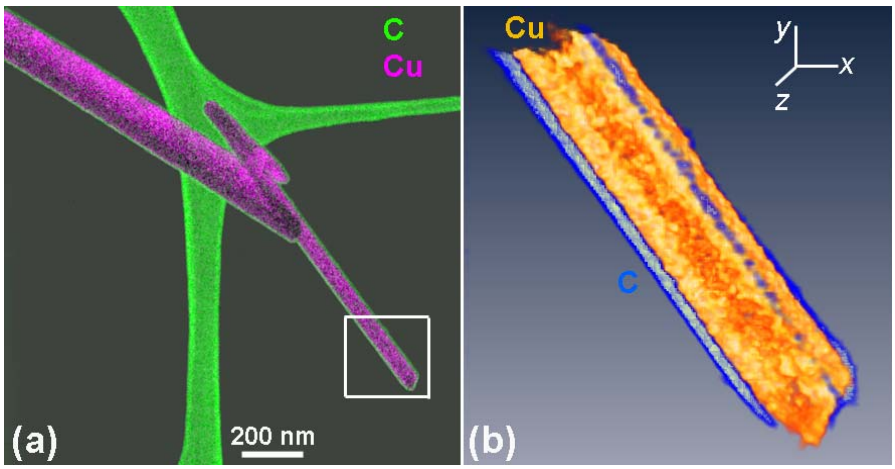

Figure 2. (a) Combined C-K (green) map and Cu-L_{23} (purple) elemental maps of a copper whisker at 0° tilt. The reconstruction volume is marked by the white rectangle. (b) SIRT reconstruction viewed along *z*.

DOI: 10.3217/978-3-85125-062-6-035,