doi:10.1017/S1431927610057302
Microsc. Microanal. 16 (Suppl 2), 2010
© Microscopy Society of America 2010

3-D Complete-Tilt Electron Tomography of Semiconductor Nanowires

Jinsong Wu*, Sujing Xie, Eric R. Hemesath, Lincoln Lauhon, Vinayak P. Dravid*

Department of Materials Science and Engineering, *The NU*ANCE* Center, Northwestern University, Evanston, IL, 60208, USA

Electron tomography approach is rapidly gaining prominence for solving 3-D morphology and structure of physical and biological materials [1]. The spatial resolution of electron tomography achieved in image-based 3D reconstructions is about 1 nm [2]. However, one of the well recognized challenges that prevents further improvement of resolution and spatial correlation is the “missing wedge” problem; conventional copper grids preclude TEM image recording at very high tilt angles. This missing wedge problem can be solved by using cylindrical specimens without any supporting grid, such that the thickness along the electron incident direction remains unchanged (or height-adjusted) upon rotation about the cylindrical axis [3]. Here, we report 3-D electron tomography of Ge nanowires by an innovative specimen mounting approach and collecting images with a full-space tilting holder.

Fig.1 shows a TEM image and diffraction pattern of a Ge nanowire, synthesized by chemical vapor deposition in a Au-catalyzed vapor-liquid-solid growth process. The usual approach for attaching single nanowires to the tip of full-space holder with a micromanipulator coupled to electron beam based welding system in the FIB is often difficult due to severe damage and contamination. We have developed an alternative sample preparation method to mount nanowires for tomographic analysis. The Ge nanowire synthesis was carried out on silicon micro-posts 50 µm in length and 5 µm in diameter. Fig. 2(a) shows an SEM image of one such micro-post. The micro-post was then transferred, as a whole, to the tip of the full-space tilting holder. The end of the micro-post, which supports many nanowires, was then welded to the end of the micromanipulator (Fig. 2b), for subsequent S/TEM. Three of the tilted STEM images are shown in Fig.3. The reconstructed 3D structure of the Ge nanowire is shown in Fig.3 (d). The satisfactory quality of specimen preparation protocol is evidenced by the visibility of clear {112} facets in the reconstructed volume, and resolution of secondary particles on the nanowire surface. The remainder artifacts in the reconstruction are attributed primarily to electron induced contamination and evaporation during data collection during the image acquisition process. Only three {112} planes can be distinguished in Fig. 3(e) due to the accumulation of contamination (amorphous carbon) during imaging. The presentation will highlight the integration of innovative specimen preparation with full-tilt tomography that will be invaluable to advancing the 3-D analysis of nanostructures. [4]

References: 1. S.J.L. Billinge and I. Levin, *Science* **316** (2007) 561-565; 2. P.A. Midgley, J.M. Thomas, L. Laffont, M. Weyland, R. Raja, B.F.G. Johnson and T. Khimyak, *J. Phys. Chem. B* **108** (2004) 4590-4592; 3. N. Kawase, M. Kato, H. Nishioka and H. Jinnai, *Ultramicroscopy*, **107** (2007) 8-15; 4. This work was performed in the EPIC facility of NU*ANCE* Center at Northwestern University. The tomography approach development is supported by US DOE-BES (VPD). The NW research is supported by a seed grant from the Center for Catalysis and Surface Science (CCSS) at Northwestern and NSF grant (DMI-0507053).

https://doi.org/10.1017/S1431927610057302 Published online by Cambridge University Press

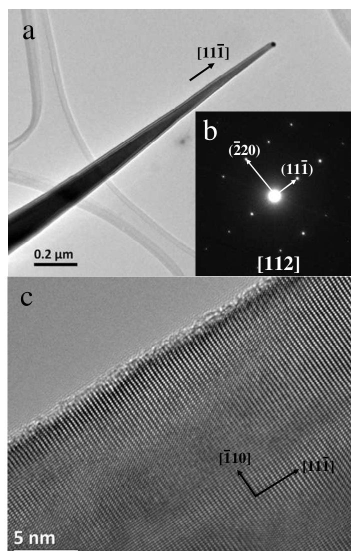

Fig.1 (a) A low magnification TEM image of a faceted Ge nanowire; (b) Its corresponding diffraction pattern, which shows the nanowire is resting on a {112} facet and the nanowire growth direction is along the <111> direction. (c) HREM image of the Ge nanowire, where {111} and {110} lattice planes are labeled.

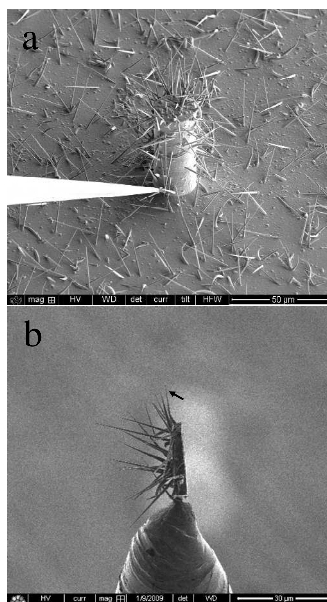

Fig.2 (a) A micropost where Ge nanowires were fabricated and then transferred. (b) A SEM image of a Si micropost with Ge nanowires welded to the tip of the Hummingbird holder. STEM tomography was carried out on the nanowire indicated by the black arrow.

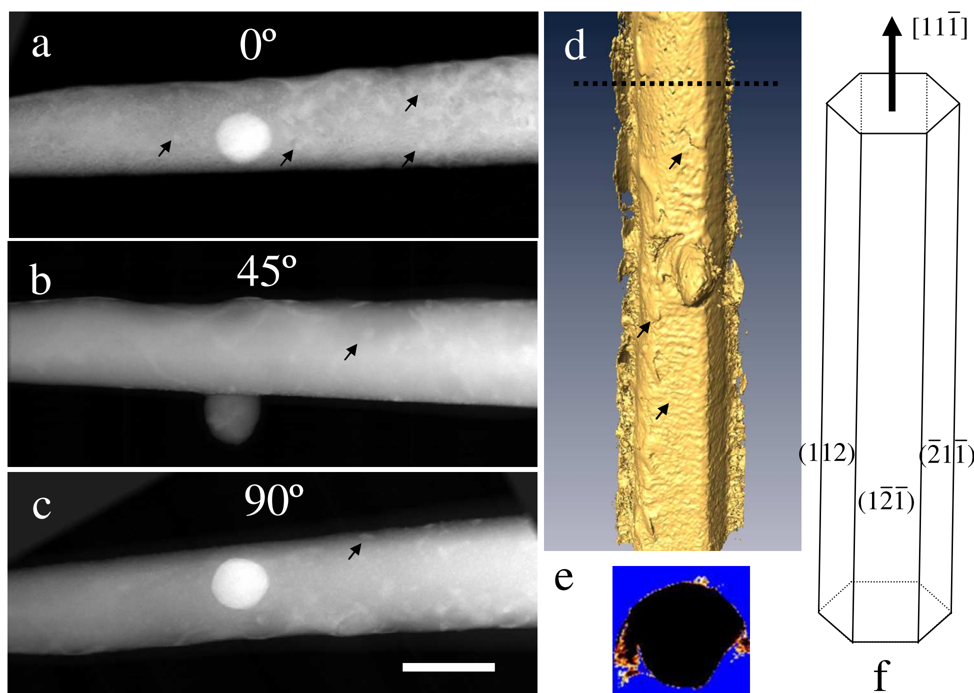

Fig.3 (a)-(c) Z-contrast STEM images of a Ge nanowire (the scale bar is 150 nm) tilted at 0, 45 and 90 degree, respectively. The small secondary particles were shown by black arrowheads. (d) The surface of reconstructed volume of the Ge nanowire. The black line shows the position where the section in (e) is generated, while black arrowheads show the particles on the surface. (e) A cross section of the reconstructed volume at the black dotted line in (d). Three {112} facets can be seen, while the other three are smoothed out by the curved surface due to carbon contamination. (f) An illustration of the facets morphology of the Ge nanowire, where three {112} facets and the growth direction are indicated.

https://doi.org/10.1017/S1431927610057302 Published online by Cambridge University Press