doi: 10.1017/S1431927609098262
Microsc Microanal 15(Suppl 2), 2009
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Electron Tomography in Materials Science

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The three-dimensional reconstruction of nanoscale objects typically encountered in materials science problems faces an additional set of challenges and opportunities from that of life sciences applications. Many materials are able to withstand electron doses many orders of magnitude beyond what would destroy biological tissue, resulting in resolution limits set by the performance of the instrument, not the dose limitations of the sample. At the same time, the presence of crystalline and strongly scattering materials can seriously complicate the reliable reconstruction of materials samples. Once dose constraints are removed, there are many methods[1-4] to improve the reliability and contrast – each with its own benefits and tradeoffs.

For materials that are both low-density and amorphous, bright field TEM is a dose-efficient and rapid option for data collection. However, even in structures as small as 5 nm artifacts can occur – such as solid Pt nanoparticles appearing to contain voids in the thickest part due to contrast reversals from dynamical scattering[1]. A basic requirement is that the signal used must be monotonic (but not necessarily linear) in projected sample thickness. If this projection requirement is not met then it is not possible to distinguish between a high-density precipitate or a void. High-Angle Annular Dark Field (HAADF) STEM greatly extends the range of samples than can be studied[3] and provides a monotonic signal in samples thinner than 50-100 nm, but it also shows contrast reversals in strongly-scattering or thicker sections. Fig 1a shows a contrast reversal in a cut from a HAADF reconstruction of Ta/TaN layer in an integrated circuit, where the densest part of the liner appears incorrectly as a void[2]. The contrast reversal in the HAADF image resulted from electrons being scattered beyond the outer diameter of the HAADF detector (Fig 1c). By instead recording the complement of the ideal HAADF signal (including the signal lost from the real detector) using an incoherent bright field (IBF) geometry the contrast reversal can be avoided (Fig 1b) and samples as thick as a micron can be reliably reconstructed[2].

Chemical information can also be obtained from energy-filtered images provided the material is not strongly diffracting. Fig 2a shows that there is little contrast between a-Si and a-SiO_2, but an EFTEM map in the 17 eV Si Plasmon (Fig 2b) reveals the a-Si nanoparticles hidden in Fig 2a. The low-loss EELS signals have very strong inelastic signals, allowing for reconstructions with a few nm spatial resolution[4], but details below 2-4 nm can be altered or lost due to delocalization of the inelastic signal[5]. For strongly diffracting materials, the EFTEM signal is no longer monotonic. The larger collection angles available in aberration corrected STEM would restore a monotonic signal (analogous to IBF), however detector readout times must be greatly reduced before it is possible to record full spectrum images at each tilt in a practical time [6].

[1] S. Bals *et al.*, Microsc. Microanal. **11**, 2118 (2005).
[2] P. Ercius *et al.*, Appl. Phys. Lett. **88**, 243116 (2006).
[3] P. A. Midgley *et al.*, Chemical Communications, 907 (2001).
[4] A. Yurtsever, M. Weyland, and D. A. Muller, Appl. Phys. Lett. **89**, 151920 (2006).
[5] M. Couillard, A. Yurtsever, and D. A. Muller, Physical Review B **77** (2008).

https://doi.org/10.1017/S1431927609098262 Published online by Cambridge University Press

[6] Plasmon tomography with A. Yurtsever and M. Weyland. Research supported by the Semiconductor Research Corporation, the Cornell Center for Materials Research, an NSF MRSEC and the Cornell Center for Nanoscale systems (NSF and NYSTAR).

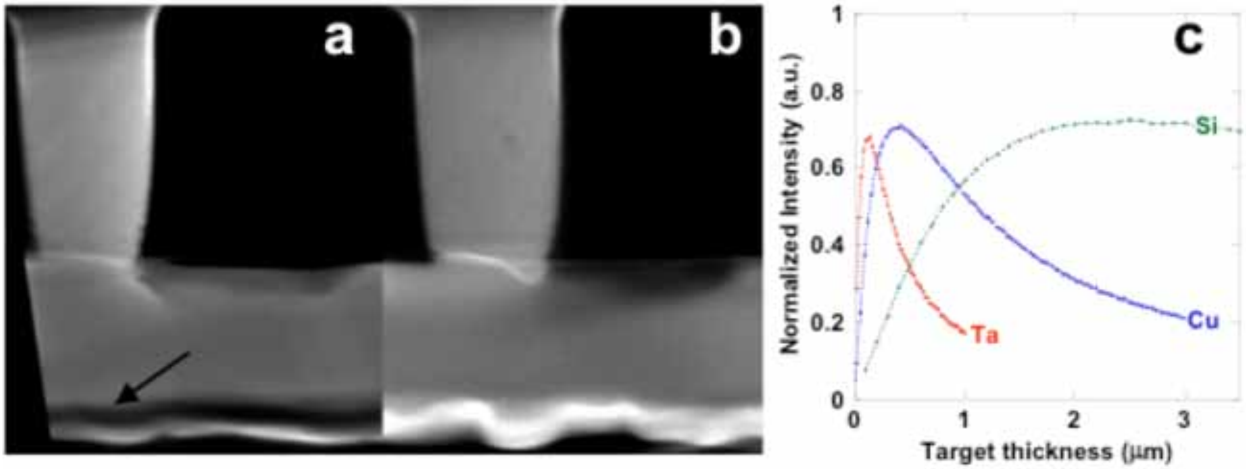

FIG. 1. Comparison of HAADF and IBF for tomography. (a) shows a cut through a 3D reconstruction of a 250 nm diameter Cu Via contacting a Cu pad recorded by HAADF. The thicker part of the Ta/TaN liner below the pad has reversed contrast and appears incorrectly as a void (arrow). (b) shows the same region recorded by IBF (with signal inverted for comparison). The contrast of the Ta/TaN liner is now correct and its roughness is apparent. The void to the right of the via is also more clearly defined. (c) HAADF signal vs sample thickness, showing a contrast reversal when electrons are scattered outside the outer edge of the detector (From [2]).

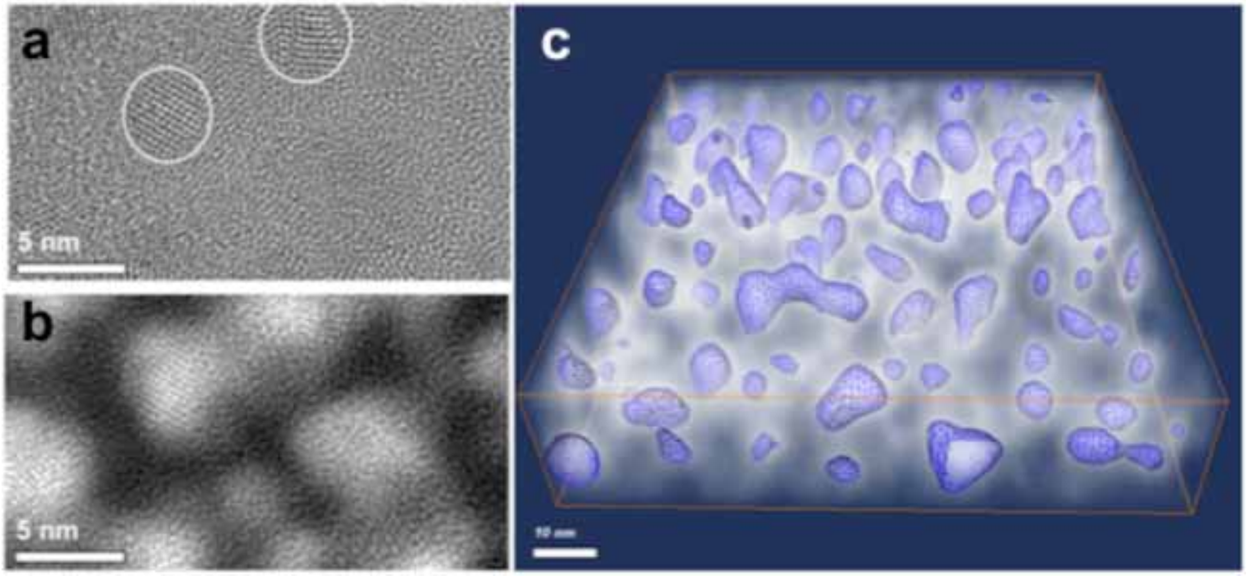

FIG. 2. Plasmon tomography of Si nanoparticles in SiO_2. (a) Bright-field HRTEM imaging shows no detectable contrast between a-Si and a-SiO_2 – only c-Si particles aligned with the beam are visible. (b) EFTEM of the same region at the 17-eV Si plasmon peak shows a high density of Si nanoparticles, most of which were invisible to elastic scattering. (c) A full 3D reconstruction of the Si nanoparticles using the Si Plasmon signal. (From [4]).

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