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IT-10-P-3102 Compressed-sensing EDX Tomography of Composite Nanowires

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The introduction of high solid angle EDX detectors has seen a renaissance in interest in EDX-based electron tomography [1]. The confined geometry of the TEM however makes the EDX spectrum prone to artefacts especially for samples tilted to high angles or mounted on copper grids [2]. Here we examine these issues further by analysing an EDX spectrum-image tilt series of a composite nanowire structure.

The sample, a Cobalt phthalocyanine (Co Pc) zinc oxide core-shell nanowire, has been studied using an FEI Tecnai Osiris equipped with a large solid angle (>0.9 srad) silicon drift detector (SDD). The ZnO nanowire shell is polycrystalline and the level of detail reconstructed in the tomogram should allow comparative assessments of spatial resolution and uniformity of grey levels in the reconstruction.

A series of EDX spectrum-images together with STEM HAADF images was recorded at 5° tilt increments with equal acquisition time (Fig 1a). The total counts in each spectrum-image for each element of interest are plotted as a function of tilt in Fig 1b. As has been seen previously the counts at high angles increase dramatically. At low angles there is a small drop in counts up to around ±20°; this is likely to be attributable to geometric shadowing of the EDX detector by the specimen holder.

The trend in the count increase is typified by the Cu signal which we believe arises primarily through the excitation (via scattered electrons) of x-rays originating from the copper support grid. As the grid is tilted the area of copper in line of sight of the scattered electrons increases by a simple geometric factor equal to approximately 1/cos(tilt angle), resulting in an increase in Cu signal. This function is plotted in Fig 1b and the fit to the Cu signal is good.

We decided to use the Zn signal in the nanowire as a test case; the increase in the signal mirrors to a large extent that seen in the Cu (and Co). As a first approximation, in order to use this tilt data for a reconstruction, we normalised the Zn signal at each tilt increment, given each map had the same acquisition time. We used compressed sensing (CS) methods [3] to reconstruct the Zn tomogram and compared the result with a more conventional SIRT reconstruction (see Fig 2). Two advantages of CS reconstruction are apparent: i) the morphology, seen in the cross-sectional slice, is more faithfully reproduced (c.f. STEM HAADF reconstruction) and ii) greyscales within the ZnO phase are more uniform.

Further work is underway to confirm the origins of the x-ray signal variation with tilt in order to move towards a true quantitative compositional tomogram.

[1] Möbus et al. Ultramicroscopy 2003,96(3-4),433-451
[2] Slater et al. Proceedings of EMAG Conference 2013
[3] Leary et al. Ultramicroscopy 2013,131,70-91

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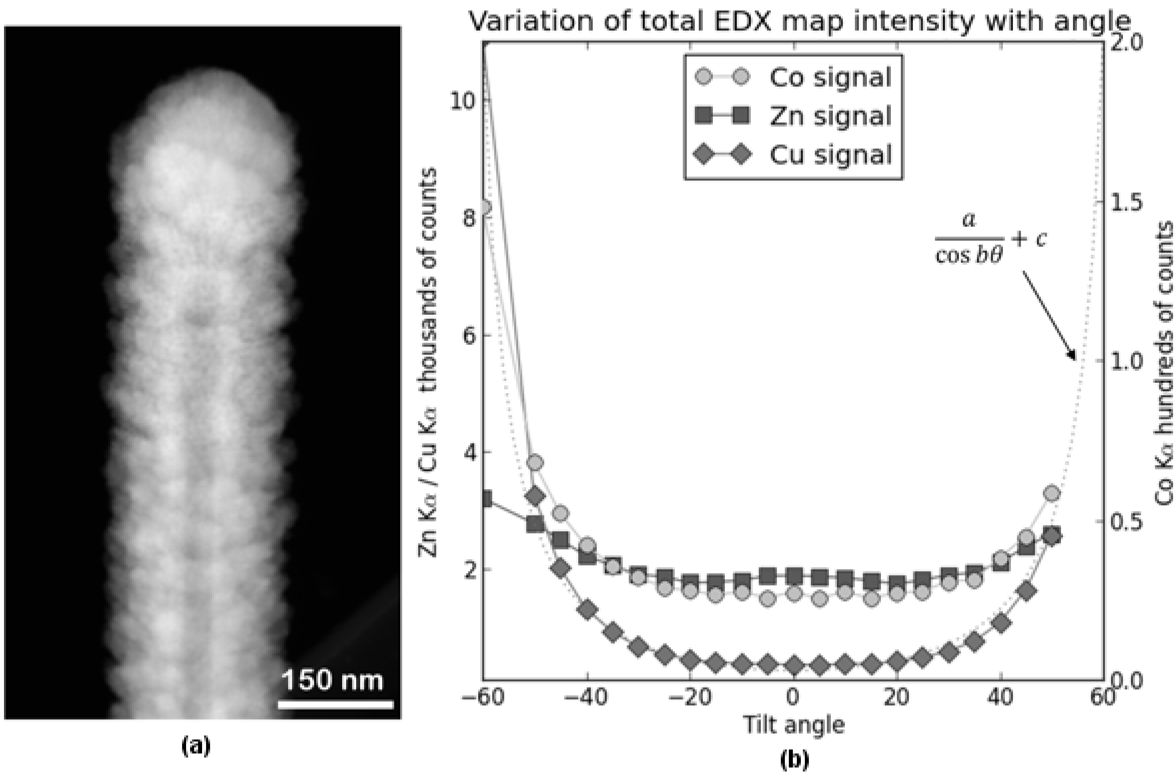

Fig. 1: (a) 0° tilt STEM HAADF image of Co Pc-ZnO core-shell nanowire mounted on 5 nm C-film, the tilt axis is vertical, (b) Total counts summed over EDX maps for Co, Cu and Zn Kα peaks for varying tilt angle with function 1/cos(tilt angle) fitted to Cu distribution. Acquired with a probe current of 0.7 nA, 45x45 pixels and dwell time of 40 ms.

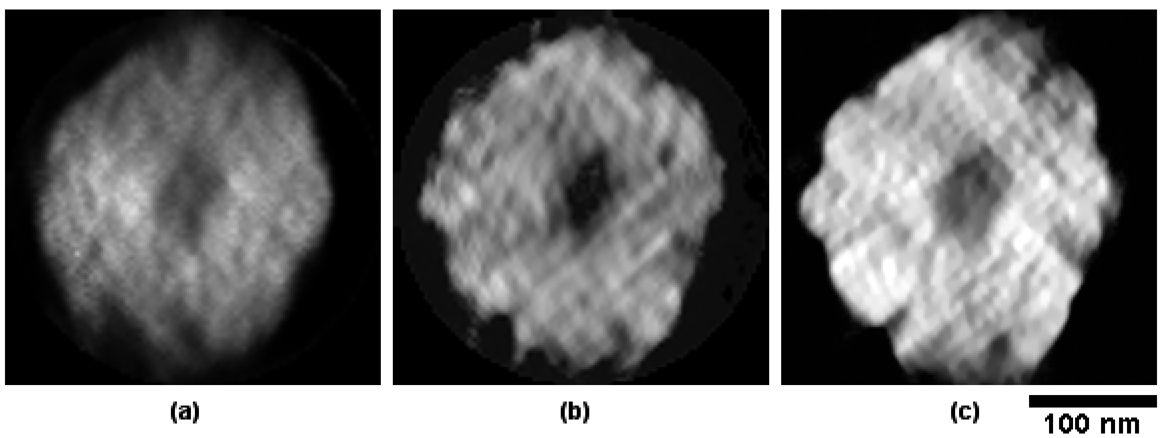

Fig. 2: (a) and (b): Slices through tomographic reconstructions of the Zn Kα peak (integrated over 8.49 – 8.79 keV): (a) 30 iterations SIRT reconstruction performed in Inspect3D, (b) CSET reconstruction. (c) Slice through tomographic reconstruction of STEM HAADF tilt series using CSET.