Mitigation of thickness effects in defocused electron probe ptychography of crystalline materials

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Ptychography determines quantitatively the amplitude and phase shift imparted to an electron beam as it passes through the specimen being observed in a transmission electron microscope (TEM). Using a defocused electron probe and a fast direct-electron detector, allows a relatively large area of the sample to be rapidly surveyed, as we show here. The time limiting step then becomes the reconstruction process, though as we show here the extended ptychographical iterative engine [1] can determine the sample complex transmission function within practical timescales. The complexity of this algorithm is reduced, and hence the speed of calculating useful reconstructions is improved, using the assumption that the sample behaves as a simple multiplicative phase object. However, many crystalline samples, particularly above certain thicknesses and in certain orientations, exhibit strong multiple scattering and thus might not be properly represented within this algorithm.

However, with some samples that have an extinction distance similar to its thickness, useful reconstructions can still be realized in some experimental configurations [2]. Aspects concerning the sample thickness, beyond the weak phase object approximation, have been treated more thoroughly within the currently dominant X-ray and optical applications of ptychography. Here it has been found that propagation within the sample is only adequately considered when the sample thickness $t \leq 5.2\ \Delta r^2 / \lambda$, where λ is the wavelength, and Δr is the image resolution [3], but the validity of this useful relationship appears to have hitherto not been experimentally investigated in electron microscopy.

Here experiments were performed and facilitated using a recently available, high-speed electron detector synchronized to the beam scan, the prior absence of which has hitherto hindered such explorations. Typical 80 nm square field of view (FOV) regions were observed and reconstructed on a range of samples having thicknesses between ~15 nm to 100 nm, using approximately 5000 overlapping, ~5 nm diameter illumination regions. Diffraction images were collected at 500 frames per seconds or greater, using a single Medipix 3RX chip and Merlin system [4] on a Hitachi HF3300V TEM operated at 300 keV, allowing data from this FOV to be collected in under 10 seconds.

Looking at example phase reconstructions of ~5 nm gold particles (Fig. 1), ~20 nm $NaHoF_4$ (Fig. 2) particles and silicon transistor devices (Fig. 3a and 3b) with total sample thicknesses up to around 80 nm allowed us to see lattice fringes with period of 0.142, 0.180, and 0.192 nm respectively, without any need for an aberration corrector in the microscope. As expected the overall reconstruction quality appeared best for the thinner samples. With the thicker samples, and particularly with beam sensitive materials, care had to be taken to both minimize the electron dose and orient the samples such to reduce the effects of dynamical diffraction appearing in the reconstructions. From this study it appears that the relationship developed by Tsai *et al* [3] proves useful guide for electron microscopy too. Further comparisons made in this work between holography and ptychography, also illustrate the capability of ptychography.

References:

[1] A M Maiden, J M Rodenburg, Ultramicroscopy **109** (2009), p. 1256

[2] J M Rodenburg, Advances in Imaging and Electron Physics **150** (2008), p. 87

[3] E H R Tsai, *et al*, Optics Express **24** (2016), p. 29089

[4] R Plackett, et al, Journal of Instrumentation **8** (2013), p. C01038

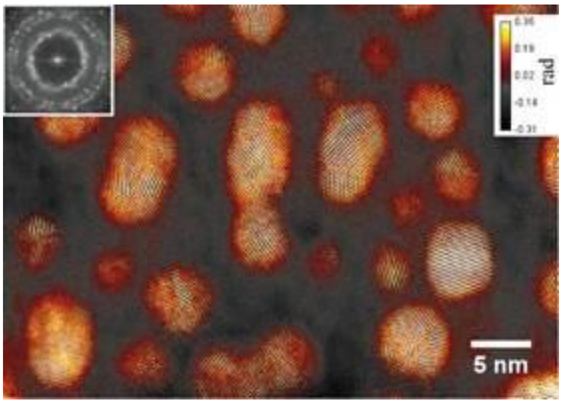

Figure 1 - Reconstructed phase image of (a) gold particles on a carbon support film. Inset: image FFT.

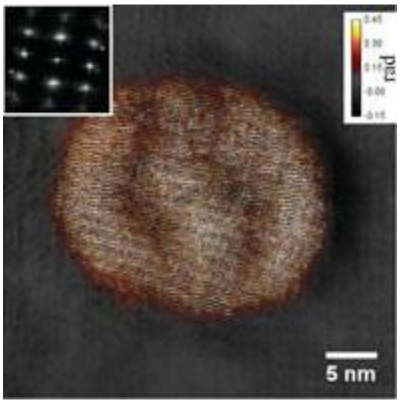

Figure 2 - Reconstructed phase image of a $NaHoF_4$ particle. Inset: image FFT.

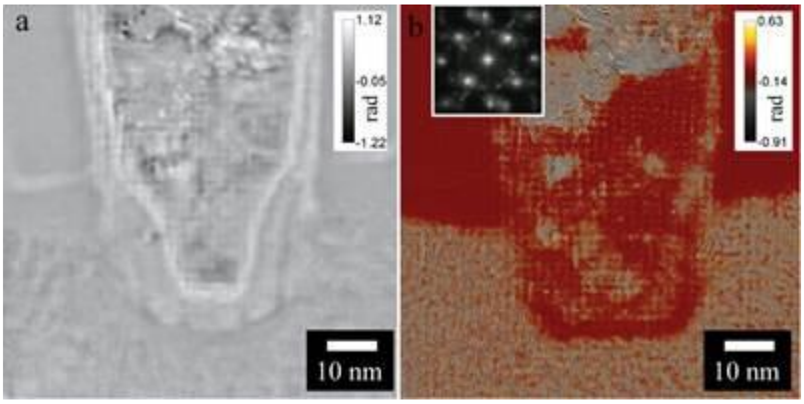

Figure 3 - Reconstructed phase from a silicon transistor region beneath a flash memory stack, separated into its (a) low and (b) high spatial frequency components. Inset: image FFT.