Transition from quantitative to geometric tomography

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Abstract. Electron tomography allows the three-dimensional (3D) quantitative characterization of nanostructures, provided a monotonic relationship is fulfilled between the projected signal and the atomic number and thickness of the specimen. This requirement is not satisfied if the micrographs are affected by (i) diffraction contrast, (ii) absorption-thickness limit or (iii) detector saturation. The effects of non-monotonic tomography acquisition have been examined using computer simulations and experimental tilt series of conical tungsten tips. It is shown that the reconstruction artefacts arising from non-linearity can be best predicted by considering geometric tomography (which reconstructs the object external shape) and quantitative tomography (which reconstructs the 3D density function) as limiting cases.

1. Introduction

Electron tomography [1] is a non-destructive technique commonly used for the 3D characterization of sub-micron sized materials with nanometer resolution. For a reliable tomography reconstruction, a monotonic relationship is required between the projected intensity and the atomic number and thickness of the specimen. Bright field transmission electron microscopy (BF-TEM) projections of common biomedical objects (carbon-based/amorphous) fulfill this requirement [2]. However, the crystalline structures encountered in materials science induce diffraction contrast in the BF-TEM projections. To overcome this limitation, incoherent imaging modes such as energy filtered TEM (EFTEM) and Z-contrast (dark field) TEM and STEM have been introduced [3-6]. However, detector saturation and/or absorption effects still fail monotony in the projected intensities. In this paper, we explore the effects of non-monotonic image acquisition and thickness limits using simulated cone-shaped objects as well as some experimental tilt series of tungsten tips, which are widely used for nanoindentation experiments. We relate the observed artifacts due to non-linearity to the ‘shape from silhouette’ technique, commonly used in the pattern recognition community [7] for the approximation of shapes from binary projections (shadows). This technique is precise under restricted object geometries and is an alternative to quantitative tomography when the latter can not be applied.

2. Tomography simulations

In a tomography experiment, the recorded intensity in every viewing direction should be linearly proportional to the integrated product of density and a function of the scattering factor (involving atomic number) of the elements present in the sample. Practically, a transfer function \( T \) (e.g. exponential absorption) modeling non-linear image acquisition (Figure 1(a)) will change the data into \( I' = T(I) \). \( T \) is divided into three simplified regions: (1) Linear (or linearizable) for \( 0 < I < I_{lim1} \), (2) saturated for \( I_{lim1} < I < I_{lim2} \), (3) non-monotonic region for \( I > I_{lim2} \). \( I_{lim1} \) and \( I_{lim2} \) depend on the density and

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atomic number of the specimen and the TEM imaging modes and settings. To identify artifacts related
to this transfer function, an Interactive Data Language IDL (RSI, USA) code has been written for
generating a cone-shaped object (Figure 1(b)), calculating its projection images, applying \( T \) and
reconstructing the 3D volume by filtered and unfiltered back-projection of the modified images.
Furthermore, the projections have been binarised and backprojected without filtering. This approach is
called ‘shape from silhouette’ (SFS) within the field of Geometric Tomography [8], and allows the
retrieval of 3D shapes of convex objects.

![Diagram](image)

**Figure 1.** (a) Intensity transfer curve for a specimen wedge, showing the linear (1), saturated (2) and
non-monotonic (3) regions; (b) a simulated cone-shaped object.

Figure 2(a) shows a linear projection of the simulated cone in the z direction. Figures 2(b) and 2(c) are
slices through the reconstruction from the simulated tilt series, showing the expected homogeneous
core.

![Images](image)

**Figure 2.** (a) Projection of the simulated cone in the z direction; Slices through the reconstructed
volume in the x-y (b) and x-z (c) planes; (d) slice through the non filtered reconstruction.

The effects of a non-linear acquisition regime were explored by applying the intensity transfer
function shown in Figure 1(a) to the tilt series of the simulated cone. Figure 3(a) shows a modified
projection at 0°, while slices through the reconstruction (Figure 3(b) and 3(c)) show an erroneous core-
shell structure introduced by inconsistencies in such tilt series. In the extreme case where the
projections are binarised (Figure 4(a)) (detector saturation), the reconstructed volume has the correct
shape but exhibits enhanced edges (Figure 4(b) and 4(c)). If the SFS is applied, whereby the binarised
projections are back-projected without applying a ramp filter, we obtain a homogeneous core and
perfect shape retrieval in this case (convex object) (Figure 4(d) and 4(e)).

![Images](image)

**Figure 3.** Modified intensity projection (a); Slices through the reconstruction (b-c) showing an
erroneous core-shell structure.
3. Intermediate Cases of Non-linearity

Unfiltered backprojection of convex objects generates an artificial density-gradient inside a constant-density object (see Figure 2(d)). This is due to the radial distribution of intensity data in polar coordinates, which become integrated during backprojection. This artificial intensity-dome is rectified by the ramp-filter in filtered backprojection. Any saturation, damping or absorption in the projections leads to missing counts and a depletion of the intensity in the dome before filtering. In mild cases, the filter then over-compensates and causes a dip in density in the core of the object. In severe saturation events, the depletion widens and only bright edges are remaining, which turns the object into an edge-enhanced (as if high-pass filtered) version. The latter case leads to SFS reconstructions with (unnecessary) ramp filter applied to the binary shadow images. These considerations still apply roughly upon generalisation from cylindrical to convex objects.

4. Experimental Applications

Tungsten tips were prepared and mounted as reported in [9]. BF-TEM and ADF-STEM tilt series of the same W tip were acquired across the range -60º to +60º with 5º increment using a JEM 2010 FEGTEM (Jeol, Japan) at 200kV. The DF-TEM tilt series was recorded using a JEM 3010 TEM (Jeol, Japan) at 300kV and tilting the specimen from -80º to +80º with 5º increment. The tilt series were then exported to IMOD [10] for the cross-correlation alignment and the reconstruction by filtered back-projection. Figure 5(a) shows a BF-TEM projection of a W tip with oxide layer and gold particles attached to it. The crystalline W core is affected by diffraction contrast. The BF-TEM reconstruction shown in Figure 5(b) is noisy but shows a constant core (line profile Figure 5(c)). The ADF-STEM projection Figure 5(d) shows a more linear projection in the core, but with slightly lower resolution than BF-TEM. The ADF-STEM tomogram in Figure 5(e) shows a well-reconstructed tip with good signal-to-noise ratio (SNR), but as of Figure 5(f) some intensity dip in the core suggests a mild saturation/thickness problem.

The tomographic reconstruction in Figure 6(b) from binarised BF-TEM projections (Figure 6(a)) shows perfect shape retrieval, although the core is lost. The line profile Figure 6(c)) shows a
homogeneous inner structure and the edge enhancement effect of the binarisation. Finally, the shape of a 500 nm thick tip is reconstructed in Figure 6(e) from DF-TEM projections (see Figure 6(d)) where the intensity is affected by the multi-scattering (i.e. absorption) effects. The edge enhancement effect is again apparent on the line profile in Figure 5(f). Although the tomogram has a low SNR, the core is quite well reconstructed. These results show that valuable information about the external 3D shape can still be extracted from corrupted projections, even if the projection requirement is not fulfilled.

Figure 6. Binarised projection of a W tip (a) and a slice through the reconstruction (b), the 3D shape is well reconstructed despite the loss of quantitative information, the line profile (c) shows the edge enhancement as a result of the binarisation; DF-TEM absorption-affected projection of a thick W tip (d), slice through the tomogram 9(e) with enhanced edges (f) and well reconstructed core.

4. Conclusion
In summary, the 3D quantitative characterization of the surface and the interior of nanostructures can be achieved to various levels by different modes of electron tomography. Artifacts related to the thickness limit, diffraction contrast and detector saturation have been studied under different imaging modes. In the case of non-monotonic image acquisition, the projections still contain valuable information about the outer shape of the structures, which can be retrieved by back-projecting the binarised projections. The accuracy of the reconstructed shape depends on the convexity of the initial object and the number of projections available.

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References
[1] Frank J 2007 *Electron Tomography*, Plenum, New York, 2nd edition
[2] Koster A J, Grimm R, Typke D, Hegerl R, Stoschek A, Walz J, and Baumeister W 1997 *J. Struct. Biol.* 120 p 276
[3] Midgley P A and Weyland M 2003 *Ultramicroscopy* 96 p 413
[4] Möbus G and Inkson B J 2001 *Appl. Phys. Lett.* 79 p 1369; and *Ultramicroscopy* 96 p 433
[5] Xu X, Saghi Z and Möbus G 2007 *Nanotechnology* 18 p 225501
[6] Kaiser U and Chuvilin A 2003 *Microsc. Microanal.* 9 p 36
[7] Laurentini A 1994 *IEEE Transactions Pattern Analysis and Machine Intelligence* 16(2) p 150
[8] Gardner R J 1995 *Geometric Tomography*, Cambridge University Press, New York
[9] Xu X, Peng Y, Saghi Z, Gay R, Inkson B J and Möbus G 2007 *J. Phys. Conf. Ser.* 61 p 810
[10] Kremer J R, Mastronarde D N, McIntosh J R 1996 *J. Struct. Biol.* 116 p 71
[11] Saghi Z, Xu X, Peng Y, Inkson B J, Möbus G, *Appl. Phys. Lett* in press.