Unconventional Electron Tomography

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Electron tomography has become established as a powerful tool not only in the life sciences, for discerning the complex 3D cellular structures, but also as a technique of great use in materials science and in particular for the study of 3D nanomaterials and devices. In the life sciences, the imaging mode of choice is bright field (BF) imaging which normally ensures the maximum signal available given a certain dose, often the limiting factor in the study of organic samples. BF imaging is simple to perform and interpretation is relatively straightforward. For life science specimens, in the most part BF images satisfy the projection requirement and thus are suited to tomographic reconstruction. In materials science, dose is normally not such an issue and this has allowed more unconventional approaches to electron tomography. Indeed, in many cases, particularly for crystalline specimens, BF imaging does not satisfy the projection requirement and cannot be used for tomography. What is becoming conventional in materials science is to use scanning transmission electron microscopy (STEM) high-angle annular dark field (HAADF) images for electron tomographic reconstructions [1]. The scattering process can be thought of as incoherent in nature and HAADF images are almost free from unwanted diffraction (and Frsenel) contrast which can plague BF images. The high angular scattering of the collected electrons ensures that the image signal is dependent on the atomic number, Z, of the species from which they are scattered, and to a first approximation can be thought of as Rutherford-type scattering and as such the image intensity is approximately proportional to Z^2. STEM tomography has now been exploited across a range of disciplines within the materials science community.

Although these conventional techniques are very powerful, over the past few years, we have also considered complementary, somewhat more unconventional, approaches to electron tomography. Here we consider a few of those:

(i) STEM tomography for biological samples. It has long been accepted that STEM is not as dose-efficient as TEM for imaging life science specimens, although with the advent of a new generation of aberration-corrected STEM instruments with large collection angles this may need to be re-appraised. Nevertheless, some life science samples are more resistant to beam damage and can be imaged with less efficient modes such as HAADF. This is only worthwhile if the image signal provides new or better information but we have found that it is very well suited to the study of cellular material with internal inorganic structures. We will show two examples of this, firstly the reconstruction of magnetite crystals within magneto-tactic bacteria in which the facetting of the cubic magnetite is revealed, and secondly the distribution of ferritin within a liver cell of a haemochromatosis patient and the remarkable 3D array of the iron-rich cores.

(ii) 3D chemical mapping using low-loss electrons and volume-spectroscopy. Combining energy-filtered imaging (EFTEM) with electron tomography has now been undertaken for some years. 3D elemental maps are possible to obtain from a variety of specimens using core-loss edges. It is also possible to use information in the low loss region of the spectrum (e.g. volume plasmons) in order to retrieve chemical information about the sample of interest. We will show one example, of multi-walled carbon nanotubes (MWNTs) covered in a nylon sheath, where conventional 3D EFTEM is not appropriate, because both components are carbon-based, and a more unconventional approach has had to be used. By forming an image from the ratio of two low-loss images it has been possible to reconstruct the 3D structure of the MWNT-nylon composite. Further, by recording an energy series of EFTEM images at every tilt, it is possible to reconstruct the 3D structure at every energy increment and therefore interrogate the energy loss spectrum at every voxel, thus enabling a form of '*volume-spectroscopy*' [2]. Such a method allows spectra to be reconstructed from isolated sub-volumes of interest, improving the interpretation of each spectrum and removing the possibility of being misled through projection artefacts.

(iii) Tomographic holography. In the absence of magnetic fields, the phase change recorded by an electron hologram of a specimen is proportional to a change in the electrostatic potential. Further, away from strong diffraction conditions, this potential is dominated by the mean inner potential of the specimen. Under these conditions, for a semiconducting p-n junction, the phase change across the junction is directly proportional to its built-in potential and to the projected thickness of the specimen. As such, the phase signal reconstructed from the hologram can be used for tomographic reconstructions. We will show an example of the 3D reconstruction of a biased and unbiased p-n junction in a silicon sample. What is revealed is the strong influence of the electronic properties of the surface and in particular the significant surface depletion regions that form when the sample has been prepared using a focused ion beam (FIB) instrument.

(iv) Weak-beam dark field (WBDF) tomography of dislocations. It is now 50 years since the first electron micrographs of individual dislocations were published. Such images have been used to explain many of the properties of metals and other materials. However, such images are 2D projections of a complex 3D arrangement and, although stereo-microscopy offers some insight, can never reveal the whole structure. Here we will show how, using weak-beam dark field (WBDF) imaging and keeping the diffraction conditions constant throughout a tilt series, it is possible to reconstruct the 3D network of dislocations with high spatial resolution. As an example we will show the defects in a GaN epilayer and reveal how threading dislocations turn over and become in-plane dislocations, how threading dislocations populate sub-grain domains and reveal the presence of dislocation bundles emanating from a crack.

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