Novel TEM techniques for the characterisation of nanostructures

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A JEOL JEM-3000F 300 kV field emission gun (FEG) transmission electron microscope (TEM) has recently been installed in the Department of Materials at Oxford University. This microscope is intended to provide a multi-user facility, whose primary aim is the characterisation of novel nanostructured materials. An ultra-high resolution objective lens polepiece, which has a low spherical aberration coefficient of 0.57 mm, a point resolution of 0.16 nm and an information limit of better than 0.10 nm, is used for the conventional high-resolution imaging of nanostructures. The analytical capabilities of the microscope include a Gatan GIF 2000™ imaging filter equipped with a 2k × 2k CCD camera, which is used to acquire electron energy-loss spectra and two-dimensional chemical maps at a resolution of ~0.5 nm. A light element-sensitive Oxford Instruments Isis 300™ and SemiSTEM™ unit are also available for the acquistion of energy dispersive X-ray spectra, linescans and maps. Microanalysis is performed using a probe that has a minimum diameter of below 0.3 nm and a current of several tens of pA. The microscope also has a nano-beam diffraction capability, which allows the acquisition of diffraction patterns from nm-sized regions with almost parallel illumination, a piezo-electric stage for automated sample drift correction and a biprism for off-axis electron holography of magnetic and electrostatic fields in materials. Automated image acquistion and microscope alignment are both available using the Gatan Digital Micrograph™ scripting language.

A wide range of nanostructured materials has been characterized using the JEOL JEM-3000F FEGTEM. These include filled and unfilled carbon nanotubes, nm-sized crystals for use in catalysis and optoelectronics, and nanoscale materials of intrerest to the recording and information storage industries. While the basic capabilities of the microscope provide an outstanding facility for the routine characterisation of such materials, many challenging problems exist for which the capabilities of the microscope have to be extended through the development of new techniques that involve lengthy experiments and considerable data analysis. One example has been the need to apply focal and tilt series reconstruction techniques to characterise crystals that have been encapsulated within single-walled carbon nanotubes. These approaches allow the complex wavefunction of the electron wave that leaves the material to be recovered. The phase of this wavefunction provides structural and chemical information that is unaffected by the aberrations of the microscope, as well as having considerably less noise and better resolution than a conventional high-resolution image. This project has required both the characterisation of the microscope and the determination of the aberrations of the microscope lenses to an unprecedented level of accuracy. The data have been used to measure the phase shifts and the positions of one, two and three atom-high columns of atoms in the encapsulated crystals for the first time, and have provided evidence of new distorted phases that form inside the nanotubes with structures that are distinct from equivalent bulk crystal lattices.