Recent results from the JEOL JEM-3000F FEGTEM in Oxford

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A JEOL JEM-3000F 300 kV field emission gun (FEG) transmission electron microscope (TEM) was installed in the Department of Materials in Oxford in 1999. The microscope is a multi-user facility, whose primary aim is the high spatial resolution structural and chemical characterisation of novel nanostructured materials. Typical problems of interest include the characterisation of filled and unfilled carbon nanotubes, nanocomposites, information storage media and optoelectronic materials. The microscope is in the ultrahigh resolution configuration, with a point resolution of below 0.17 nm and an information limit of below 0.10 nm. The spherical aberration coefficient of the objective lens is nominally 0.60 mm and specimen tilts of approximately ±25° can be achieved. The analytical facilities on the microscope include a Gatan imaging filter (GIF) 2000™, which is equipped with a 2k × 2k charge coupled device (CCD) camera. An additional 1k × 1k CCD camera, which is located before the GIF, has proved to be particularly useful for the routine acquisition of high-resolution electron microscope (HREM) images that do not require energy-filtering. An Oxford Instruments ISIS 300™ system with an ultrathin window Si(Li) detector and a SemiSTEM unit are used for acquiring energy dispersive X-ray (EDX) spectra, linescans and maps using a probe that can have a diameter of below 0.3 nm with a current of several tens of pA. A piezoelectric stage provides automated sample drift correction, and an electrostatic biprism is used for off-axis electron holography of magnetic and electrostatic fields in materials. Both image acquisition and external microscope alignment and control are performed using the Digital Micrograph™ scripting language and software. Figure 1a shows a photograph of the microscope. Figures 1b, c and d show the results of three of the microscope's acceptance tests, which were performed at 300 kV and demonstrate a point resolution of better than 0.17 nm, a probe size of below 0.3 nm and an energy resolution of better than 0.7 eV, respectively. Other measurements (not shown here) include a sample drift rate of below 0.4 nm/ min and a focused probe drift rate of below 0.15 nm/ min.

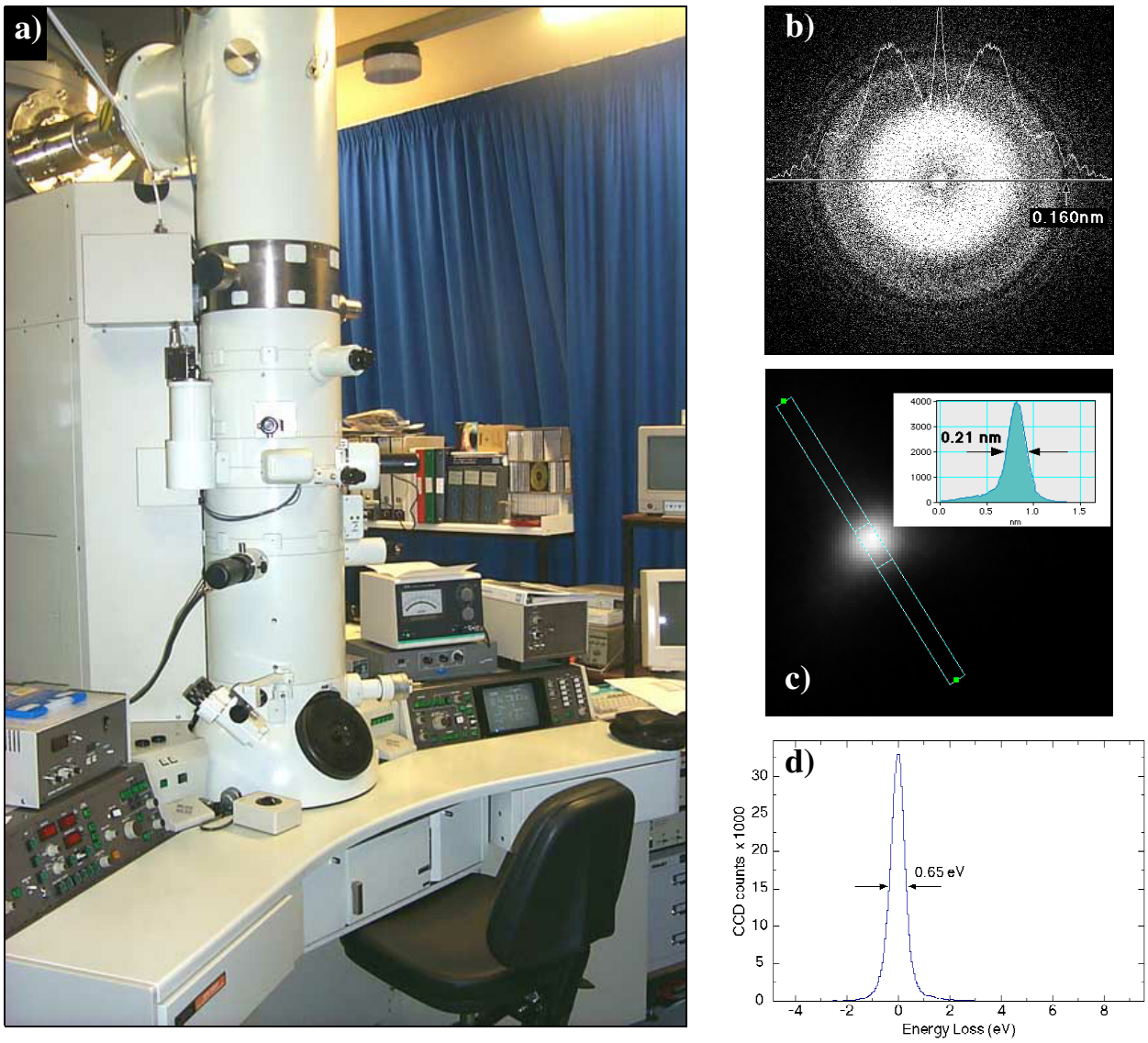


Fig. 1. a) The JEOL JEM-3000F in Oxford; b) Diffractogram obtained at 300 kV close to Scherzer focus confirming point resolution of between 0.16 and 0.17 nm; c) Minimum measured probe size of 0.21 nm, obtained with an acquisition time of 0.25 s. In order to achieve this condition, free lens control was used to saturate the first condenser lens, increase the current in the second condenser lens and decrease the current in the objective lens slightly. d) Zero-loss peak showing minimum energy spread of 0.65 eV for an emission current of 20 μA and an acquisition time of 0.1 s.

The basic capabilities of the 3000F provide an outstanding facility for the characterisation of a wide range of nanostructured materials. For example, representative HREM images of single walled carbon nanotubes filled with crystals of metal halides and sulphides are shown in Fig. 2, alongside EDX data obtained from a nm-sized section of filling material. Figure 3a shows the predicted resolutions of energy-loss images in the 3000F when using a 20 eV energy-selecting slit width.

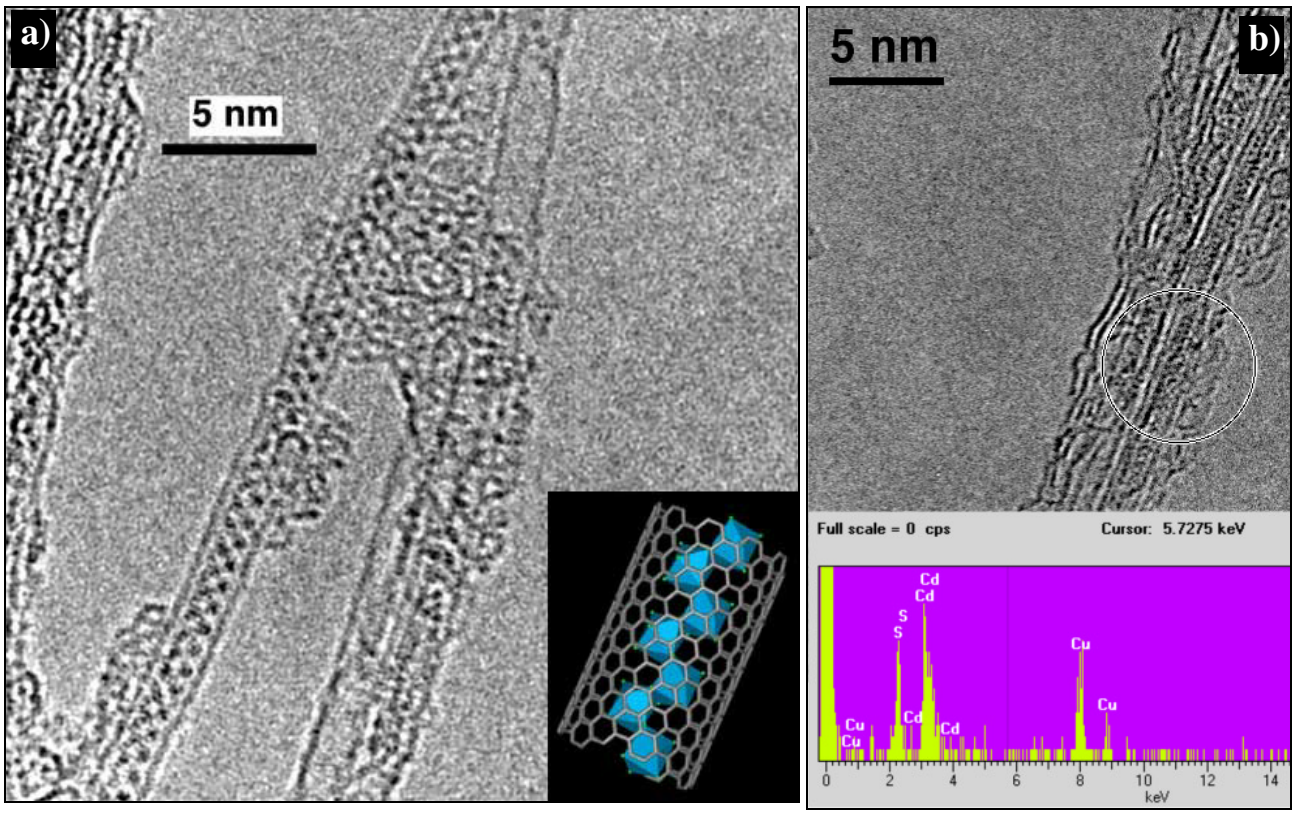
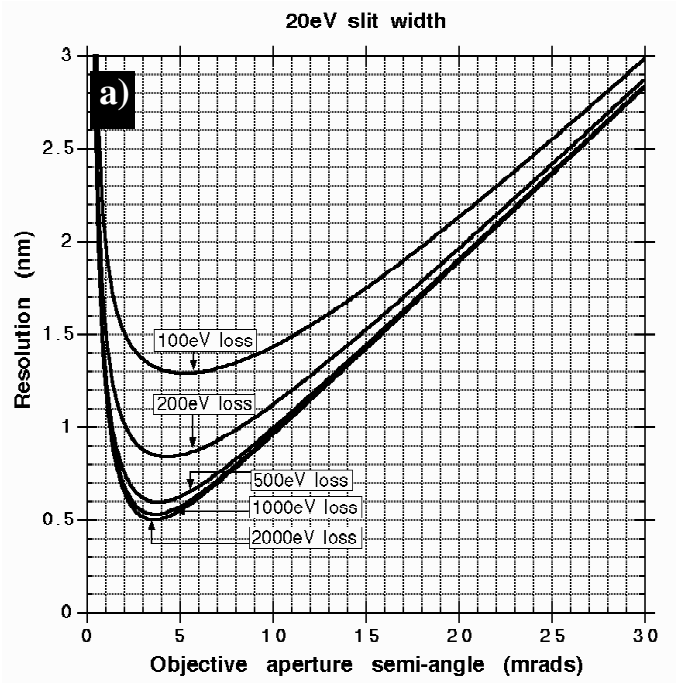


Fig. 2. a) High-resolution image of a 1.4 nm diameter single walled carbon nanotube filled with a single crystal of thorium chloride. Each black dot is associated with the contrast of one thorium atom. The inset shows the corresponding structural model. b) High-resolution image of a single walled carbon nanotube filled with cadmium sulphide, together with an EDX spectrum obtained from the area marked by the circle.

The microscope has objective apertures of semi-angle 1.2, 4.1, 12.2 and 24.6 mrad. Although Fig. 3a would suggest that the 4.1 mrad aperture should always be used, in practice the 12.2 mrad aperture is usually chosen in order to achieve an optimal number of accounts from thin regions of samples. Typical examples of chemical maps obtained under these conditions are shown in Figs. 3b and 3c. Many challenging problems also exist for which the capabilities of the microscope have to be extended through the development and application of new techniques. One example has been the need to apply focal and tilt series reconstruction techniques to allow the full characterisation of 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 information that is unaffected by the aberrations of the microscope, as well as having less noise and better resolution than a conventional HREM image.



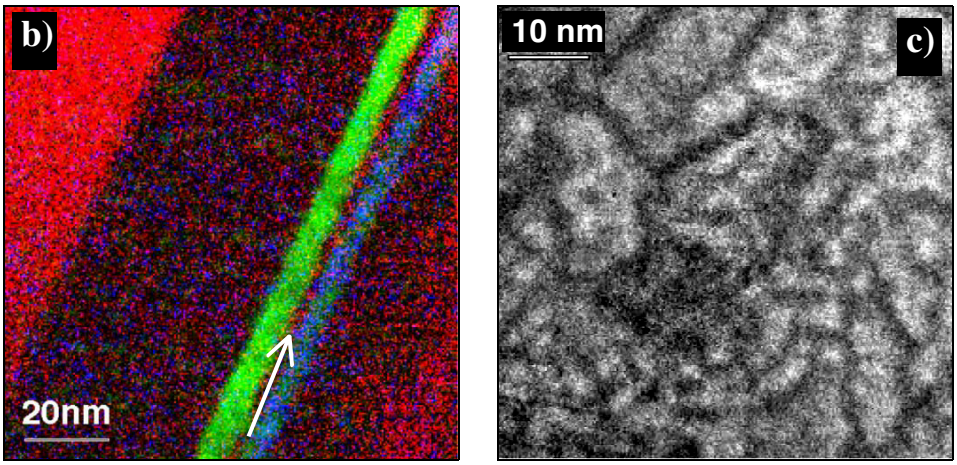


Fig. 3. a) Predicted resolution of energy-loss images as a function of objective aperture size for an energy-selecting slit width of 20 eV and the energy losses indicated; b) Greyscale version of composite image formed from Fe (708 eV), Co (779 eV) and Al (1560 eV) three-window elemental maps for a cross-sectional magnetic tunnel junction grown on sapphire, using an objective aperture of semi-angle 12.2 mrad. The tunnel barrier of width 2 nm (arrowed) is clearly resolved. c) As for b) but showing Cr (574 eV) grain boundary segregation in a Co-rich (779 eV) magnetic recording medium in plan-view. The dark contrast at each boundary has a width of approximately 1.2 nm.

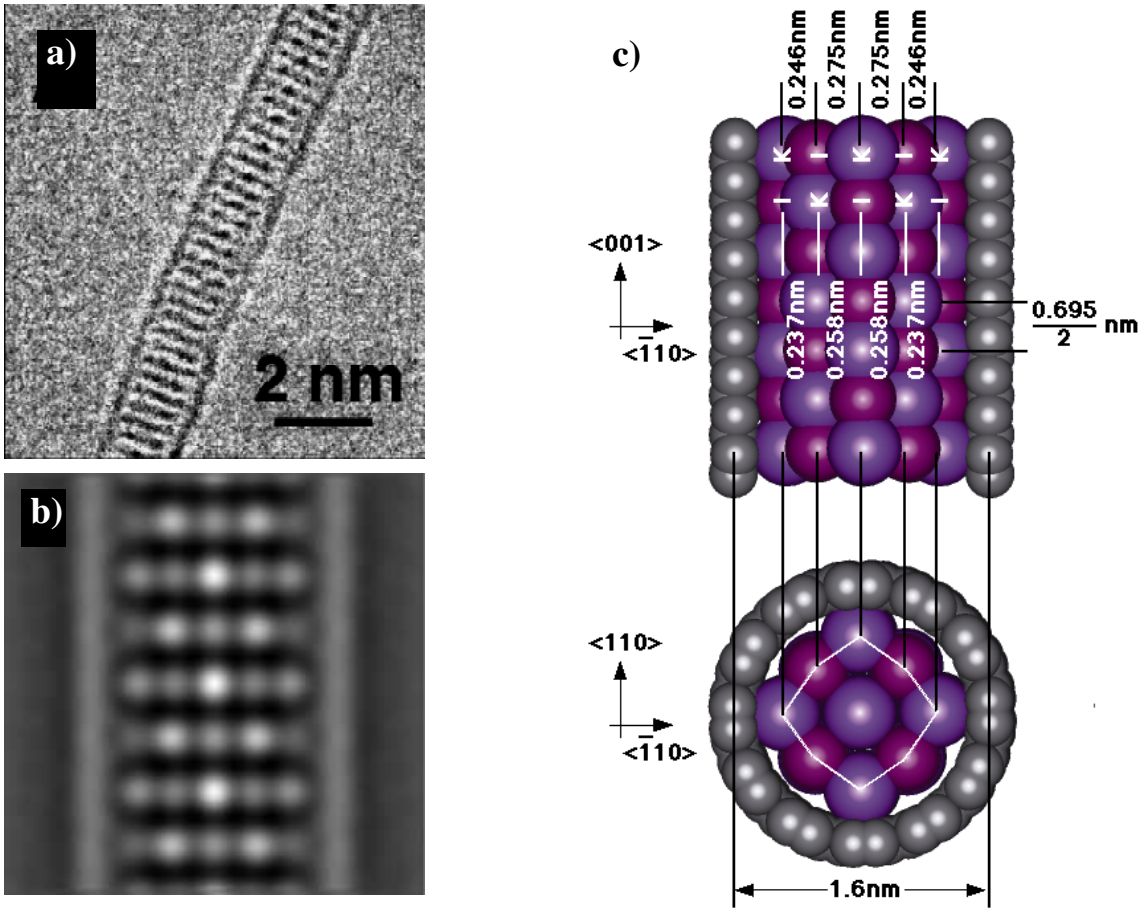


Fig. 4. a) Sherzer focus high-resolution image of a 1.6 nm diameter single-walled carbon nantoube filled with a single crystal of potassium iodide, viewed along the <110> direction of the parent crystal structure; b) Phase image obtained from a through-focal series reconstruction of 20 images of the crystal shown in a). The row of dots adjacent to the nanotube walls corresponds to alternating single atoms of potassium and iodine; c) Measurements of lattice spacings from phase image in b), showing expansion of crystal structure perpendicular to chain axis, and contraction along axis.

The data have been used to measure the phase shifts and the positions of one, two and three atom-high columns of atoms in one-dimensional crystals of potassium iodide for the first time, as shown in Fig. 4. Far higher quality information about the crystal can be obtained from a phase image reconstructed from a through-focal series of twenty images (Fig. 4b) than from a single image taken at Scherzer focus (Fig. 4a). The data have also provided evidence for new distorted phases that form inside nanotubes with structures that are distinct from the bulk crystal lattice of potassium iodide.

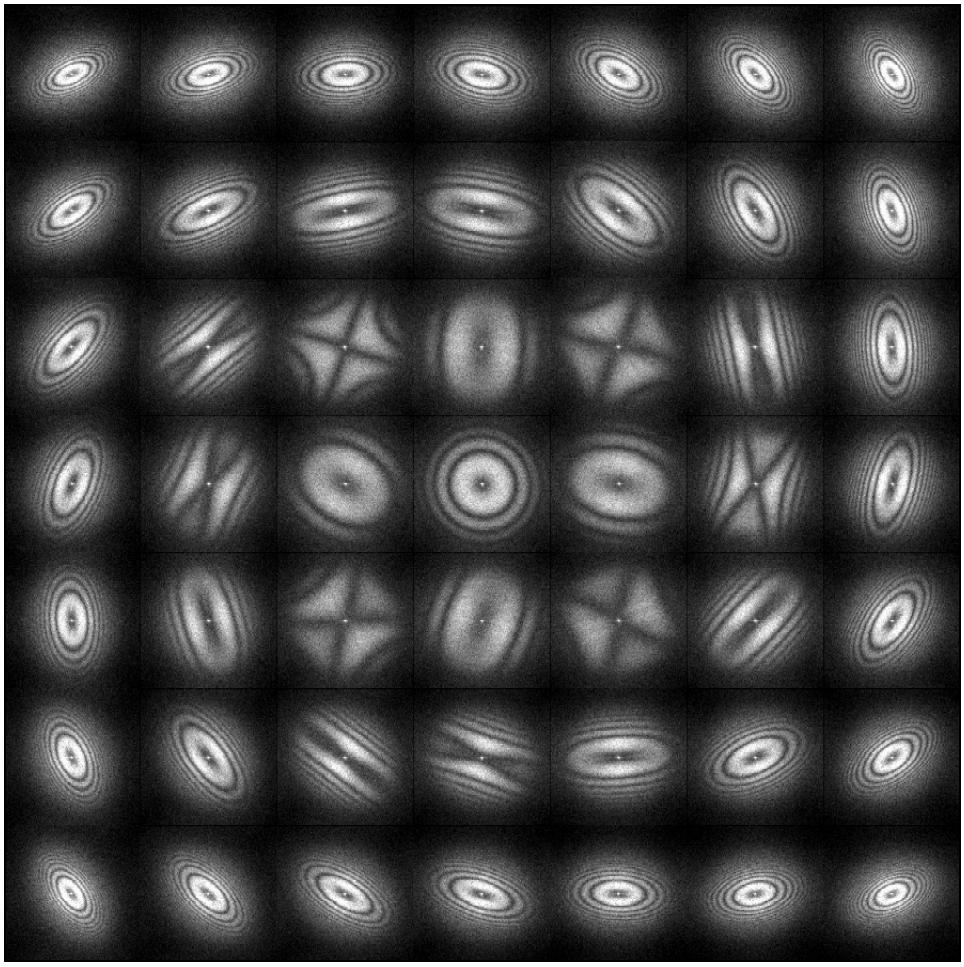


Fig. 5. Tilt tableau of diffractograms obtained from amorphous carbon for measuring objective lens aberrations. Tilts were ±30 mrad in steps of 10 mrad, and the starting defocus was -210 nm.

The work has required both the characterisation of the microscope and the determination of the aberrations of its lenses to high accuracy. Tilt tableaus such as that shown in Fig. 5 have been used to determine that the coefficients of spherical aberration and three-fold astigmatism in the 3000F are 0.57 mm and 850 nm, respectively, as well as to measure the beam divergence (0.10 mrad) and the focal spread (5 nm) of the microscope and to calibrate the magnitude and direction of the beam tilt coil strengths. Further details about this project and about the microscope can be found in the papers of Meyer *et al*. (2000) and Hutchison *et al*. (1999), respectively.

References

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