Advanced transmission electron microscopy of nanoscale materials and devices

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The Department of Materials Science and Metallurgy in Cambridge contains an extensive suite of advanced transmission electron microscopes (TEMs), including a JEOL 4000EXII dedicated high-resolution TEM and two state-of-the-art field emission gun (FEG) microscopes (Figs. 1a and b). Research within the Electron Microscopy Group in the Department centres on the development of new techniques for the characterisation of nanoscale materials and devices. Two techniques that are of particular interest are electron tomography for the characterisation of the microstructure and chemistry of materials in *three* dimensions, rather than simply in projection, and off-axis electron holography for measuring magnetic and electric fields in materials. Each technique has a spatial resolution approaching 1 nm.

Figure 1c shows a chemical map of a chain of crystalline $Fe_{55}Ni_{45}$ nanoparticles, which have an average diameter of 50 nm and are of interest for magnetic recording applications. The image was obtained by using a Gatan imaging filter in the Philips CM300ST FEGTEM (Fig. 1a) to record Fe, Ni and O three-window background-subtracted elemental maps of the sample. The magnetic microstructure in such nanoparticle chains is sensitive to their composition (which affects the exchange length in the alloy), as well as to the sizes and the positions of the particles. A reconstruction of the three-dimensional shapes and positions of particles in a similar chain, obtained in the FEI Tecnai F20 (Fig. 1b) by acquiring a series of high-angle annular dark field images over a sample tilt range of ±70°, is shown in Fig. 2a. Figure 2b shows the remanent magnetic state of a 75 nm $Fe_{55}Ni_{45}$ particle sandwiched between two smaller particles, recorded in zero-field conditions using off-axis electron holography on the Philips CM300ST FEGTEM. The contours reveal the in-plane magnetic induction in the sample integrated in the electron beam direction, which is strongest where the contours are most closely spaced. The contours are observed to run through all three particles in a channel of width 22 nm. Comparisons with simulations suggest that the largest particle contains a vortex whose axis lies parallel to the chain axis, as shown schematically in Fig. 2c. The large diameter of the flux channel (the vortex core) results from magnetic dipole interactions along the chain.

The application of off-axis electron holography to the characterisation of electrostatic (rather than magnetic) fields is illustrated in Fig. 3. Figure 3a shows an electron holographic phase image of a Si p-n junction specimen of uniform thickness, which was prepared for TEM examination using focused ion beam milling. The contrast in this image reveals the position of the semiconductor junction directly, and is directly proportional to the variation in potential across the depletion region in the sample. Line profiles obtained from similar phase images (Figs. 3b and c) show that the measured electron holographic phase shift increases with both specimen thickness (Fig. 3b) and applied reverse bias voltage (Fig. 3c). In order to obtain the line profiles shown in Fig. 3c, an external voltage was applied to the sample *in situ* in the electron microscope using a custom-built biasing holder.

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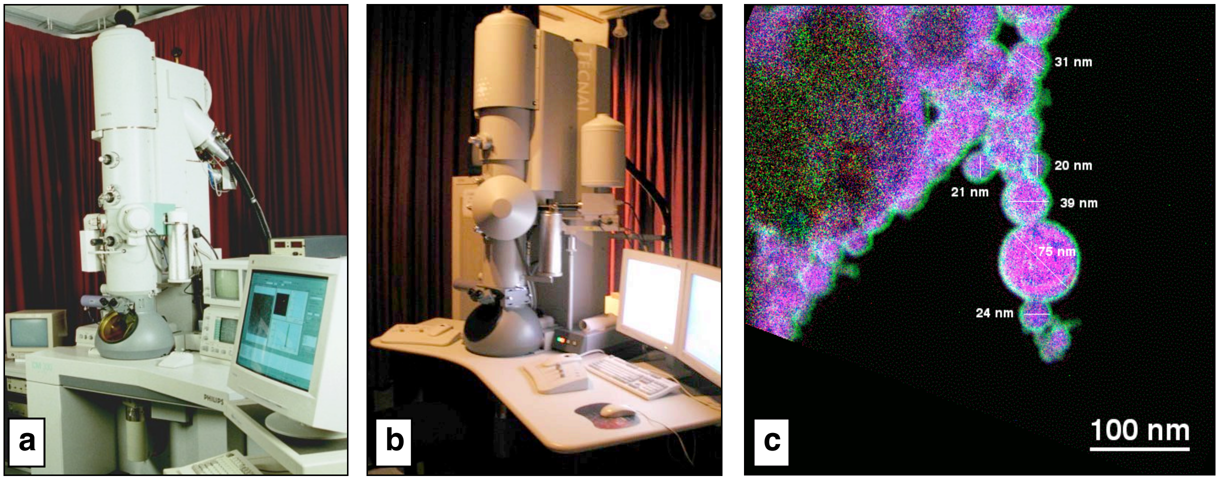

Figure 1. (a) Philips CM300ST and (b) FEI Tecnai F20 field emission gun transmission electron microscopes in the Department of Materials Science and Metallurgy in Cambridge. Each microscope is equipped with a Gatan imaging filter and a high-angle annular dark field detector. The CM300 also has a Lorentz lens for imaging magnetic materials in field-free conditions and an electron biprism for off-axis electron holography. (c) Representative chemical map obtained using the Gatan imaging filter on the Philips CM300ST microscope from a sample containing chains of crystalline ferromagnetic $Fe_{55}Ni_{45}$ nanoparticles (purple), which have an average diameter of 50 nm and are each coated in an oxide shell (green).

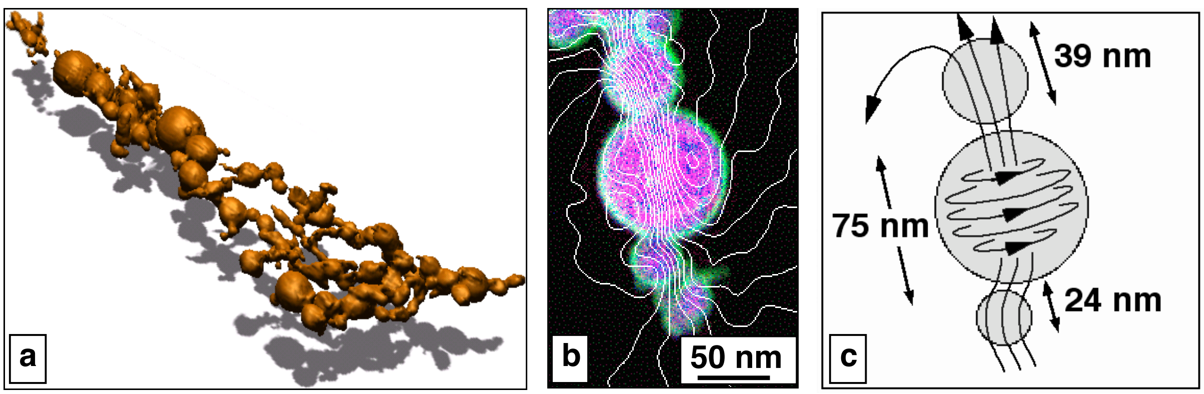

Figure 2. (a) Electron tomographic reconstruction of the three-dimensional positions and shapes of FeNi nanoparticles in a single chain, calculated from an ultra-high-tilt series of high-angle annular dark field images. (b) Electron holographic magnetic phase contours, which have been overlaid onto a chemical map, showing the in-plane induction (integrated in the electron beam direction) in a chain of $Fe_{55}Ni_{45}$ particles, recorded with the microscope objective lens switched off. The largest particle in the chain has a diameter of 75 nm. The contour spacing is 0.083 radians. The mean inner potential contribution to the phase shift has been removed from the image. (c) shows a schematic diagram of the magnetic microstructure in the chain, in which a vortex spins around the chain axis.

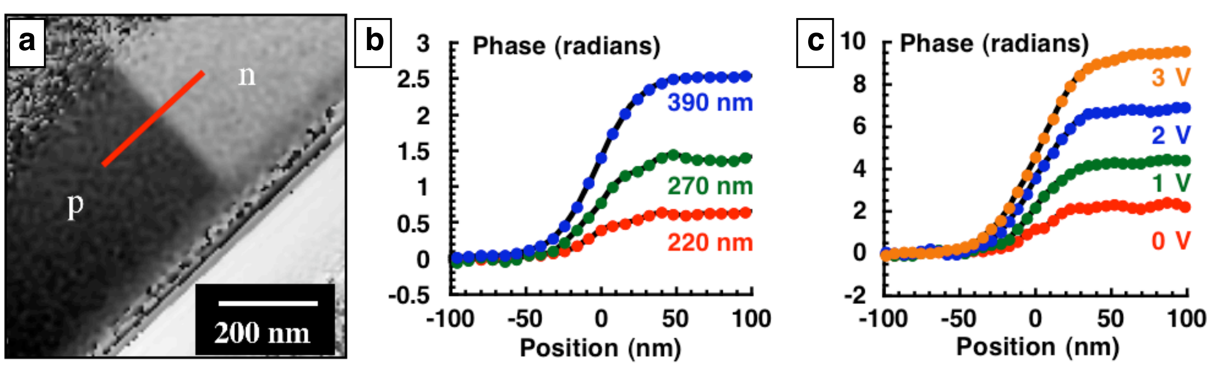

Figure 3. (a) Electron holographic phase image acquired from a Si p-n junction prepared for electron microscopy using focused ion beam milling. (b) Phase profiles along the line marked in (a) obtained from three unbiased specimens whose thicknesses are indicated. (c) Line profiles across a p-n junction in a specimen of thickness 390 nm, acquired as a function of applied reverse bias using a home-built electrical biasing sample holder.