In situ electrical biasing for electron holography of magnetic and electric fields in working devices

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Off-axis electron holography can be used to characterize magnetic and electrostatic fields in materials in the transmission electron microscope (TEM) at a spatial resolution that can approach the nanometer scale. The technique involves applying a positive voltage to an electron biprism to overlap a coherent electron wave that has passed through the specimen with a part of the same electron wave that has passed only through vacuum. We have designed a cartridge-based side-entry TEM specimen holder that allows electron holography to be used to examine a sample that has two independent electrical contacts applied to it. A third electrical contact can then be brought towards it using micrometers and piezoelectric drives. Tilts of ±70° can be achieved before the central 500 µm of the specimen begins to be shadowed by the edges of the holder. Specimens that are at different stages of examination or preparation can be stored in separate cartridges, or transferred to a scanning electron microscope, a focused ion beam workstation, a plasma cleaner or an Ar ion miller. Two primary cartridge designs allow electrical contacts to be made either by clamping the sides of the specimen (typically a cleaved square of semiconductor wafer) between a conducting block and a spring or by applying spring contacts to its surface. New specimen geometries can be accommodated by redesigning the cartridge rather than the entire specimen holder.

Figure 1 shows a conceptually straightforward application to the measurement of electrostatic fields at the ends of nanowires and nanotubes, with the aim of understanding the details of field emission on a nanometer scale. A gold needle was brought towards bundles of single-walled carbon nanotubes, and a voltage was then applied between the needle and the nanotubes. Unexpectedly, above a critical voltage, the nanotube bundles began to grow additional branches at their ends. These branches were subsequently identified to consist primarily of amorphous carbon. Their growth did not depend on the presence of the electron beam, and was most rapid when the microscope vacuum was poor. Their formation is therefore likely to result from electric-field-induced migration of carbon-based species within the microscope. Electron holographic amplitude and contoured phase images of a nanotube bundle, recorded before any branches have grown, are shown in Figs. 1a and b, respectively. In Fig. 1b, the recorded electric field is strongest where the contours are most closely spaced, close to the end of the bundle. Corresponding images are shown in Figs. 1c and d after the formation of branches. A wide variety of nanowires or needle-shaped specimens can be used to form the moveable contact in the nanopositioning holder in order to probe the mechanical, electrical or magnetic properties of a specimen placed in the cartridge.

Figure 1. a) Amplitude and b) contoured phase images obtained from an off-axis electron hologram of a carbon nanotube bundle with a voltage applied to it in the TEM. c) and d) show similar images obtained from a nanotube bundle that grew branches of amorphous carbon above a critical applied voltage.