Prospects and challenges for the quantitative measurement of magnetic and electric fields in nanoscale materials in the TEM

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Abstract: Recent applications of off-axis electron holography and Lorentz transmission electron microscopy to the study of magnetic and electric fields in nanoscale materials and devices are presented and discussed.

Introduction: Two of the most powerful techniques for providing quantitative information about magnetic fields and electrostatic potentials in nanoscale materials with sub-10-nm spatial resolution are off-axis electron holography and Lorentz transmission electron microscopy [1]. Here, we describe several recent developments in their application to the study of specimens examined as a function of temperature and applied bias *in situ* in the electron microscope. We also describe new quantitative approaches that allow the measurement of magnetic moments and charge densities in individual nanostructures.

Magnetic fields and magnetic moments: We have recently applied both off-axis electron holography and the Fresnel mode of Lorentz transmission electron microscopy to study extended crystals and sub-100-nm nanoparticles of magnetite (Fe_3O_4) at room temperature and below the Verwey transition, at which the magnetocrystalline anisotropy increases by an order of magnitude and the magnetic easy axis switches from the <111> directions of the parent cubic phase to the [001] direction of the low temperature monoclinic phase. The easy axis of the monoclinic phase can lie along any of the three <100> directions of the cubic phase. In extended crystals examined at low temperature micrometer-scale monoclinic twin domains that are separated from each other by jagged twin walls are then observed. The twin domains are further subdivided by lamellar twins, which join at their ends to form needle twins that move in response to thermal or magnetoelastic stress by the advancement and retraction of their needle tips. Lateral motion of twin walls is only observed within a few degrees of the phase transition temperature [2]. In individual nanocrystals that can only a single magnetic domain, we observe competing effects on the magnetic microstructure of shape anisotropy and magnetocrystalline anisotropy that are different above and below the Verwey transition. In order to measure the magnetic moments of such nanoparticles quantitatively, we developed an algorithm that relies on the relationship between the volume integral of the magnetic induction and the magnetic moment. The approach requires the use of circular boundary around the nanostructure to evaluate either a contour integral of the phase shift measured using electron holography around a circular path or an integral of the phase gradient bounded by the same loop. It provides two orthogonal components of the magnetic moment, which are free of most artifacts if the measurements are extrapolated to a circle of zero integration radius [3].

Electrostatic potentials and charge densities: In direct analogy to the measurement of the magnetic moment of an individual nanocrystal, we have also developed an algorithm that can be used to measure the charge distribution in a nanoscale object from a recorded electron holographic phase image. Figure 1 illustrates the application of the approach to the measurement of the charge distribution along a carbon nanotube that has a voltage applied to it *in situ* in the TEM. Figures 1a and 1b show defocused bright-field images of several nanotubes, while Fig. 1c shows a phase image of one of the nanotubes acquired using electron holography. The charge distribution is determined by evaluating either a contour integral of the phase gradient around a closed path or the Laplacian of the phase within the area bounded by the same loop. The charge distribution along the nanotube in Fig. 1c was inferred to have two different charge densities along its length (2.3 and 4.5 e/nm). The approach is inherently unaffected by the presence of charges outside the field of view or any linear phase ramps that may be present [4].

Prospects and challenges: Off-axis electron holography presently allows the study of magnetic fields and electrostatic potentials in materials with sub-10-nm spatial resolution, as well as the quantitative measurement of parameters such as magnetic moments and charge densities. The experiments can be complicated by the sensitivity of phase images to the effects of dynamical diffraction (i.e., small changes in specimen tilt), by charging of the specimen as a result of electron beam irradiation and by changes over time in the thicknesses of overlayers or adsorbates on the specimen surface. If such effects can be minimized, then our calculations suggest that it may be possible to measure the magnetic moments of nanoparticles that are below 100 μ_B, both in projection and in three dimensions. The combination of electron holography with environmental (gas reaction) transmission electron microscopy and the application of chromatic aberration correction to improve the spatial resolution of measurements obtained with the specimen in a magnetic-field-free environment are also presently being explored.

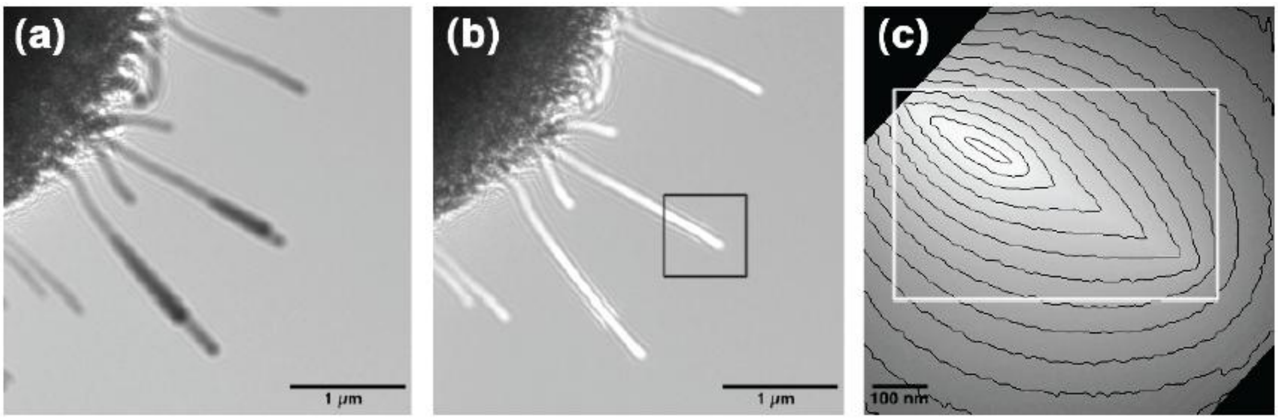

Fig. 1. (a) and (b) Bright-field images, acquired at defocus values of ±3 mm, of carbon nanotubes protruding from a nanotube bundle examined under an applied bias in situ in the electron microscope. (c) Reconstructed phase image acquired from the region indicated in (b), with 1.5 rad phase contours superimposed. The outline in (c) marks the region used to determine the charge on the nanotube.

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