The prospect of 3-dimensional induction mapping inside magnetic nanostructures by combining electron holography with electron tomography

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There is an increasing need to combine analytical techniques in the transmission electron microscope with microscope control and image analysis to obtain unique information about nanostructured materials.

Here, we examine the prospect of characterizing magnetic vector fields *inside* nanocrystals in three dimensions by combining electron tomography with electron holography, a technique that allows the phase shift of an electron wave that has passed through a specimen to be measured.

When characterizing three-dimensional magnetic fringing fields <u>in vacuum</u> , the acquisition of two ultra-high-tilt series of electron holograms about orthogonal axes can be used to provide the three-dimensional distribution of two of the three components of the induction B_x and B_y within and around a sample, where x and y are directions perpendicular to the incident electron beam direction z.

If each phase image is differentiated in a direction perpendicular to the tilt axis, then standard tomographic reconstruction algorithms can be used to calculate the three-dimensional distribution of the component of B that lies parallel to the tilt axis.

After determining B_x and B_y in three dimensions in this way, B_z can be evaluated by making use of the criterion that $\nabla.B = 0$.

The application of this approach to the characterization of magnetic fields <u>inside</u> nanostructured magnetic materials is complicated by the fact that the (often dominant) mean inner potential contribution to the measured phase shift must be removed at *each* sample tilt angle.

This requirement can be achieved if each tilt series is recorded both before and after reversing the direction of magnetization in the specimen (e.g., using the microscope objective lens).

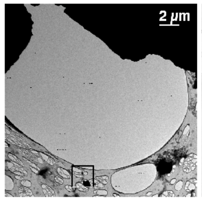
Subsequently, half of the difference between pairs of reversed images acquired at <u>each</u> tilt angle can be used to provide the desired magnetic contribution to the phase shift.

In practice, many additional difficulties must be overcome if this combined technique is to be applied successfully:

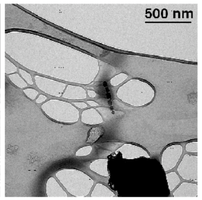
The region of interest must lie close enough to a large enough hole in the thin specimen support film to allow electron holograms to be acquired at high sample tilt angles about two tilt axes, without the region of interest (or the hole) being shadowed by other parts of the specimen. For a sample of magnetic nanocrystals on a carbon support film on a Cu grid, only regions that are both close to the centre of a grid square and near the center of the 3 mm sample are suitable for analysis.

In general, the distribution of nanocrystals imaged must be isolated and small, as the magnetic field from them should ideally decrease to close to zero at the edge of the field of view in each hologram. The difficulty of finding such a region is illustrated in the figures shown on the right and below. A chain of six magnetite nanocrystals is obscured by dirt on the specimen at high tilt angles even though it is located close to a 20 μm hole in a carbon support film.

The need to record images at tilt angles of up to ±80° to avoid reconstruction artifacts is also <u>much</u> more strict than for applications of tomography to the characterization of microstructure or chemistry. This difficulty results from the fact that the desired magnetic vector field may decay slowly towards (and indeed outside) the edges of the reconstructed volume, and each component of the vector field is likely to change sign within the field of view.



Low magnification bright-field image showing the location of a chain of magnetite crystals adjacent to a hole in a carbon support film.

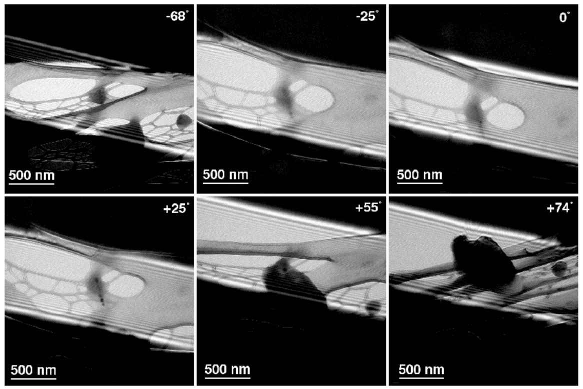


Higher magnification image of the same chain of six magnetite nanocrystals obtained from the marked region.

In addition, the diffracting condition of the crystals must not change between each pair of reversed images, and the sample shape must remain unchanged over the time required to record four tilt series of holograms.

The registration of many undersampled phase images, which have been acquired over a large range of tilt angles, presents one of the <u>most</u> substantial difficulties to be overcome.

Finally, an appropriate choice of tomographic reconstruction algorithm is essential. The most suitable algorithm for reconstructing a vector field that decays slowly and changes sign across the field of view is very different from the algorithms that are used to characterize microstructure or composition in three dimensions using tomography, in either the physical or the biological sciences, for which the positions and the shapes of nanoparticles are the parameters of interest.



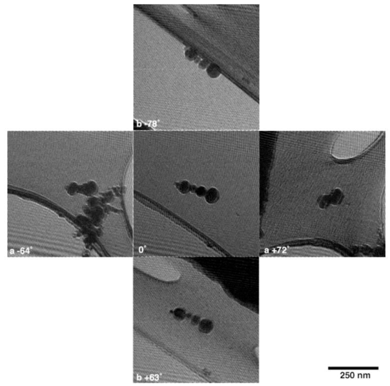
High tilt series of off-axis electron holograms of the chain of magnetite nanocrystals, acquired at 300 kV with the sample in magnetic-field-free conditions using a Philips CM300ST field emission gun transmission electron microscope.

Each image was acquired at the tilt angle indicated. The tilt axis is approximately horizontal.

The images illustrate the fact that at high tilt angles the hole in the support film becomes smaller in projection, the support film becomes thicker in projection, and the region of interest may be shadowed by parts of the specimen that appear far away at small tilt angles.

Few of these difficulties are insurmountable if sufficient resources are invested in the development, and ultimately automation, of this technique.

The figure on the right shows holograms taken from a tilt series of a chain of ferromagnetic FeNi particles acquired successfully about orthogonal tilt axes.



<u>References:</u>

1. G. Lai et al., *J. Appl. Phys.* 75 (1994) 4593.
2. S. McVitie et al., *J. Appl. Phys.* 89 (2001) 3656.

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