Off-axis electron holography of nanomagnet arrays fabricated by interferometric lithography

R E Dunin-Borkowski[1], S B Newcomb[2], M R McCartney[3], C A Ross[4] and M Farhoud[4]

[1] Department of Materials Science and Metallurgy, Pembroke Street, Cambridge CB2 3QZ
[2] Materials and Surface Science Institute, University of Limerick, Limerick, Ireland
[3] Center for Solid State Science, Arizona State University, Tempe, AZ 85287-1704, USA
[4] Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139, USA

ABSTRACT: Electron holography has been used to characterise the magnetic microstructure of arrays of 100 nm diameter Co dots and 57 nm diameter Ni pillars, which were grown onto Si substrates and prepared for transmission electron microscopy (TEM) using focused ion beam milling (FIB). The technique allows magnetic interactions between adjacent dots and pillars to be observed directly.

1. INTRODUCTION

Arrays of sub-micron sized magnetic elements are of interest for future high-density magnetic recording applications. Here, we use off-axis electron holography in the TEM to characterise square arrays of submicron-sized Co and Ni nanomagnets that have been patterned onto Si using interferometric lithography. Electron holography involves the use of a field emission gun TEM to provide a coherent source of electrons and an electrostatic biprism to overlap an electron wave that has passed through the sample with one that has passed only through vacuum. Information about magnetic fields in the sample is recorded in the local position (the phase) of the holographic interference fringes that form in the overlap region.

2. EXPERIMENTAL DETAILS

We examined both evaporated 100 nm diameter 20 nm thick Co dots that were arranged in square arrays of side 200 nm and electrodeposited 57 nm diameter 200 nm high Ni pillars that were arranged in square arrays of side 100 nm. The dots and pillars had been fabricated on Si using interferometric lithography (Ross, 2001) and were prepared for TEM examination using focused ion beam milling in plan-view and cross-sectional geometries, respectively. Holograms were recorded digitally at 200 kV in a Philips CM200-ST field emission gun TEM. This microscope is equipped with a Lorentz lens, which allows magnetic materials to be examined in close-to-field-free conditions with the conventional objective lens switched off. The objective lens can also be excited slightly and the sample tilted in a known vertical magnetic field to perform *in situ* magnetisation reversal experiments. The field of view in each hologram was 1.26 μm and the acquisition time was 4 s. Reference holograms were always recorded to remove artefacts associated with the imaging and recording process. Subtraction of the mean inner potential contribution to the measured phase was essential for successful characterisation of the magnetic microstructure (Dunin-Borkowski et al., 2001).

3. RESULTS

The holograms described below were all recorded in zero applied field, with the samples tilted slightly away from zone axis orientations of the underlying Si substrate to minimise diffraction contrast.

Figure 1a shows a scanning electron microscope (SEM) image of the Co dot sample. A bright-field image of a plan-view TEM membrane that was prepared from this sample by FIB is shown in Fig. 1b. It is possible (although not certain) that the elliptical shape of the dots results from damage sustained during sample preparation by FIB. The representative electron hologram shown in Fig. 2a was obtained by using a biprism voltage of 160 V to overlap a region of vacuum onto dots that were close to the edge of the film. Figure 2b shows an enlargement of the holographic fringes in Fig. 2a, which have a spacing of 2.48 pixels (corresponding to a distance of 3.05 nm on the sample).

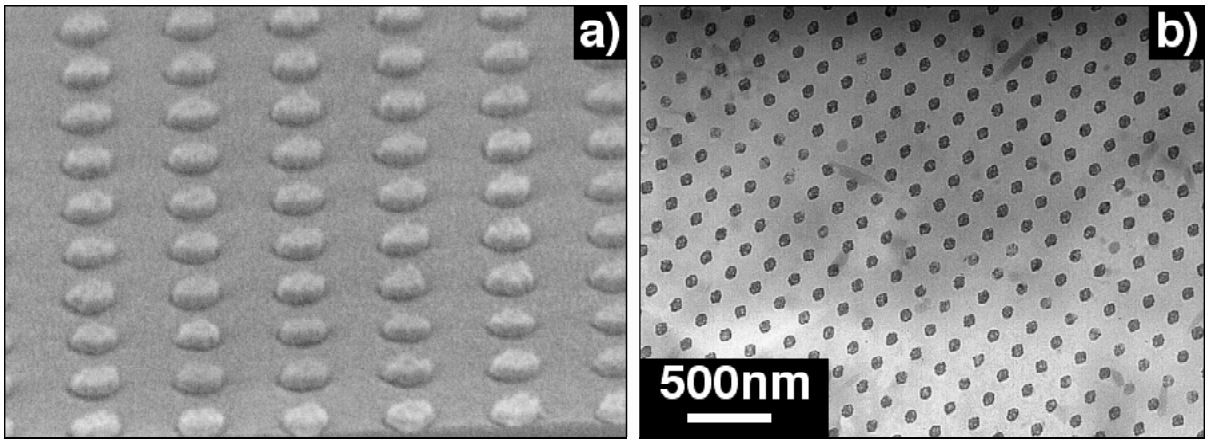

Fig. 1 a) SEM image of 100 nm diameter 20 nm thick Co dots fabricated on Si in square array of side 200 nm using interferometric lithography; b) Bright-field image of part of $10 \times 12 \mu$m electron-transparent membrane prepared using FIB. Membrane contains ~3250 Co dots.

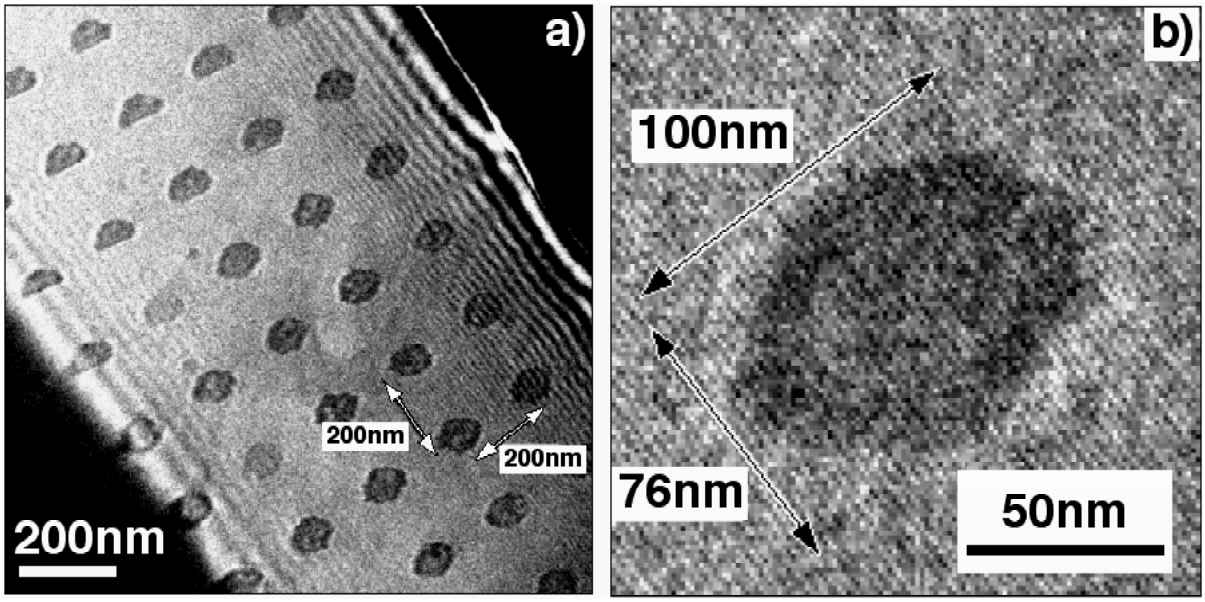

Fig.2 a) Off-axis electron hologram of part of membrane. Overlap width is 1.04 μm. Sample edge is towards bottom left; b) Enlargement of holographic fringes at position of one Co dot.

Figure 3 shows contours that have been added to the (slightly smoothed) magnetic contribution to the holographic phase for two different remanent magnetic states of the Co dots. Such images provide semi-quantitative maps of the strength of the local magnetic field in the sample. (Strictly, the contour spacing is inversely proportional to the in-plane component of the magnetic induction integrated in the incident beam direction). In Fig. 3a the dots are oriented magnetically in the same direction, whereas in Fig. 3b they are magnetised in a range of directions. The experiments show that the dots are sometimes magnetised out of the plane (e.g. at the bottom left of Fig. 3b), and also that shape anisotropy does not seem to dominate their behaviour. The measured saturation magnetisations are smaller than expected for pure Co, possibly because of oxidation or damage sustained during sample preparation.

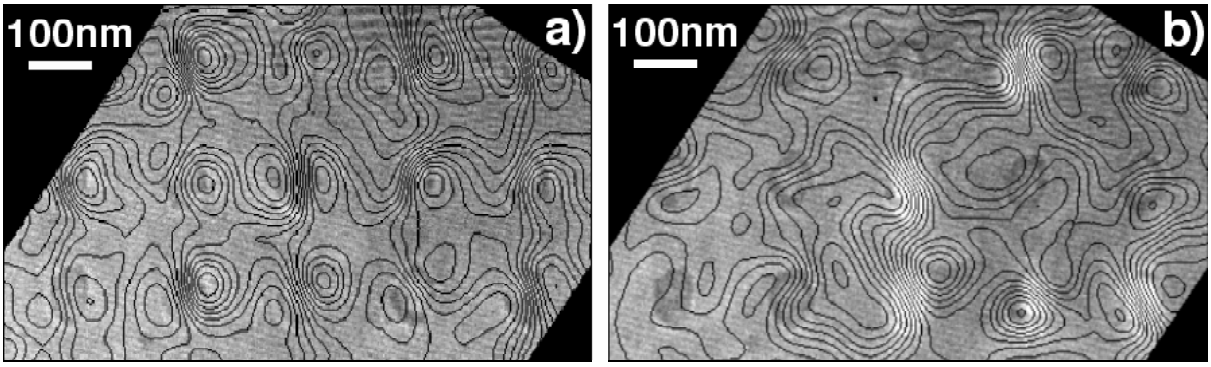

Fig.3 Magnetic contribution to measured electron holographic phase of Co dots for two remanent states. Contour spacing is 0.033 ≈ π/94 radians. a) was formed by saturating dots upwards and then removing external field; b) was formed by saturating dots upwards, applying 382 Oe downward field and then removing external field.

An SEM image of the Ni sample is shown in Fig. 4a, and a bright-field TEM image of an FIB-prepared membrane of a single row of pillars is shown in Fig. 4b. (Note the thickness corrugations in the Si substrate). A corresponding FIB image taken at the final stage of milling is shown in Fig. 5. Figure 6 shows contours added to the measured magnetic contribution to the holographic phase for two different remanent states. Despite their shape, not all of the Ni pillars are magnetised parallel to their long axes. Just as for the Co dots, the pillars, which were initially thought to be magnetised along their length, interact with each other strongly, with two, three or more adjacent pillars frequently combining to form vortices. The measured saturation magnetisations are again smaller than expected for pure Ni.

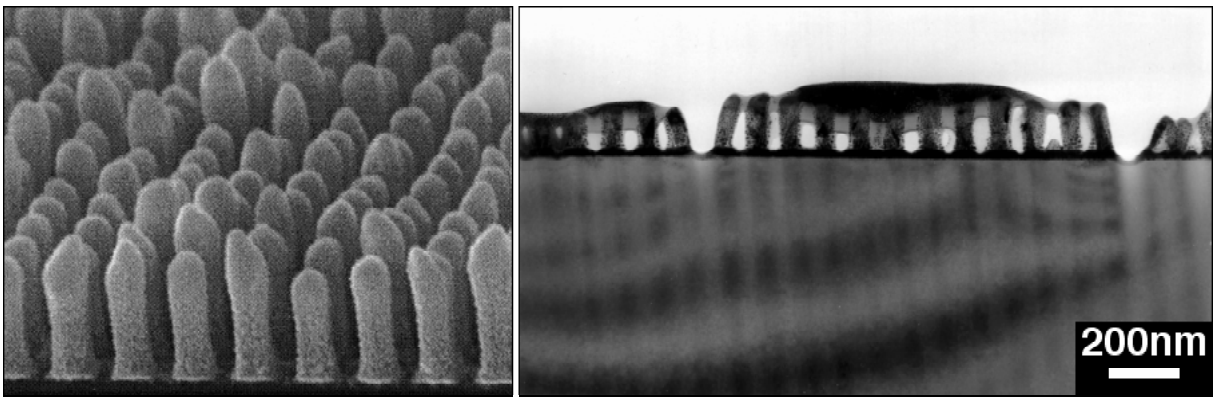

Fig.4 a) SEM image of 50 nm diameter 200 nm high Ni pillars fabricated on Si in square array of side 100 nm using interferometric lithography. b) Bright-field image of membrane containing single row of Ni pillars prepared in cross-section using FIB.

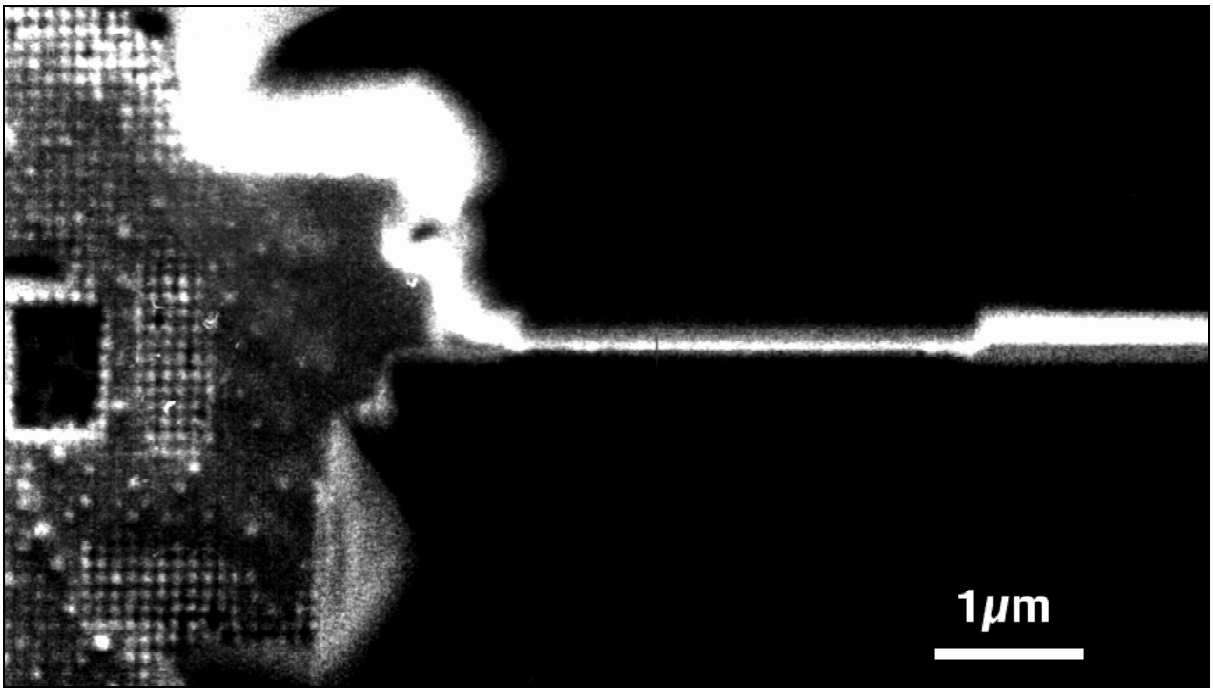

Fig.5 FIB image of final membrane containing single row of Ni pillars. Note square regions that have been milled away during alignment of milling direction parallel to rows of pillars.

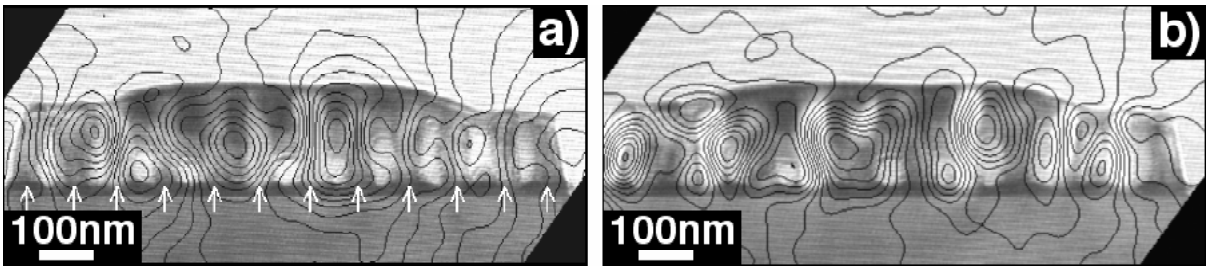

Fig. 6 Magnetic contribution to holographic phase of Ni pillars for two remanent states. Contour spacing is $0.05 \approx \pi/63$ radians. In each case pillars were first saturated upwards. Field of a) 404 Oe and b) 382 Oe was then applied downwards, before removing external field.

4. CONCLUSIONS

Electron holography has been used to characterise the magnetic microstructure of square arrays of Co dots and Ni pillars that were prepared for TEM examination by FIB and examined in plan-view and in cross-section, respectively. Magnetic interactions between adjacent dots and pillars were observed directly for a range of remanent states.

ACKNOWLEDGMENTS

RED thanks the Royal Society for support. Electron microscopy was carried out at the Center for High-Resolution Electron Microscopy, Arizona State University.

REFERENCES

Dunin-Borkowski RE, McCartney MR, Kardynal B, Parkin SSP, Scheinfein MR, Smith DJ 2000, J. Microsc. **20**, 187
Ross CA 2001, Annu. Rev. Mater. Res. **31**, 203