Quantitative Measurements of Magnetic Fields and Moments of Nanoparticles by Off-axis Electron Holography

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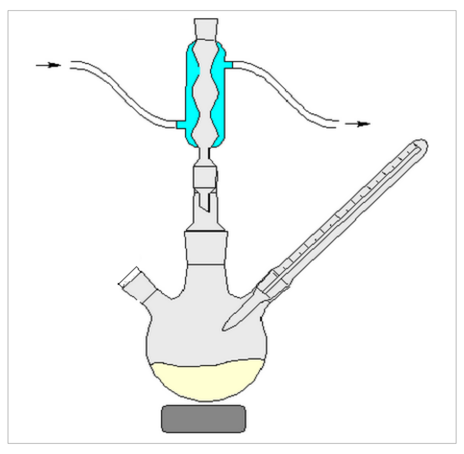
Motivation

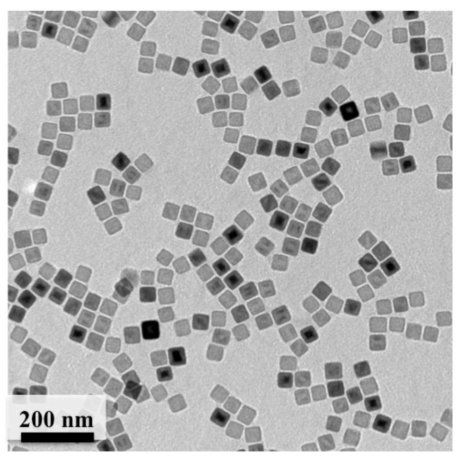
- Local variations in magnetic fields of nanostructures
- Quantitative measurement of the magnetic moments of nanostructures

Experiment

Here, we map the local variations in magnetic induction within and around magnetite nanoparticles using off-axis electron holography with nanometer spatial resolution. We then apply a contour integration method developed by Beleggia et al. [1] to measure their magnetic moments with an estimated precision of better than 1%. The origins of possible statistical and systematic errors in such measurements are discussed.

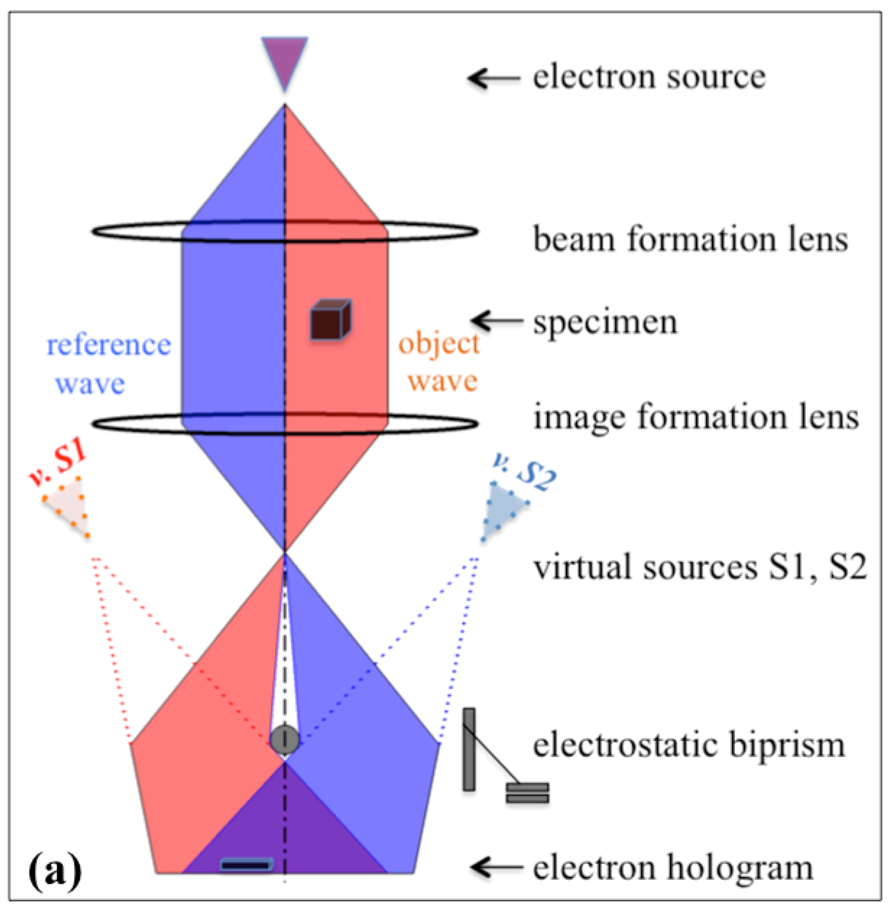
Synthesis & Morphology



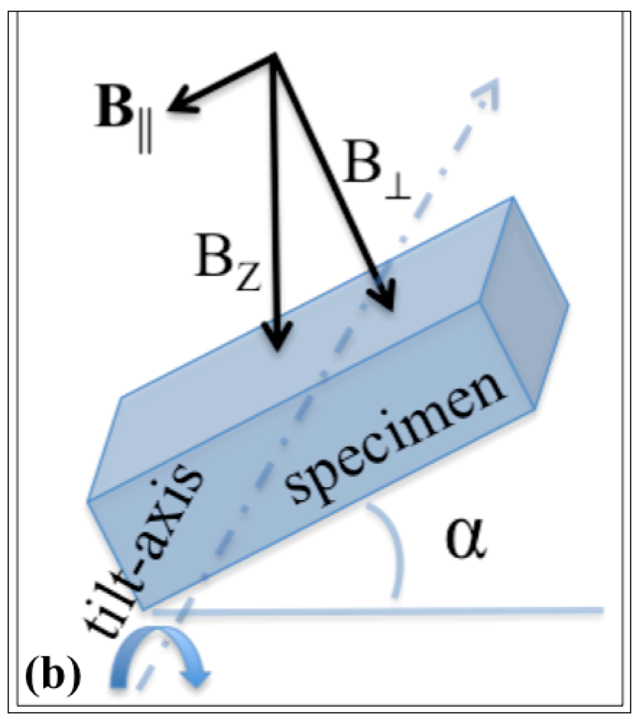


Thermal decomposition of iron(III) acetylacetonate with benzyl ether and oleic acid; Under magnetic stirring and Argon flux

Electron Holography Setup

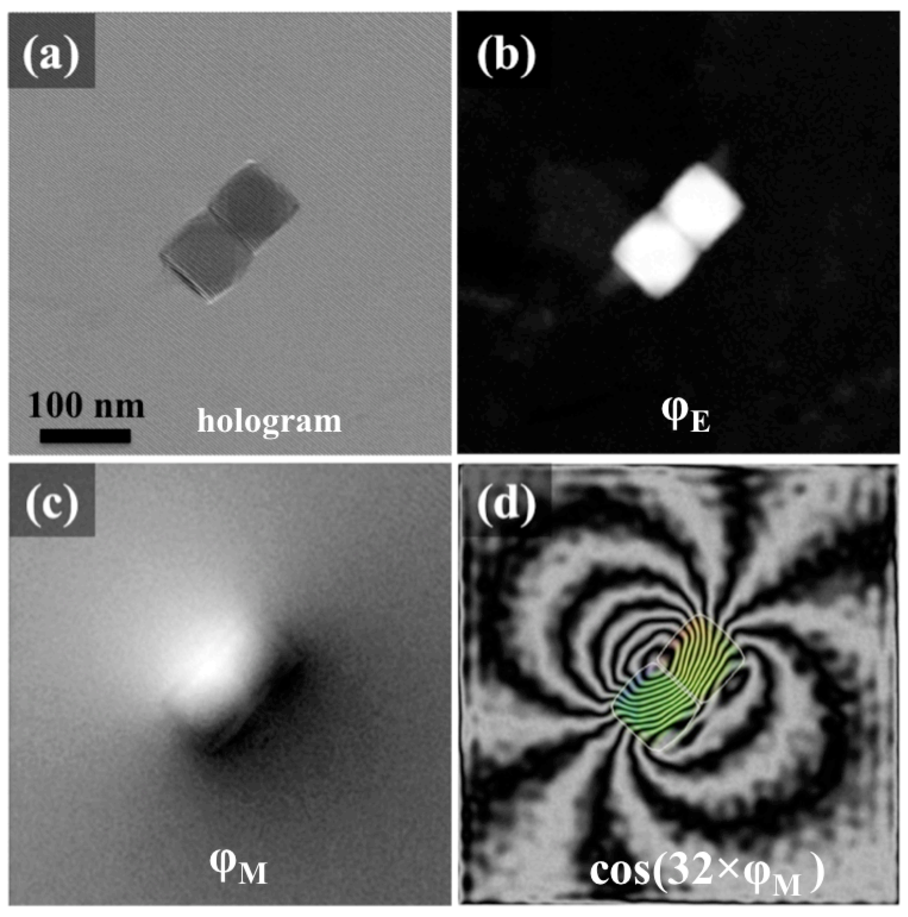


In-situ magnetization using the field of the objective lens and specimen tilt



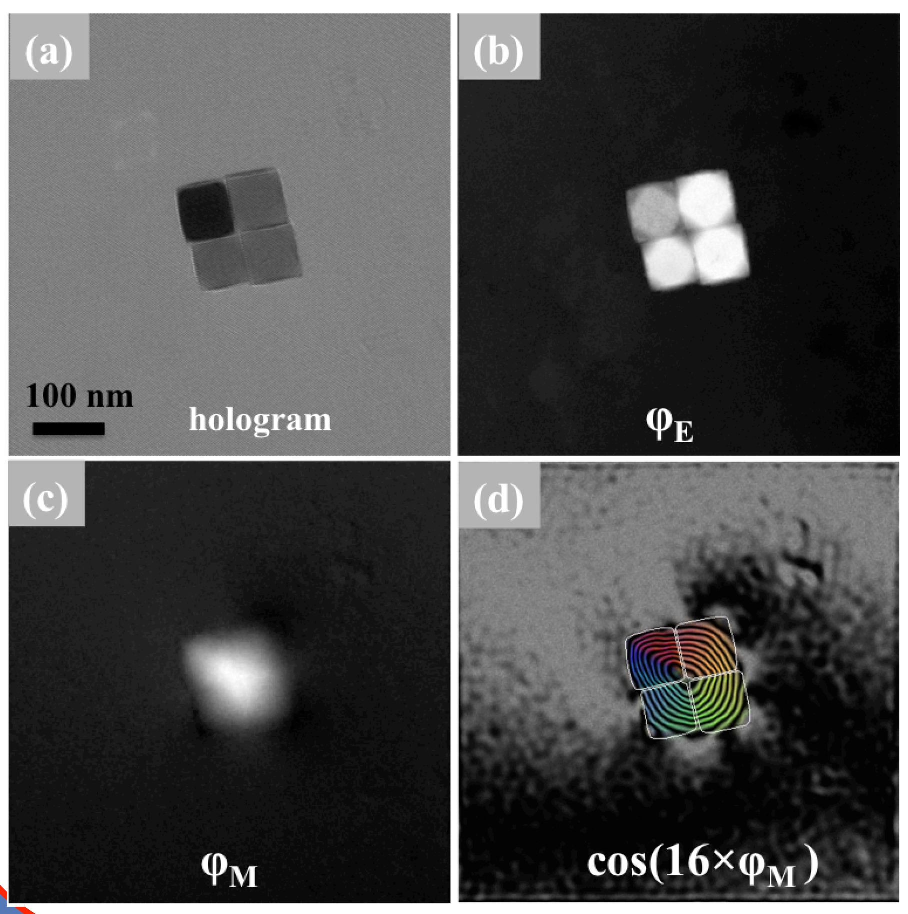
Schematic representations of (a) experimental setup for off-axis electron holography, and (b) in-situ magnetization[2] in a TEM involving specimen tilt and objective lens switch on.

Magnetic Induction Field Distributions



Magnetite *particle dimer*. (a) Recorded hologram. Reconstructed phase images of (b) projected electrostatic potentials and (c) magnetic flux. (d) The cosine of the amplified magnetic phase map.

The contours in both (d) images trace the directions of projected magnetic induction field lines.

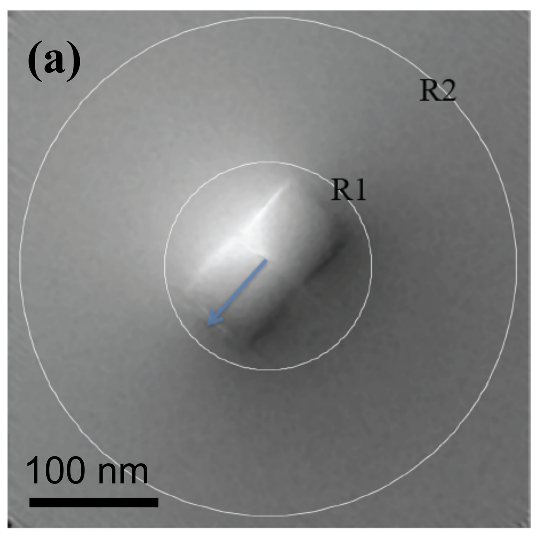


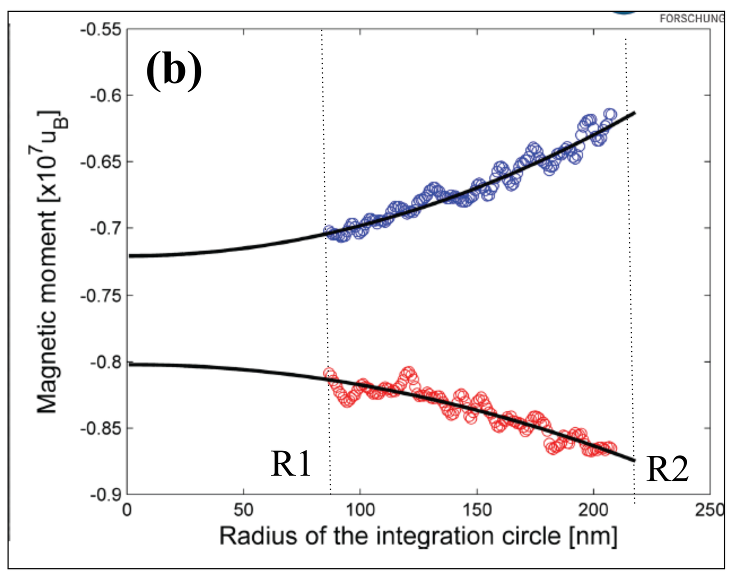
Magnetite *particle tetramer*. (a) Recorded hologram. Reconstructed phase images of (b) projected electrostatic potentials and (c) magnetic flux. (d) The cosine of the amplified magnetic phase map.

Measurement of Magnetic Moments

Measurement scheme proposed by M. Beleggia et al.[1]

- The magnetic moments of nanoparticles or nanostructures $\mathbf{m} = \iiint \mathbf{M}(\mathbf{r})\,d^3\mathbf{r}$ can be measured from phase images.
- The phase gradient is proportional to the magnetic induction projected along the electron trajectory. $\frac{\phi_0}{\pi}\left[\hat{\mathbf{z}} \times \nabla\varphi(\mathbf{r}_\perp)\right] = \int_{-\infty}^{+\infty} \mathbf{B}(\mathbf{r})\,dz$
- If the phase gradient is integrated over a portion of the field of view, then a proportionality can be established between the integrated phase gradient and the volume integral of the induction, $\frac{\phi_0}{\pi}\left[\hat{\mathbf{z}} \times \iint \nabla\varphi(\mathbf{r}_\perp)\,d^2\mathbf{r}_\perp\right] = \iiint \mathbf{B}(\mathbf{r})\,d^3\mathbf{r}$ a quantity denoted "inductive moment" $\mathbf{m}_B$. $\mathbf{m}_B = \frac{1}{\mu_0}\iiint \mathbf{B}(\mathbf{r})\,d^3\mathbf{r}$
- The relationship between the magnetic moment **m** and the inductive moment $\mathbf{m}_B$. If the region of integration is a circle encompassing all magnetized elements, the relationship is: $\mathbf{m}_B = 1/2\ \mathbf{m}$.
- To improve statistics and reduce artifacts, the integration is carried out over circles of decreasing radius until the integration loop touches the particle boundary, and the obtained curve is extrapolated to zero radius using a parabola function.





(a) Magnetic phase map of particle-dimmer for moment analysis with inner circle R1 and outermost circle R2. The blue arrow denotes the orientation. (b) The plot of integrated phases for moment estimation by parabolic extrapolation to zero-radius.

$\mathbf{m}_B = 1.078\times10^7\mu_B$

$\delta\mathbf{m}_B = \pm\,3.352\times10^4\mu_B$

The measurement errors estimated from the parabola-fitting with 95% confidence interval are less than ~1%

Discussion

- With the particle volume V, magnetization $\boldsymbol{M} = 2\boldsymbol{m}_B/V$, the $\mu_0\mathbf{M}$ is about 0.45T that falls short to the 0.6T for bulk Fe_3O_4. The discrepancy can be ascribed to non-uniformly magnetized particle-dimer and/or surface maghemitization.
- The moment analysis by contour-integration method is robust against phase ramps and phase errors associated with perturbed reference wave or phase image processing.

Conclusion

- Magnetic fields of magnetic nanostructures can be obtained with nanometer resolution by off-axis electron holography in the TEM operated in Lorentz mode.
- Magnetic moment of magnetic nanostructures can be directly measured by the contour-integration method on the retrieved magnetic phase images with better than 1% accuracy.
- The sources of phase errors, e.g., image misalignment, diffraction contrast and magnetic reversibility, affecting the accuracy of moment measurement have been addressed experimentally and using simulations.

References

[1] M. Beleggia, T. Kasama, R. E. Dunin-Borkowski. Ultramicroscopy, **110**, 425-432, (2010)

[2] R.E. Dunin-Borkowski, M.R. McCartney, D.J. Smith, S.S.P. Parkin. Ultramicroscopy, **74**, 61-73, (1998)

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