Experiment Brief

K2 IS Camera

Title

Application of off-axis electron holography in measurements of magnetism in nanoscale magnetite mesocrystals.

Gatan instrument used

K2® IS camera is the fastest and highest performance *in-situ* camera to resolve dynamic details in heating, catalysis, mechanical deformation, STEM diffraction, electrical testing, and chemical reaction experiments.

Background

Mesocrystals are defined as superstructures based on the crystallographically ordered assembly of nanoparticles, which are indistinguishable by diffraction techniques from the real single crystal of the same phase. Magnetite (Fe_3O_4) single crystals of ~30 nm in size typically display a stable single domain behavior, whereas smaller entities are superparamagnetic. The magnetic characterization of such small crystals represents a significant challenge due to very low effective magnetic phase shift values. Here the magnetic induction within and around magnetite nanocrystals arranged in rings and chains was used to determine how the mesocrystalline nature of the particles and their interaction influence their magnetic properties.

Materials and Methods

Off-axis electron holograms were acquired at 300 kV in Lorentz mode with a K2 IS camera in Summit configuration using the linear, counting and super-resolution modes. The direction of magnetization in the crystals was reversed at room temperature in-situ in the TEM by tilting the sample by ±75° and turning on the conventional microscope objective lens to apply a magnetic field of 1.5 T to the sample, parallel to the direction of the electron beam. The objective lens was then turned off and the sample tilted back to 0° for hologram acquisition in field-free conditions (residue field is <0.1 mT). The different holograms were recorded with the specimen magnetized in opposite directions and the mean inner potential was separated from the magnetic potential.

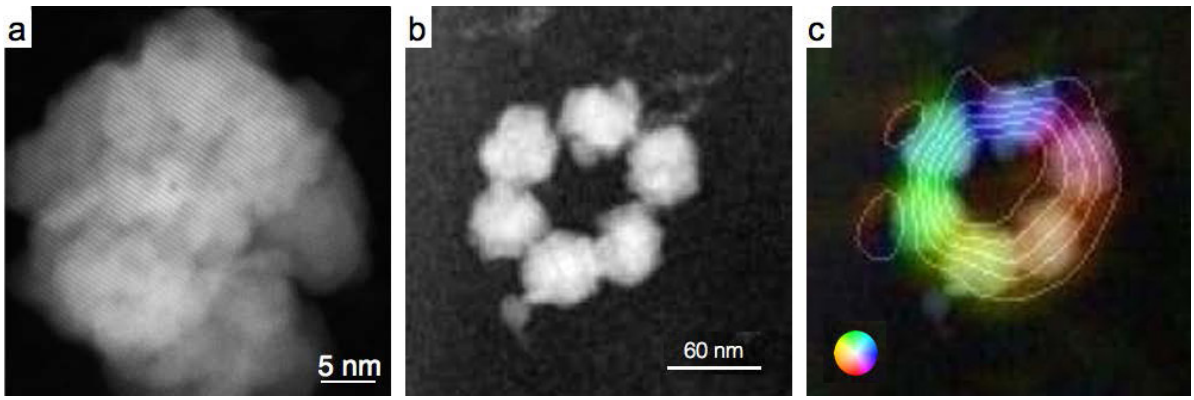

Figure 1. (a) High resolution HAADF STEM image of a single magnetite mesocrystal, showing each particle is composed of smaller subunits. (b) Mean inner potential of a magnetic ring of magnetite mesocrystals and the corresponding (c) magnetic induction map (showing that each particle has a single magnetic domain). The contour spacing is 0.06 radian.

Summary

Relationship between the structure and magnetic properties of magnetite mesocrystals was studied in this research. Using the K2 IS camera, very clear magnetic induction maps could be recorded on several particles. These maps had very small amount of noise from the substrate and proved that these magnetite nanocrystals can be regarded as mesocrystals, and their magnetic properties are dominated by their superstructure.

Credit(s)

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Gatan, Inc. is the world's leading manufacturer of instrumentation and software used to enhance and extend electron microscopes—from specimen preparation and manipulation to imaging and analysis.

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