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Experiment Brief

K3 IS Camera

Title

Magnetite nanoparticle orientation mapping from a 4D STEM dataset

Gatan Instrument Used

The K3® IS camera delivers simultaneous **low-dose imaging** via real-time electron counting, fast continuous data capture, and a large field of view. In a 4D STEM experiment, the STEMx® system precisely synchronizes the speed of the scanning probe to the camera frame rate to enable high-speed data acquisition and eliminates the potential for data loss.

Background

Magnetite (Fe_3O_4) nanoparticles are promising magnetic materials for hyperthermia, MRI contrast agents, and as magnetic cores in high switching frequency inductors and transformers. In each of these applications, a strong magnetic saturation (M_{sat}) is necessary. Recent studies show that the presence of antiphase boundaries (internal defects) can significantly reduce this value. Identifying optimal synthetic conditions is useful to produce high-quality NPs with M_{sat} approaching maximum bulk values.

Materials and Methods

Magnetite nanoparticles were observed using 4D STEM on a Titan ETEM, where a K3 IS camera was used to capture counted CBED diffraction patterns during the STEM scan. The full dataset consists of an array of 70 x 70-pixel positions with a 1440 x 1023-pixel diffraction pattern acquired at each pixel position. The dataset took approximately 100 s to acquire, and was captured with a low beam current; only 0.3 pA ($1.742*10^6$ e^-/s) was measured by the camera. Virtual SAED apertures were applied to the data using standard tools in Gatan Microscopy Suite® (GMS). Python scripting in GMS was then used to process the data to produce a map of pattern orientation and visibility by measuring the direction and magnitude of the strongest spot in each pattern. A maximum pattern, where each pixel is the maximum value of that pixel over all of the patterns in the dataset, was also produced using Python scripting.

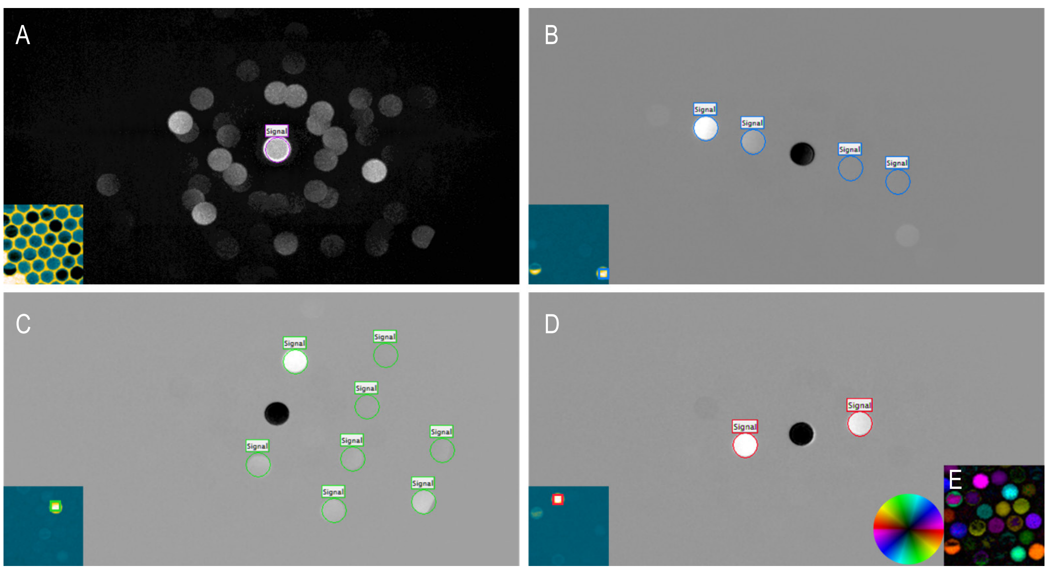

Figure 1. Virtual apertures and orientation mapping. A) Maximum diffraction pattern, where each pixel is the maximum value of that pixel over all patterns in the dataset. Inset: Virtual BF Image from the virtual aperture indicated in A. B – D) Diffraction patterns summed from the square regions specified in the corresponding insets. B – D Insets: Virtual DF images from the virtual apertures indicated in B – D. E) Map of brightest spot orientation and intensity. The color hue in the map specifies the direction from center of the strongest spot in the diffraction pattern, while the brightness corresponds to the intensity of this peak. The average pattern over all pixels was subtracted from the data.

Summary

The map of lattice spacing orientation can be used to determine which particles are similarly oriented, as well as the number of particles where there is significant structural variation within a single particle. The K3 IS, therefore, provides statistically relevant information on the presence of particle defects, which can then inform batch synthesis to produce high quality, single-crystal magnetite nanoparticles. The observation of any correlated orientation between particles is important, as interparticle interactions can adversely affect the desired magnetic behavior.

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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