Application Note

Aduro 500™

Off-axis electron holography under high temperature and oxidizing environment

Introduction

Conventional TEM imaging uses mass-thickness and phase information to create an image of the sample. Electron holography measures the phase shift of electrons after it passes through the sample due to electric and magnetic fields. Off-axis holography is the most common form of holography where a biprism — consisting of a conductive wire — is located close to the image plane and is positively biased. Overlapping of a "reference" electron wave passed through vacuum and the electron wave that has passed through the sample forms the off-axis hologram. Using this technique one can map the magnetic flux density and electrostatic fields in a sample with nanometer resolution. Spintronic devices such as magnetic random access memory and magnetoresistive read/write heads in hard disk drives, magnetotactic bacteria, and multi- or single-domain magnetites are among some of the applications that can take advantage of this technique.

The TEM excels at high-resolution imaging, chemical and structural analysis, and mapping electric and magnetic fields using holography. When combined with *in situ* systems capable of applying an electrical bias and high temperature, researchers can study material behavior under real-world conditions. The Protochips Aduro system heats or applies an electrical biasing inside the TEM. It is based on patented MEMS devices called E-chips, which are available in a variety of geometries that suit many types of experiments. The ultra-stable thermal E-chips heat to 1200 °C, and the electrical E-chips enable low noise measurements in the picoAmp range. The E-chips are robust and result in minimal degradation — a highly desired property when researchers require multiple experiments and correlative measurements with different instruments and techniques. Aduro includes a powerful and user-friendly workflow-based software, which makes experiments reproducible, consistent and precise. This enables valuable correlation between atomic resolution images and videos and the material's structural response to applied stimuli.

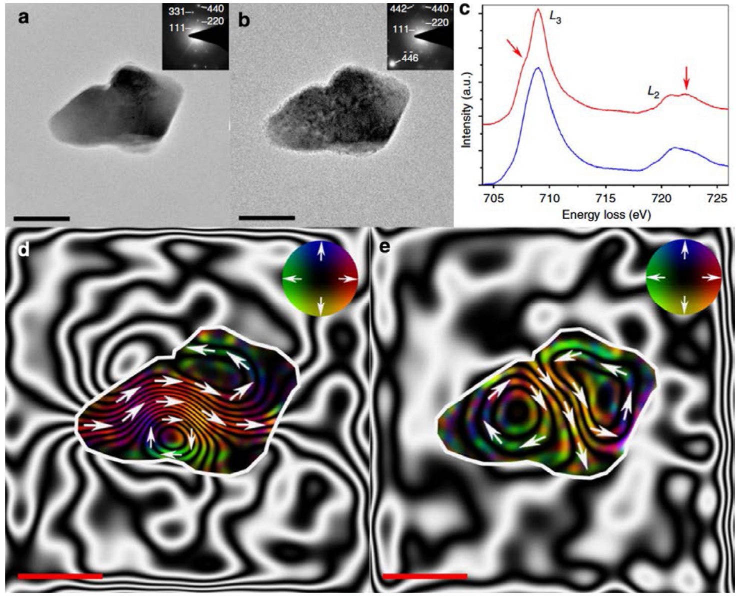

Figure 1: (a-c) bright-field images and EELS analysis of a magnetite particle before and after oxidation. (d, e) Magnetic induction maps evolution determined from the magnetic contribution to the phase shift reconstructed from holograms.

Experiment

The experiments were preformed by multiple groups including Almeida, et al., at Imperial College London, Dunin-Borkowski, et al., at the Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons (ER-C) and the Peter Grünberg Institute and Hansen, et al., at Denmark Technical University (DTU) Center for Electron Microscopy. The Aduro system was used inside a Cs-corrected FEI Titan, operating at 300 kV at ER-C, and an FEI Titan ETEM operating at 300 kV at DTU. All images were taken in bright-field TEM mode. Off-axis electron holograms were acquired in Lorentz mode, with a CCD camera and an electron biprism operating at 160 V. The magnetic fields of individual magnetite (Fe_2O_3) particles were observed upon heating from room temperature to 700 °C at 1 °C/s in vacuum at ER-C. The structural changes of Fe_2O_3 were studied in the ETEM at DTU under 9 mbar of O_2 after 8 hours to correlate chemical alteration to magnetic evolution of Fe_2O_3 particles. Electron energy loss spectra were collected to confirm any phase transitions and structural evolution.

Discussion

Fig. 1a and 1b represent bright-field TEM images of a pristine and heated Fe_2O_3, respectively, where physical degradation was observed (Fig. 1b). Development of new peaks in the EEL spectra (indicated with red arrows in Fig. 1C) confirmed oxidation and chemical alteration to γ- or α-Fe_2O_3. The corresponding magnetic induction map, Fig. 1d, indicates the presence of two vortices, which are distinctly separated after oxidation, Fig. 2e, flowing in opposite directions around a central transverse axis. The magnetic contour spacing also widened indicating a weaker remnant magnetic field. Upon heating individual Fe_2O_3 nanoparticles under vacuum, similar behavior was observed where the spacing between the contours increased. However, upon cooling the magnetic contours narrowed, indicating a recovery of the remnant magnetic field, proving permanent structural and magnetic change occurs upon chemical alteration. This study demonstrates the capabilities of Aduro combined with off-axis electron holography to better understand the magnetic redistribution upon heating and chemical changes in magnetite particles.

Applications

Combined with holography capabilities, one can investigate local magnetic and electrostatic maps of nanoparticles such as Co or CoPt in addition to Fe_xO_y, magnetotactic bacterium, and doped semiconductor devices, under the presence of external stimuli. The Aduro system is an important component of such accurate and precise measurements while atomic resolution and quantitative analysis are required. It delivers image stability for EELS acquisition and provides consistent interference fringe resolution in the acquired electron holograms. In addition to thermal experiments, Aduro is ideal for investigations where applied electric fields result in structural or property changes. The system Aduro is compatible with major microscope manufacturers and features a powerful workflow-based software, which is a key component to applying consistent stimuli and eliminating thermal and electrical deviation in the experiment.