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Direct visualization of chemical and thermo-remanent magnetization of pseudo-single-domain magnetite grains and the implications for reliable paleomagentic signal acquisition

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In order to reliably interpret paleomagnetic measurements, the mechanisms of chemical remanent magnetization (CRM) and thermoremanent magnetization (TRM) must be fully understood. Currently, most models of CRM and TRM processes only exist for the smallest, uniformly magnetized grains, termed single domain (SD). However, the magnetic signal from rocks is often dominated by slightly larger grains containing non-uniform magnetization states, termed pseudo-SD (PSD) grains.

Magnetite (Fe_3O_4) is the most magnetic naturally occurring mineral on Earth, carrying the dominant magnetic signature in rocks and providing a critical tool in paleomagnetism. The oxidation of Fe_3O_4 to other iron oxides, such as maghemite (γ-Fe_2O_3) and hematite (α-Fe_2O_3), is of particular interest as it influences the preservation of remanence of the Earth's magnetic field by Fe_3O_4. Further, TRM in Fe_3O_4 grains is acquired in the direction of the ambient geomagnetic field as they cool below their Curie temperature (T_C) of ~ 580 ˚C. The latest transmission electron microscopy (TEM) techniques like electron holography and environmental TEM (ETEM) allows for the imaging of magnetization in nano-scale minerals during *in situ* heating under vacuum and controlled atmospheres.

In the present study, synthetic Fe_3O_4 particles in the PSD size range (< 200 nm) were heated *in situ* in an ETEM under an O_2 atmosphere. Close examination of Fe_3O_4 particles after *in situ* heating revealed surface degradation, whilst electron energy-loss spectroscopy confirmed their oxidation. The effect of CRM was visualized using electron holography, in the form of reconstructed magnetic induction maps, where the oxidized grains exhibited a loss of overall remanence and change in remanent direction.
The thermomagnetic behavior of Fe_3O_4 particles in the PSD size range is also investigated using off-axis electron holography. Magnetic induction maps, which are recorded during *in situ* heating up to above the T_C, reveal the PSD nature of several Fe_3O_4 grains by visualizing their vortex domain states. The vortex states in small Fe_3O_4 grains (Fig. 1a & b) are shown to rotate or collapse into a single-domain state close to its unblocking temperature (Fig. 1c), rather than remaining thermally stable as seen in the vortex states of larger Fe_3O_4 grains.