Unique Views of Catalysis and Magnetism at the Nanoscale in the Transmission Electron Microscope

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Modern environmental transmission electron microscopes (ETEMs) can be equipped with aberration correctors and monochromators to improve spatial resolution and spectral sensitivity during dynamic studies of chemical reactions and growth processes. We have recently installed an FEI Titan 80-300 ETEM, in which seven different gases can be introduced into the microscope at pressures of up to 1500 Pa and additional gases can be connected when required. In an attempt to understand fundamental processes in catalysis that include shape changes and sintering, we have studied metal nanoparticles on BN, graphene and silica supports in oxidizing and reducing environments at elevated temperature. We observe sintering both by migration and coalescence and by Ostwald ripening, as well as rapid changes in nanoparticle morphology, orientation and crystallography [1].

The addition of an electron biprism to a TEM allows the phase shift of the electron wave that has passed through a thin specimen to be recorded. Interpretation of the phae shift then allows magnetic fields in materials and in working devices to be mapped quantitatively with a spatial resolution approaching 1 nm. A typical 2 nm ferromagnetic particle is associated with a step in phase of $\sim 2\pi/1000$ radians, and the prospect of achieving a phase sensitivity of this magnitude with sub-nm spatial resolution is now offered by ultra-stable electron microscopes that are equipped with high brightness electron sources, aberration correctors and sophisticated software for the automation of lengthy experiments. It is also possible to measure certain physical quantities without the need to interpret the local phase shift itself. For example, the magnetic moment of a nanocrystal can be measured quantitatively from a phase image by making use of the relationship between the volume integral of the induction and the true magnetic moment. This relation can be utilized to study particles of arbitrary shape and magnetization state to yield a measurement of the magnetic moment that is free of most artifacts [2]. Our assessment of the relationship between the phase noise and the error in the measurement of the magnetic moment suggests that it may be possible to measure magnetic moments with magnitudes of below 100 μ_B, both in projection and in three dimensions, if the effects of dynamical diffraction, charging of the specimen due to electron beam irradiation and the presence of adsorbates on the specimen surface are minimized.

We are presently combining ETEM and electron holography experiments by studying the reduction of single crystalline 15 nm Fe oxide cubes to Fe at elevated temperature in hydrogen *in situ* in the electron microscope, and their subsequent reoxidation to polycrystalline Fe oxide. The magnetic properties of the cubes are measured quantitatively using electron holography after each stage of reduction and oxidation. I will discuss the degree to which the presence of the high-energy electron beam affects each of these experiments.

[1] T.W. Hansen, J.B. Wagner, and R.E. Dunin-Borkowski, Materials Science and Technology **26**, 1338 (2010).
[2] M. Beleggia, T. Kasama, and R.E. Dunin-Borkowski, Ultramicroscopy **110**, 425 (2010).