Nanomaterial Transformations Captured by Atomic Resolution 3D Electron Microscopy

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Electron tomography is a powerful tool to explore the morphology, 3D structure, and composition of a broad range of (nano) materials. Although these experiments are already at the state-of-the-art, several open questions remain. These questions are often related to the fact that 3D characterization by TEM is typically performed using the conventional conditions of a TEM: ultrahigh vacuum and room temperature. Since it is known that the morphology and consequently, the activity of nanomaterials will transform at higher temperatures or pressures, this poses a fundamental limitation. It is therefore not surprising that much effort has been devoted to monitoring nanoparticle transformations upon application of external stimuli by TEM.

For irreversible transformations, nanomaterials can be exposed to such stimuli outside the microscope and investigated afterward, allowing even for a direct comparison of the same nanoparticle before and after the stimuli was applied. This approach is particularly interesting for measuring nanoparticle transformations under stimuli that are challenging to introduce inside the TEM, for instance, laser excitation [1]. For example, we performed atomic resolution electron tomography on the same mesoporous-silica-coated gold nanorod, before and after femtosecond laser irradiation. Combined with molecular dynamics simulations based on the experimentally determined 3D atomic-scale morphology, the complex atomistic rearrangements, causing shape deformations and defect generation, could be unravelled [2].

For reversible processes and to obtain dynamic information, *in situ* TEM characterization can be performed either using a dedicated environmental TEM or through a wide variety of holders based on MEMS devices. However, understanding the complex changes for anisotropic nanosystems in 3D rather than in 2D remains very challenging. Our recent experiments demonstrate the progress that can be obtained by accelerating both the acquisition and reconstruction during electron tomography [3,4]. In this manner, we were able to perform a dynamic characterisation of shape and compositional changes of (bi)-metallic nanoparticles at high temperatures, even with atomic resolution [5]. Finally, by combining aberration corrected electron microscopy with a quantitative interpretation [6–8], we can provide quantitative measurements of the coordination numbers of the surface atoms of catalytic nanoparticles at high temperatures and in gaseous environments [9].

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