

In-situ liquid and gas experiments in TEM

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Transmission electron microscopy (TEM) is one of the key experimental methods for characterization of nanomaterials, providing direct structural and chemical information down to the atomic scale. The conventional TEM allows us only a post-mortem analysis, where samples are analyzed only before and/or after synthesis, treatment, or operation. In-situ TEM overcomes this limitation by confining liquids or gases between electron-transparent membranes, enabling the direct observation of transformations and transient states of materials under controlled environments. Such experiments are especially valuable for following nanoparticle nucleation, growth, reshaping, dissolution, oxidation/reduction, and catalytic activation in real time.

In our contribution we will present fundamentals, limitations, and practical examples of in-situ liquid and gas experiments in TEM performed at F6-JSI. In liquids, particular attention will be given to liquid-flow cells, radiolysis, beam-induced chemistry, and the balance between spatial resolution, dose, and realistic reaction conditions, on example of anisotropic gold nanoparticles. For gas experiments, we will discuss in-situ reduction experiments on Fe-oxide nanoparticles exposed to hydrogen gas at elevated temperatures, where morphological and structural changes can be correlated directly with the surrounding reactive atmosphere. The comparison of liquid and gas-cell approaches show that both have common challenges, such as electron-beam effects and window scattering, while the gas-cell is especially useful for operando studies of functional nanomaterials.