In-Situ TEM on (De)Hydrogenation and Oxidation/Reduction of Pd at High Pressures

Yokosawa, Tadahiro[1)] and Zandbergen, Henny W. [2)]

1) Institute for Nuclear Waste Disposal, Karlsruhe Institute of Technology, Eggenstein - Leopoldshafen, Germany.
2) Kavli Institute of Nanoscience, Delft University of Technology, Delft, Netherlands.

Abstract:
In order to understand the mechanism of chemical reactions based on catalytic effects, such as, hydrogen (de)absorption and oxidation/reduction on a metallic crystal, it is important to study the processes under realistic operating pressures. Combining the high vacuum demands of transmission electron microscopy (TEM) with in-situ investigations at industrial relevant gas pressures is a challenging task. Here, we show that a setup based on a micro electro mechanical system (MEMS) based environmental cell (nanoreactor) mounted in a custom made holder, which can be operated till at 4.5 bar in a standard TEM. The cell contained thin electron transparent SiN windows and incorporated a heating spiral, which was controlled externally. The holder was connected to a custom made external gas supplier that was operated manually. We demonstrate the feasibility of the approach by observing the reversible lattice expansion and shrinkage of palladium (alpha-phase to beta-phase and vice versa) due to hydrogenation and dehydrogenation at known hydrogen pressures and temperatures. The same technique using the nanoreactor is applied to in-situ oxidation/reduction of palladium as well, showing appearance and disappearance of the oxides in the individual palladium particles at oxygen and hydrogen pressures, respectively. These results show that this is a fast method to investigate metal hydrides and oxides with information at the nanometer scale.