In-situ TEM on (de)hydrogenation of Palladium

Abstract

In order to understand the mechanism of chemical reactions based on catalytic effects, such as, hydrogen (de)absorption and oxidation on a metallic crystal, it is important to study the processes at the atomic level 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 mounted in a custom made holder could be operated at 4 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. This result shows that this is a fast method to investigate metal hydrides with information at the nanometer scale.