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TEM HOLDER FOR SAMPLE TRANSFER UNDER REACTION CONDITIONS

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Abstract

Environmental transmission electron microscopy (ETEM) studies are usually performed using conventional sample holders in a dedicated ETEM [1] or in a traditional TEM by use of a dedicated high-pressure cell sample holder [2]. In both cases, the setup defines the conditions regarding gas, pressure and temperature, which usually are far from the operando conditions of e.g. heterogeneous catalysis. Our efforts focus on bridging these gaps by establishing *in situ* sample transfer between complementary measurement techniques.

To fully exploit the capabilities of ETEM complementary experiments and characterization techniques are beneficial. Normally, the complementary measurements are done in parallel with experiments separated in time and space [3] or by mimicking a reactor bed by changing the feed gas composition according to reactivity and conversion measured in dedicated catalyst set-ups [4]. Furthermore, dedicated transfer holders have been used to transfer catalyst samples between reactor set-ups and TEM at room temperature in inert atmosphere [5]. To take the full advantage of complementary *in situ* techniques, transfer under reactions conditions is essential.

This study introduces the *in situ* transfer concept by use of a dedicated TEM transfer holder capable of enclosing the sample in a gaseous environment at temperatures up to approx. 900°C. By oxidation and reduction experiments of Cu nanoparticles it is shown possible to keep the reaction conditions during transfer outside the microscope.

This opens up for the possibility to do complimentary *in situ* experiments of the exact same sample without changing the sample condition during transfer. As an example a commercially used methanol catalyst $Cu/Zn/Al_2O_3$ [6], was reduced at 1 bar in 2% H_2 in He at 220°C and in an *in situ* x-ray diffractometer (XRD) setup and subsequently successfully transferred under reaction conditions to the ETEM.

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