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In situ Characterization of Catalysts: Combining X-ray and electron microscopy

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Fundamental insight into structure-functionality relationships is required to develop and improve properties of heterogeneous catalysts. As catalysts may change their structure with respect to the environment, it is essential to investigate the catalysts under reaction conditions. Furthermore, structural and compositional information have to be acquired on different length scales[1] and such *in situ* studies require dedicated complementary techniques. Traditionally, nanoscale imaging and spectroscopy of catalysts in a gaseous environment is performed in an environmental transmission electron microscope (ETEM). TEM gives insight in the atomic changes during reaction, however it is restricted to relatively low pressure (<~1 kPa) and a thin sample (<~100 nm)[2]. Spatially resolved information on the meso scale (50 nm–1 µm) can be obtained by X-ray microscopy, which enables *in situ* studies at both ambient and elevated pressure[3]. This contribution elucidates catalyst properties by combining X-ray and electron based microscopy.

One example highlighted combines X-ray imaging with ETEM studies of a bifunctional Cu/ZnO@zeolite core-shell catalyst for direct production of methanol[4]. A special designed *in situ* cell was used to enable *in situ* hard X-ray ptychography. The cell is based on a TEM heater chip from Protochips. The complementary nature of *in situ* hard X-ray ptychography and electron microscopy was applied to study the stability of the core-shell catalyst in a hierarchical manner at different length scales during reduction and oxidation treatments. *In situ* ptychograms of the Cu/ZnO@zeolite core-shell catalyst are shown in Figure 1. The study reveals a stable core-shell interface at 250°C, although reduction of the Cu containing core material led to a shrinkage of the particles on the nanometer scale. At further heating to 350°C changes on the µm scale were observed.

The results underline the need for complementary techniques and highlight the potential of these for application in catalysis.

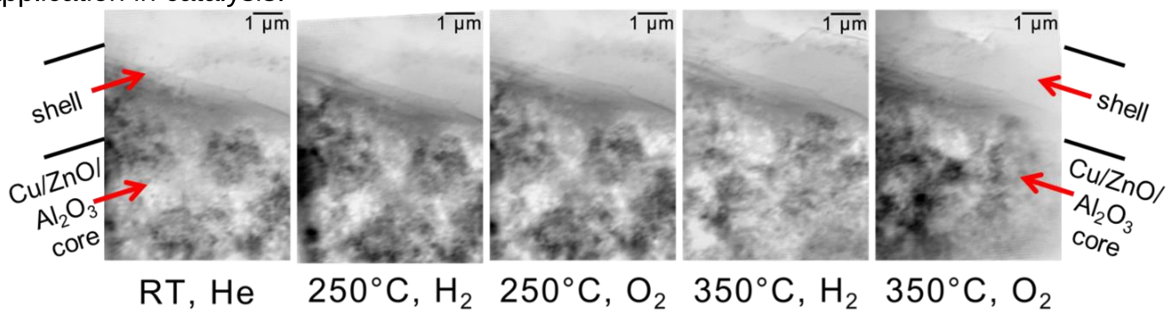

Figure 1. In situ ptychograms (phase contrast) of a thin slice of a Cu/ZnO@zeolite core-shell catalyst at room temperature in He, 250°C in H_2, 250°C in O_2, 350°C in H_2, and 350°C in O_2, respectively [4].

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