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# Micro-Electro-Mechanical Systems for Electron Microscopy in Catalysis

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It has long been realized that the state and functionality of solid catalysts may depend intimately on the reaction conditions. The fact that, strictly speaking, the active state only exists during catalysis emphasizes the need for studying catalysts *in situ* under relevant reaction conditions. *In situ* studies by means of transmission electron microscopy (TEM) are particularly challenging due to the stringent vacuum requirements for the electron source and imaging conditions.

With developments of differentially pumped vacuum systems and sample stages, reactive gas environments can be confined on a millimeter-scale to the vicinity of the TEM specimen. The instrumentation enables TEM of catalysts *in situ* during exposure to gas environments at pressures of 1-20 mbar and at temperatures of up to about 900$^{o}$C, while an image resolution of about 1.0 Å is retained, depending on the microscope and its operation [1]. While the instrumentation in itself opens up for new insight into the surface structure and reactivity of catalysts, parallel developments of micro-electro-mechanical systems (MEMS) may be beneficial for targeted sample preparation and provide complementary functionality. In this presentation, I will outline recent work that employs MEMS for *in situ* TEM of Pt nanoparticles in relation to the catalytic abatement of environmental harmful emissions from diesel engines.

The first example considers MEMS fabrication as a route for the preparation of well-defined two-dimensional model catalyst systems to facilitate studies of the Pt nanoparticle stability [2-4]. Figure 1 outlines a model catalyst, which initially consisted of a mono-dispersed and uniform ensemble of Pt nanoparticles on a highly electron-transparent oxide surface. Upon exposure to oxidizing conditions, the Pt nanoparticles sintered into larger sizes. Time-resolved TEM image series acquired *in situ* unequivocally reveal that sintering of the Pt nanoparticles was mediated by the Ostwald ripening process. Furthermore, the time-resolved image series allow kinetic information to be extracted and compared with predictions made from ripening models [2-4].

The second example demonstrates a nanoreactor concept (Figure 2) [5-6]. Made as MEMS, the nanoreactor integrates a micrometer-sized gas-flow channel with a microheater and an array of electron-transparent windows of silicon nitride. The nanoreactor allows for the introduction of gasses at ambient pressure levels and elevated temperatures. The ambient pressure level is unique as it directly matches conditions of relevance for catalyst testing. Still, the nanoreactor enables high-resolution TEM imaging under those conditions. The nanoreactor performance is demonstrated by a study of Pt nanoparticles catalyzing the oxidation of carbon monoxide. Specifically, during the catalytic reaction, time-resolved and high-resolution TEM image series were acquired of the Pt nanoparticles concurrently with mass spectrometry of the gas phase. The possibility to directly correlate the atomic-scale structure and reactivity of nanoparticles at conditions relevant to catalysis will be discussed.

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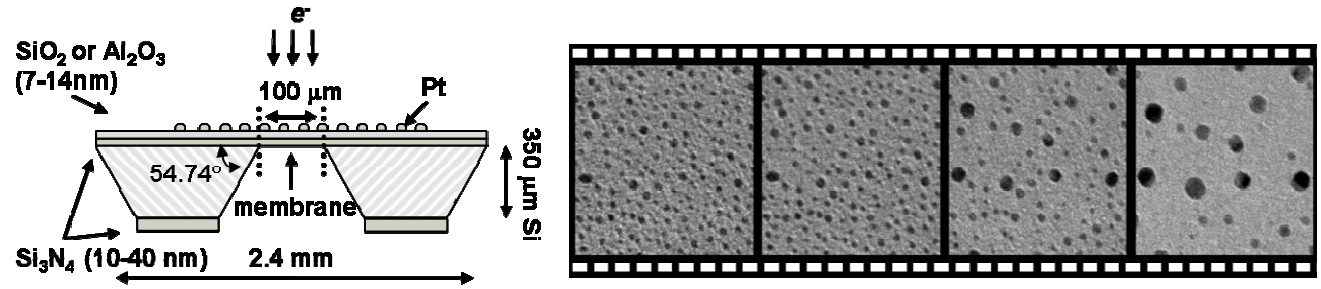


**Figure 1.** Left: Cross-sectional sketch of the two-dimensional model catalyst. Right: Time-resolved TEM images (130 nm x 130 nm) recorded *in situ* at 10 mbar technical air at 650 $^{o}$C [2-4].

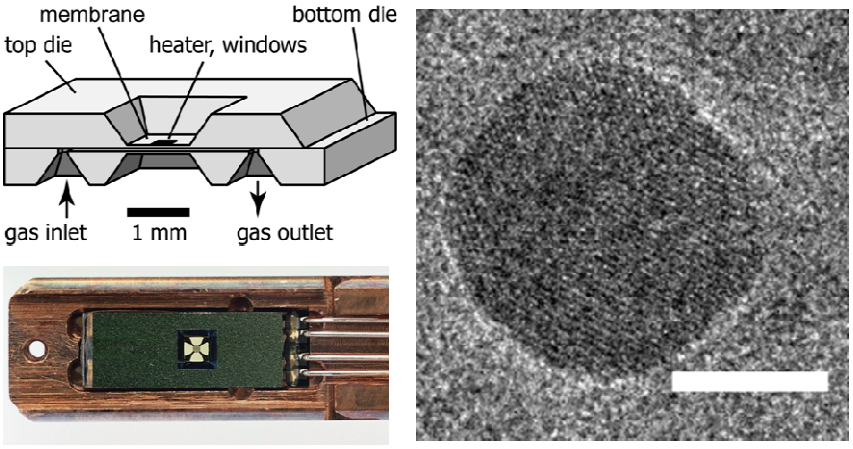


**Figure 2.** Left: Sketch and photo of the nanoreactor. Right: High-resolution TEM image of a Pt nanocrystal acquired *in situ* at 1000 mbar of CO/$O_2$/He at 400 $^{o}$C. Scale bar, 5nm [5-6].

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