Synchrotron nanotomography studies of hierarchical nanoporous gold catalysts

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Catalysts are a class of functional material whose structural features and chemical reactive behaviour are closely interlinked. Structural imaging of such functional materials, especially catalysts, with high spatial resolution is crucial to investigate their structural properties, including porosity, elemental composition and morphological changes. While two-dimensional (2D) imaging techniques such as electron microscopy and microspectroscopy are mainstream in catalysis research, three-dimensional (3D) techniques based on tomography are getting more attention for the greater quality of extractable information. So far, electron-based methods are well-known for reaching spatial resolutions down to the atomic scale. However, X-ray tomography is also being increasingly utilized for the capability to run beyond ambient atmospheres, non-invasive sample preparation, and extended field of view with additional capability to be combined with nanofocussed X-ray absorption or emission spectroscopy.

This study exploited electron tomography (ET), focused-ion-beam ("slice-and-view"), and ptychographic X-ray computed tomography (PXCT) to visualize the hierarchical pore structure of pure- and ceria-incorporated nanoporous gold (np-Au) as the probed material. np-Au is a catalytically active material with a unique porous structure spanning from nm to µm scale, which is of great interest for catalysis and sensing applications. Moreover, a quasi *in situ* study of thermal annealing of pure and ceria-containing np-Au was also carried out via PXCT to investigate the thermal stability of the catalysts. Samples were synthesized via dealloying and submersion process (University of Bremen) and were cut into particular shapes using focused ion beam (Institute of Nanotechnology, KIT). A thin lamella, cuboid and cylinder were made for ET, "slice-and-view", and PXCT, respectively. ET was performed using Titan 80-300 (Institute of Nanotechnology, KIT), "slice and view" using EsB 1540 dual-beam FIB-SEM, and PXCT using a customized setup at the cSAXS beamline (Swiss Light Source, PSI).

The 3D-rendered results from three different techniques exhibited a similar sponge-like structure. The high spatial resolution achieved permitted quantitative measurements to compute the volume fraction, surface area, and pore size distribution of the samples, together with the pore network topology in 3D. Furthermore, the resulting isotropic spatial resolution of 23 nm via PXCT is the highest so far known for 3D X-ray imaging of a catalytic material.

All in all, synchrotron-based PXCT is highlighted as a technique with unique advantages in catalysis research, combining large fields of view, high spatial resolution, with the potential to perform under quasi *in situ* or *operando* conditions. The use of synchrotron X-ray tomography together with X-ray spectroscopies allow to gain detailed information on porosity, chemical composition and stability in a non-invasive manner is widely applicable in the field of catalysis. Such data may be further applied for computational modeling of mass and fluid transport, and inspire the design of more efficient catalysts in future.

This work was supported by Bundesministerium für Bildung und Forschung project(05K16VK1).