Designing a user platform for *in situ* 2D&3D X-ray ptychography at DESY-P06

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In heterogeneous catalysis, *in situ* and *operando* studies involve performing spectroscopic or microscopic analysis under typical reaction conditions, and with simultaneous acquisition of catalytic activity data. Online monitoring of catalyst structure and performance helps to reveal the structure-activity relationships and to design more efficient catalysts. Although beneficial, the construction of proper sample environments for *in situ* studies is challenging and even more if the cell volume has to be downscaled into mm or μm scales, i.e., to compensate the limitation in the probed area and the penetration depth of beam sources as in spectroscopy and microscopy studies, and to maintain small internal volumes to allow residual product gas analysis. Nevertheless, the ability to combine realistic catalytic environments with high resolution X-ray imaging combined with spectroscopy is of fundamental interest in catalysis and material science.

Herein, a series of *in situ* cells for 2D X-ray ptychography (2G cell) and ptychographic X-ray computed tomography with limited tilting angles (3G cell) were developed and optimized at KIT, then tested at the nanoprobe station of beamline P06 at the synchrotron radiation source PETRA III (DESY). The integral design concept is based on commercial MEMS-based chips for electron microscopy, allowing integrated heating and sample positioning with nanometer precision. The MEMS-chips were integrated into a stainless-steel body with gas connections, forming a functional 'nanoreactor'. The cell bodies were produced via mechanical (micro)structuring process (Institute for Micro Process Engineering, KIT). Optimization was carried out using ANSYS AIM Student with fluid flow module to simulate the gas flow and with thermal module to simulate the heat generation. Tests for imaging were conducted using size selected metal nanoparticles and colloidal solutions.

The 2G and 3G cells are major upgrades from the past *in situ* cell for X-ray ptychography, but with a bulky design in cm scale[1]. These new cells allow use of a flexible range of sample types using membrane-type MEMS chips and through-hole MEMS chips, a wide range of temperatures up to 1000 °C, and gas pressures up to 1 bar. This covers a wide range of conditions relevant for catalysis. The optimization showed that (i) 2G cell exhibited flow-over gas movement and 3G cell showed flow-through gas behaviour, (ii) heat localization within 1 mm diameter from the center point and a few μm height from the surface of the heating source. The former result was corroborated by experimental tests on the beamline using an IR-thermography camera. Tilting tests for the 3G cell at P06 revealed a potential tilting angle up to ±35°. The cell geometry additionally allowed collection of complementary fluorescence data in addition to ptychographic images.

All in all, the combination of simulation and practical experiments are versatile to optimally develop *in situ* cells. While the 2G cell is planned for *in situ* high-resolution X-ray ptychography, the 3G cell is intended for *in situ* ptychographic X-ray computed tomography. This approach combining extremely small flow volume (5 and 20 μL, respectively), with the

[1]Baier, S., et al. Microsc. Microanal. 22, 178–188, 2016).

ability to perform electron or X-ray microscopy is required for future *in situ* and *operando* studies at the synchrotron.

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

[1]Baier, S., et al. Microsc. Microanal. 22, 178–188, 2016).