Downloaded from orbit.dtu.dk on: Sep 25, 2021

DTU Library

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

Damsgaard, Christian Danvad; Baier, Sina; Grunwaldt, Jan-Dierk

Published in:
69th Annual Meeting of the Nordic Microscopy Society

Publication date:
2018

Document Version
Publisher's PDF, also known as Version of record

Link back to DTU Orbit

Citation (APA):
Damsgaard, C. D., Baier, S., & Grunwaldt, J-D. (2018). *In situ* Characterization of Catalysts: Combining X-ray and electron microscopy. In *69th Annual Meeting of the Nordic Microscopy Society*

General rights
Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

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

Christian Danvad Damgsaard *[1,2], Sina Baier[2,3], and Jan-Dierk Grunwaldt[3,4].

[1] *DTU Danchip/Cen, Technical University of Denmark, Kgs. Lyngby, Denmark.*
[2] *DTU Physics, Technical University of Denmark, Kgs. Lyngby, Denmark.*
[3] *Institute for Chemical Technology and Polymer Chemistry, Karlsruhe Institute of Technology, Karlsruhe, Germany.*
[4] *Institute of Catalysis Research and Technology, Karlsruhe Institute of Technology, Karlsruhe, Germany.*
*E-mail: christian.damsgaard@cen.dtu.dk

Keywords: In situ, Environmental TEM, Ptychography, Catalysis, X-ray microscopy

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]. The study reveals (Fig. 1) 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.

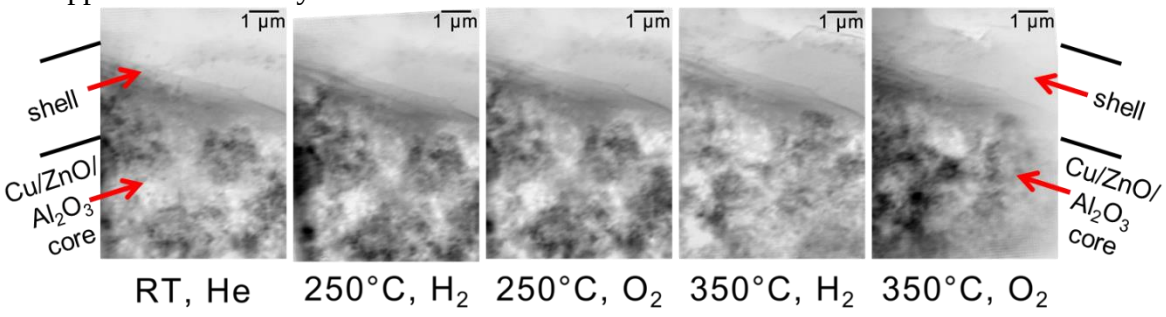

Figure 1: *In situ* ptychograms (phase contrast) of a thin slice of a Cu/ZnO@zeolite core-shell catalyst at room temperature in He, 250°C in H_2, 250°C in O_2, 350°C in H_2, and 350°C in O_2, respectively [4].

[1] J. D. Grunwaldt *et al.*, *ChemCatChem*, vol. 5, no. 1, pp. 62–80, 2013.
[2] T. W. Hansen *et al.*, *Science*, vol. 294, no. 5546, pp. 1508–1510, 2001.
[3] S. Baier *et al.*, *Microsc. Microanal.*, vol. 22, no. 1, pp. 178–188, 2016.
[4] S. Baier *et al.*, *Microsc. Microanal.*, vol. 23, no. 3, pp. 501–512, 2017.