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# Correlative 3D-Characterization of Liquid Metal Catalysts (LMC) utilizing X-ray and Analytical Electron Microscopy

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X-ray microscopy (XRM) allows non-destructive 3D investigations of materials across multiple length scales. At the University of Erlangen-Nürnberg a high-resolution XRM/NanoCT instrument (ZEISS Xradia 810 Ultra) has been recently installed and put into operation. The instrument uses Fresnel zone plate optics to achieve 3D resolutions down to 50 nm and can be equipped with a Zernike phase ring enabling phase contrast in addition to standard absorption contrast imaging. While the latter is well suited to image materials containing regions of sufficiently different densities the former can be employed to study weakly absorbing materials and to discriminate materials exhibiting similar densities. Due to the high resolution capabilities and flexible contrast the ZEISS Xradia 810 Ultra can be combined with Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) techniques for correlative 3D studies of functional materials.

In this contribution we report correlative 3D studies of Pd-Ga liquid metal catalysts which have been recently shown outstanding performance in alkane dehydrogenation and, in particular, high resistance against coking [1]. Such liquid metal catalysts exhibit a complex material structure featuring a molecularly defined, catalytically active liquid film/droplet layer adsorbed on meso- or macroporous silica. High-resolution 3D characterization across different length scales is required to gain deeper insight into the structure and microscopic mechanisms of the catalyst system. SEM imaging (Fig. 1a) and corresponding Energy-Dispersive X-ray Spectroscopy (EDXS) analysis (not shown) reveal the presence of larger Ga-rich particles on the surface of the porous silica but do not allow to clarify to which extend Ga and Pd is incorporated in the bulk of the porous network. By combining absorption and phase contrast imaging in the XRM the metal droplets and the macroporous silica network can be independently resolved. Tomographic reconstruction enables non-destructive characterization of the 3D distribution of metal droplets in the porous network structure (Fig. 1b).

In order to study the composition of individual metal droplets inside the porous network site-specific sample preparation has to be combined with high-resolution analytical TEM techniques. Fig. 1c) exemplarily shows a Scanning Transmission Electron Microscopy (STEM) image of a mesoporous silica network containing a single Pd-Ga particle. The corresponding EDXS mapping clearly shows that the particle has a bimetallic structure with Pd-rich and Ga-rich parts occurring in direct contact to each other. Currently, we perform *in situ* TEM experiments in order to reveal phase transformations and melting behavior of such bimetallic particles upon heating to typical process temperatures of ~ 500 °C.

References:
[1] N. Taccardi et al., Nat. Chem. **9** (2017), 862-867
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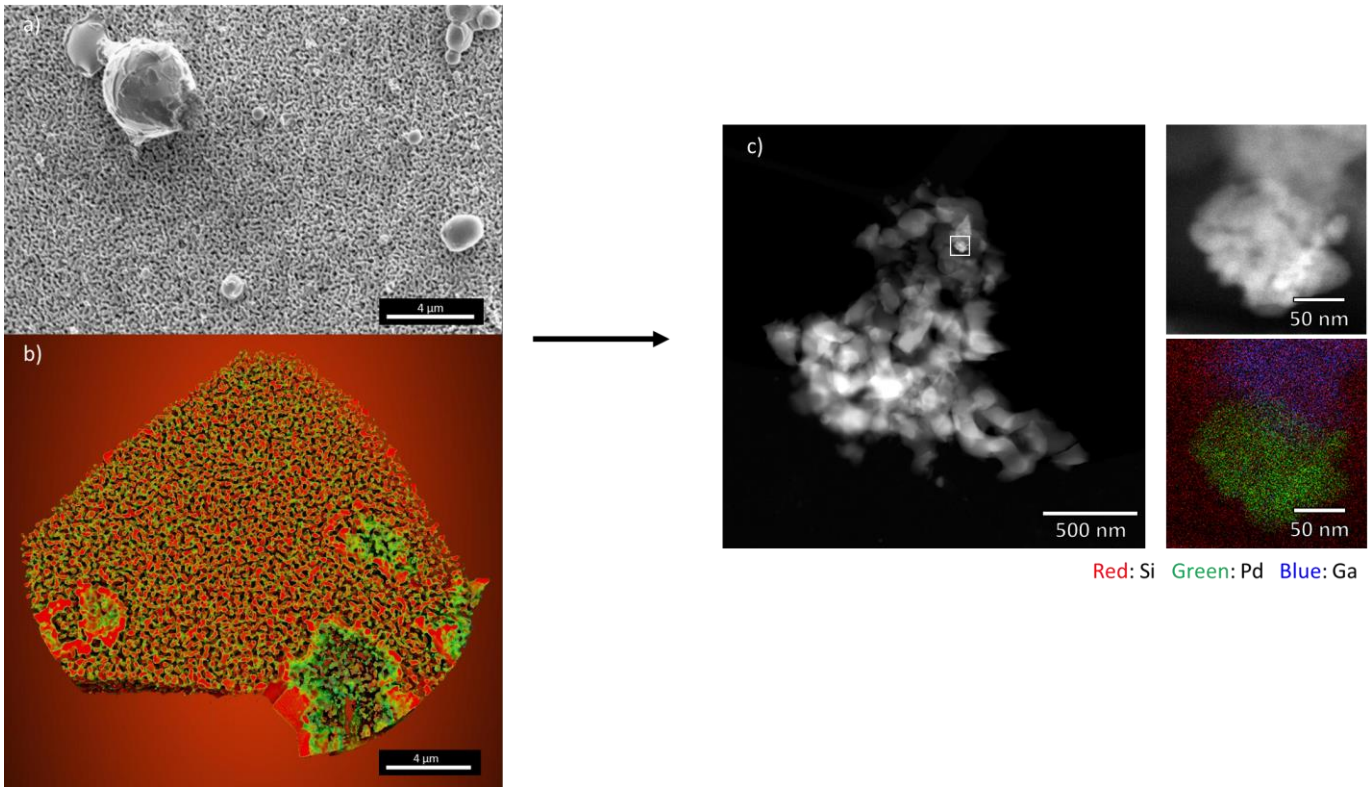


Figure 1: a) SEM image of material system surface: Ga droplets on porous glass network. b) Virtual slicing of 3D volume generated by high-resolution X-ray tomography: higher intensity zones (green) represent Ga phases. c) STEM image of glass ligament (particle composition (Ga:Pd ratio) and size varies from particle to particle inside 3D volume) with corresponding EDX scan: Pd within close proximity to Ga phase.

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