ESRF	**Experiment title:** Humidity induced deformation of ordered mesoporous silica particles with nanodiffraction and 2D- Ptychography	**Experiment number**: SC-4197
Beamline: ID13	**Date of experiment**: from: 13.11.2015 to: 17.11.2015	**Date of report**: 22.02.2016
Shifts: 12	**Local contact(s)**: Dr. Britta Weinhausen, Dr. Manfred Burghammer	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Oskar Paris, Lukas Ludescher*, Institute of Physics, Montanuniversität Leoben, AUSTRIA

Florian Putz*, Michael Elsaesser*, Nicola Hüsing, Department of Materials Science and Physics, Universität Salzburg, AUSTRIA

Gudrun Reichenauer, Christian Balzer, Division Energy Efficiency, ZAE Bayern, GERMANY

Report:

In this proposal we have investigated amorphous silica with hierarchical porosity. The system consists of a macroporous network of struts, which are composed of of cylindrical mesopores ordered in a 2D hexagonal pore lattice. The lattice parameter is around 8 nm, which means that the material diffracts like a crystal at small angles (Fig. 1). The aim of the proposal was twofold:

1) To use X-ray nanodiffraction at small angles to map the local lattice strains and the change of these strains with changing humidity due to water sorption induced deformation.
2) To explore whether 2D ptychographic imaging can be used to detect the bending of struts due to sorption induced deformation.

Single struts and struts connected to joints were prepared by ultrasonic grinding of the monolithic materials and dispersing the particles on Si_3N_4 membranes. These where mounted in the ID13 inhouse slow-flow humidity chamber on a hexapod in the nanofocus hutch of the ID13 beamline. An energy of 14.85 keV was selected and the beam was focussed by the beamline optics to a beam size of approximately 150 x 180 nm. Particles on the membrane were identified with the optical microscope and scanned with a step size of 100 nm while collecting diffraction patterns using the Eiger 6M detector with typical measurement times of 0.1 - 0.5 s per single frame. The samples were scanned at different relative humidities (rH) with humidified nitrogen. We managed to measure several particles of a calzined (~500°C) and a sintered (~750°C) silica monolith at rHs of 20%, 30%, 50% and 70%.

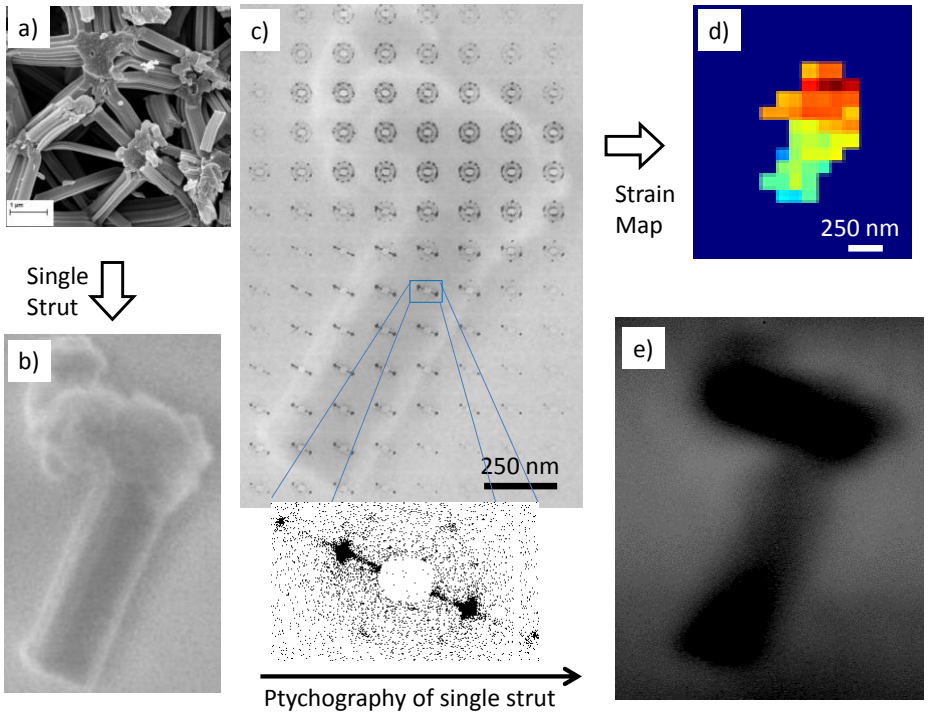

Fig. 1. Single struts (b) were prepared from monolithic samples (a) by ultrasonic milling, and dispersed on silicon nitride membranes. c) shows a composite map of SAXD patterns measured on the strut shown in b), demonstrating the single crystalline nature of the strut and the more disordered structure towards the joint. From the positions of the strong (10) reflection of the 2D hexagonal pore lattice, a relative strain map of the whole particle can be constructed (d). Local strain differences up to 3% are present between the strut and the joint. e) shows a ptychographic image of the strut in b).

Figure 1 shows a preliminary result of a strain measurement on a single particle. Special care in the data evaluation has to be taken to correct for geometrical effects in the peak positions due to the single crystalline nature of the samples; this is still work in progress.

We could see changes of the strain maps with humidity, however, two problems occurred in the attempt to quantitatively evaluate these strains:

i) radiation damage in the samples increased due to the humid environment, and
ii) the samples moved due to the take-up of water.

From systematic tests concerning problem i) we estimated that structure changes due to radiation damage were negligible for measurement times ≤ 0.5 s, even for high sample humidity. A first effort to resolve problem ii) was to use the integrated intensity maps to quantify the translation and in-plane rotation of the particle due to water sorption. This turned out to be quite successful, as the outer particle shape could be roughly matched for different humidities. Still, the quantitative numbers remain doubtful, since movements or rotations out of the observation plane are not covered by this procedure. This problem can only be resolved when in addition also sample rotation with several discrete rotation angles for each humidity step is employed, which would allow to reconstruct the 3D translation and rotation matrix of the particle directly from the experiment.

Finally we have tried to obtain a ptychographic image of one of the struts by keeping the very same experimental setup as for nanodiffraction, simply moving the sample out of the nanobeam focus. Data were reconstructed using the software available at the ID13 beamline. Fig. 1e) demonstrates the principal feasibilty of such measurements. Unfortunately, there was no more time to to follwow this attempt for different humidities, as was originally planned. Also, the quality of the reconstructed image was not as good as expected and definitey not good enough to detect small changes in particle shape. Moreover, similar to above, quantitative information on particle deformation will only be possible, when 3D Ptychographic data are available. It turns out that combining the two methods (nanobeam diffraction and 2D ptychography) in an single in-situ experiment is currently by far too ambitious for our system. Two independently optimized experiments will at present certainly be more successful.