Electronic Supplementary Material (ESI) for RSC Advances.


# Influence of gas atmosphere and ceria on the stability of nanoporous gold studied by environmental electron microscopy and *in situ* ptychography

Sina Baier [1], Arne Wittstock [2], Christian D. Damsgaard [3,4], Ana Diaz [5], Juliane Reinhardt [6], Federico Benzi [1], Junjie Shi [4], Torsten Scherer [7,8], Di Wang [7,8], Christian Kübel [7,8], Christian G. Schroer [6,9] and Jan-Dierk Grunwaldt [1,10,*]

[1] *Institute for Chemical Technology and Polymer Chemistry, Karlsruhe Institute of Technology, 76131 Karlsruhe, Germany*

[2] *Institute of Applied and Physical Chemistry, University of Bremen, 28359 Bremen, Germany*

[3] *Center for Electron Nanoscopy, Technical University of Denmark, 2800 Kgs. Lyngby, Denmark*

[4] *Center for Individual Nanoparticle Functionality, Technical University of Denmark, 2800 Kgs. Lyngby, Denmark*

[5] *Paul Scherrer Institut, 5232 Villigen PSI, Switzerland*

[6] *Deutsches Elektronen-Synchrotron DESY, Notkestr. 85, 22607 Hamburg, Germany*

[7] *Institute of Nanotechnology, Karlsruhe Institute of Technology, 76021 Karlsruhe, Germany*

[8] *Karlsruhe Nano Micro Facility (KNMF), Karlsruhe Institute of Technology, 76344 Eggenstein-Leopoldshafen, Germany*

[9] *Department Physik, Universität Hamburg, Luruper Chaussee 149, 22761 Hamburg, Germany,*

[10] *Institute of Catalysis Research and Technology, Karlsruhe Institute of Technology, 76344 Eggenstein-Leopoldshafen, Germany*

*Electronic mail: grunwaldt@kit.edu, Tel.: +49 721 608-42120, Fax: +49 721 608-44820

This electronic support information gives additional information on the results obtained in the study of the annealing behavior of nanoporous gold.

Figure S1 presents the ligament size distributions before and after the study during *in situ* ptychography presented in Figure 6 and 8. The ligament diameter was obtained by manually determining the diameter between connecting knots in the ligaments [1]. For the evaluation before the annealing SEM images taken before the annealing experiment were used, whereas after annealing STEM images were used for evaluation. Ptychographic images could not be used, as the resolution of 20 nm does not give enough precision. Figure S1a) presents the distribution of the ligament diameter for np-Au, while the stabilized $CeO_2$/np-Au ligament diameter distribution is presented in Figure S1b). Both size distributions show similar diameters before (black bars) and after (red bars) the annealing, revealing that no coarsening occurred. Compared to the pure np-Au sample, the distribution was narrower for the $CeO_2$ stabilized sample and the ligaments were slightly smaller.

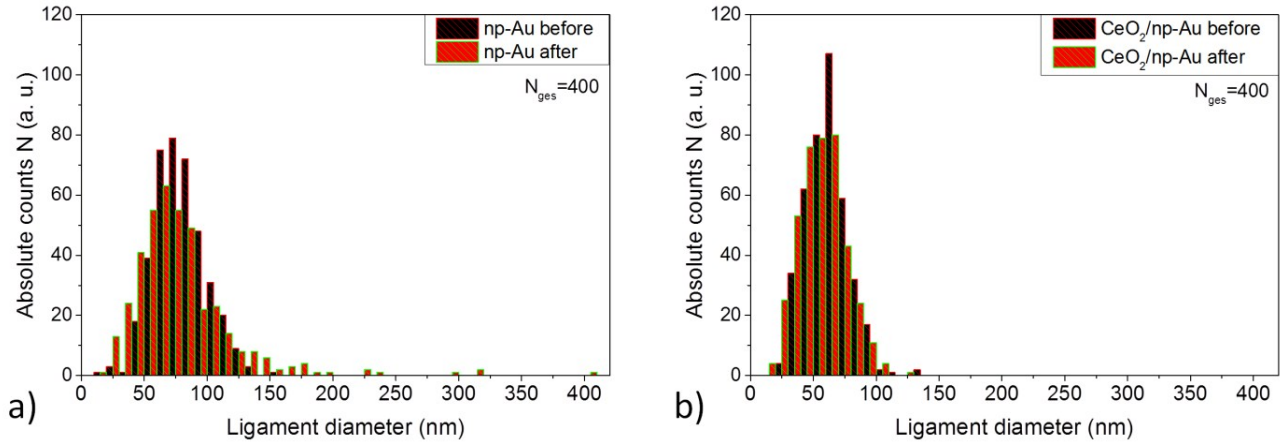


**Figure S1:** Ligament diameter distributions before and after the annealing treatment performed during *in situ* ptychography. The distribution gained for the np-Au sample is depicted in a), b) shows the distribution for the $CeO_2$/np-Au. The distribution before the annealing treatment is shown by black bars, whereas after the annealing treatment by red bars.

Magnified images of the bottom left part of the np-Au sample studied by *in situ* ptychography in Figure 6 are presented for 275°C-355°C in Figure S2. Comparably, magnified images taken from the top right corner of the $CeO_2$/np-Au sample in Figure 8 are presented in Figure S3. Like the

distribution of the ligament diameter both magnified image series reveal that no coarsening occurred. However, at 385°C (Figure S3), a slight material loss can be observed, as some areas become brighter.

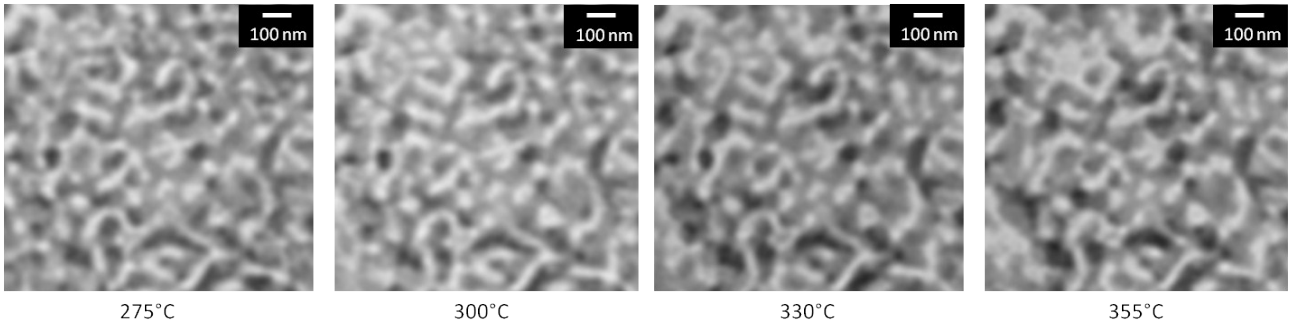


**Figure S2:** Magnified area of the bottom left part of the phase contrast images of np-Au at different temperatures presented in Figure 6. The magnified images reveal that no coarsening occurred.

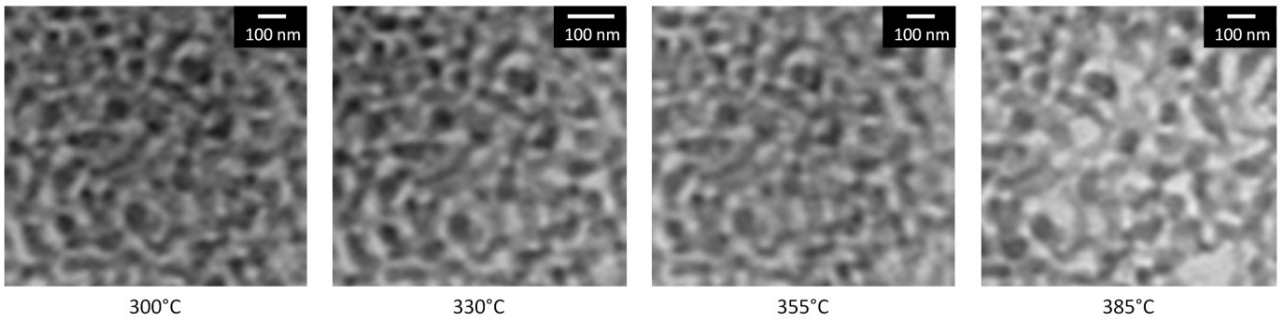


**Figure S3:** Magnified area of the top right part of the phase contrast images of $CeO_2$/np-Au at different presented in Figure 8. The magnified images reveal that no coarsening occurred, instead, at 385°C, a slight material loss becomes visible in the bottom right corner and the center of the studied area.

Figure S4 shows STEM-HAADF images and a background subtracted EELS spectrum in an area of the $CeO_2$/np-Au sample after it was studied by *in situ* ptychography. EELS analysis of the weak residue, which was left behind, when the material was lost reveals the presence of Si, Ce, slight amounts of C, N and O. Si and N can be attributed to the $Si_3N_4$ membrane below the sample and the small amount of C might be explained by impurities caused by the FIB process. Ce and O can be attributed to the $CeO_2$ particles used for stabilization.

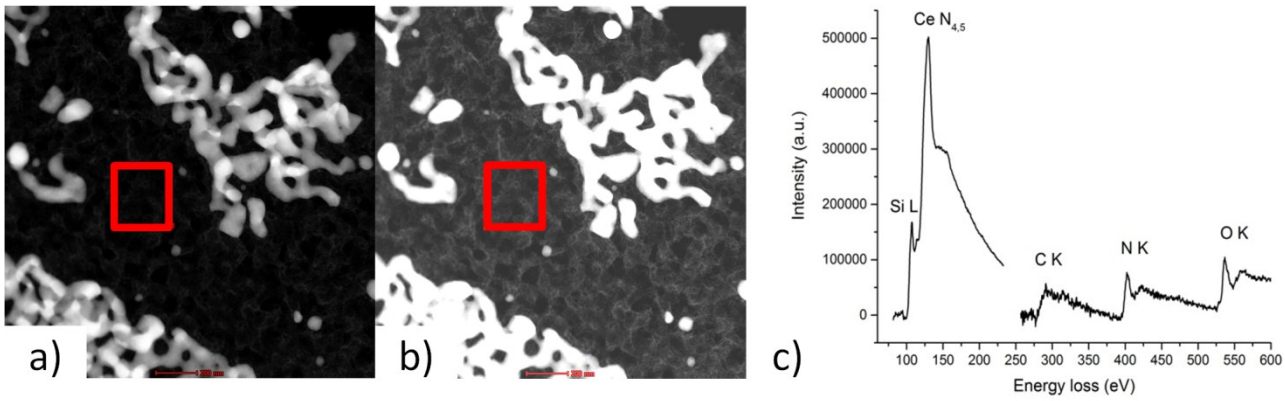


Figure S4: STEM-HAADF images of $CeO_2$/np-Au showing the area of EELS measurements (red box) and EELS spectrum performed after annealing during the *in situ* ptychography. a) STEM-HAADF image with normal contrast to show the position for the EELS measurement with respect to the remaining np-Au (bright material), b) STEM-HAADF image with increased contrast to visualize the remaining residue, c) Background subtracted EELS spectrum of the selected area marked by the red box in a) and b).

**References:**

[1] Z. Li, S. Zhou, H. Li, W. Si, Y. Ding, J. Nanosci. Nanotechnol., 2009, 9, 1651-1654.