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TEM Characterisation of Au Nanoclusters on SBA15

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Nanoclusters are used in a wide field of applications as building blocks in functional materials. In heterogeneous catalysis noble metal nanoclusters, such as Au, Pt, Rb and their intermetallic compounds are used on various oxide substrates for the conversion of matter. Au nanoclusters are used for converting CO to CO_2. The chemical nature of the substrate is of importance for the redox reaction, and SiO_2 as well as TiO_2 can be used. The cluster size is crucial to obtain a maximum catalytic rate. In order to avoid coalescence of the noble metal nanoclusters and to achieve a maximum surface for the gas reaction, we use an SBA15-analogue, a porous amorphous SiO_2 with periodically arranged mesopores as a model system for a substrate material. The pores act as cages for the clusters and allow reactive gas flow.

Complex characterisation of porous amorphous substrates for heterogeneous catalysis is essential for the development of novel efficient catalytic materials. Dependent of the growth process of the amorphous SiO_2, SBA15 grows in different micromorphologies: a) continuous framework structure, b) as platelets or c) in connected roundish particles. Electron tomography is used as a tool for characterisation of the pore arrangement in these three sample geometries. In addition, CTEM and HRTEM are used for conventional imaging in two dimensions.

The SiO_2 was prepared by sol-gel-processing using tetrakis(2-hydroxyethyl)orthosilicate as an ethylene glycol modified precursor and an amphiphilic molecule as the structure-directing agent. The mesoporous particles can be formed in a cooperative self-assembly process. The surfactant is then removed by subsequent annealing. The Au nanoclusters were deposited onto the SiO_2 by using tetrachlorogold-acid. The nanoclusters were characterised in two dimensions by HRTEM and HAADF. The results clarify the location of the Au nanoclusters inside of the mesoporous SiO_2 matrix.

Dependent on the morphology of the SBA15 material, framework structure, platelets or roundish particles, we characterised the arrangement of the pores concerning diameter, arrangement and connectivity. For later applications it is of major importance to see whether the pores are straight or curved and whether they are open on two sides in order to allow gas flow. The structure of the pores was characterised in two dimensions by CTEM, HAADF and in three dimensions by electron tomography. The results show the differences in the pore structure dependent of the location in the sample, i.e. in the framework structure the difference between arms and the crosslinks. The results further allow a comparison of the pore structure within the framework structure, the platelets and the roundish particles.

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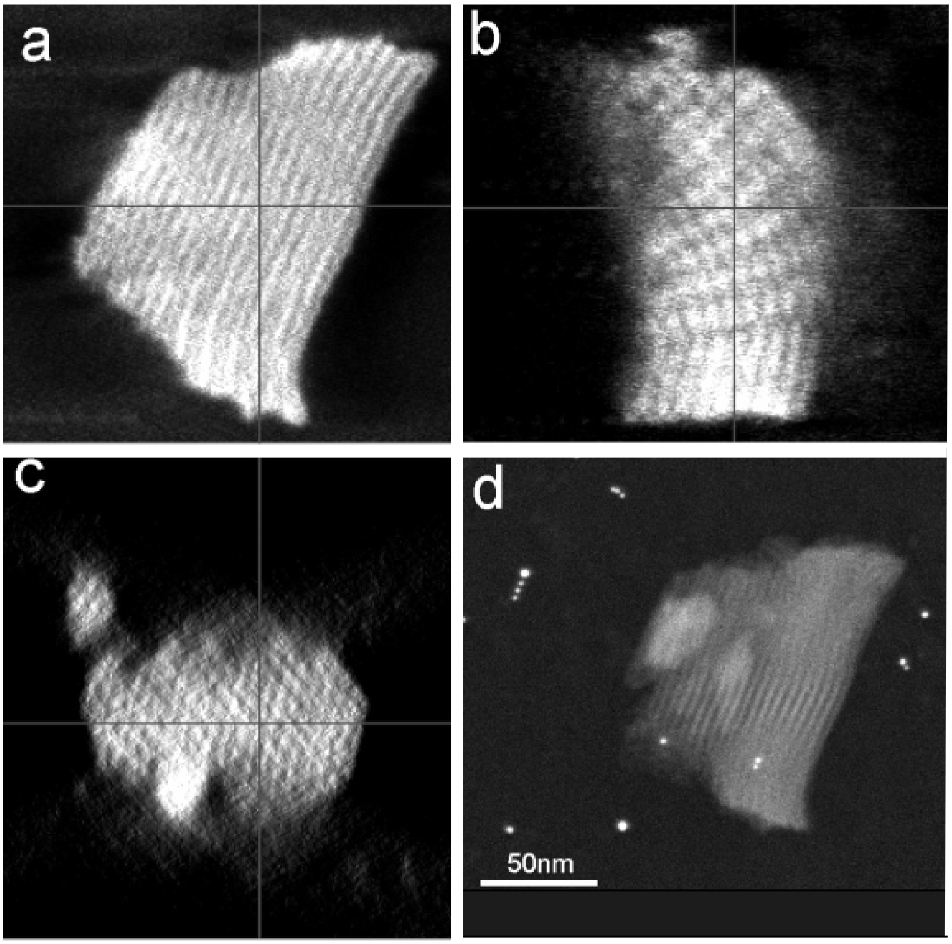

Fig. 1: Fig. a – c show section through a reconstructed particle in different projections. Fig. d shows the same particle as a HAADF micrograph. The bright spots are Au markers which are used for the reconstruction of the volume.

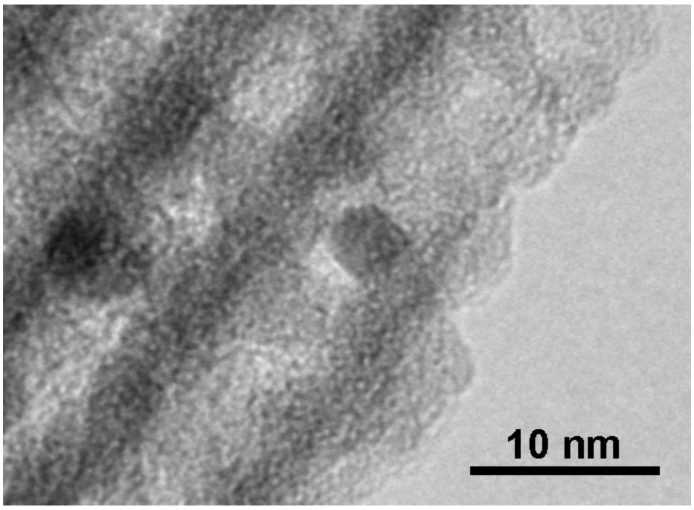

Fig. 2: HRTEM micrograph of a Au particle in mesoporous SiO_2. From interface analysis it can be concluded that this particle is located inside of the pore.