A.

ESRF	**Experiment title:** Structure study of nanodisperse Metal-metaloxide particles	**Experiment number:** HS-639
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Report:

Aim: The aim was to detect the internal structure of metal-nanoclusters by WAXS. These nanoclusters are produced by depositing di-block copolymer micelles containing metal-salts on different substrates and subsequent treatment with oxygen-plasma [1-4]. The size of the clusters can be adjusted uniformly from 1 nm to 20 nm and the distance from 20 nm to 150 nm depending on the parameters of micelles. The nanoclusters may show a new class of material and they will play an important part in homogeneous and heterogeneous catalyses. In this first attempt we studied Au-nanoclusters with different sizes deposited on glass, mica and silicon.

Set-up: We used grazing incidence (with an angle of incidence of about 0.05°), so the whole sample consisting of nanoclusters is illuminated, the scattering volume is enlarged and the scattering of the substrate is acceptable. It turned out that the substrate for these experiments must be glass, because mica or silicon are showing too high scattering. By carefully varying the angle of incidence we could detect mostly isotropic WAXS of the nanocrystals with the 2D-detector. The intensity along the reflection – circles was integrated.

Results: Most samples with Au-nanocrystals as prepared showed no or only a very weak isotropic WAXS scattering with broad Au reflections. After annealing at T = 700 °C (2h) for all samples sharper reflections like in bulk Au could be detected. (fcc, the lattice constant may be slightly larger as in bulk) (figure 1). The results are consistent with the findings by AFM. In figure2 the results of a sample with Au-nanoclusters before and after annealing are shown. The distance between the clusters is the same, but the size and the height (e.g. 15,7nm at T=20°C, 12,8nm at 600°C, 7,8nm at T=900°C) are smaller. Regarding the results gained by WAXS and AFM we conclude that before annealing each nanocluster consists of small Au nuclei (size about 1 to 3nm, depending of parameters of preparation). After annealing the Au-nuclei are growing, forming small Au-crystals (3nm to 6nm) resulting in observable X-ray reflections and in smaller size and height of the nanoclusters. This result is very important for the understanding of the efficiency of catalysts and should be investigated systematically also for real systems (Au/Al_2O_3, Au/TiO_2, Au/Fe_2O_3).

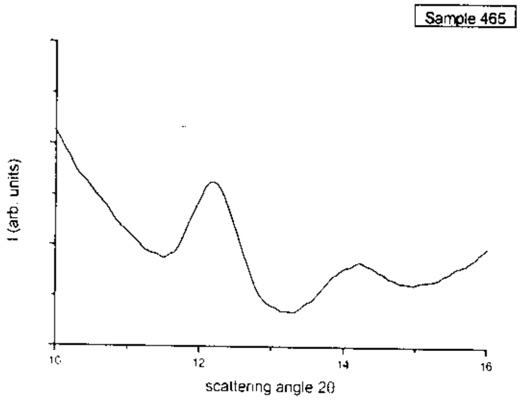

Figure 1a: Integrated WAXS-intensity of Au – nanoclusters before annealing

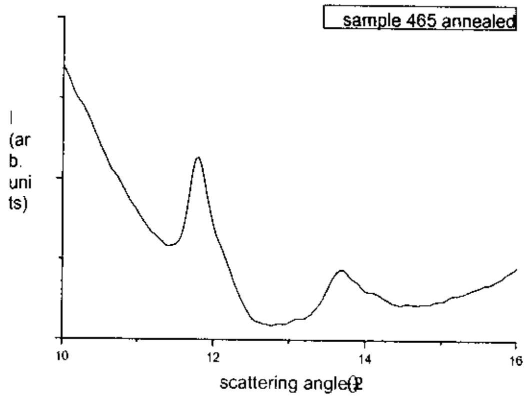

Figure 1b: Integrated WAXS-intensity of Au – nanoclusters after annealing at T =700°C (2h)

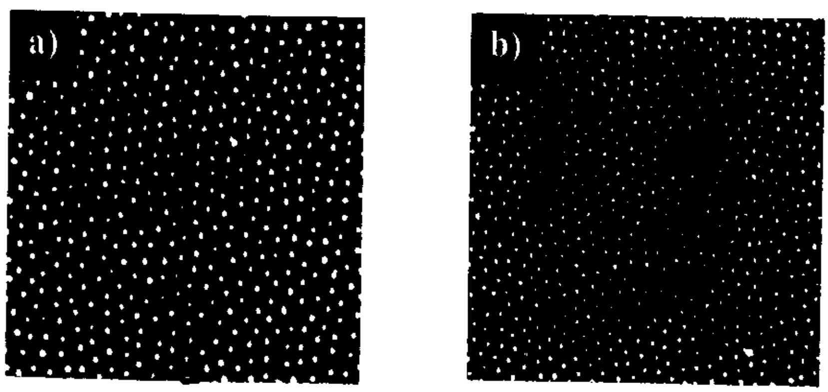

Figure 2: AFM pictures of Au-nanoclusters, (size of pictures 2µm x 2µm a)before annealing, b) after annealing T= 900°C (4h)

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