TRANSMISSION ELECTRON MICROSCOPY OF SILICA-SUPPORTED PT PARTICLES

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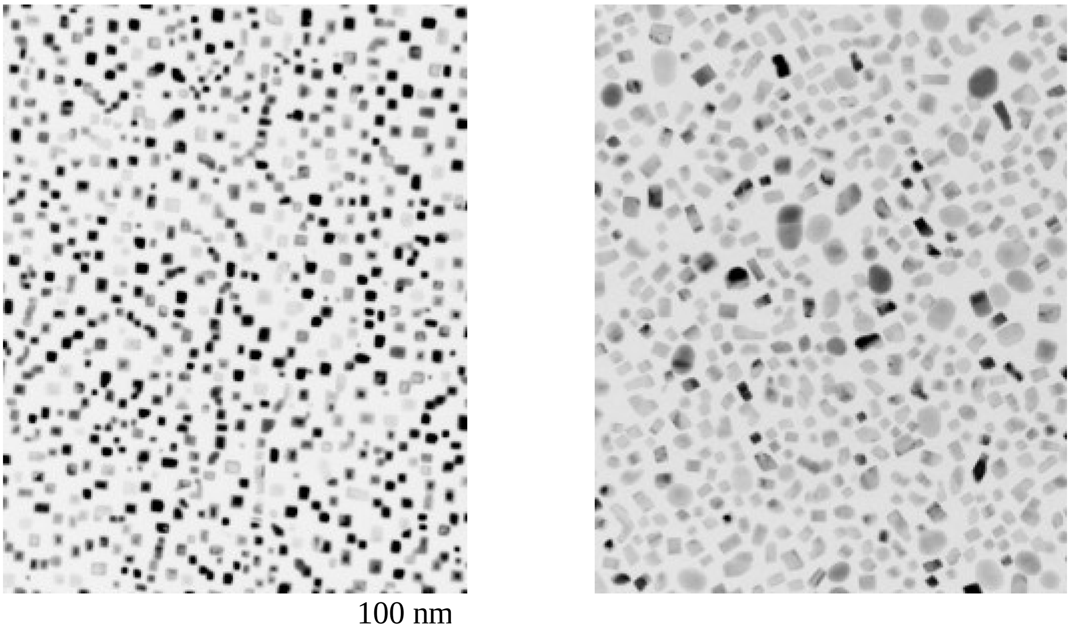
In heterogeneous catalysis studies, the catalyst surface needs to be well defined during the catalytic process. However, it is difficult to apply UHV compatible methods and electron microscopy to a „real" catalyst. Therefore, model systems are necessary to elucidate the mechanism of structure-sensitive reactions. Metal particles grown on planar thin supports, thus facilitating the characterisation by electron microscopy, were extensively studied as model systems for supported catalysts. In the present work, Pt particles epitaxially grown on (001) NaCl single crystals were covered with a supporting film of amorphous silica. Subsequently, the NaCl was dissolved in distilled water and the films were mounted on gold grids for electron microscopy [1]. After an oxidising treatment (O_2, 673K, 1h) the samples were exposed to H_2 at 873K for 1h. The morphology and structure of the samples were then examined by HRTEM and compared to the as-prepared state.

Fig. 1(a) is a low-magnification image of the as-prepared sample; Fig 1 (b) shows the reduced catalyst. Both images were taken with a Philips CM200 FEG electron microscope. Most of the as-prepared Pt particles exhibit a square or rectangular shape [1]. In contrast, after the reductive treatment, a considerable amount of Pt particles have undergone a change in either shape or orientation. Those with round edges also have increased in size, obviously due to the agglomeration of some metal particles. Further information about the structure modification can be deduced from the diffraction patterns of the sample before and after the reductive treatment, shown in Figs 2 (a) and (b). The sharp diffraction spots in Fig 2(a) reveal that, due to their orientation with respect to the original NaCl substrate, most of the as-prepared Pt particles are in their [001] zone, and exhibit parallel lattice planes. In contrast, the diffraction pattern of the sample after the reductive treatment shows series of rings together with some broadened, but distinct spots. Thus, some particles have changed in azimuthal orientation, although they are still in [001] zone. Apart from (200) and (220), also (111) and (311) spots belonging to a [110] zone can be detected which suggests that the base planes of some particles have changed during the treatment. Moreover, weaker spots are observed corresponding to (100) and (110) reflections (denoted in Fig. 2(b) by arrows), which are forbidden for an fcc structure. Also in the high resolution image a double periodicity of the lattice fringes (3.92 Å) is observed in many particles.

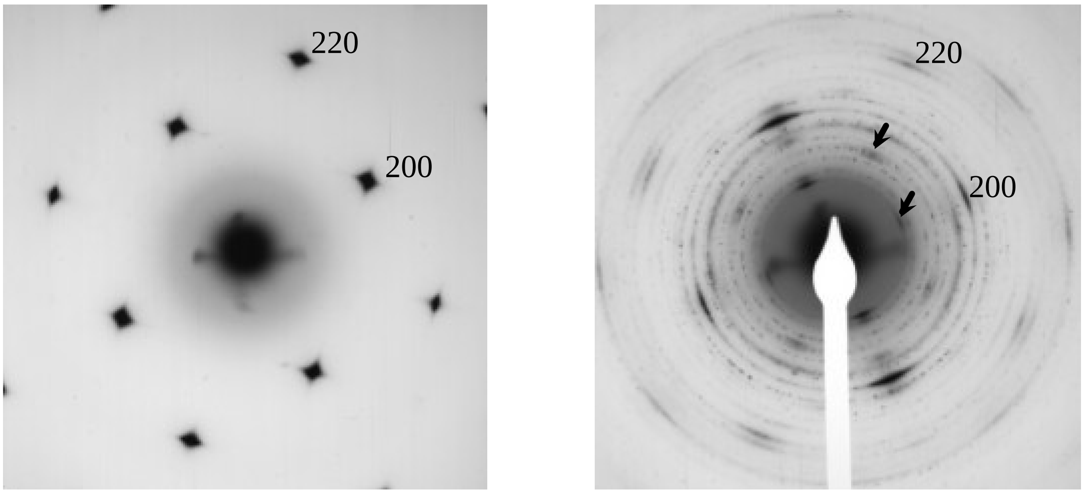
Hence, the shape and microstructure of the particles differ considerably before and after treatment. Changes in particle structure after reductive treatments under comparable conditions have been explained previously by the approach to a hydrogen-induced equilibrium shape [2], but also a platinum silicide formation has to be taken into account [3]. Since the diffraction pattern is not consistent with silicide formation an explanation in terms of a modulated superstructure is favoured in this case. The formation mechanism of this structure and its relevance for catalytic activity are still under investigation.

Figure 1. Low magnification image of Pt supported on SiO_2 (a) before and (b) after the treatment in H_2 at 873 K.



(a) (b)

Figure 2. Electron diffraction patterns of Pt supported on SiO_2 (a) before and (b) after the treatment in H_2 at 873 K.



(a) (b)

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