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Characterization of Catalytic Supports by Means of High-Resolution TEM and SEM in Combination with Shadow-Casting and Decoration Procedures

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In view of the well-known fact that the nature of a solid support can have a profound influence on the performance of a heterogeneous catalyst, it is of specific interest to understand the correlation between structural details of the support, and size and distribution of the supported metal particles as well as their catalytic activity [1].

With respect to better understanding of this correlation we applied electrochemical studies and high-resolution TEM and SEM investigations in combination with shadow casting and decoration procedures on Pt/C catalysts with various carbon supports. In order to study the influence of the surface structure on the preservation of the particle size and distribution, tungsten was evaporated under a flat angle on different types of carbon supports. A special electron impact evaporator developed by us allows the reproducible evaporation of tungsten [2]. This vacuum deposited tungsten layer formed a uniform film at a thickness of 0.2 - 0.3 nm and showed sufficiently contrast for imaging the finest surface structural details. The tungsten deposited catalysts were studied by means of HRTEM (Hitachi HF 2000) and HRSEM (Hitachi S-5200). Results are summarized in HRSEM micrographs of Fig. 1.

The surface roughness depends on the preparation of the carbon black supports. The Vulcan XC72 (Carbot Corp.) reveals a quite smooth surface whereas the Printex XE2 (Degussa) shows a rough texture. In order to proof the existence of differences in the surface roughness between the carbon blacks we carried out gold-decoration experiments. For this purpose the carbon blacks were evaporated with gold under temperature controlled conditions. The gold formed island structures in thickness of 0.3 - 1 nm. The size, density and distribution of gold cluster on the surface depends on the temperature and is highly influenced by the roughness of the surface. After evaporation procedure the carbon black surfaces are decorated by Au clusters as shown in Fig. 2.

The smallest Au cluster and the highest particle density were achieved in case of Printex XE2.

We observed that the surface roughness influences the particle size, distribution and preservation of carbon supported Pt nanoparticles. The HRTEM studies of the various carbon blacks before and after platinumoxide loading and after electrochemical reduction to platinum confirmed the results obtained by the roughness studies [3]: Under operation conditions undesired sintering of Pt nanoparticles in case of Printex XE2 support is much less pronounced if compared to the Vulcan XE72 support because Printex XE2 has a much rougher surface (Fig. 3).

In our investigations the Pt/Printex XE2 material proves to be the best electrocatalyst for the oxygen reduction reaction [3, 4].

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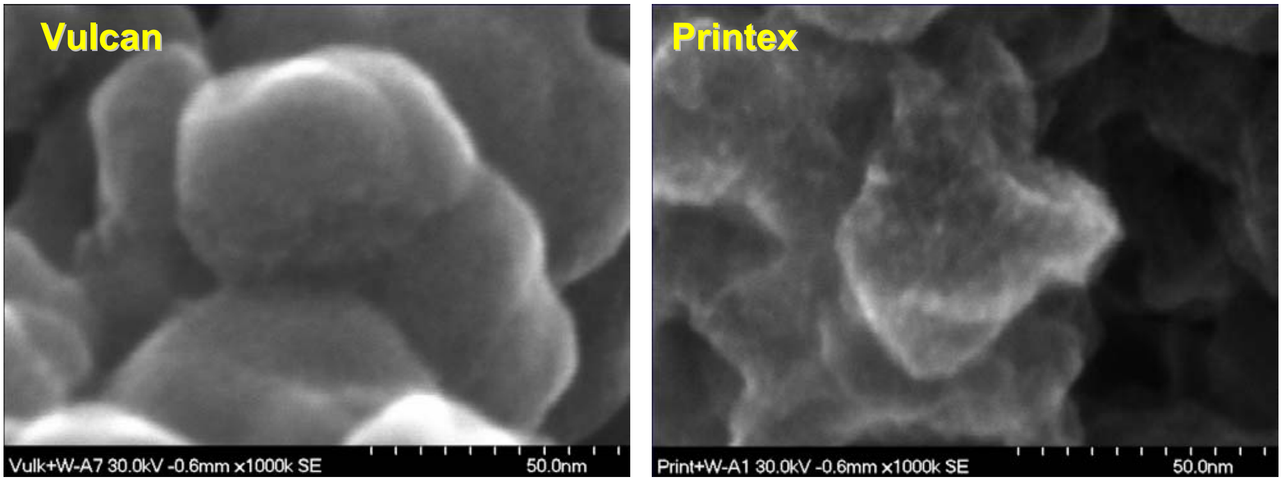

Fig. 1: HRSEM images from carbon supports Vulcan XC72 and Printex XE2 after schadow casting with tungsten.

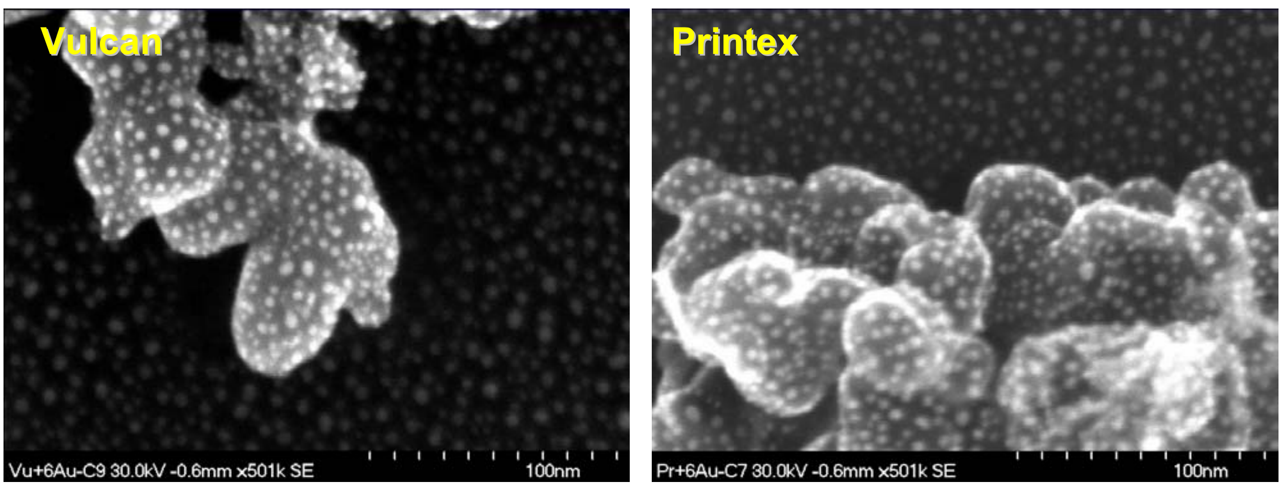

Fig. 2: HRSEM images from gold decorated carbon supports Vulcan XC72 and Printex XE2.

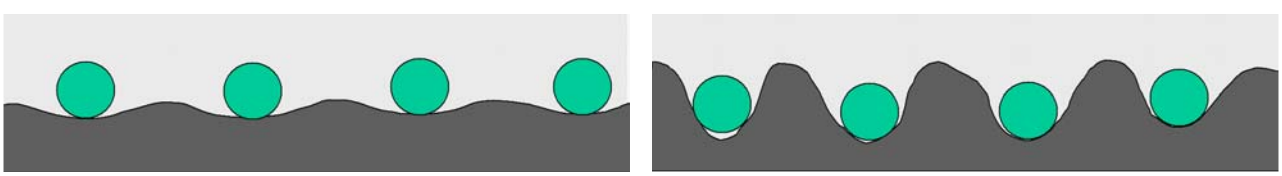

Fig. 3: Influence of smooth (left) and rough surface structure (right) on the fixation of particles (schematic).

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