ID-10-P-2183 Controlled TEM specimen preparation of supported nanoparticle catalysts for quantitative analysis

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Obtaining meaningful statistical information from supported nanoparticle catalysts using TEM can be a challenge. Figure 1 shows an SEM image of a Pt/Pd/alumina catalyst, revealing a complicated three-dimensional mesoporous structure and alumina particle sizes of several micrometers. The specimen preparation method most commonly used for these samples involves crushing the catalyst powder in a mortar, dispersing it in alcohol and depositing some droplets onto a support film, usually carbon. While this approach has its advantages due to simplicity and speed, the obtained specimens are usually very inhomogeneous, making it difficult to find representative areas for the investigation. Also, information about differences in nanoparticle distributions along the alumina particle is lost when crushing the powder.

In this work, specimens have been prepared using the lift-out technique in a combined scanning electron / focused ion beam microscope. Prior to this, the catalyst powder has been embedded in an acrylic resin. The specimens obtained by this method contain regions which originally were inside of the alumina particle, as well as regions of the outer layer of the particle.

Figure 2 shows a STEM image of the outer edge of an alumina particle, revealing the existence of a near surface layer with a higher Pt/Pd nanoparticle density compared to the inner region, especially when looking at nanoparticles larger than 1 nm. Separate particle size distributions for these two regions have been obtained. Additionally, large nanoparticles with diameters above 10 nm are exclusively seen very close to the outer edge. This information cannot easily be extracted using the traditional crushing method.

Acknowledgement: We acknowledge financial support by the Swedish Energy Agency, AB Volvo, Volvo Car Corp, AB, Scania CV AB, Haldor Topsøe A/S, ECAPS AB, K. & A. Wallenberg FDN and Vetenskapsrådet.
Fig. 1: Secondary electron SEM image of a Pt/Pd/alumina catalyst particle. The alumina support has a complicated mesoporous structure and is several micrometers in diameter, making TEM specimen preparation challenging.

Fig. 2: HAADF STEM image of a Pt/Pd/alumina catalyst prepared by the lift-out technique, showing the outer edge of an alumina particle. Pt/Pd nanoparticles are seen as bright features. A higher density of nanoparticles with diameters above 1 nm can be seen close to the lower edge.