Investigation of Supported Catalyst Nanoparticles by Transmission Electron Microscopy

<u>A. Pfrang</u>¹, A. Pitois¹, G. Tsotridis¹, G.J.M. Janssen², E.F. Sitters² and J.A.Z. Pieterse²

1. European Commission, Joint Research Centre, Institute for Energy, Cleaner Energy Unit, PO Box 2, 1755 ZG Petten, The Netherlands

2. Department of Hydrogen and Clean Fossil Fuels, Energy research Centre of the Netherlands ECN, P.O. Box 1, 1755 ZG Petten, The Netherlands

andreas.pfrang@ec.europa.eu

Keywords: Catalyst, Energy filtered TEM, Proton-exchange-membrane fuel cell, Steam reforming

Catalyst nanoparticles are of major interest in various fields of technical applications. Transmission Electron Microscopy (TEM) is a suitable technique for the investigation of nanoparticles as TEM does not only allow the visualization of these particles but also their characterization in terms of element composition or lattice structure. For some of the technical applications restriction of the degradation of the catalyst is one of the most relevant issues for a further improvement of the respective processes. In this study, TEM has been used to characterize supported catalyst nanoparticles before and after use in two types of applications.

Firstly, carbon supported Pt nanoparticles in proton-exchange-membrane fuel cell (PEMFC) were investigated. It has become clear in the last years that the PEMFC durability should be significantly improved before its large-scale introduction. One of the major reasons for fuel cell degradation is the degradation of Pt catalyst particles in the electrodes, an effect which can be investigated by TEM [1]. When using hydrogen produced from fossil sources, the tolerance of the catalyst to CO poisoning is an important issue. Figure 1 shows TEM bright field images of a carbon supported Pt catalyst before and after use together with the corresponding particle diameter distributions. A clear increase in average diameter from 2.9 nm to 5.4 nm after cycling was observed.

Secondly, Ni catalysts on $MgAl_2O_4$ support for steam reforming in membrane reactors were investigated. Hydrogen membrane reactors are being studied for power production with pre-combustion decarbonisation. The membrane reactor produces hydrogen from natural gas at a low pressure for electricity production and a CO_2 rich stream at high pressure for storage. Cost-effective catalysts for these reactors are based on supported nickel. Figure 2 shows a TEM bright field image of such a Ni catalyst before use and a corresponding Ni map which was acquired using energy filtered TEM. Using this combination, the Ni particles can be identified. This allows a further investigation of the influence of the reforming process parameters and of the type of preparation of the catalyst and the preparation parameters on the stability of the catalyst.

1. G.J.M. Janssen et al., J. Power Sources (2009), in press, doi:10.1016/j.jpowsour.2009.02.027.



Figure 1. Pt catalyst particles on carbon support (Hispec 9100), a) Before use, average diameter 2.9 nm, b) Catalyst particles from used fuel cell cathode (30000 cycles between 0.7 and 0.9 V at 80 °C), average diameter 5.4 nm.





Figure 2. Nickel steam-reforming catalyst on $MgAl_2O_4$ support, a) TEM bright field image, b) Ni map of the same location, energy filtered TEM image, three-window method, Ni L_{23} edge.