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A. Pfrang

A. Pitois

G. Tsotridis

G.J.M. Janssen

E.F. Sitters

J.A.Z. Pieterse

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A. Pfrang[1], A. Pitois[1], G. Tsotridis[1], G.J.M. Janssen[2], E.F. Sitters[2] and J.A.Z. Pieterse[2]

[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

Transmission Electron Microscopy (TEM)

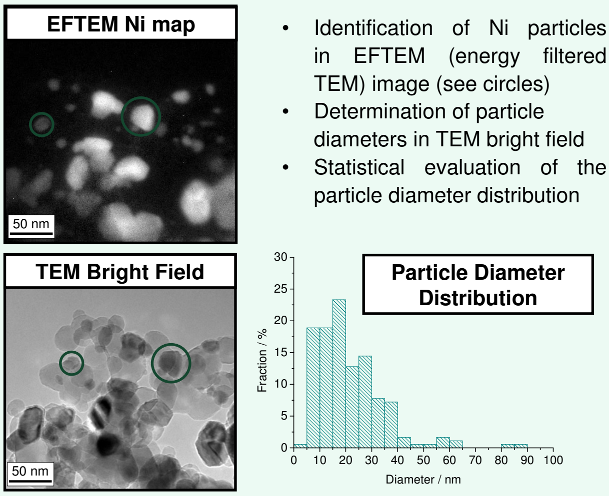
Transmission Electron Microscopy does not only allow the visualization of catalyst nanoparticles but also their characterization in terms of elemental composition and particle diameter distribution.

Experimental:
Standard copper grids were dipped into a dispersion of the supported nanoparticles in ethanol and then dried in air. A FEI Tecnai 20 TEM equipped with a LaB_6 filament and a Gatan GIF200 energy filter was operated at 200 kV.

Ni catalysts for steam reforming of methane

Hydrogen membrane reactors are being studied for power production from natural gas with pre-combustion carbon capture. To assess the potential of Ni as catalyst material, Ni was deposited onto $MgAl_2O_4$ by impregnation of nitrate salts and by homogenous deposition precipitation (HDP) and Ni particle diameter distributions were determined.

- Identification of Ni particles in EFTEM (energy filtered TEM) image (see circles)
- Determination of particle diameters in TEM bright field
- Statistical evaluation of the particle diameter distribution



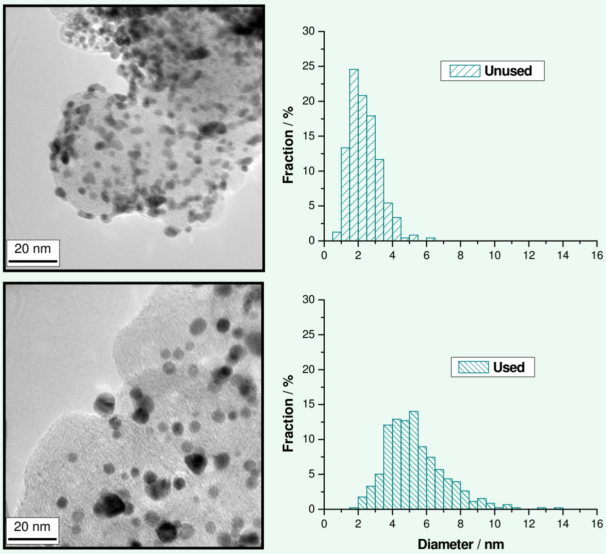
TEM results of a nickel steam-reforming catalyst prepared by impregnation on $MgAl_2O_4$ support; EFTEM: three-window method, Ni L_{23} edge; average diameter of Ni particles 21.4 nm.

Summary

- EFTEM in combination with TEM bright field allows the discrimination of Ni catalyst from the $MgAl_2O_4$ support for both preparation methods and the determination of the catalyst particle diameter distribution

Pt-based catalysts for PEM fuel cells [1, 2]

Carbon supported Pt-based catalysts are used as standard in proton-exchange-membrane fuel cells (PEMFC) and one of the major reasons for fuel cell performance decay is the degradation of Pt catalyst particles. A commercially available catalyst (Hispec 9100) was investigated as received and after cycling in PEMFC cathodes.



Pt catalyst particles on carbon support (Hispec 9100), top: before use, average diameter 2.4 nm, bottom: catalyst particles from used fuel cell cathode (30000 cycles between 0.7 and 0.9 V at 80 °C), average diameter 5.4 nm.

Summary

- Visualization of catalyst particles allows investigation of catalyst degradation and its quantification by determining changes in particle diameter distribution
- Degradation characterized by a higher average particle diameter as well as a broader distribution of particle diameters

References

[1] G.J.M. Janssen, E.F. Sitters, A. Pfrang, Proton-exchange-membrane fuel cells durability evaluated by load-on/off cycling, Journal of Power Sources 2009, 191(2), 501-509

[2] A. Pitois, J.C. Davies, A. Pilenga, A. Pfrang, G. Tsotridis, Kinetic study of CO desorption from PtRu/C PEM fuel cell anodes: Temperature dependence and associated microstructural transformations, Journal of Catalysis 2009, 265, 199-208.

Contact

Dr. Andreas Pfrang
European Commission • Joint Research Centre
Institute for Energy • Cleaner Energy Unit
Tel. +31 224 56-5047 • Fax +31 224 56-5623
E-mail: andreas.pfrang@ec.europa.eu

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