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ETEM characterization of NiGa model catalysts for CO₂ hydrogenation to Methanol

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Metal nanoparticles (NPs) dispersed on a high surface area support are widely used as catalysts for heterogeneous catalysis. The atomic configuration of the active NPs is highly linked to the performance of the catalyst. An Environmental Transmission Electron Microscope (ETEM) equipped with a differential pumping system to confine a controlled gas flow around the specimen offers a unique tool to investigate individual NPs at the atomic level in a gaseous environment. However, the morphology of the high surface area support often complicates the interpretation of TEM micrographs on the atomic level and blurs the spectroscopic information due to limited depth of field multiple scattering events. One way to circumvent this is to synthesize NPs on a low surface area support representing the "real" high surface area supported catalyst.

 $\delta\text{-Ni}_5Ga_3$ catalysts prepared by incipient wetness impregnation on a high surface area SiO_2 support (Figure 1A), have shown promising for CO_2 hydrogenation to methanol with comparable turn-over frequencies to the preferred commercial $Cu/Zn/Al_2O_3$ catalyst system[1-2]. This study presents TEM investigations of NiGa NPs supported on 200 nm SiO_2 nano-spheres (Figure 1B) and 20 nm thick SiO_2 membranes (Figure 1C)[4], respectively.

By studying the morphology (surface structure, facets, NP size, crystal structure, material composition) of these model catalysts during synthesis and CO₂ hydrogenation to methanol, we aim to describe the catalyst formation process and the pre-dominant deactivation mechanism at the atomic level. Complementary observations have been acquired using *in-situ* X-Ray Diffraction (XRD) and catalytic activity measurements using a Gas Chromatograph (GC) or Mass Spectrometer (MS).

The nano-sphere supported model catalyst features numerous NPs that can be illuminated directly with the electron beam. This enables atomic resolved structural (HRTEM) and spectroscopic information (EELS) of the individual NPs and was used to characterize phase, and surface structure during CO_2 hydrogenation.

The membrane supported model catalyst features numerous NPs with similar size distribution as the high surface area supported catalyst. This enables investigations of changes in size distribution, facet formation, and NP orientation (HRTEM) during synthesis and CO_2 hydrogenation.

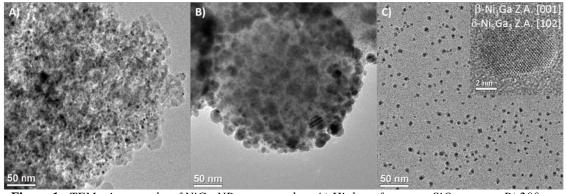


Figure 1. TEM micrographs of NiGa NPs supported on A) High surface area SiO₂ support B) 200 nm SiO₂ spheres, and C) SiO₂ membrane.

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