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Synthesis and Activation of Catalysts for Biofuel Synthesis in an Environmental Transmission Electron Microscope

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The synthesis of transportation fuels from sustainable resources requires new and better production paths. Our approach is to use biogas to synthesize alcohols, such as methanol or higher alcohols for fuel and other chemical products. For the production of methanol a reduction of processing temperature and pressure to lower the process cost and make the product more competitive is desired. Higher alcohols are in general favorable over methanol due to their high energy density and ease of use in current internal combustion engines. However, better catalysts for this reaction are needed to increase the poor yield of higher alcohols in present production routes [1].

The Catalysis for Sustainable Energy Project (CASE) at the Technical University of Denmark aims at discovering new and improved catalysts based on density functional theory (DFT) and testing the chemical reactivity of the most promising candidates experimentally. Transmission electron microscopy (TEM) is used for microstructural characterization and provides feedback for both theory and synthesis.

We have studied the catalysts close to their working conditions in an environmental transmission electron microscope (ETEM) equipped with a differential pumping system to confine a controlled gas flow around the specimen, allowing observation in a gaseous environment. Using heating holders, dynamic information about catalysts in their working state can be gained using a variety of TEM techniques *in situ*. [2,3]. Here, we present recent ETEM studies of CuNi and NiGa catalysts for alcohol synthesis using High-Resolution TEM (HRTEM), energy electron-loss spectroscopy (EELS), Energy-Dispersive X-ray Spectroscopy (EDX). Complementary observations have been done using *in-situ* X-Ray Diffraction (XRD). We focus on structural changes during the catalysts synthesis and activation in a reducing atmosphere at elevated temperature. Changes in phase and particle size distribution with respect to the temperature can be directly observed and correlated to catalytic activity and integral phase information from the *in-situ* XRD.

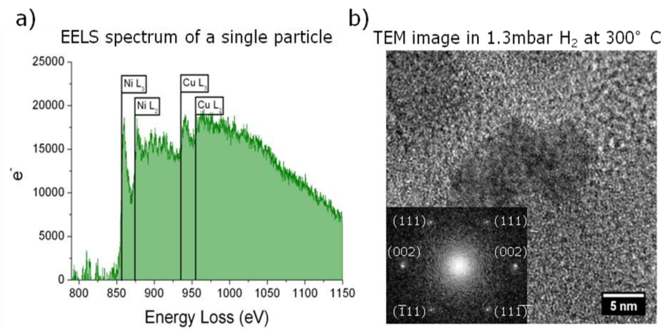

Figure 1: a) EELS spectrum and b) HRTEM image with FFT of a single CuNi particle taken at 300°C in 1.3mbar H_2 atmosphere.

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