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Publication date:
2012

In situ investigation of catalysts for alcohol synthesis

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The need for studying catalyst under realistic conditions is emphasized both by academic and industrial research. Acquiring highly resolved local information from materials under realistic environments by means of Transmission Electron Microscopy (TEM) has been found to be essential in connecting microscopic and macroscopic properties of materials, e.g. relating catalytic performance with crystal structure and morphology.

This study presents extensive characterization of NiGa and CuNi alloys during catalyst formation, alcohol synthesis, and accelerated aging experiments [1,2]. The characterization platform consists of three complimentary in situ techniques: (1) Activity measurements based on a reactor connected to a gas chromatograph (GC), (2) In situ x-ray diffractometer (XRD) measurements based on a reactor cell connected to a mass spectrometer (MS), and (3) environmental TEM (ETEM) that allows for observation in a gaseous environment. By using heating holders, dynamic information about catalysts in their working state can be gained using a variety of TEM techniques [3,4].

The presented platform successfully illustrates the capability of correlating the dynamic changes in structural phase and particle size distribution, measured both macroscopically (XRD) and microscopically (ETEM), with the catalytic activity.

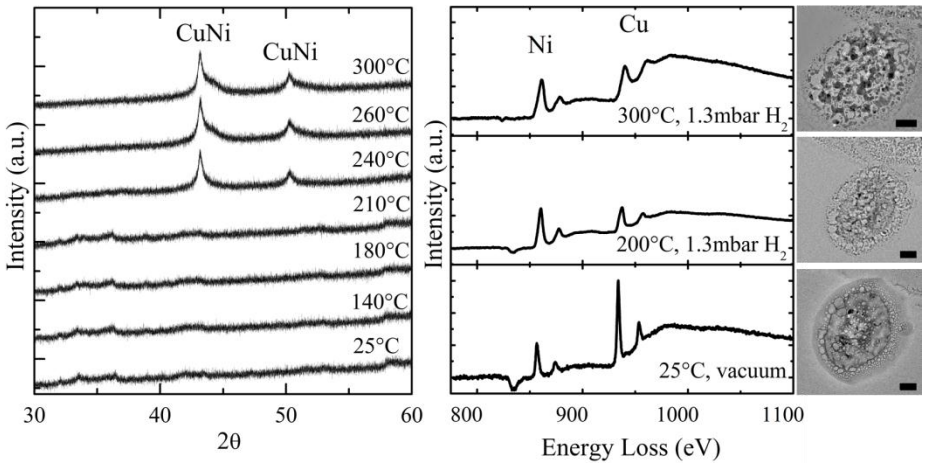

Fig. 1: In-situ XRD and TEM investigation of the synthesis of a CuNi catalyst out of their nitrate salts. The XRD peaks show the formation of CuNi alloy while the EELS spectra show the reduction of the CuO to Cu as the Cu onset peak becomes weaker. The bright field images show the nanoparticle formation (scale bars are 100nm).

[1] I. Sharafutdinov et al., Technical University of Denmark (to be published).
[2] Q. Wu et al., Technical University of Denmark (to be published).
[3] A.K. Datye, Journal of Catalysis **216**, 144-154 (2003).
[4] T.W. Hansen et al., Science **294**, 1508-1510 (2001).