$\overline{\mathrm{ESRF}}$	Experiment title: The State of Nickel as Promoter in Fischer-Tropsch Catalysts	Experiment number: CH-3595	
Beamline: BM01b	Date of experiment:from: October 31, 2012to: November 6, 2012	Date of report: November 27, 2012	
Shifts: 18	Local contact(s): Wouter van Beek	Received at ESRF:	
Names and affiliations of applicants (* indicates experimentalists):			

Magnus Rønning	Department of Chemical Engineering, NTNU
Georg Voss	Department of Chemical Engineering, NTNU
Nikolaos Tsakoumis	Department of Chemical Engineering, NTNU
Kristin Høydalsvik	Department of Physics, NTNU
Anita Reksten	Department of Material Science, NTNU

Report:

This study focused on the state of promotors in Fischer-Tropsch catalysts. Especially at reaction conditions, the nature and location of the promoter itself is unclear which leads to the utilization of in situ techniques. The experiment involves collection of XAS data on different Co-Ni catalysts during typical Fischer-Tropsch conditions in order to identify the state of Ni in such.

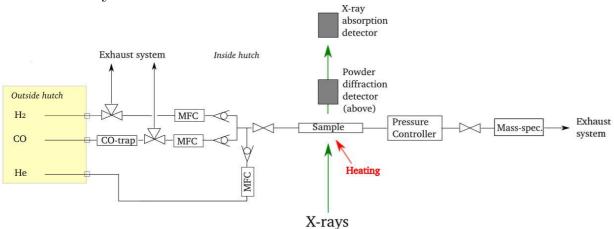


Figure 1: Experimental setup scheme.

During the experiment Co-Ni catalysts supported on γ - and α - alumina have been investigated. Our setup (Figure 1) enabled us to follow the catalyst reduction and subsequent reaction *in situ* at typical FT conditions (reduction at 400 °C and reaction at 210 °C and 18 bar) and realistic synthesis gas composition.

High CO conversion is essential in order to obtain a realistic partial pressure of steam. The Co and Ni K-edges (7709 eV and 8333 eV, respectively) were studied by XAS. HR-XRPD was performed at $\lambda = 0.5$ Å in order to minimise interference with the K-edges. For the in situ measurements a quartz capillary reactor heated by an air blower was used together with a gas distribution system and a mass spectrometer (MS) for product monitoring. The catalysts passed through the following procedures: reduction, pressurisation, reaction for 24 h, accelerated deactivation.

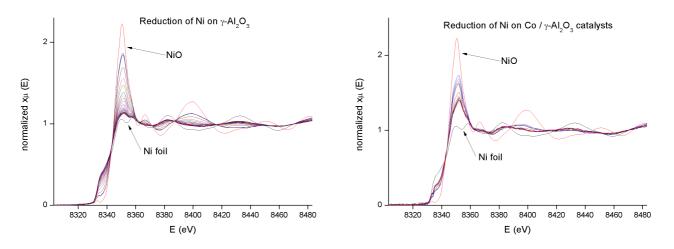


Figure 2: In situ XANES for the reduction of Ni on γ -alumina catalysts.

In Figure 2 in situ XANES of two catalyst show preliminary results of the measurements taken during the allocated beamtime. Briefly, the results show the difference in reduction of Ni on γ -alumina support with and without the catalytic active metal (Co). In the first case Ni is clearly reducable. In the second case, the catalyst was impregnated with Co, and Ni has no longer direct access to the surface. Both edges, for Co and Ni, where measured and the reduction of Co (not shown here) was followed. As can be seen, the reduction of Co seems to affect Ni somehow. Further analysis of the data while determine more information about the interaction. The study was extended to α -alumina as support material and various loadings of the metals.

Comments:

The study aimed also at investigating some catalysts at real Fischer-Tropsch conditions. Due to malfunction of the heatblower, several measurements where taken at wrong temperaturs during the beamtime and are dissmisable. In addition, due to trouble at the beamstation BM01a the ESRF beamline technicians closed the frontend without notification, which resulted in loss of beamtime. Finally, there have been some problems at the refill, which also reduced the active beamtime.