Beamtime Report
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From the application deadline (April 2016) to the actual beamtime in (January 2017) we changed our focus from ammonia synthesis to hydrogen production from the decomposition of ammonia. CoRe-systems supported on silica aerogels were investigated and compared with Co supported on silica aerogel. *In situ* XAS data was successfully collected at the Co K- and the Re L_{III}-edge at BM31 January 2017. The samples were reduced in 75% H_2 in Ar at 600°C and thereafter the hydrogen production properties were investigated with the decomposition of 5% ammonia in helium between 200°C - 700°C. The exhaust from the in situ cell was monitored using the mass spec available at the SNBL.

The different XANES endpoints after one hour in 75% H_2 in Ar are shown in Figure 1 and compared with Co foil and a bimetallic reference. From the pre-edge feature and the E_0 it was found that the supported CoRe-system is in a bimetallic state, and the reducibility of the supported CoRe-system is better than the supported Co.

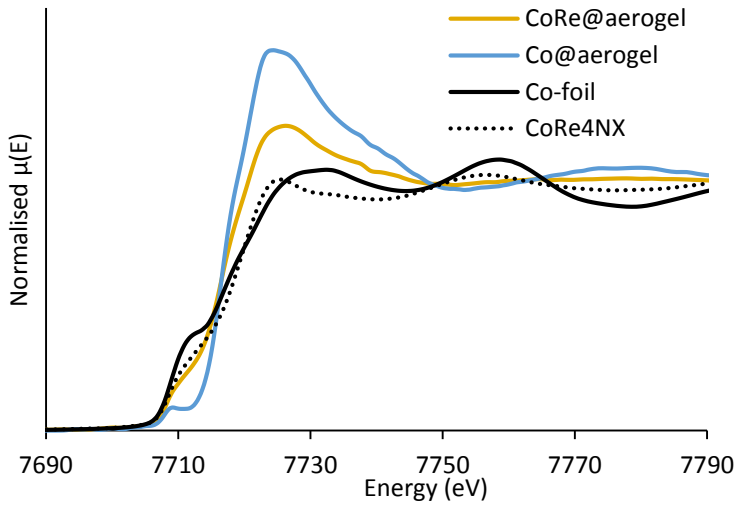

Figure 1. XANES of the reduced supported Co and CoRe-systems at aerogel. Compared with Co-foil and a CoRe-bimetallic reference.

It was found that the supported Co- and the supported CoRe-system starts to reduce at the same temperature, but from 400°C the reduction of Co@aerogel stagnates. The CoRe@aerogel has a better reducibility and continues its reduction. The reduction profiles are shown in figure 2.

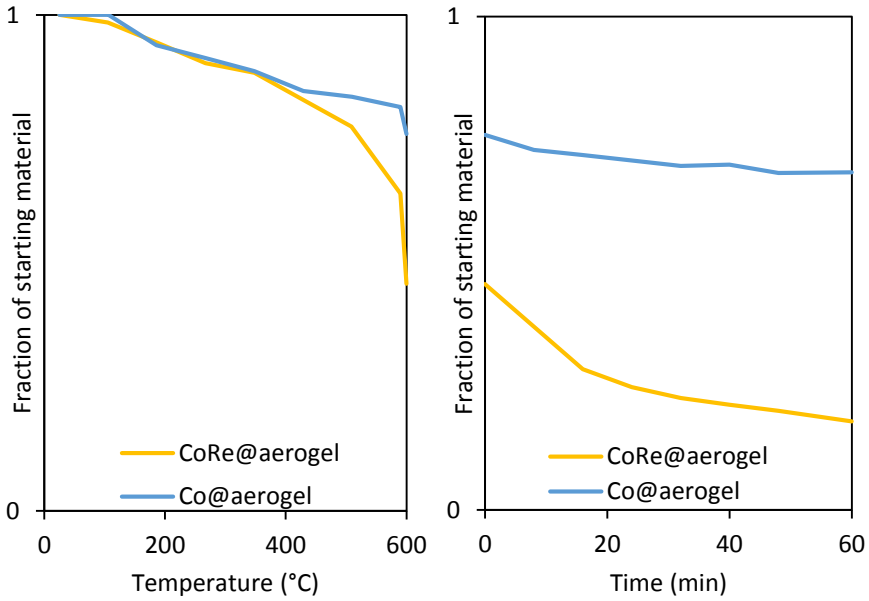

Figure 2. Reducibility of supported Co and CoRe at aerogel.

Under flow of 5% NH_3 in He the supported CoRe-system partially re-oxidises between 200°C - 400°C and possibly forming a nitride phase in the reducing atmosphere. From 400°C - 700°C the supported CoRe-system is again partially reduced.

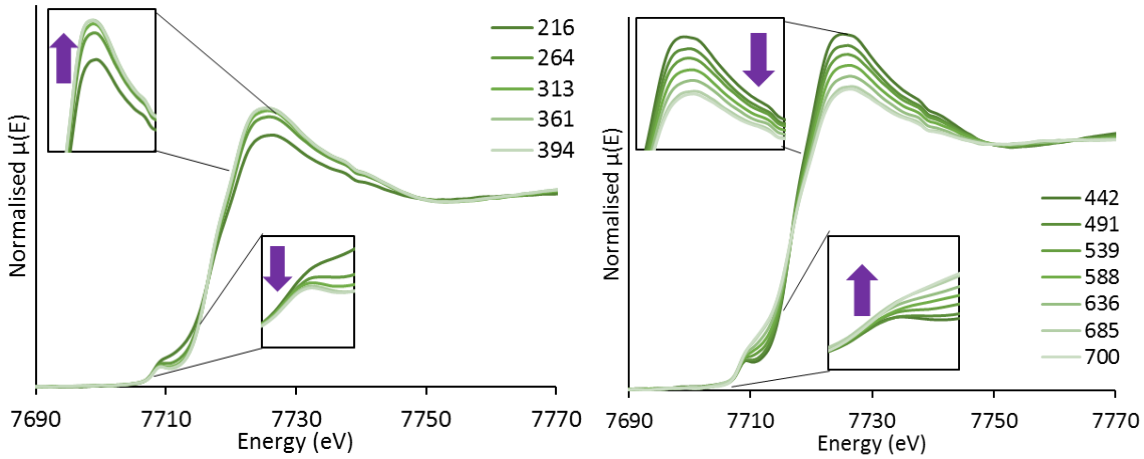

Figure 3. XANES from Co K-edge during decomposition of ammonia. Left: From 200°C - 400°C. Right 400°C -700°C.

The performance of the supported CoRe-system is better than the supported Co. The CoRe-system has an earlier onset temperature and has a higher conversion than supported cobalt itself as seen in Figure 4, showing results from online MS measurements during *in situ* XAS data collection.

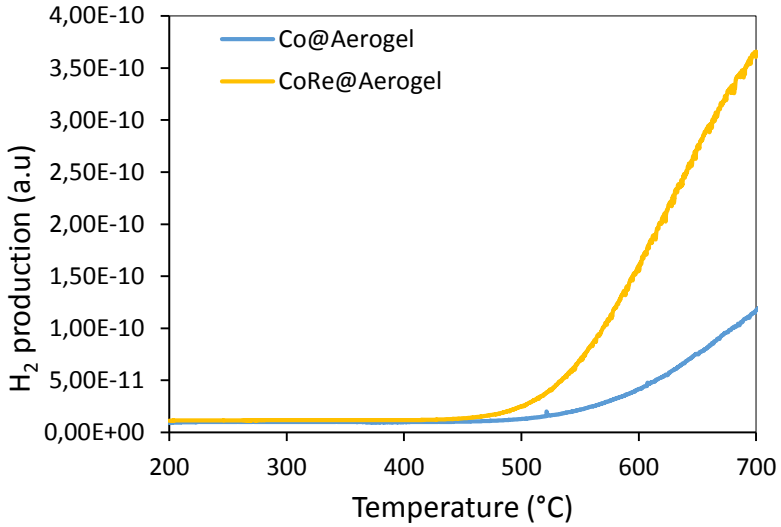

Figure 4. Hydrogen production for the supported Co and the supported CoRe system.