ESRF	**Experiment title:** Mixed-metal materials: The solution for safe hydrogen storage?	**Experiment number**: CH 6439
Beamline: BM30	**Date of experiment**: from: 25.09.2022 to: 03.10.2022	**Date of report**: 28.02.2022
Shifts: 15	**Local contact(s)**: Antonio Aguilar, Abdallah Nassereddine	*Received at ESRF:*

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Report:

In the carried-out experiments, different iron oxide-based materials were investigated using XAFS during their oxidation and reduction processes in the hydrogen storage reaction. Our group provided the catalysis setup (capillary holder, quarzt-tube reactor, gas-dosing system), the furnace was provided by the ESRF (see. Fig. 1). Thanks to the support of our local contact, we were able to build a perfectly fitting holding system for the furnace, which allowed the combination of our catalysis setup with the heat blower.

In course of one cycle, the material is reduced at 600°C in a hydrogen flow producing water. After flushing of the atmosphere with argon, in a second step, vaporized water was lead through the catalyst, oxidizing the reduced iron species under formation of hydrogen. Every material was followed through two cycles using XANES spectra to follow the changes of the oxidation state and EXAFS spectra to investigate the final states (initial sample, reduced and oxidized species). The reaction was followed using water sensors in front and after the capillary reactor.

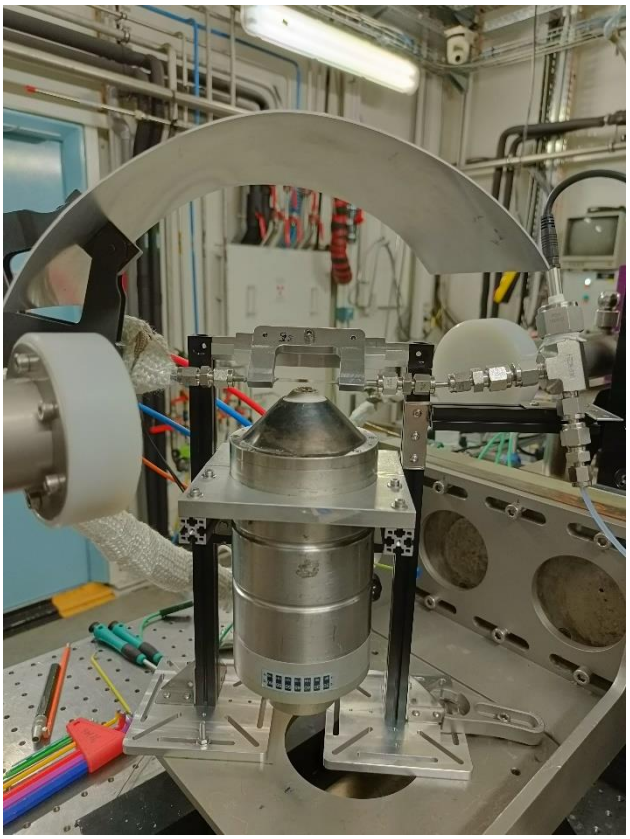

Figure 1: Furnace provided by ESRF used for heating the quarzt-tube reactor with the sample.

In the beginning of the *in-situ* measurements some technical issues occurred (problems with the synchrotron beam and with our setup), which led to the necessity to remeasure the first sample. After solving these problems, four samples could be measured during two cycles on the Fe K-edge. Due to step scan mode of the monochromator it was not possible to measure the proposed amount of *in-situ* samples. Since the capillaries have been prepared in advance, the measurements could be carried out continiously.

Ex-situ samples of the as-prepared storage materials and metal oxide references have been measured in transition mode as pellets without any issues. During this beamtime we focussed on iron-based samples with molybdenum as additive, so we could be sure to measure the whole set of molybdenum samples. The Fe/Mn and Fe/Cr should be measured at upcoming beamtimes.

In

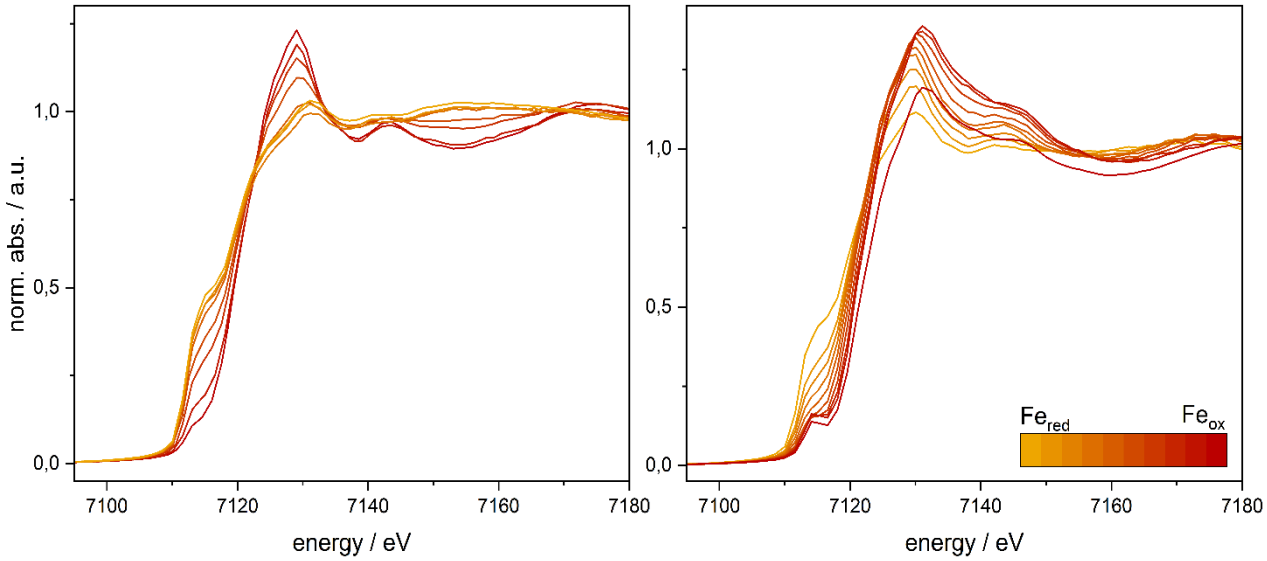

Figure 2:Tracking the reduction (left) and oxidation (right) via XANES of the F-K-edge.

detail, the reduction was performed with hydrogen, the changes were tracked with XANES and EXAFS was collected of the final state. In the second step, the material was oxidized by water vapour with home lab setup and the changes were again tracked with XANES and the final state with EXAFS (see fig. 2).