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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

fill in a separate form for each project or series of measurements.

type your report, in English.

include the reference number of the proposal to which the report refers.

make sure that the text, tables and figures fit into the space available.

if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Structural Studies of Functional Oxides and their Calatytic Reactivity for CO_2 Decomposition – in situ XAS Studies of Nanophase Ferrites having different particle sizes	**Experiment number**: 01-01-696
Beamline: SNBL	**Date of experiment**: from: 16.11.05 to: 21.11.05	**Date of report**: 01.02.06
Shifts: 15	**Local contact(s)**: Hermann Emerich	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

XAS studies on iron, cobalt, nickel, copper and zinc-containing spinels (nanophase) and their catalytic reactivity for CO_2 decomposition and NO reduction

Functional materials e.g. the oxygen-deficient functional oxide ferrite spinels (AFe_2O_4 where A is here a dipositive metal ion; Co, Ni and Zn) have been synthesised according to procedures reported in the literature [1,2] and identified by XRD. The average particle size were estimated to be in the range of 2-15 nm and calculated from the Debye-Scherrer equation [3].

XAS data were collected in November 2005 (01-01-696). The structure of the nanophase oxide ferrite spinels; cobalt, nickel and zinc ferrites, were studies in the catalytic decomposition of CO_2 and the reduction of NO. The XAS spectra were measured in the transmission mode at the Fe, Co, Ni and Zn K-edges. Both the Exafs and the Xanes spectra are used and are currently being processed. The XAS spectra clearly show the structural transformation of the ferrites during the different steps in the catalytic reaction.

It can be seen that the different ferrites are completely or partially reduced to metallic iron, nickel, cobalt or zinc in hydrogen, and reoxidized during the reduction of NO. Similar observations are made in the case of decomposition of CO_2, illustrated in the figure below. Nickel ferrite is reduced to metallic nickel and iron in hydrogen, and reoxidized to

the spinel structure after the CO_2 treatment, but with traces of unreacted nickel metal. The nickel-oxygen shell multiplicities are 6 both before and after the catalytic reaction.

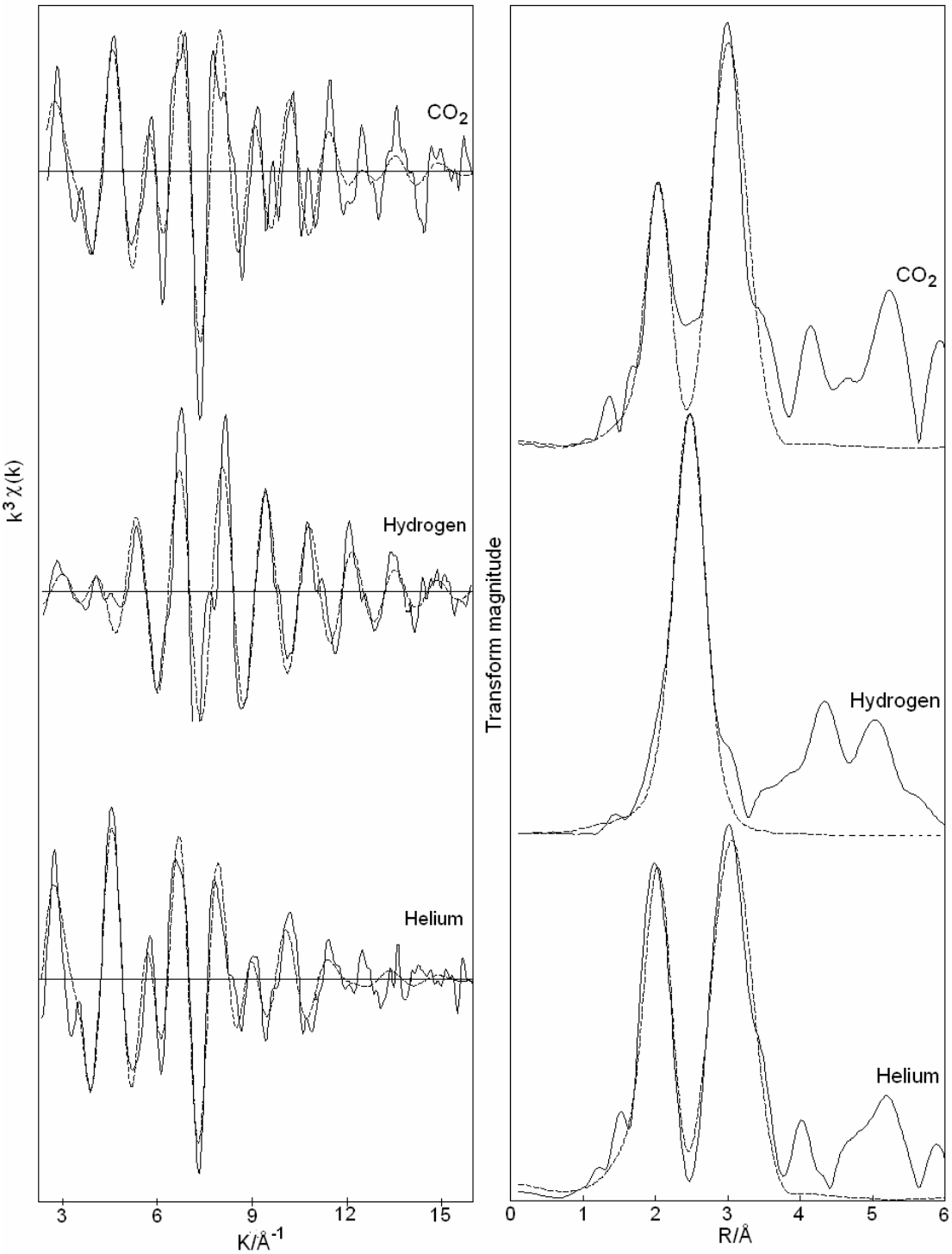

Figure 1. Experimental (-) and calculated (--) Fourier filtrated (1-25) k^3-weighted EXAFS and its Fourier transform for nickel ferrite heated in helium at 500°C, in hydrogen at 500°C, and in CO_2 at 500°C at the Ni k-edge.

References

1. A.S. Albuquerque, J.D. Ardisson, W.A.A. Macedo, J.L. Lopez, R. Paniago, A.I.C. Persiano, *J. Magnetism and Magnetic Materials*, 226-230 (2001), 1370-1381.
2. Q. Chen, A.J. Rondinone, B.C. Chakoumakos, Z.J. Zhang, *J. Magnetism and Magnetic Materials*, 194 (1999) 1-7.
3. S.R. Davis, A.V. Chadwick and J. Wright, *J. Mater. Chem.*, 8(9) (1998) 2065-2071