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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.

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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:** **A Programme of XAFS Studies on a variety of Functional and Advanced Materials** -XAFS studies on some iron, cobalt, nickel, copper and zinc-containing spinels (nanophase)	**Experiment number**: 01-01-654
Beamline: SNBL	**Date of experiment**: from: 15. June to: 20. June 2005	**Date of report**: 31.08.05
Shifts: 15	**Local contact(s)**: Denis Testemale	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

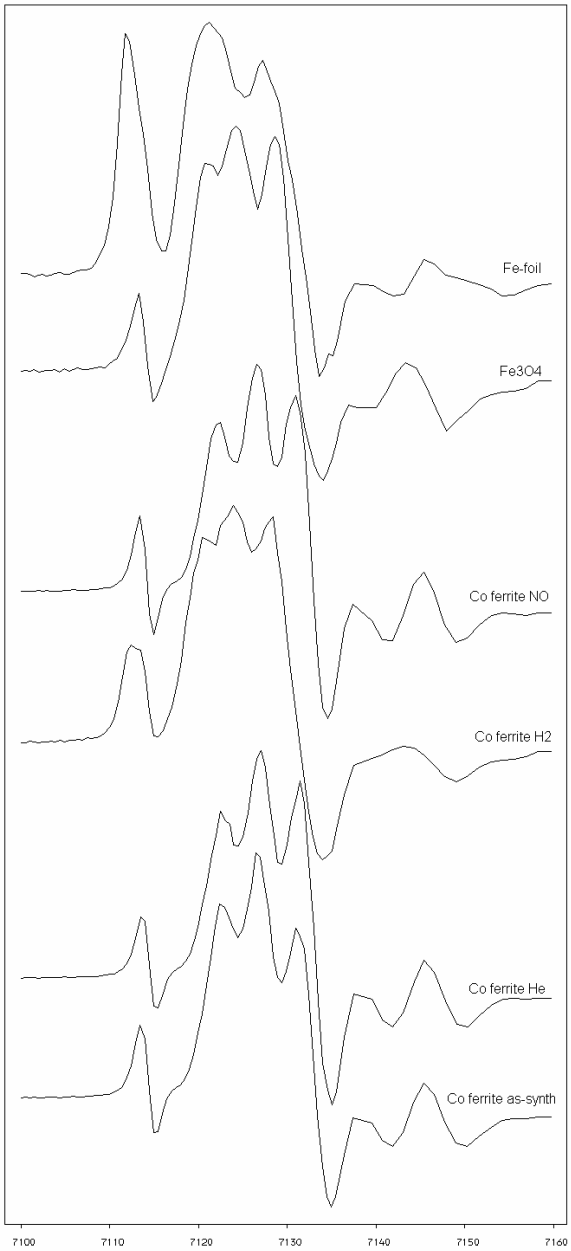
XAFS studies on some iron, cobalt, nickel, copper and zinc -containing spinels (nanophase)

Functional materials e.g. the oxygen-deficient functional oxide ferrite spinels (AFe_2O_4 where A is here a dipositive metal ion; Co, Ni or Zn) have been synthesised according to procedures reported in the literature [1,2] and identified by XRD. The average sizes of the nanoparticles have been calculated using the Debye-Scherrer equation [3] from the (311 & 440) line widths of the XRD, and they were estimated to be in the range 2-15 nm.

XAS data (XAFS and XANES) were collected in June (01-01-654). The XAS spectra were measured in the transmission mode at the Fe, Co and Ni *K*-edges, and the data are of good quality.

In addition, *in situ* XAS data were collected at the Fe, Co and partly the Ni K-edges. The data are now under processing. We are currently comparing the XAFS data for the different nanophases. The catalytical behaviour of oxygen deficient cobalt and nickel ferrites towards the selective reduction of NO_x was investigated. The XAS data were collected after the different steps in the catalytical procedure: heating the sample in helium, reduction of the ferrites in hydrogen and the reduction of NO. The gas treatments were done at 300 and 500ºC while the XAS data collection was done at room temperature.

The XAS data indicate that the reduction of the ferrites by hydrogen at 300 ºC gave a partially reduced nickel ferrite while no structural change was observed in the case of cobalt ferrite. At 500ºC, the cobalt ferrite was partially reduced by hydrogen, and the nickel ferrite completely reduced to metallic iron and nickel.



After the selective reduction of NO, the reduced ferrites were oxidised back to its original spinel structure. XANES data are very useful for establishing the A or Fe sites occupations because these affect the valence states. The 1st derivative XANES spectra of an iron and cobalt-containing spinel reduced and treated by NO gas at 500ºC are given in Figure 1 below.

The results are interesting and more beamtime is needed for making the data sets complete.

Figure 1: The 1st derivative XANES spectra of an iron and cobalt-containing spinel as synthesised, heated in helium, reduced by hydrogen and treated by NO gas, the Fe_3O_4 model and the iron foil.

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. G.H. Stout, *X-ray Structure Determination: A Practical Guide.* 2nd ed., 1989