



## 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.



	<b>Experiment title:</b> Magnetic coupling between the layers of bi-magnetic core-shell nanoparticles	<b>Experiment number:</b> HE-3263
<b>Beamline:</b> ID08	<b>Date of experiment:</b> from: 30 juny 2010 to: 04 jul. 2010	<b>Date of report:</b> 10 Feb. 11
<b>Shifts:</b> 12	<b>Local contact(s):</b> Pardeep kumar Thakur	<i>Received at ESRF:</i>

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**Report:**

Core-Shell nanoparticles composed of soft( $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>)-hard(Mn<sub>3</sub>O<sub>4</sub>) ferrimagnets (FiM) were studied through X-ray absorption spectroscopy (XAS) and X-ray magnetic circular dichroism (XMCD). Two different types of particles with different core size and equal shell thickness were analyzed, Sample1 [ $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>(7 nm)/Mn<sub>3</sub>O<sub>4</sub>(2nm)] and Sample2 [ $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>(11nm)/Mn<sub>3</sub>O<sub>4</sub>(2nm)].

Measurements were carried out at the ID08 beam line in total electron yield (TEY) and fluorescence (FY) modes, using the 5 T cryomagnet setup. Note that the FY mode was mainly used to prove that TEY mode could penetrate enough to get information on the whole particle.

The XAS spectra the O K-edge and at the Mn and Fe L<sub>2,3</sub>-edges were recorded to get further insight into crystallographic structure of the core and the shell. On the other hand, XMCD at the Fe and Mn L<sub>2,3</sub> edges were measured to selectively address the magnetization of the two FiM layers. Figure 1 shows absorption spectra (XAS) measured using TEY mode at the Mn and Fe L<sub>2,3</sub> and O K edges at 5 T field at 10K, after field cooling in 5T. Confirming our X-ray diffraction data, the Mn and Fe spectra are consistent with the expected for  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> and Mn<sub>3</sub>O<sub>4</sub> phases. Nevertheless, interestingly, the Mn edge spectrum seems to somewhat deviate from the theoretical one for Mn<sub>3</sub>O<sub>4</sub>. From the relative intensity of the peaks the spectra can be understood as a mixed signal from pure Mn<sub>3</sub>O<sub>4</sub> and another Mn-oxide phase formed mainly by Mn<sup>2+</sup> ions. The XMCD spectrum confirms that Mn is not in a single phase but it is formed

by a mixed contribution of  $Mn_3O_4$  and another Mn oxide phase. This additional contribution can probably be ascribed to an intermediate, inner, shell between the core and the outer shell formed by a mixed Mn-Fe oxide. The XMCD also shows that the Fe- and Mn-based phases are magnetic both at 10 K and at room temperature, Figure 2, confirming the presence of another Mn-based phase with  $T_C > T_{RT}$ , different from  $Mn_3O_4$  ( $T_C \sim 40K$ ).

Hysteresis loops, Figure 3, were acquired by recording the field dependence of the XMCD signal at the energies corresponding to three main peaks of Fe-edge and for the main energy of the Mn-edge, after field cooling in 5T. The loops were carried out up 2 T to saturate the hard magnetic part. Remarkably, apart from a linear background in the Mn-edge loop, the loops at the Fe- and Mn-edges are very similar (e.g., with the same coercivity). This is somewhat unexpected and requires further experiments to fully understand this behavior. Moreover, both nanoparticles (small and large  $\gamma$ - $Fe_2O_3$  core) exhibit very similar behavior with Fe-edge and Mn-edge loops showing very similar features. The results may indicate that the soft and hard phase are strongly coupled to each other, thus reversing coherently.

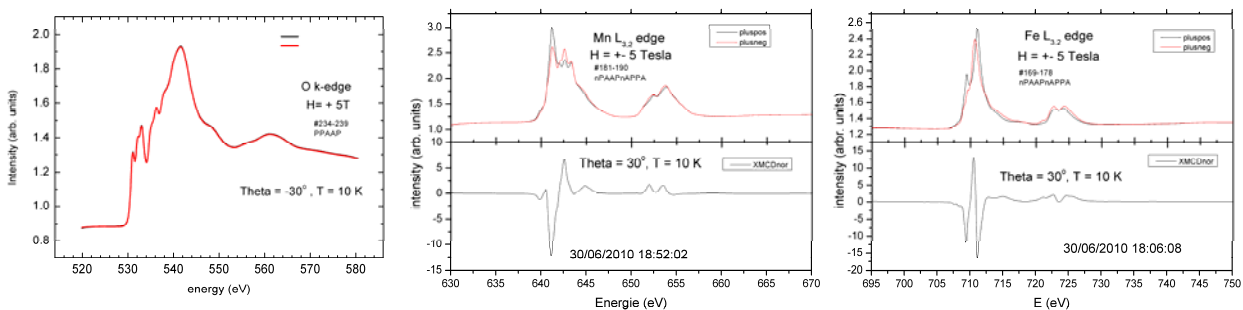


Fig.1,

XAS spectra for O K ,Mn and Fe  $L_{2,3}$  edges at 10 K in 5T after field cool in 5 T for Sample1

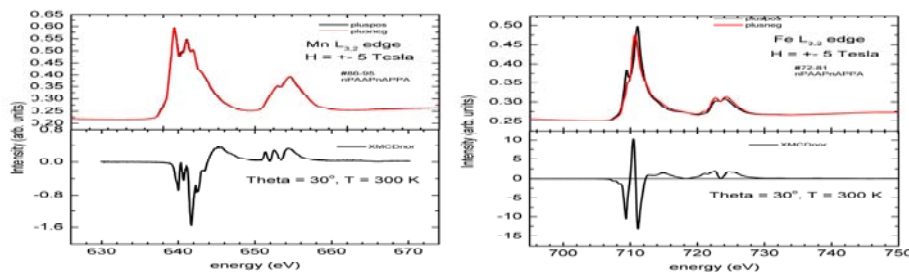


Fig.2, XAS spectra for O K ,Mn and Fe  $L_{2,3}$  edges at 300 K in 5T for Sample1

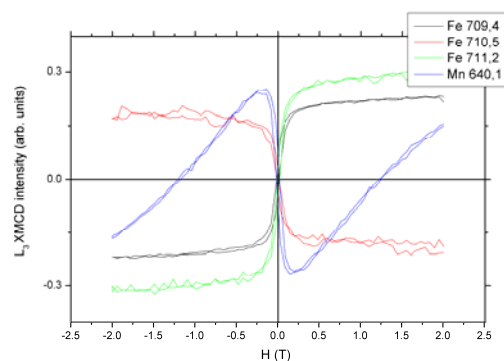


Fig. 3, Hysteresis loops recorded at 10K after field cool in 5T, the loops were acquired for the main energie peaks of Fe (3) Mn (1) XMCD spectra for Sample1