EUROPEAN SYNCHROTRON RADIATION FACILITY

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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 via the User Portal:

Reports supporting requests for additional beam time

Reports can 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:** Nuclear Resonant SAXS by Magnetic Nanoparticles	**Experiment number**: MA 1825
Beamline:	**Date of experiment**: from: 11 March 2013 to: 13 March 2013 from: 16 July 2013 to: 20 July 2013	**Date of report**:
Shifts: 15	**Local contact(s)**: BESSAS, Dimitrios	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

We proposed to investigate the structural and magnetic properties of Fe containing nanoparticles using Nuclear Resonance Small Angle X-ray Scattering (NRSAXS). The application of the method to the samples of scientific interest becomes possible only nowadays due to the strong increase of the flux at 3rd generation synchrotrons. The method was applied to study both the magnetic structure of γ-Fe_2O_3 nanoparticles with canted spins in the surface region and dimer nanoparticles γ-Fe $_2O_3$/FePt. We aimed to extract both the structural and the magnetic form factor, which would provide insight into the difference between each other. This new method would offer an unique way for the analysis of magnetic structures in nanoparticles and the single particle shape of heterogeneous complexes of nanoparticles.

We planed to investigate enriched γ-$^{57}Fe_2O_3$ and partially-enriched γ-$^{57}Fe_2O_3$/FePt dimer nanoparticles by NRSAXS using Nuclear Forward Scattering (NFS) and the Synchrotron Mössbauer Source (SMS). Both methods have advantages and drawbacks. While NFS provides higher flux by ~3-5 times, SMS allows for directly measuring NRSAXS, related to the particular hyperfine structure. For the γ-Fe_2O_3/FePt nanoparticles the SMS setup is the most promising one, because the signal arising from the iron in the Fe_2O_3 is separated from the signal of the iron in the FePt by different velocities of the Mössbauer drive. The γ-Fe_2O_3 only give a 6 peak absorption spectrum, which implies the usage of NFS due to the higher flux.

The first part of the measurement was performed with NFS in March 2013, selected data is depicted in Fig.1. On the first glance, there is no difference between the electronic (prompt) and nuclear (delayed) scattering. This can be reasoned by different facts. First, there might be no difference in the electronic and the magnetic form factor. Which would mean that the expected canted spins are not canted or they are not at the surface. Second, every resonant scattered photon can also be scattered electronically. If we assume that the electronic scattering cross section is very large, the delayed signal would be the product of a large electronical part and a small nuclear part. Thus, the difference between the nuclear and electronic form factor might not be visible.

The second part of the measurements were performed with SMS in July 2013. We observed already in a early stage of the experiment that the flux is too low to get reliable results over a large enough Q-range, thus we switched to investigate the behaviour of spherical γ-$^{57}Fe_2O_3$ nanoparticle with full enrichment in comparison to dimer $^{57}Fe_2O_3$/FePt nanoparticles with only the iron oxide sphere enriched in NFS geometry. The obtained data is depicted in Fig 2 and 3. The applied magnetic field strongly influence the dimer nanoparticles and align them parallel to the field, in contrast the single spheres of iron oxid are only slightly influenced. This behaviour is quite unexpected, because magnetometry suggest for the γ-$^{57}Fe_2O_3$ nanoparticles nearly complete alignment of the spins in a 1 T field, i.e., the shape of zero-field cooled and field cooled curves is equal and nearly complete saturation in the hysteresis loop is found. We have obtained NFS data on the γ-$^{57}Fe_2O_3$ nanoparticles in magnetic fields up to 7.5 T from the first part of the experiment, which are depicted in Fig. 4. The oscillation frequency changes in the highlighted regions with increasing field. This is related to the expected alignment of the spins parallel to the magnetic field, which we observed for the dimers at much smaller fields. Even at 7.5 T no complete alignment seems to be established.

In summary, we were able to measure NRSAXS with quite good statistics in a NFS setup. Preliminary results reveal no difference between normal SAXS and NRSAXS. Nevertheless, we proved that the flux is high enough in order to perform the measurement. The SMS option exhibits a too small flux for NRSAXS investigations, roughly speaking a factor of 5 or 10 enhancement would be needed. We obtained field dependent NFS and SMS data of the used nanoparticles, which exhibit unexpected behaviour. A corresponding manuscript is under preparation.

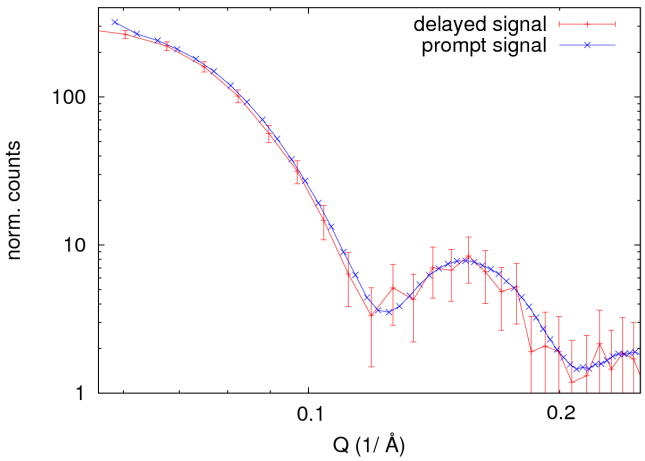

Fig 1.: Delayed and prompt signal, obtained on $^{57}Fe_2O_3$ Nanospheres in 1 T magnetic field.

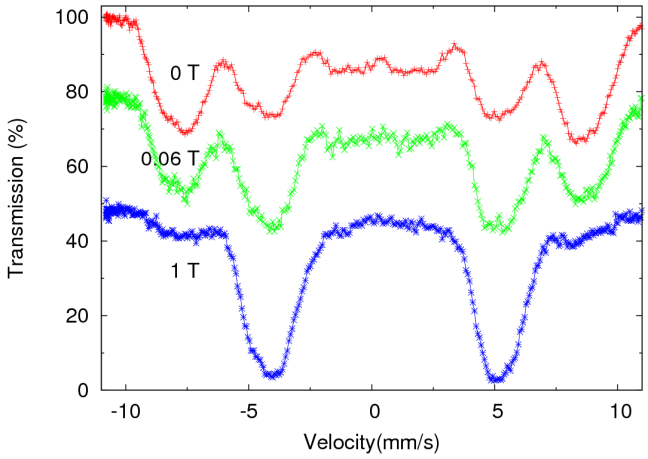

Fig 2.: $^{57}Fe_2O_3$/FePt Nanodimeres with different applied magnetic fields orthogonal to beam direction.

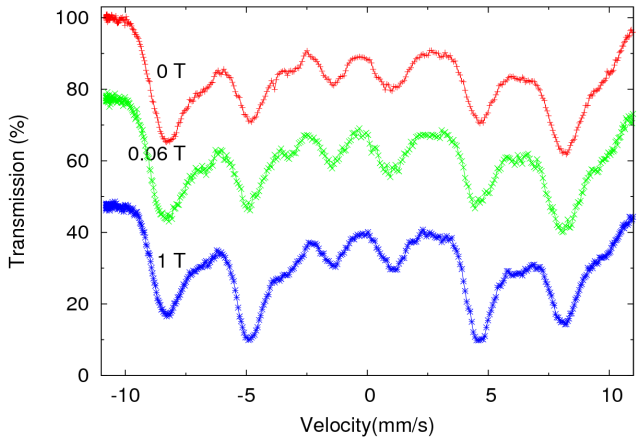

Fig 3.: $^{57}Fe_2O_3$ Nanoparticles with different applied magnetic fields orthogonal to beam direction.

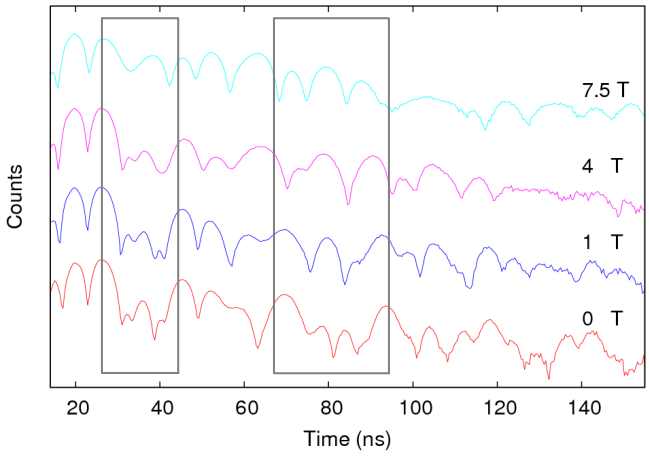

Fig 4.: NFS data of $^{57}Fe_2O_3$ *Nanoparticles with different applied magnetic fields orthogonal to beam direction.*