EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

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:** Determination of the "hidden" Fe structure in Fe_xM_{100-x} (M=Au, Ag and Cu) nanogranular thin films.	**Experiment number**:
Beamline:	**Date of experiment**: from: 15/07/15 to: 20/07/15	**Date of report**: 22/07/2015
Shifts:	**Local contact(s)**: María Vila Santos	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Diego Alba Venero*: Universidad de Cantabira

Jesús Ivan da Silva*: ISIS neutron and muon source

Pilar Ferrer Escorihuela: Diamond Light Source

Luis Fernández Barquín: Universidad de Cantabria

Report:

We have produced a diluted (less than 10 atomic %) Fe-nanoparticled system with very interesting magnetic properties. In this samples, the Fe structural data has been masked by a huge contribution coming from the noble metal matrices (Au, Ag or Cu). The aim of the experiment was to extract the crystalographic Fe information by tunning the incoming X ray energy to the matrix absorption to reduce their contribution to the XRD pattern steaming from the matrix and therefore been able to extract the "hidden" Fe structure.
The experiment began with some beamline work originated by a compleat blackout of the current the previous day. The first 8 hours were used to reconfigurate the beamline and setting up the experiment conditions, since some of the encoders were out of position due to the power cut. We started the experiment tunning the X-ray beam energy out of the resonance of any of the absorption edges of any of the metals present in our samples (i.e., Au, Ag, Cu and Fe), E = 16.148 keV, which produces a wavelenght of $\lambda = 0.7677$ Å. After setting up the desired energy, the diffractometer was aligned and the first sample (Fe_7Au_{93}) was set in place and the off resonance X-ray diffraction pattern was recorded, ending with the first day of experiment. During the second day we swap samples (Fe_7Ag_{93} and Fe_9Cu_{91}) . It is worth mentioning that we count for long times (around 12h for each spectra) because the low wavelenght used for the off-resonant experiment grant access to a d-space where there is a sole peak of Fe. However, despite the long counting time we were not able to observe it.
During the third day we start to swap energy, initially to the L_3-Au edge. For stabilising clearly the beam energy we run a transmission experiment through a Au refence foil. We test it with the Fe_9Cu_{91} sample (since all the films have a 3 nm Au capping layer), and try to

observe if tunning more precisely the energy will produce a drop in the peak steaming from the Au capping layer, failing to observe any difference. Since the Ag K energy edge was out of the beamline capabilities our next step was moving to the Cu K-edge. The effect in the K edge was much stronger than in the L3 edge, observing a big decrease in the Cu peaks, as can be observed in the Fig. 1. We adquire statistics for the rest of the beamtime.

The CCD camera was not used by strong recomendation from the beamline responsible Germán Castro, so we did not check the possible preferential orientations in the film.

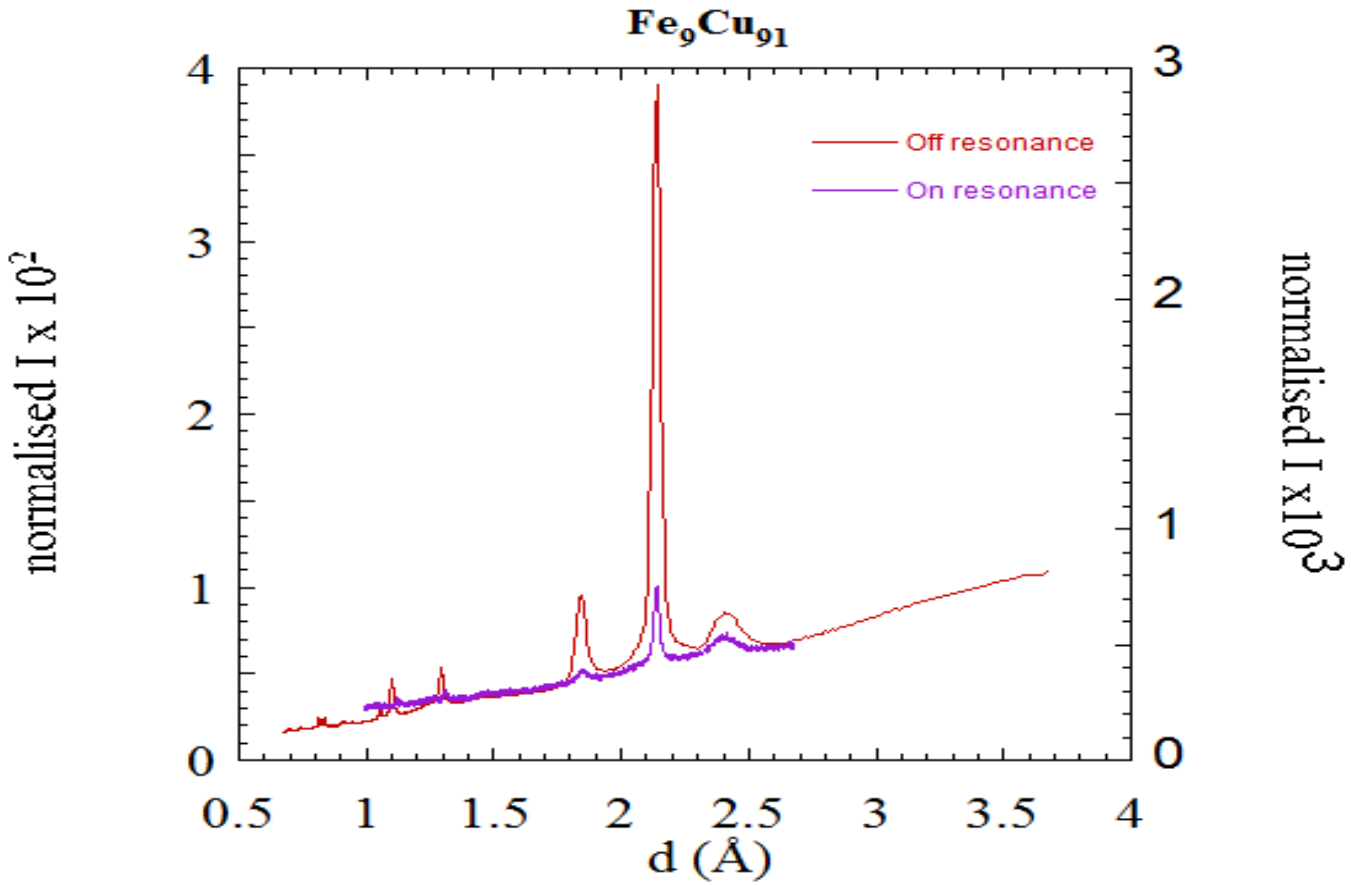

Fig 1. *XRD pattern obtained off and on resonance. The relative intensity of the Cu peaks is greatly diminish in the on resonance energy.*