EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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:

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:** Nanophase identificaiton in artificial multiferroics	**Experiment number:** 20-02 728
Beamline: BM20B	**Date of experiment:** from: 11 April 2014 to: 15 April 2014	**Date of report:** 25-02-2015
Shifts: 11	**Local contact(s):** Carsten Baehtz	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Prof. Lino Pereira (KU Leuven)
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*Koen van Stiphout (KU Leuven) – (experimentalist, not applicant)

This experiment was devoted to two types of artificial multiferroics. In the following, we report on the two independent sets of measurements.

1) Ferromagnetic Fe and Fe-Co thin films on ferroelectric $LiNbO_3$ (LNO)

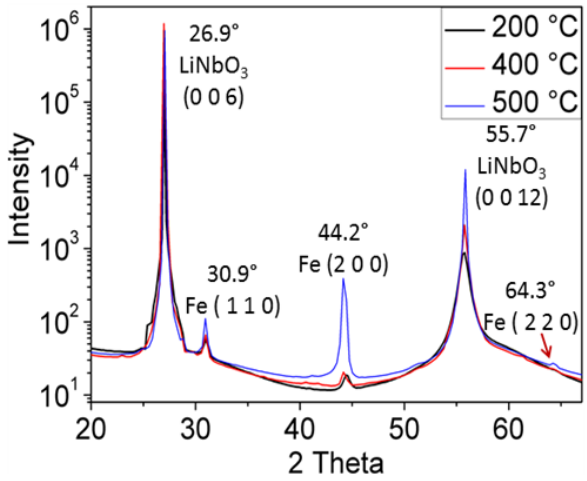

Fig. 1 - θ/2θ scans for Fe/LNO samples annealed at 200°C, 400°C and 500°C.

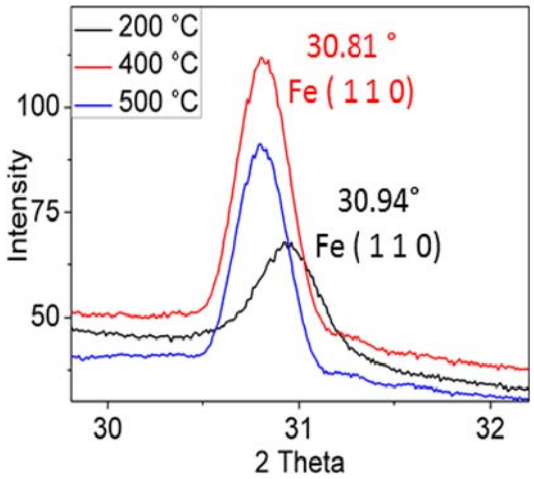

Fig. 2 - Rocking curve for the Fe (110) peak on samples annealed at 200°C, 400°C and 500°C. Similar results were obtained for FeCo/LNO.

This part of the experiment contributes to a project focusing on the magnetoelectric coupling between a **ferroelectric substrate and a ferromagnetic thin film**. Our previous studies had shown

magnetoelectric (ME) coupling in Fe/LNO and FeCo/LNO systems. In particular, we observed that the ME coupling increased monotonously with post growth annealing (PGA) temperature. SR-XRD was performed on these samples to correlate the changes in ME coupling with changes in crystallinity of the Fe / FeCo films. The samples consisted of Fe and FeCo thin films (20 nm) on LNO, as-grown and annealed at 200°C, 400°C, and 500°C. All samples were capped with 10 nm Ge films to avoid oxidation of the metal films. These results show that the post growth annealing improves the crystallinity of the metallic films which in turn leads to the stronger magnetoelectric coupling between the ferromagnetic film and the ferroelectric substrate. Quantitative analysis (ongoing) will provide a more detailed insight on the dependence of the ME coupling strength on the texture of the ferromagnetic films.

2) Ferromagnetic nanoparticles embedded in a multiferroic oxide ($BiFeO_3$ thin films)

This part of the experiment contributes to a project focusing on the magnetoelectric coupling between a **multiferroic matrix and embedded magnetic nanoparticles** wich are formed after Fe/Co implantation and adequate thermal annealing. The samples were prepared by Fe and Co implantation into $BiFeO_3$ thin films (epitaxial and policrystalline), with varying ion fluence and implantation energy. Epitaxial $BiFeO_3$ films were grown by pulsed laser deposition on $SrTiO_3$ substrates. Polycrystalline $BiFeO_3$ films samples were grown by sol-gel methods on $Si/SiO_2/TiO_x/Pt$ structures (suitable for ME coupling measurements). In polycrystalline $BiFeO_3$, after Co-implantation and annealing, we were unable to detect any sign of formation of magnetic secondary phases (Fig. 1). On the other hand, for epitaxial $BiFeO_3$ films implanted with Co, we were able to identify the implantation and annealing conditions leading to the formation of cobalt ferrite $CoFe_2O_4$ (Fig. 2). We are currently investigating the magnetoelectric coupling established between multiferroic $BiFeO_3$ matrix and the embedded ferromagnetic $CoFe_2O_4$ nanoparticles.

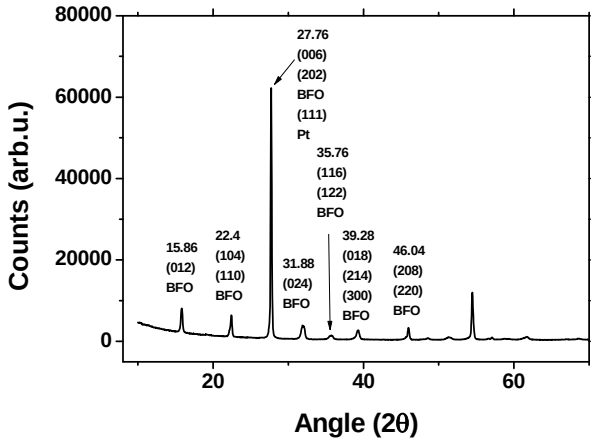

Fig.1 - GI-XRD on Co-implanted polycrystalline $BiFeO_3$ on $Si/SiO_2/TiO_x/Pt$, showing only $BiFeO_3$ peaks.

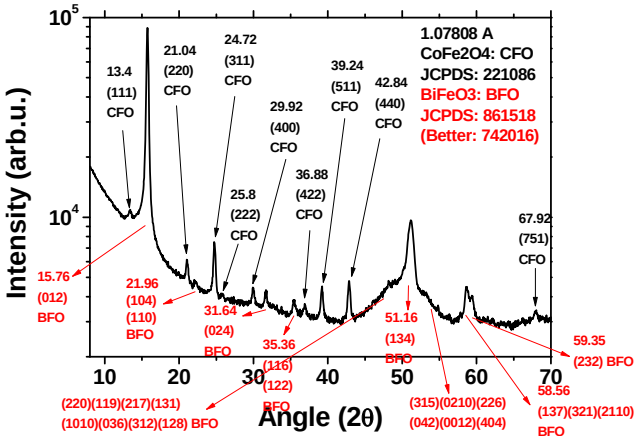

Fig. 2 - GI-XRD on Co-implanted epitaxial $BiFeO_3$ on SrTiO3 substrate, showing the formation of $CoFe_2O_4$.