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

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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.

	Experiment title: Structural characterization of Nd-Fe-B ultrathin films: towards the understanding of the origin of spin reorientation in $Nd_2Fe_{14}B$	**Experiment number**: A252-1047
Beamline: BM25	**Date of experiment**: from: 8th November to: 14th November	**Date of report**: 17/01/2023
Shifts: 18	**Local contact(s)**: Juan Rubio Zuazo	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Jimena Soler*, **Iciar Arnay***, Andrés Martín-Cid, Paolo Perna, Cristina Navío, Alberto Bollero

IMDEA Nanoscience Institute, C/Faraday 9 28049 Madrid, Spain

Report:

Proposal summary and main objectives: The study performed was focused in the structural and compositional characterization of epitaxial Nd-Fe-B (NdFeB) thin films by means of x-ray diffraction (XRD) and high energy x-ray photoelectron spectroscopy (HAXPES). Rare earth transtion metal thin films present high magnetic anisotropy, being the preferred choice for a large number of applications [1-5]. In this particular case, our research is framed under the H2020 FET-OPEN project UWIPOM2 (Ref. 857654) and focuses on the development of wireless NdFeB-based microactuator for microsurgery, with additional potential applications in transport and aerospace applications. The experiment was divided in two differenciated parts, detailed below, taking advantage of all the resources provided by the BM25-SpLine beamline in order to optimize the use of the shifts allocated.

I-XRD measurements with temperature dependence (GIXRD experimental station)

XRD measurements were performed in the Grazing incidence XRD experimental station, using a beam energy of 15 keV (0.826 Å). NdFeB films were prepared by DC sputtering by ablating a Fe target with Nd and B pieces attached to it. A Mo layer was deposited as a buffer to favor the adhesion of the NdFeB film to the substrate. Fig. 1a shows out of plane XRD θ-2θ measurement showing [001]-oriented growth of Mo and NdFeB films on top of MgO. Characterization of several Mo and NdFeB reflections prove an epitaxial growth of Mo on MgO with a 45 degrees in plane rotation. NdFeB grows also epitaxially, axis on axis respect to MgO lattice and consequently with a 45 degrees in plane rotation respect to the Mo buffer layer. Thus, following epitaxial relations are found: NdFeB[001]||Mo[001]||MgO[001] and NdFeB[100]||Mo[110]||MgO[100]. Calculated lattice constants show a relaxed growth of Mo with bulk-like lattice parameter and a cubic symmetry (a=3.15±0.03 Å) within the experimental error. Despite the presence of the Mo buffer, NdFeB shows a conmensurate growth with MgO, as can be seen in the alignment of MgO and NdFeB contributions in reciprocal space maps (RSM) (fig. 1b). This results in a compresive in plane strain of -3.4% in the NdFeB lattice. Simultaneously, an expansion of 1.4% of the lattice occurs out of plane, resulting in lattice parameters of a=8.48±0.03 Å and c=12.04±0.05 Å. RSM around MgO[111] reflection also shows additional contributions related to Fe and Nd_2O_3 polycrystalline phases (fig. 1b), which was also observed by energy dispersive x-ray (EDX) analysis in our home laboratory.

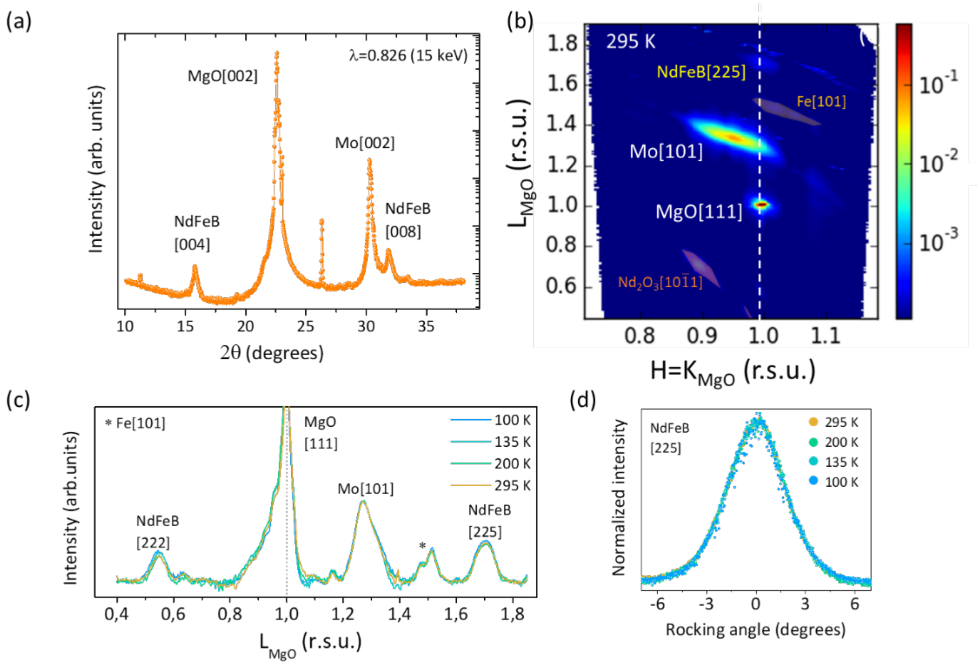

Figure 1: XRD characterization of NdFeB/Mo/MgO stacks performed at beam energy of 15 keV (λ=0.826Å). (a) Out of plane θ-2θ scan. (b) RSM around MgO[111] and Mo[101] reciprocal space posititons. (c)Evolution of substrate CTR 11L as a function of sample temperature. (d) Evolution of rocking curves around NdFeB[225] reciprocal space position as a function of temperature.

In order to decrease the temperature, an ultra-high vacuum (UHV) baby chamber was installed in the diffractometer, with a pumping system and a Cryostat coupled to it. Temperature was varied in a range between 295 K and 100 K. Fig 1c. shows the evolution of the MgO[11L] crystal truncated rod as a function of the temperature. Background has been sustracted and intensity normalized using Mo[101] intensity. No changes in the relative intensity or position of NdFeB[222] and NdFeB[225] reflections are observed as the temperature decreases. Additional rocking scans around NdFeB[225] reflections were recorded. No modification of the peak profile is observed under temperature variation. This preliminary results point to the absence of structural changes associated with the spin reorientation transition.

II- HAXPES measurements at different beam energy (SXD+HAXPES experimental station)

Second part of the experiments consisted of HAXPES measurements in the Surface and X-ray diffraction and X-ray photoelectron spectroscopy station. Kinetic energy ranges corresponding to Mo1p, B1s, Fe2p, Nd3d, O1s and Ta3p edges were scanned using photon energies of 7, 10 and 12 keV. Figure 2 shows spectra recorded in Nd 3d, Fe 2p and Mo $1p_{1/2}$ core level energy range at the three photon energies. At the highest energy we can already observe the Mo contribution. Therefore, by changing the incoming photon energy, the whole depth of the film has been probed: from the outer surface to Mo/NdFeB interface. Preliminary results indicate no variations of the lineshape profile with photon energy variation, which at first indicate no variations in the oxidation state of Fe and Nd along the z-direction of the film. Further analysis will provide more accurate information about the stoichiometry and homogeneity of the film.

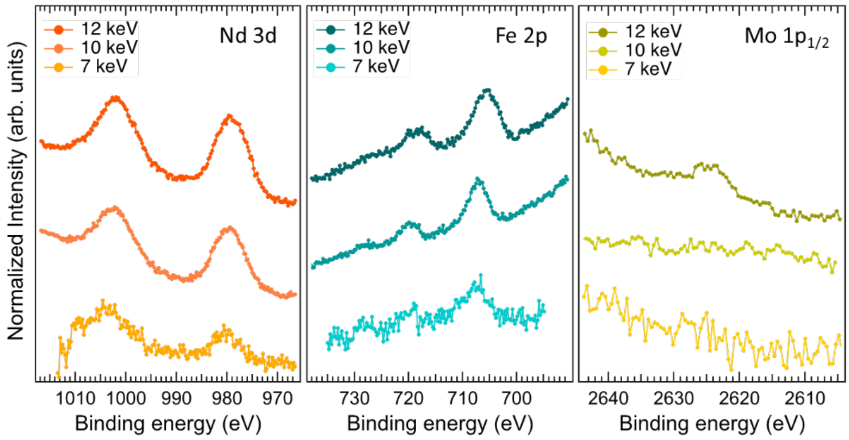

Figure 2: From left to right Nd3d, Fe2p and Mo1p1/2 core level spectra measured on a NdFeB/Mo/MgO stack at photon energy of, from up to bottom. of 12 keV, 10 keV and 7 keV

Relation with the current research:

Acquired data at the ESRF is being complemented with SQUID measurements at low temperature in order to determine spin reorientation temperature for the epitaxial NdFeB/Mo/MgO[001] stacks. Given the strain observed in our epitaxial system a modification of the transition temperature is expected [6,7], which will be confirmed by the evolution of magnetization as a function of temperature. Vibrating-sample magnetometry (VSM) measurements prior to the experiment, already indicated the presence of a strong perpendicular magnetic anisotropy as well as large coercive fields at room temperature. Structural strain will also be related to NdFeB films stoichimetry by a more thoughtful analysis of the photoemission spectra.

References:

[1] A. Morisako *et al., J. Magn. Magn. Matter*. 304, 46-50 (2003)
[2] X. Liu et al., J. Appl. Phys. 97, 10K301 (2005)
[3] T.-S. Chin, J. Magn. Magn. Matter. 209, 75-79 (2000)
[4] A. Bollero et al., Nanoscale, 12, 1155-1163 (2020)
[5] O. Gutfleisch et al., Adv. Mater. 23, 821–842 (2011)
[6] A. Handstein et al., J. Magn. Magn. Matter. 101, 382-384 (1991)
[7] L. M. García et al., Phy. Rev. Lett. 85, 2 (2000)