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

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.

<table>
<tr><td>ESRF</td><td>Experiment title: Crystallographic study of Fe_3O_4/MNM/Fe_3O_4 (MNM=Ag, Au) plasmonic spin valve systems tailoring the plasmonic response of the Metallic Non-Magnetic nanostructured layer</td><td>Experiment number:
25-02-940</td></tr>
<tr><td>Beamline:
BM25-SpLine</td><td>Date of experiment:
from: 10 November 2020 to: 15 November 2020</td><td rowspan="2">Date of report
30-11-2020

Received at ESRF:</td></tr>
<tr><td>Shifts:15</td><td>Local contact(s): RUBIO ZUAZO Juan and LOPEZ SANCHEZ Jesus</td></tr>
<tr><td colspan="3">Names and affiliations of applicants (* indicates experimentalists):
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& Main proposer #Remote users *Experimental users</td></tr>
</table>

Report:

Fe_3O_4/Au/Fe_3O_4/NiO and Fe_3O_4/Ag/Fe_3O_4/NiO multilayers grown by Pulsed Laser Deposition (PLD) on MgO(001) substrates have been studied by Grazing Incidence Surface X-Ray Diffraction (GIXRD) experiments at 15 keV at the BM25-SpLine beamline at The ESRF (Grenoble, France). Metallic Non-Magnetic (MNM) Au and Ag layers were prepared with different growth conditions in order to modify the non-continuos character of the layers and their effective thickness for the plasmonic spin valve systems. Figure 1 displays the scheme of these spin valve systems identifying the different layers. Analyzed samples are shown in Table 1.

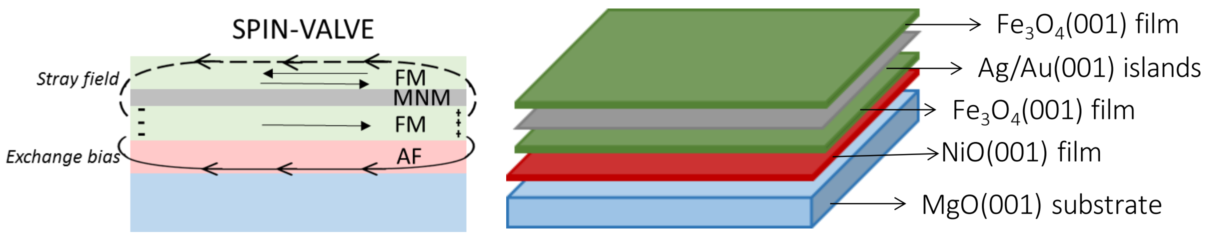

Figure 1. Spin valve design based on using the half metallic ferrimagnet Fe_3O_4 (FM). NiO serves as the antiferromagnetic (AF) exchange biasing layer and Ag/Au nanostructured layer would be the nonmagnetic (MNM) spacer.

Table 1. Description of the growth condition of Fe_3O_4/Au/Fe_3O_4/NiO and Fe_3O_4/Ag/Fe_3O_4/NiO multilayers grown by PLD on MgO(001) substrates.

Sample	Metallic Non-Magnetic (MNM) layer	Growth temperature of MNM at the PLD	Post-annealing
Au 1	Au	150 ºC	-
Au 3	Au	350 ºC	-
Au 2	Au	550 ºC	-
Ag 1	Ag	200 ºC	-
Ag 4	Ag	200 ºC	400 ºC
Ag 3	Ag	200 ºC	500 ºC

Several crystallographic measurements (CTRs, RODs, low and high angle XRR, among others) have been performed in order to determine the crystalline structure of the layers, elucidate the presence of strain on the thin films and analyze the presence or absence of chemical inter-diffusion at the interface between the different layers.

Figure 2 displays an example of the low angle XRR resutls for two samples with Ag as the non-magnetic spacer: multilayers Ag 1 and Ag 4. As the Ag layers is post-annealed at 400 ºC (Ag 4), a clear modification of the low angle XRR signal is noted and associated with the Ag nanostructuration during the post-annealing with respect to the sample Ag 1.

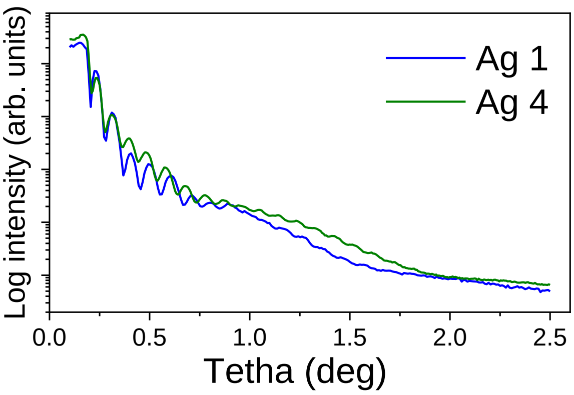

Figure 2. Low angle XRRs of epitaxial Fe_3O_4/Ag/Fe_3O_4/NiO system grown on MgO(001) substrate (sample Ag 1) and that with the Ag layer post-annealed at 400 ºC (sample Ag 4).

For all multilayer systems, irrespective of the growth conditions of the non-magnetic layers (Au and Ag), the high angle XRR spectra revealed the presence of single-phase epitaxial NiO, Fe_3O_4 and Au/Ag with (001) orientation. Besides, it should be noted that Kiessig fringes around thin film Bragg peaks were observed, indicating the occurrence of high-quality and smooth surfaces and abrupt NiO-substrate, Fe_3O_4-NiO, Au/Ag-Fe_3O_4 and Fe_3O_4-Au/Ag interfaces. These findings can be observed in the out-plane reciprocal space spectra presented in Figure 3. In addition, for the plasmonic spin valves with Au a greater crystallintiy and effective thickness (height of Au nanostructures) are obtained as the Au growth temperature increases, related to a larger Au nanostructuration. For the Ag systems a shift of Bragg's peaks for Ag layer is identified towards larger L values as increases the post-annealing temperature, which can associate with a lattice contraction.

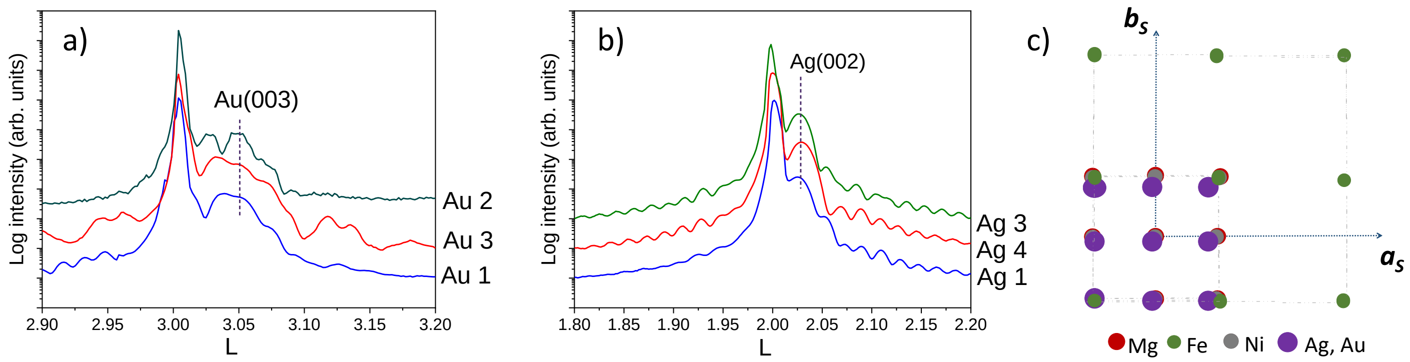

Figure 3. L reciprocal space scans for plasmonic spin valve with a) Au as MNM layer and b) Ag as MNM layer prepared under different growth conditions. c) Representative scheme in real space of the lattice coupling for the Fe_3O_4/MNM/Fe_3O_4/NiO system on MgO(001) substrate. a_s and b_s represent the in-plane lattice paramenters of MgO substrate.

From the Reciprocal Space Maps (RSM) measurements, the coupling behavior between the different lattices and the corresponding substrate lattice is achieved. In all cases, the crystallographic axes of the lattices of multilayers are identified as collinear with those of MgO(001) substrate, as represented in Figure 3c. In all cases, layers grow incommensurate, based on the noncoincidence of the in-plane diffraction maxima from different layers and the MgO substrate. These findings can observe in Figure 4 where two representative RMS for Ag 3 and Ag 4 are displayed. Here, in addition to Ag peaks, those related to NiO and Fe_3O_4 layers are found.

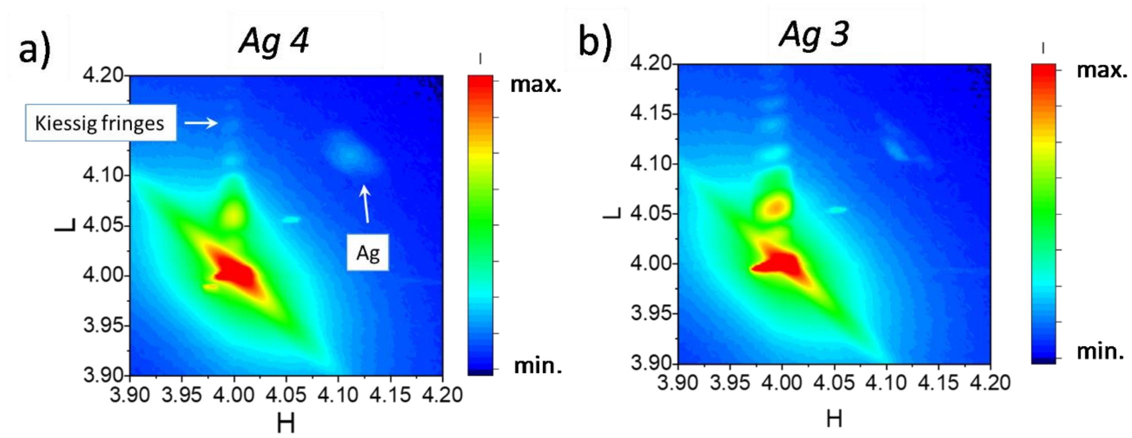

Figure 4. LH reciprocal space maps (RSM) for Fe_3O_4/Ag/Fe_3O_4/NiO multilayers varying the post-annealing temperature for Ag layer. From the in-plane diffraction measurements, we identify a noncoincidence of layers and substrate peaks, evidencing an incommensurate epitaxial growth. The color scale corresponds to the signal intensity.