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.

	Experiment title: THE ROLE OF THE INTERFACIAL ATOMIC STRUCTURE IN THE EMERGENCE OF PERPENDICULAR MAGNETIC ANISOTROPY IN Co/Pd MULTILAYERED SYSTEMS	**Experiment number**: 25-02-1002
Beamline: BM-25	**Date of experiment**: from: 29/09/2021 to: 5/10/2021	**Date of report**: 01/02/2023
Shifts: 6	**Local contact(s)**: Juan Rubio Zuazo	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

This experiment has allowed us to elucidate the relationship between Perpendicular Magnetic Anisotropy (PMA) in Co/Pd heterostructures and the structure and composition of the Co/Pd multilayer. We explored the effect of low energy ion bombardment, that can increase interfacial roughness, induce intermixing or even clustering and alloying. We have performed a combination of XRD, XRR, supported by energy-dependent HAXPES and XAS to get a complete structural and compositional map of the multilayers, and we have correlated it with the perpendicular magnetic anisotropy.

We have characterized the compositional and chemical depth-profile of Co/Pd heterostructures grown at the same conditions, before and after being modified by low energy ion bombardment. We have measured 1 pristine sample and 5 ion-bombarded samples, treated with different ion doses at an ion (Ar^+) energy of 3 keV. The samples (formed by 20 Pd/Co bilayers) were fabricated by magnetron sputtering in Madrid at a base pressure better than 10^{-7} torr by sequential deposition of Co and Pd.

We have determined, by XRR, the thickness of each sample and found results matching our expected primarily thicknesses. XRD results show a preferential growth in the (111) out-of-plane direction for the Pd. But they also show a lack of clear structural transformations with increasing ion doses, even though our magnetic measurements showed a trend, were the PMA is clearly reduced with the increasing dose. At the highest dose the PMA completely disappears, and the easy magnetization direction is the in-plane direction, typical of ordinary thin films. So the transition from out-of-plane (PMA) to in-plane easy magnetization is not accompanied by clear structural transformations, at least clearly detected with the techniques available during the beamtime. The disorder induced by ion bombardment at the topmost interfaces is not evidenced by XRD, but clearly unbalances the delicate equilibrium which stabilizes the PMA.

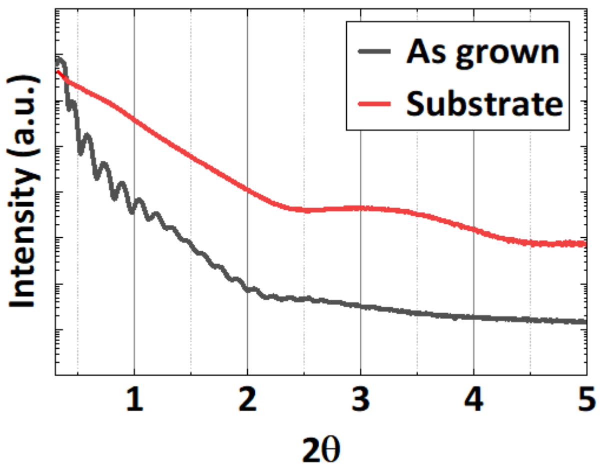

Figure 1. a) An X-ray Reflectometry (XRR) of a pristine multilayer, before ion bombardment. Also, an XRR of a bare Si(001) substrate as the ones used for the growth of the multilayers is shown, showing the oscillation from the native oxide thin film (with a thickness around 2 nm).

HAXPES and XAS measurements show that the metallic Co deposited in the multilayers is in fact not fully metallic, but approximately 50% of it was oxidized, also in the pristine sample. We have not found a trend relating the ion dose to the degree of oxidation of the samples. The nature of the oxide is probably closer to CoO than to any other phase. The existence of a satellite in the HAXPES spectra is a clear hint supporting the presence of octahedral CoO. XAS measurements also support the presence of the monoxide CoO as the most compatible phase.

XPS spectra show that the presence of the Cobalt oxide is more evident at the surface (where XPS is more sensitive) than in the bulk (Figure 2).

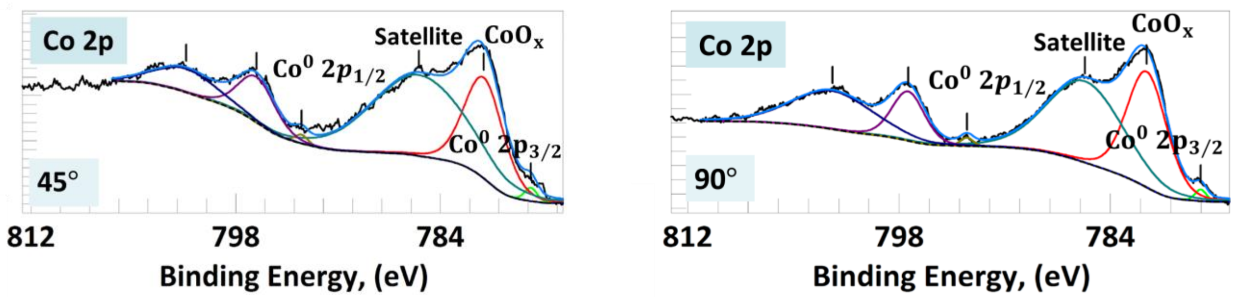

Figure 2. XPS spectra of Co 2p core level of the as-grown multilayer, at two different angles of the detector with respect to the sample surface (45º and 90º).

Probably, the most interesting and surprising result of the beamtime has been to get the evidence that Cobalt is oxidized to a high extent. So, the study of the nature of the magnetic interactions present must necessarily include this fact. To check that the presence of the oxide has not been induced by the synchrotron beam, we have checked, after the beamtime, in Madrid, that the pristine sample keeps the PMA untouched and that it is essentially the same as before taking the sample to the ESRF.

Figure 3 shows some of the other most relevant preliminary results of the measurements taken (HAXPES, XAS and XRD). HAXPES has allowed us to get compositional information from the deepest layers of the heterostructure. This is evident from the Si 1s core level, where we detect both the Si and SiO_2 phases (that later coming from the native oxide of the substrate before growing).

In summary, the beamtime has been fruitful and we have obtained interesting results, which are now being summarized and discussed in a manuscript which will be soon submitted for publication.

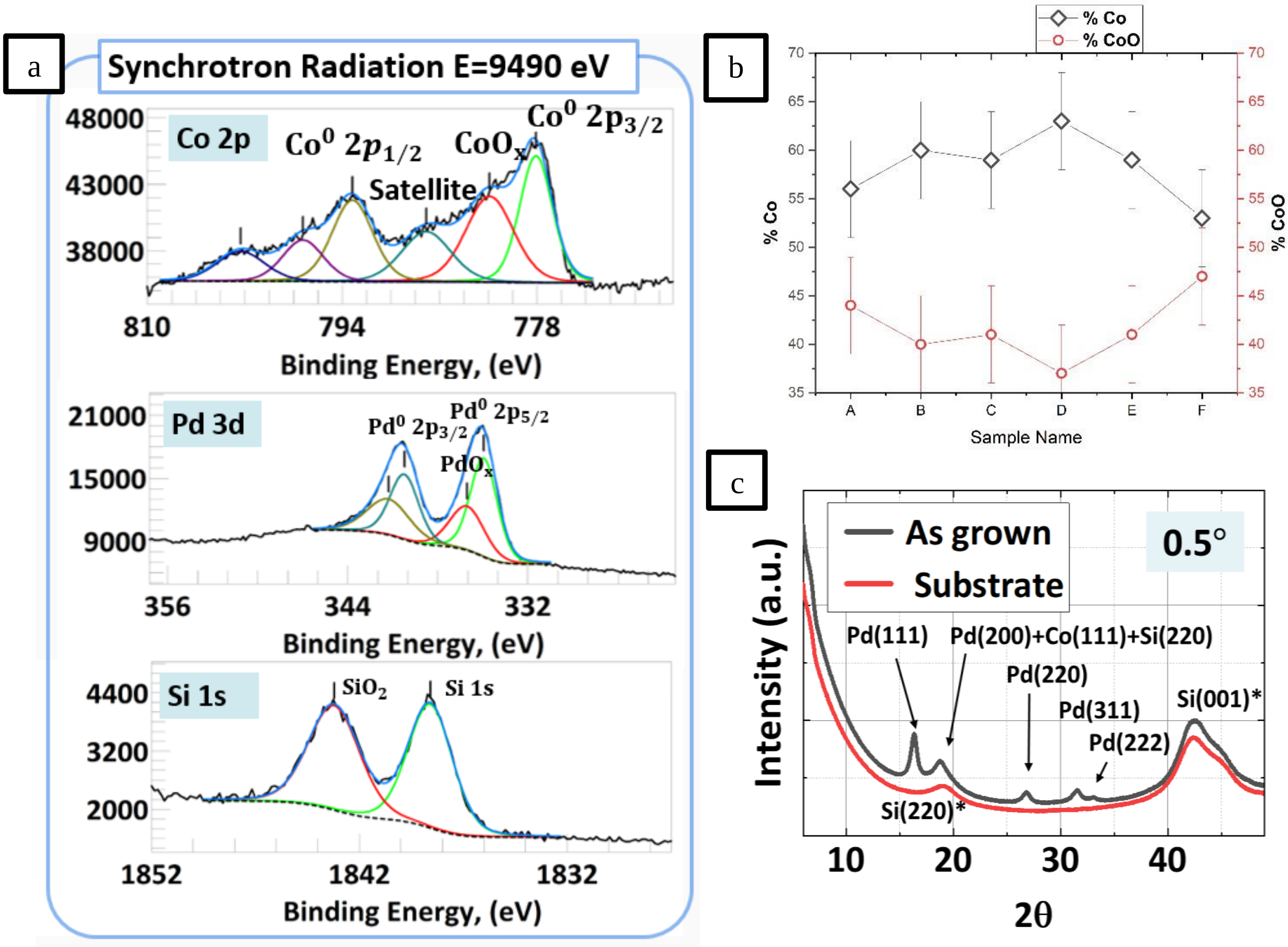

Figure 3. a) HAXPES mesaurements at 9490 eV. Co 2p, Pd 3d and Si 1s regions for the pristine sample. The deconvolution shows the preence of Co oxide b) XAS results showing the Co vs CoO concentration for each sample. The samples are named and ordered by the irradiation dose, being A the pristine sample and F the sample irradiated with the highest dose. c) XRD of the pristine sample (black) and the bare substrate (red).