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.

<table>
<tr><td> </td><td>Experiment title:
Morphological, compositional and electronic study of ultra-thin silicon oxide film embedded at the interface between Fe3O4 and Si</td><td>Experiment number:
25-02-1008</td></tr>
<tr><td>Beamline:</td><td>Date of experiment:
from: 31/05/2022 to: 06/06/2022</td><td rowspan="2">Date of report:
16/02/23
Received at ESRF:</td></tr>
<tr><td>Shifts:</td><td>Local contact(s):
Juan Rubio Zuazo</td></tr>
<tr><td colspan="3">Names and affiliations of applicants (* indicates experimentalists):

Laboratory SpLine CRG c/o ESRF 71 avenue des Martyrs CS 40220 FR - 38043 GRENOBLE Cedex 09
Dr RUBIO ZUAZO Juan
Dr LOPEZ SANCHEZ Jesus
Ms. SANCHEZ PAGE Beatriz</td></tr>
</table>

Report:

The aim of the this experiment was to study the viability of introducing an ultra-thin oxide layer at the interface between the ferromagnet and the semiconductor used on non-volatile memories with the objective of removing the large impedance mismatch present between ferromagnets and semiconductors that limits the spin injection from high conductive ferromagnetic material to high-resistive non-magnetic semiconductor. The ferromagnetic material provides a unique ability to switch rapidly their magnetization and the semiconductor provides a unique ability to modify its properties by doping, light or voltage.

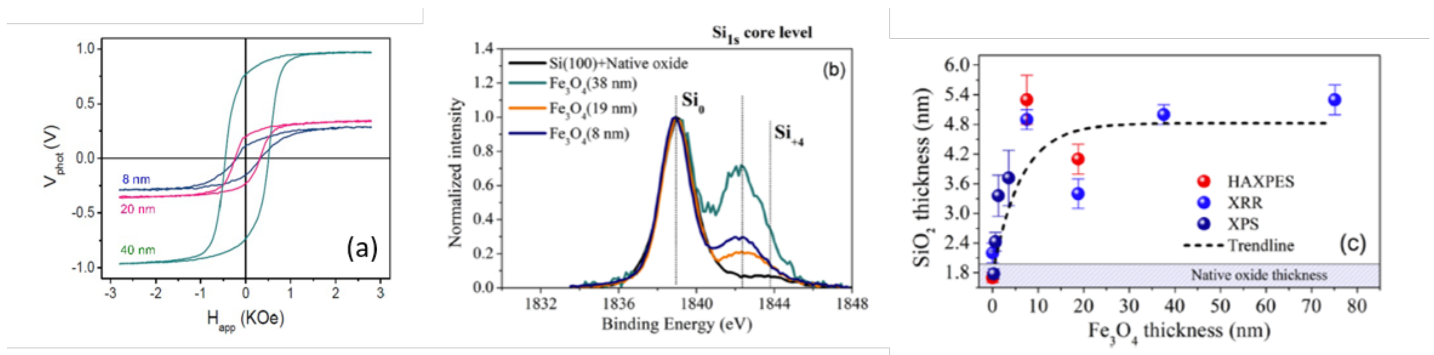
It is well known the ability of SiO2 to rapidly grow on the Si surface when the substrate is heated to evaporate the ferromagnetic material. Hence, in this experiment we explored the possibility of using the native SiO2 layer from Si(100) substrates to integrate Fe3O4 ferromagnetic oxide known the difficulty present in stabilizing extremely thin layers of SiO2 on silicon substrates while deposition of the ferromagnetic material. Also it is well known the ability of SiO2 to oxidize the interface of the ferromagnetic metal frustrating the magnetic contribution.

We measured 3 samples grown by PLD, thin layers of Fe3O4 on Si(100)/SiO2 substrates (with dimensions 10x10 mm2) formed by Si(substrate)/SiO2/Fe3O4 (8, 20 and 40 nm thick) by combination of X-ray Reflectivity, X-ray Diffracion and Hard X-Ray Photoelectron Spectroscopy. The samples showed an increase of the coercive field and saturation values as a function of Fe3O4 thickness as revealed by MOKE (See figure a). Standard XPS revealed the presence of pure Fe3O4 phase at the heterostructure surface.

We performed X-ray reflectivity, X-ray Diffraction and Hard X-ray Photoelectron Spectroscopy measurements to find the physical properties of the buried Fe3O4/SiO2 interface and to be able to analyse the morphology of the SiO2 interface (layer thickness and interface roughness) and of the presence or absence of non-magnetic iron oxide phases or silicate phases. A complete compositional and electronic depth profile was performed by

following the evolution of the photoemission signal from the Fe1s, Fe2p, Fe3s, Si1s and Si2p orbitals as a function of the photon energy, i.e., electron kinetic energy and hence sampling depth and a complete X-ray reflectivity pattern was obtained using 12 keV photons.

In a first insight, we have seen a competitive interplay between silicon and iron oxidation. During the first stage of iron oxide evaporation the SiO2 thickness increased while the iron was unable to get fully oxidized. From a certain iron oxide thickness, the SiO2 layer is stabilized in thickness and the iron was fully oxidized. This is explained by oxygen transport through the iron layer. While the iron layer has low thickness, the oxygen is able to diffuse until the Si oxidizing its surface, forming SiO2 and avoiding oxidation of the iron. For larger iron layer thickness, the oxygen cannot travel through it, limiting the Si surface oxidation and enabling iron oxide formation. This scenario is shown in Figure b in which Si vs SiO2 HAXPES signal is represented for different iron oxide layer thickness. Taking into account the HAXPES, XRR and standard XPS measurements we determined a stabilized SiO2 thickness of 4.8 nm (See figure c)



Further data analysis is needed to find the morphological, compositional and electronic correlation of this ultra-thin silicon oxide layers embedded at the interface between Fe3O4 and Si.