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.

ESRF	**Experiment title:** The influence of strain on the magnetic anisotropy of epitaxial ε-Fe_2O_3 films on rigid and flexible substrates	**Experiment number**: A25-2-1021
Beamline:	**Date of experiment**: from: 31/01/2023 to: 06/02/2023	**Date of report**: 13/07/2023
Shifts:	**Local contact(s)**: Juan Rubio Zuazo	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Although all shifts were used, the ESRF beam dropped several times and there were several optics stabilization windows due to those inconveniences. On the other hand, we had a leak search for low temperature baby chamber measurements.

1) Epsilon iron oxide ε-Fe_2O_3 thin film on $MgAl_2O_4$ (111) substrate

An epitaxial epsilon iron oxide ε-Fe_2O_3 thin film on $MgAl_2O_4$ (111) substrate was measured on cooling to low T. We aquired HKL scans and rockings at temperatures -193ºC, -163 ºC, -133 ºC, -103 ºC, -73 ºC, -23 ºC, and RT.

Several long range H and L scans were tested to find suitable regions were film and substrate can be probed probed together. Specific diffraction spots were defined to aquire rockings for peak width assessment. A composition of acquired H,L scans is shown in Fiugre 1(a) of the mapped region in the [1-10](111) substrate plane. The first impresion is matching the expectation and no traces of other iron phases (like gamma or alpha) are identified. For the temperature dependence L-scans around H=2 were acquired for different temperatures, this scan includes 2 substrate and 8 film difraction peaks, which should allow a precise determination of the lattice expansion of substrate and film, respectively. Note that the map shown in (a) is acquired without baby chamber. Nevertheless, in the measured L-scan sufficient film (eFO – epsilon Fe_2O_3) and substrate (S – MAO – $MgAl_2O_4$) reflections are labelled in Figure 1(b) and in an amplified region in figure 1(c). Slight variation of the peak position with temperature is observed. Extraction of lattice parameters and combing the results from H,L and rockings is in progress.

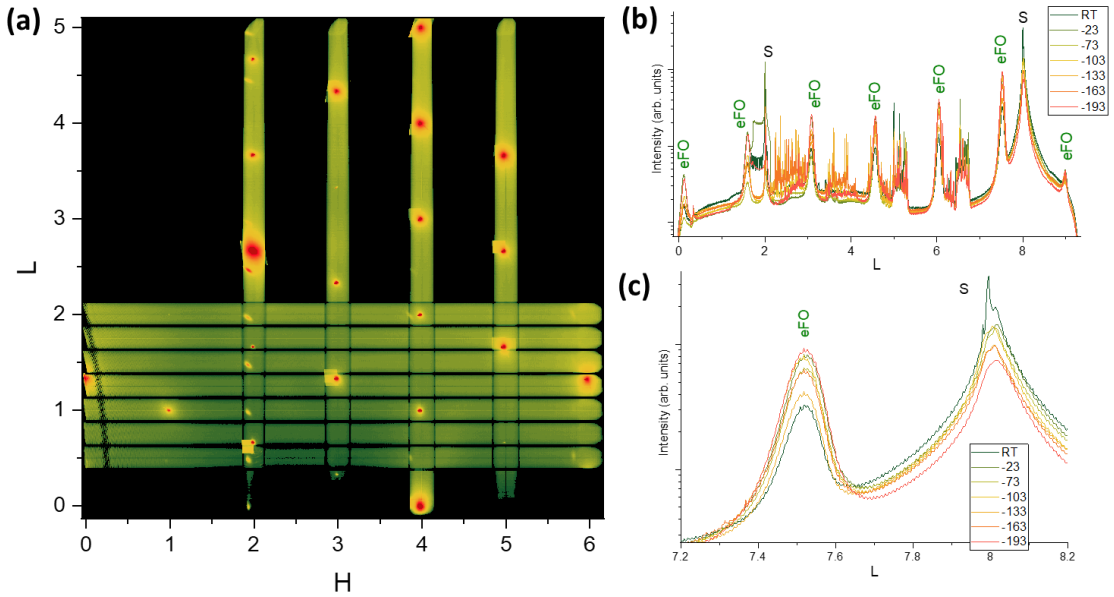

Figure 1 (a) HL-scan for eFO/MAO(111) thin film at room temperature. (b) Temperature dependence measured at via L-scan at H=2, and an amplified region around L=8 (c).

2) Epsilon iron oxide ε-Fe_2O_3 thin films on flexible mica (001) substrate

In-situ bending experiments for ε-Fe_2O_3 thin films on Mica substrates are intended to reveal the degree of mechanic coupling in these complex Van-der-Waals heterostructures, and consequently the possible effect of mechanical deformation on the magnetic anisotropy. These layered substrates can be thinned by cleaving and are flexible below 30 µm thickness. The aim is to probe the mechanical deformation of the epitaxial layer by bending the substrate in-situ. Nevertheless, the alignment of the thinned mica substrate, resulted to be rather difficult. Flourflogopyte mica is a layered silicate commonly with a high amount of stacking faults and often presents in-plane twinning. To allow handling and bending, the mica susbtrates were thinned (cleaved) to about 20 µm thickness and taped between Kapton tape, inevitably inducing additional defects.
Figure 2a shows a selected detector frame of a main substrate reflection, used to calibrate the sample orientation. A large range HL map was acquired (Figure 2b) and multiple split and deformed substrate peaks are observed. A selected rocking was acquired for substrate (Figure 2c,d) and epitaxial thin film (Figure 2e,f) The multiple split and deformed substrate spots are reflected in the thin film maps, too. We deduced that this is due to overall surface waviness (20 µm thick susbtrate on Kapton tape) and partial substrate cracking during the sample preparation. Several intents to align along the corresponding 90º film direction failed, previously determined lattice parameters (by laboratory powder diffraction) might be not exact enough to set a precise orientation matrix. We know from experience that these substrates present a much wider spread in quality, compared to single crystalline oxide or semiconductor substrates.

Finally, only one direction could be measured and both the temperature and the bending procedure were suspended in favor of an alternative sample grown on a rigid single crystal $BaTiO_3$ substrate. The results are exposed in the next section.

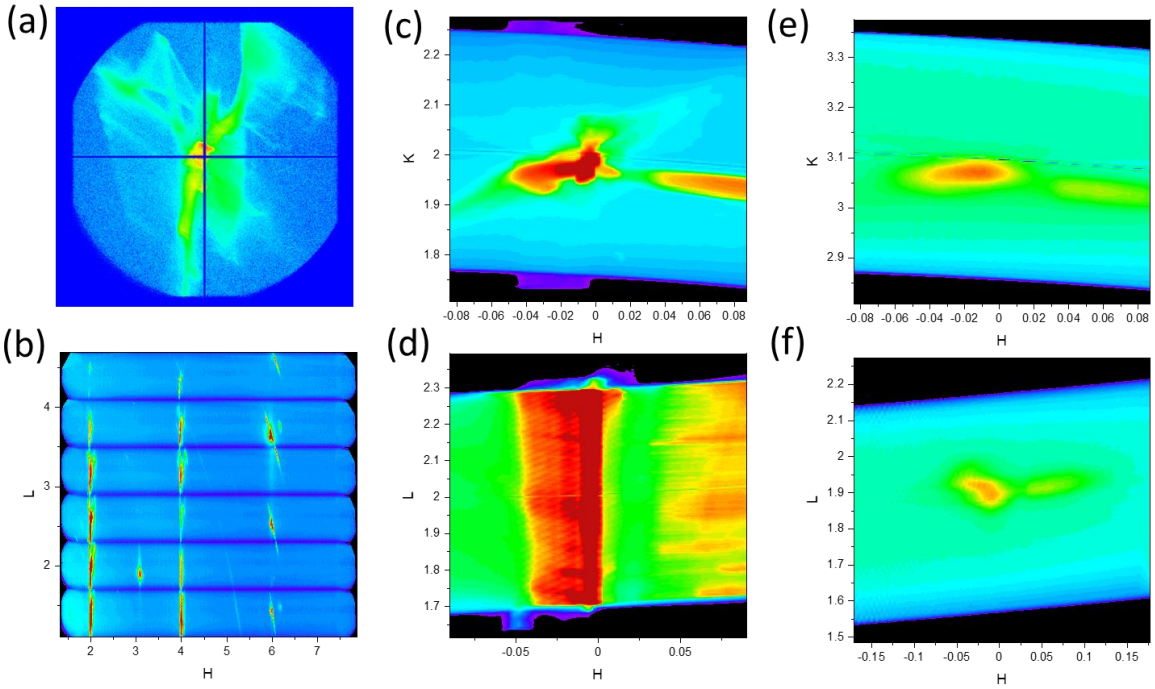

Figure 2 (a) Detector frame of mica peak during substrate calibration. (b) Large range HL map for the optimized conditions. HK, HL maps from rocking (c,d) substrate and (e,f) film peak.

3) Alternative sample of epsilon iron oxide ε-Fe_2O_3 thin films on $BaTiO_3$ (111) ferroelectric substrate

To answer the question if mechanical deformation is an effective route to strain the functional oxide layer, as an alternative sample set, we tried to determine the effect of elastic strain transfer to the film during temperature-dependent structural phase transitions of $BaTiO_3$. BTO is a ferroelectric substrate with several structural phase transitions once close to -90ºC, 0ºC and +120ºC. Due to the configuration used, we tried to assess the transitions at -90ºC and 0ºC. We measured RT → -30ºC → -70ºC → -130ºC. Thus the tetragonal → orthorhombic → rhombohedral BTO phases. The substrate peak shifts to lower 2θ values and thus the lattice spacing slightly increases (Figure 3a), while transiting from tetragonal to orthorhombic structure, this is compatible with reported structural data. On the other hand, the film peaks (Figure 3b) show a shift to higher 2θ values and thus reflect a compression with respect to RT. This suggests mechanical coupling of film and substrate, as one of the aims is to clarify if the mechanical strain is effective to modify the magnetic anisotropy of the epsilon phase. The lower T transitions from orthorhombic to rhombohedral phase is also well visible in the substrate reflection, although 2θ values of the film appear to change to a lesser amount than in the tetragonal to orthorhombic phase transition. This is an important experimental detail, as it is a first step to interpreting the very small change in magnetic anisotropy observed for epsilon iron oxide phase grown on both flexible mica and $BaTiO_3$ single crystals. Further detailed analysis of multiple reflections and rocking curves may shed further light on this system. Also in comparison to thin mangetic films in spinel phase, which in both substrates, show significant changes of the magnetic properties due to mechanical deformation in contrast to epsilon iron oxide phase.

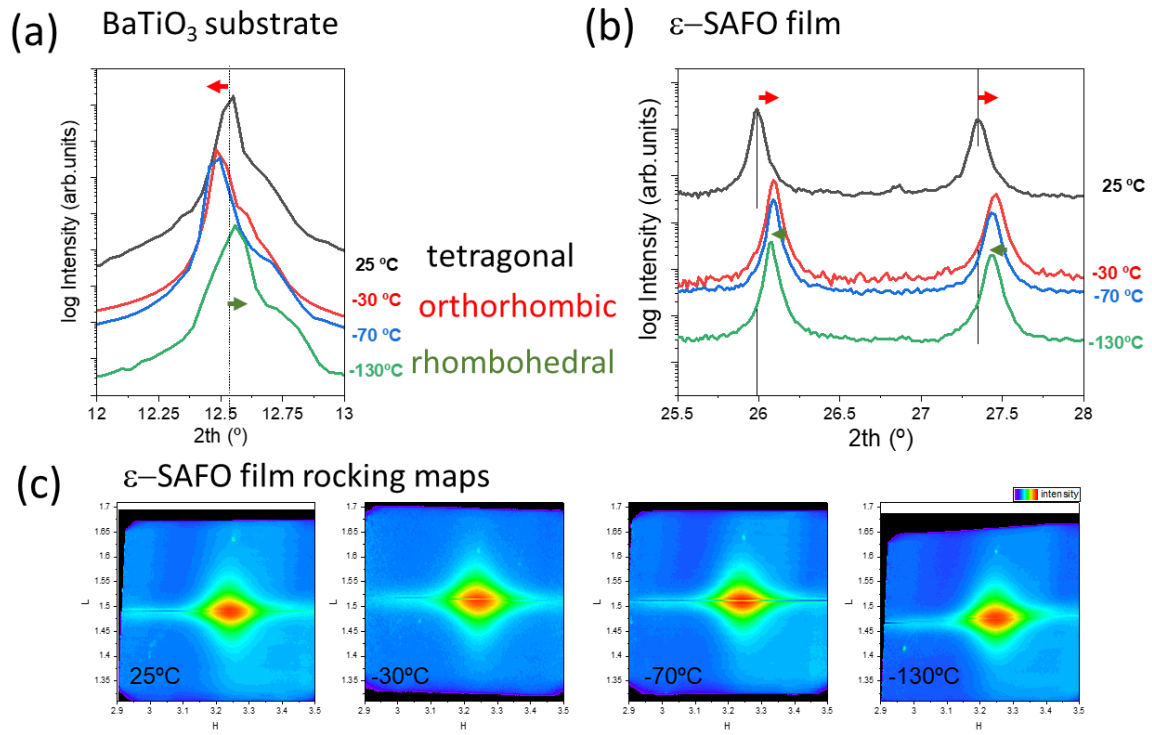

Figure 3 Temperature dependence of (a) 2th-scan for BTO(111) (b) espilon SAFO peak in substrate proximty. (c) HK-rocking maps of eSAFO film peak vs temperature.

4) epsilon iron oxide ε-Fe_2O_3 thin films on YSZ substrates with different surface orientation

Three samples of epsilon phase on YSZ substrates were explored to check the feasibility of determining the structural differences of epsilon phase with different surface directions and amount of formed domains. Similar to the previous samples we acquired large range reciprocal space maps. YSZ(100) and (111) show (00L) oriented with multiple in-plane domains forming, the observed pattern match the expected domain formation of 6 and 3 respectively. The epsilon film on (110) oriented susbtrate is clearly different: the observed pattern matches 2 domains forming with (00L) epsilon being oriented along {001} crystal directions. For further studies, the acquisition conditions should be improved to allow better overlap in the reciprocal space maps. The bulk YSZ substrates lattice parameters have to be revised, as on rotation of the sample the desired diffraction planes could not be found, we conclude that the supposed YSZ bulk lattice parameters were not precise enough (provider depending Y substitution might be the origin).

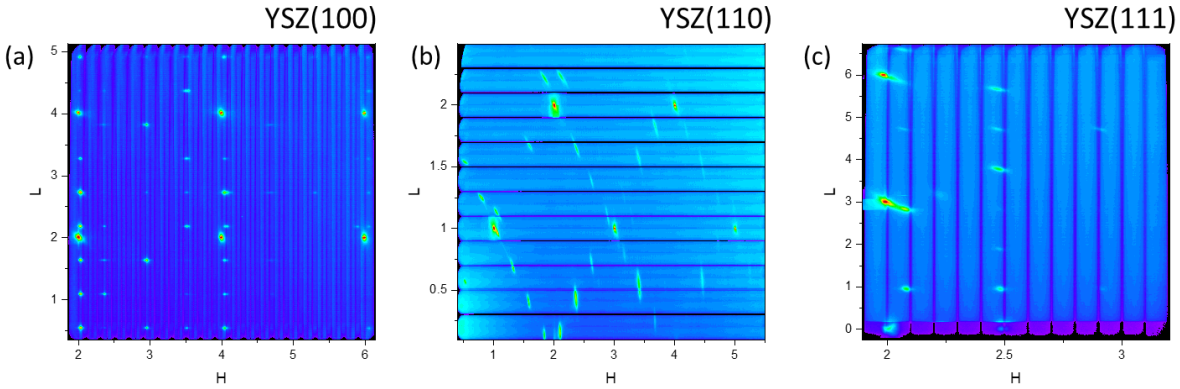

Figure 3 HL-maps for epsilon iron oxide films on (a) YSZ(100) (b) YSZ(110) and (c) YSZ(111).