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

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

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.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal 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:** Correlation between crystal and magnetic structure in epitaxial films of exotic eFe2O3 iron oxide on GaN(0001)	**Experiment number**: HC-3323
Beamline:	**Date of experiment**: from: Sep 27 2017 to: Oct 01 2017	**Date of report**:
Shifts: 12	**Local contact(s)**: Nicholas Brookes	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report (preliminary):

The XMCD experiment at ID32 was conducted on a series of epitaxially grown ε-Fe_2O_3 / GaN / Al_2O_3 samples of different thickness (40 nm, 5 nm, 2 nm) prepared using different growth conditions to ensure different shape of the in-plane hysteresis curve. The 40 nm and 5 nm samples were precharacterized by vibrating sample magnetometer to conform that magnetization loop is always double component consisting of a hard and a soft magnetic components. The 2 nm sample was not measured by VSM as its small thickness precludes accurate magnetization measirements.

SHOW LOOPS

Main results

Dichroism at Fe edge of εFe_2O_3 films

- For the first time high quality XMCD spectra of a series of εFe_2O_3 films have been acquired.

- The spectra were measured in total electron yield (TEY) mode with circular polarized light propagating parallel to the applied magnetic field. The sample was positioned in the cryomagnet with the beam incidence angle of 30 deg.
- The XMCD spectra at iron L_3 edge were shown to have a characteristic three peak structure ascribed to the octahedral and tetrahedral iron 3+ sites. The shape of the XMCD spectra was found to be thickness dependent. A drastic variation of the XMCD spectral shape was observed for the thinnest 2 nm εFe_2O_3 film. The XMCD spectrum for the thin 2 nm film shows a very pronounced Oh peak at high energies. SHOW SPECTRA.
- The magnetization curves have been measured for all the samples in two different ways to look for the traces of the soft and hard magnetic components.
- The first and the most traditional way was to set the energy to one of the dichroism maxima (708.5 eV, 709.7 eV, 710.3 eV) and to measure the TEY signal as a function of magnetic field for two opposite photon helicities. This method was shown to produce quite smooth M(H) loops except near zero field where TEY experiences special conditions.
- A more advanced method of measuring M(H) loops has been tried. The shape of the L_3 absorption peak in the range 705-715 eV has been measured with two opposite photon helicities and as a function of magnetic field. In this way the post processing analysis can be used to integrate any part of the XMCD spectrum to form a magnetization curve of a particular subalttice.
- Surprisingly it has been observed that the M(H) loops measured by TEY XMCD always show a single component shape. If the VSM loop is decomposed into soft and hard component, the hard component is reproduced in TEY XMCD loop while the soft component cannot be observed. To make sure that this behavior is not dependent on which dichroic peak or peaks are used to plot the loop the post processing of the data measured by the second loop measuring technique have been applied.
- Any of the main three absorption peaks as well as their combinations always show a hard magnetization behavior. The hard magnetic component coercivity was shown to depend on the measurement temperature and sample thickness in agreement with SQUID/VSM data. The loops are wider for thick samples and lower measurement temperatures.
- The most important result is the in 5-50 nm εFe_2O_3 the hysteresis at no energy position shows an indication of the soft component! This likely means that the soft magnetic component resides in the interface region and is not accessible in the TEY regime.
- The idea of the interfacial origin of the soft component is supported by the observation of the soft magnetic behavior in the 2 nm εFe_2O_3 film. Here we made advantage of the surface sensitive XMCD synchrotron technique as the SQUID/VSM is not sufficiently sensitive to such a thin film.
- The reason for soft magnetic behavior at the interface may be the small size of domains (superparamagnetic behavior) or the mixture of εFe_2O_3 with isostructural $GaFe_2O_3$ at the interface due to Ga diffusion during growth.

Absorption at Ga edge

- A negative response at the Ga edge was found in 80 nm αFe_2O_3 and 40 nm εFe_2O_3 films. The observed effect is quite unusual and may be related to the light being absorbed on Ga (either in the film or in substrate) and causing decrease of TEY high energy tail of Fe and oxygen.
- In a 2 nm εFe_2O_3 a strong Ga absorption edge has been detected. This can be due to GaN substrate as the 2 nm thickness is semi-transparent from the point of view of TEY.

εFe_2O_3 vs αFe_2O_3

- The XAS spectra of εFe_2O_3 thick and thin films on GaN have been compared the spectra of somewhat similar αFe_2O_3 films on GaN. The αFe_2O_3 phase is close counterpart during growth of εFe_2O_3 on GaN, so comparison was an important thing to be done, especially of the interface layer.

- The Fe L_{23} absorption spectrum was found to be very different for εFe_2O_3 and αFe_2O_3 in agreement with the data known from literature. **SHOW SPECTRA.** A distinctive feature of the αFe_2O_3 spectrum is the absence of the intensity at the energy corresponding to the Td site.
- The O 1s absorption spectrum is very different too for εFe_2O_3 and αFe_2O_3 films. **SHOW SPECTRA.** While the hematite shows a distinct splitting of the O peak at the absorption edge, in εFe_2O_3 only a broad peak is detected both for thin and thick films. The same broad features are observed for thin εFe_2O_3 and αFe_2O_3 nucleation layers suggesting that these layers have nothing to do with αFe_2O_3 lattice structure.

Linear dichroism in αFe_2O_3 films

- A preliminary magnetization experiment has been conducted on a single αFe_2O_3 / GaN film.
- No measurable circular dichroism has been detected in αFe_2O_3 films at grazing incidence in agreement to the extra weak ferromagnetic nature of hematite.
- Interestingly a strong linear dichroism has been detected in αFe_2O_3 films measured as a difference in absorption of vertically and horizontally polarized light at almost normal incidence. Moreover the XMLD difference showed a pronounced shape dependence on the magnetic field applied in-plane. **SHOW SPECTRA.**
- The absorption shape spectrum variation with field was different for H and V polarized light. In high applied field the peak and the satellite shift in energy towards each other as the polarization is changed from H to V. In zero field the effect disappears. This suggests that the effect is not as simple as the non-linear dependence of the TEY on the applied field and has a true linear dichroism nature.
- We had no time to measure temperature dependence of the XMLD effect that is expected to be switched on/off at Morin temperature of hematite.
- Further investigation of this effect would be desirable. Above Morin transition hematite is a non collinear antiferromagnet. Therefore it exhibits a small ferrimagnetic moment perpendicular to the large antiferromagnetic moment. The applied field is able to rotate the ferromagnetic moment in the basal plane of hematite and therefore rotates the antiferromagnetic moment as well. The antiferromagnetic moment can be sensed by XMLD.