Experimental report

09/02/2020

Proposal: 5-41-990 **Council:** 10/2018

Title: Magnetic structure of epsilon-Fe2O3 films

Research area: Physics

This proposal is a new proposal

Main proposer: Victor UKLEEV

Experimental team: Sergey SUTURIN
Victor UKLEEV
Jonathan WHITE

Local contacts: Bachir OULADDIAF
Ketty BEAUVOIS

Samples: Fe2O3/GaN/Al2O3

Instrument	Requested days	Allocated days	From	To
D10	7	6	05/09/2019	11/09/2019

Abstract:

Among rich family of iron oxides, the metastable e-Fe2O3 is the most intriguing phase, demonstrating room-temperature ferroelectricity, ferrimagnetism and large magneto crystalline anisotropy. In nanocrystalline form e-Fe2O3 exhibits a low-temperature phase transition accompanied by about 5% diminution in the saturation magnetization a large decrease in the coercivity field between 200 and 100 K. On the other hand, such transitions have never been observed in the epitaxial films by hysteresis loop measurements. However, temperature dependence of magnetization of 50 nm-thick epitaxial film indicates a magnetic phase transition suppressed by the field in field-cooling measurement. This would be interesting to track this transition by diffraction. Noteworthy, the magnetic structure of the e-Fe2O3 epitaxial films has never been explored by means of neutron or resonant X-ray scattering and hence remains unknown. We propose to determine the magnetic structure and track a possible magnetic phase transition of the epitaxial film e-Fe2O3 grown on GaN(0001) buffer.

Experimental report on single crystal neutron diffraction on ε-Fe2O3/GaN(0001) thin film

D10 at ILL (Grenoble)
Date: 5th-11th September 2019
Participants: S.Suturin, J. White, V. Ukleev
Local contact: Bashir Ouladdiaf, Ketty Beauvois
Proposal number: 5-41-990
Title: Magnetic structure of epsilon-Fe2O3 films

The iron oxides form a big family of magnetic materials exhibiting a rich variety of outstanding physical properties. Among these the metastable ε-Fe_2O_3 is the most intriguing phase. Single crystals of ε-Fe_2O_3 do not exist in the bulk form have been only once synthesized as the epitaxial layers. In nanocrystalline form ε-Fe_2O_3 exhibits a low-temperature phase transition accompanied by about 5% diminution in the saturation magnetization a large decrease in the coercivity field H_c, from 22.5 to 0.8 kOe between 200 and 100 K [1]. On the other hand, such transitions have never been observed in the single-crystalline films. Present neutron diffraction experiment was aimed to elucidate the difference between the low-temperature magnetic structures of the ε-Fe_2O_3 nanostructures.

Experimental details and preliminary results

The single crystal epsilon ferrite film of 100 nm thickness and are of 10x10 mm^2 was grown by means of laser molecular beam epitaxy on GaN(0001) as described in Ref. [2]. The film crystal structure and lattice orientation were confirmed by in-situ reflection high-energy electron diffraction (RHEED) and ex-situ X-ray diffraction (XRD) studies. In the grown films the polar ε-Fe_2O_3 [001] axis gets oriented normal to the surface while the easy magnetization ε-Fe_2O_3 [100] axis lies in plane parallel to GaN [1-10]. The film was mounted onto 4-circle D10 diffractometer equipped with the He cryostat (Fig. 1). Room-temperature neutron measurement with the analyzer option has already shown some difference between the diffraction peaks in the powder [1] and single crystal samples. Despite the same symmetries (space group #230 $Pna2_1$) and lattice constant values, intensities of the peaks differ significantly (Fig. 1), presumably, due to the difference in the oxygen atomic positions in the unit cell. Unfortunately, the statistics and number of the measured peaks does not allow to accurately refine the crystal structure of the film.

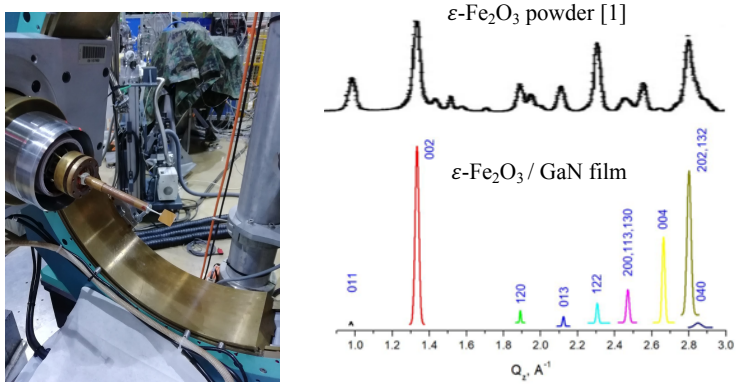

Figure 1 Left panel: ε-Fe_2O_3 film mounted onto 4-circle diffractometer at D10. Right panel: integrated intensities of the ε-Fe_2O_3 nanocrystals in the powder diffraction experiment [1] and present data measured from the single-crystalline thin film.

Temperature-dependent neutron diffraction data measured for the most intense (002) peak is shown in Fig. 2. Interestingly, we have found non-monotonous evolution of the diffraction intensity, that exhibits a dome-like shape with a maximum at T = 130 K. This behavior correlates

with the magnetization trend measured by SQUID [3]. Therefore, the neutron diffraction experiment has directly shown this magnetization change is inherently corresponds to ε-Fe_2O_3 and not the other iron oxide polymorphs that might be present in the film. Intensity change of (002) peak can be ascribed to 1) re-orientation of the magnetic moment from the (ab)-plane towards the c-axis, or 2) antiferromagnetic canting of the spins in the (ab)-plane. Assuming the strong shape anisotropy in the film, the second scenario is more plausible. However, more detailed investigation is required to draw a conclusion. Such study will be possible with thicker ε-Fe_2O_3 films allowing the measurement of the less intense peaks.

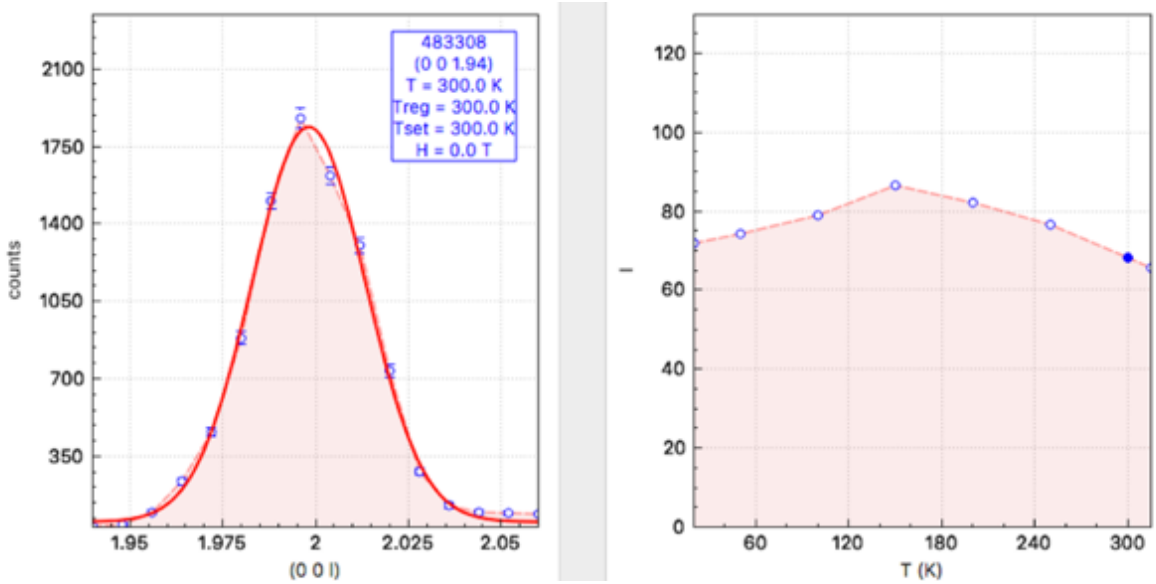

Figure 2 Left panel: (002) diffraction peak measured from 100 nm-thick ε-Fe_2O_3 film at 300 K. Right panel: temperature dependence of the integrated intensity of (002) peak.

References

[1] Gich, M., et al. *Applied Physics Letters* 96, no. 11, 112508 (2010).
[2] Suturin, S. M., et al. *Physical Review Materials*, 2, 7, 073403 (2018).
[3] Ukleev, V., et al., *Scientific reports* 8.1: 8741 (2018).