Experimental report

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Proposal: **5-54-226** **Council:** 10/2016

Title: Neutron reflectometry of exotic epsilon-Fe2O3 thin films

Research area: Physics

This proposal is a new proposal

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Samples: Fe2O3

Instrument	Requested days	Allocated days	From	To
D17	8	3	11/02/2017	14/02/2017
SUPERADAM	8	0		

Abstract:

The magnetic layers of various iron oxides on the GaN semiconductor surface are supposed to present interest from the point of view of potential use in design of novel (opto-) electronic and spintronic devices. In present work we propose to investigate exotic metastable iron oxide epsilon-Fe2O3 phase grown on GaN layer. This films exhibit interesting magnetic and electric properties, such as high coercivity at room temperature and ferroelectric behaviour. The magnetisation loops measured by SQUID leaving us to suggest the existence of two magnetic sublayers presented in the film. A pilot measurement of neutron reflectometry in zero magnetic field revealed the formation of 10 nm-thick interfacial layer between the iron oxide film and GaN. Now we want to obtain layer-specific magnetization loops by polarized neutron reflectometry to specify the origin of double-hysteresis behaviour. This knowledge will help us to improve the homogeneity of epsilon-Fe2O3 layers which are extremely promising for future applications.

5-54-226 Experimental report

Neutron reflectometry study of metastable iron oxide ε-Fe_2O_3 layers produced by laser beam epitaxy

Polarized neutron reflectometry (PNR) experiment has been performed at D17 instrument in the polarized time-of-flight (TOF) mode. The temperature and magnetic field at D17 instrument was controlled by 7 T vertical field cryomagnet with from single-crystalline sapphire windows. Neutrons with wavelengths $4 < \lambda < 16$ Å were used in the time-of-flight mode to provide the maximal incoming flux but avoid the possible problems with polarization efficiency. Polarization of the direct beam was measured by analyzer for each value of magnetic field used in the experiment. It was found that the polarization changes from P_0=98.8% at B = 50 mT to P_0=97.5% at B = 2 T. The experiment was performed without polarization analysis, i.e. R^+ and R^- components of reflectivity were acquired. Reflected beam was detected by two-dimensional He^3 position-sensitive detector. The amplitudes of PNR curves were corrected for polarization efficiency, reflected beam divergence and wavelength resolution.

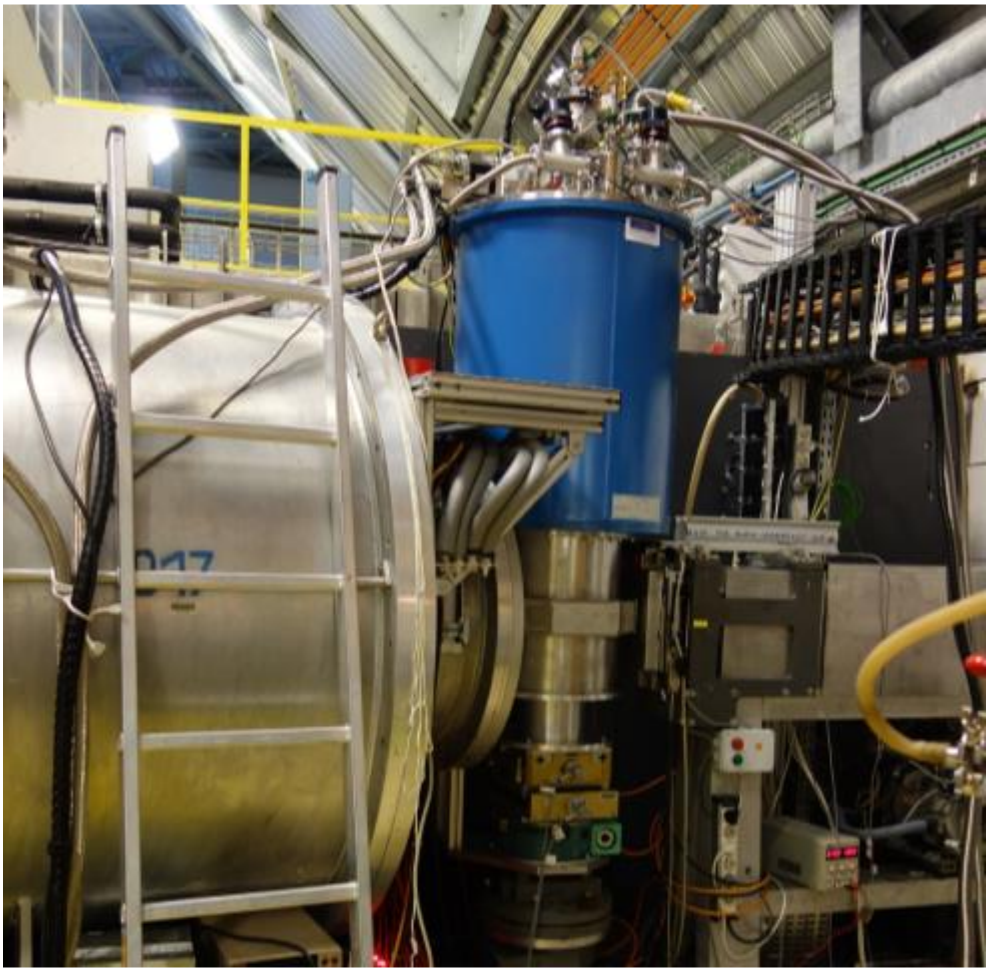

Figure 1 View of the D17 polarized neutron reflectometer equipped with the 7T vertical field cryomagnet.

PNR experiment has been performed for layer-resolved measurement of magnetization distribution inside the heterostructure. PNR curves $R(Q_z)$ measured at temperatures T=300 K and T=10 K are shown in Fig. 1a are multiplied by Q_z^4 to compensate Fresnel decay and provide an access to all the features of reflectivity function. Note, that all the curves are shifted along vertical axis for clarity: the multiplication factor is indicated for each curve by the text of the same color. The splitting of $R^+(Q_z)$ and $R^-(Q_z)$ is proportional to the net magnetization of the film along the field direction. Because of the relatively small magnetic moment of ε-Fe_2O_3 the splitting is hardly distinguishable at T=300 K, but clearly visible at the lowest temperature T=10 K in applied magnetic field of B = 2 T.

For neutron reflectometry the buffer GaN layer with thickness of 6 μm can be considered as a bulk substrate. Thus, reflectivity data was fitted assuming the model consisted of GaN, transition layer (interface) and ε- Fe_2O_3 layer, consequently. The initial model contained the table values of scattering lengths and densities and nominal thickness of iron oxide layer; then the densities and thicknesses of Fe_2O_3 and interface layer were allowed to vary freely in the fitting routine. Iron oxide density is reduced by 5% and GaN density is reduced by 1.5% compared to the calculated values. The minimum of the fitting algorithm (goodness of the fit χ^2=1.97) corresponds to the model containing interface layer with thickness of d_i=18 ± 3 Å and roughness σ_i=36 ± 10 Å between iron oxide and GaN buffer.

The initial input if magnetic moment of iron oxide layer was estimated from magnetometry data. PNR curves measured at applied magnetic fields 50 mT, 500 mT, 1000 mT and 2000 mT were fitted simultaneously with the same structural parameters while only magnetic contribution to SLD was ρ_m varied. Both nuclear and magnetic SLDs at the interface are reduced compared to ε-Fe_2O_3 layer (Fig. 1). Interface layer is also exhibiting reduced magnetization compared to the main part of the iron oxide layer (Fig. 2).

Temperature dependence of the absolute value of magnetic moment measured by PNR is corresponds to the magnetization data measured by SQUID magnetometry giving saturation moments for the main layer of $\mu=0.7\mu_B$ and $\mu=0.3\mu_B$ at T=10 K and T=300 K, respectively.

We suppose, that the interfacial layer consist of the $Ga_{(1-x)}Fe_xO_3$ due to the thermal degradation of the GaN during the ε-Fe_2O_3 growth at the temperature 850° C, which is close to the temperature of the thermal degradation of gallium nitride films.

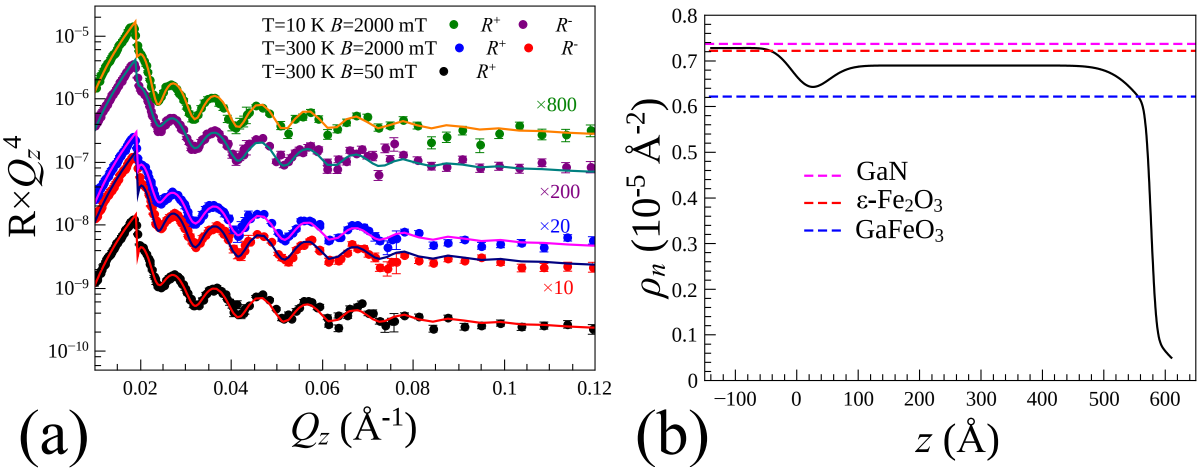

Figure 2 (a) PNR curves measured at (a) T=300 K B = 50 mT, B = 2000 mT, and at T=10 K B = 2000 mT; (b) T=10 K B = 50 mT and T=10 K B = 100 mT. The data is represented in form RQ4z to provide the smallest features of reflectivity function and outline the quality of the fit. Symbols are representing the experimental data while the solid lines are calculated. For clarity of curves are shifted by factors mentioned by the same color above or under the curve. (b) Nuclear SLD profile obtained from the fitting routine. Coordinate z = 0 Å corresponds to the top surface of GaN layer.

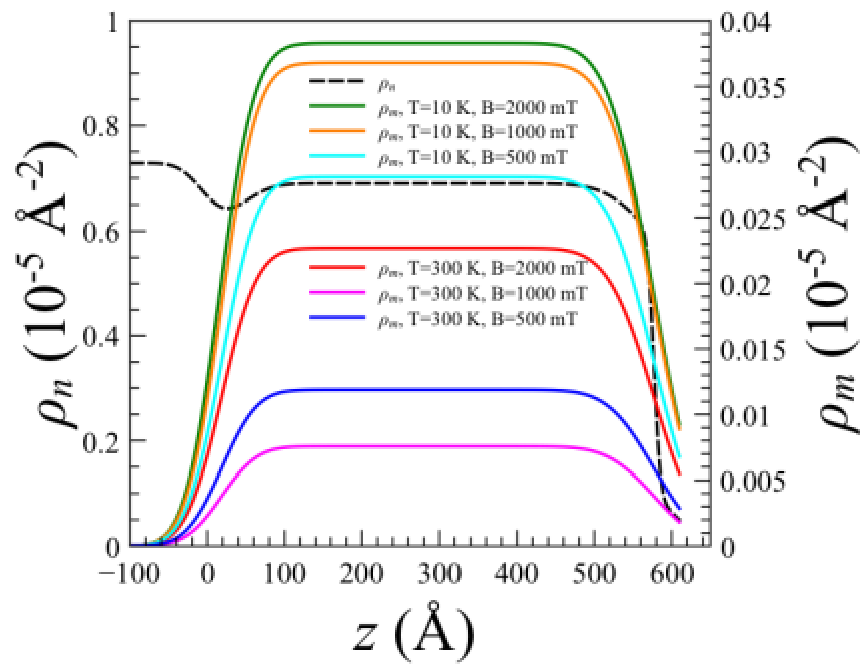

Figure 3 SLD profiles obtained from the fit of experimental PNR data. Left axis corresponds to the nuclear SLD (black profile line), right axis corresponds to the magnetic SLD (color lines).