Experimental report

Proposal: CRG-2507 Council: 4/2018

Title: Layer Integrity in Composite Magnetoelectric Thin Film Stacks Studiedby

Polarized Neutron Reflectivity

Research area: Materials

This proposal is a resubmission of 5-54-271

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Samples: NiFe2O4 on LAO

| Instrument | Requested days | Allocated days | From | To |
|------------|----------------|----------------|------------|------------|
| SUPERADAM | 7 | 5 | 20/10/2018 | 22/10/2018 |
| | | | 20/07/2019 | 23/07/2019 |

Abstract:

We propose to use specular polarized neutron reflectometry (PNR) to study the structural and magnetic integrity of the ferromagnetic component in composite magnetoelectric multilayer stacks. The stacks consist of a varying number of layers of ferromagnetic NiFe2O4 and ferroelectric (K,Na)NbO3 (KNN), deposited by atomic layer deposition (ALD). The thickness of the individual layers are from 5 - 20 nm.

The samples are made to study magnetoelectric coupling in composites of magnetostrictive/ferromagnetic and ferro-/piezoelectrics. This is an alternate way of producing multiferroic materials systems that can have enhanced properties compared to single-phase multiferroic materials, where either the magnetic- or electric response is too low for application.

A crucial parameter in a composite system such as this is the integrity of the individual layers, both magnetically and morphologically.

How are the magnetic layers ordered in relation to each other? How defined is the morphology of the interfaces between the layers? These are questions we hope to answer by this PNR-study.

Layer Integrity in Composite Magnetoelectric Thin Film Stacks Studied by Polarized Neutron Reflectivity

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In this study we have investigated the magnetic properties of NiFe₂O₄ thin films deposited by atomic layer deposition (ALD) on various substrates. Polarized neutron reflectometry (PNR) at SuperADAM @ ILL was used to probe the magnetic properties of the thin films and to investigate the interface region towards the substrates. Our investigations show that the thin films are of high quality, with low roughness, and with expected magnetic properties, Figure 1. We found a one-layered model to best describe our samples, indicating a homogenous magnetization though the films, Figure 2. Further, the magnetic properties was observed to vary with substrate and orientation. These properties are for some samples listed in Table 1. Especially the good magnetic properties of (111)-orientated NiFe₂O₄ on (001)-orientated Al₂O₃ is an influential result for our coming research. In addition, we conducted field scans near the critical edge where the *uu*- and *dd*-splitting is most prominent to determine the cohesive field and the remanence of the magnetic thin films, Figure 3. The results from this beamtime is of great value for our research towards magnetoelectric thin film stacks, and will serve as input for design of future model systems. We also expect to publish these results in a soon upcoming paper detailing the magnetic properties of ALD NiFe₂O₄ epitaxial films.

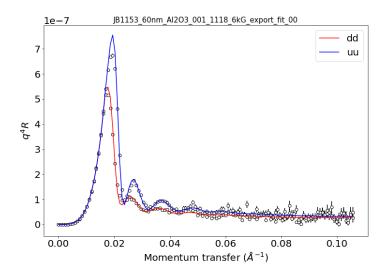


Figure 1: Fitted PNR profile of 60 nm NiFe₂O₄ on (001)-orientated Al₂O₃.

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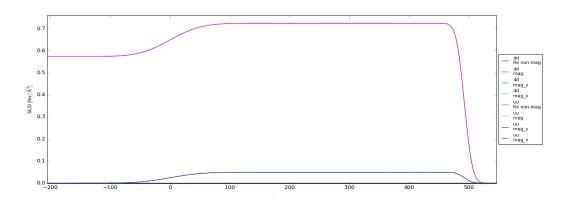


Figure 2: SLD profile of 60 nm NiFe₂O₄ on (001)-orientated Al₂O₃ from PNR profile fitting.

Table 1: Extracted magnetic assets of magnetic NiFe₂O₄ thin films with various thickness. Magnetization under various fields are given in μ_B per atom. * indicate values from field scan.

| Thickness | 60 nm | 60 nm | 60nm | 11nm |
|----------------|---------------|-----------|-------|-----------|
| Substrate | Al_2O_3 | Al_2O_3 | MgO | Al_2O_3 |
| Orientation | (001) | (012) | (001) | (001) |
| 6000 G | 1.34 | 1.36 | 0.32 | 0.62 |
| 50 G | 0.81 | | 0.01 | |
| 230 G | 0.56 | | | |
| 270 G | | 0.14 | | |
| Coercive field | 230 G | 270 G | | |
| Remanence | 60 %, (81 %)* | (83 %)* | 3 % | |

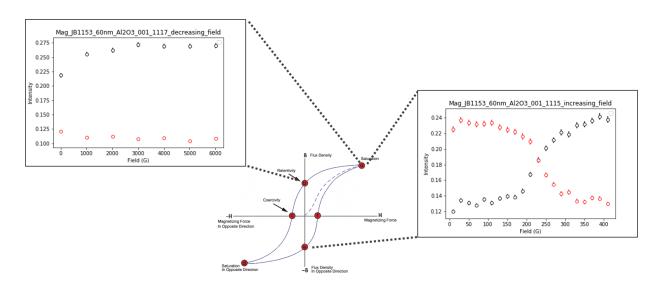


Figure 3: Illustration of conducted magnetization scans to determine the cohesive field and remanence of the samples. The figures shows measured data for $60 \text{ nm NiFe}_2O_4 \text{ on } (001)$ -orientated Al_2O_3 .