Experimental report

05/02/2016

Proposal: **5-54-196**

Council: 4/2015

Title: Interfacial spin clusters in CoFe/IrMn exchange bias multilayers

Research area: Physics

This proposal is a new proposal

Main proposer: **Thomas SAERBECK**

Experimental team: Thomas SAERBECK

Local contacts: Thomas SAERBECK

Samples: [NiCr(6nm)/IrMn(10nm)/CoFe(3nm)]x30
[Cu(6nm)/IrMn(10nm)/CoFe(3nm)]x30
[Cu(6nm)/IrMn(10nm)]x30
[NiCr(6nm)/IrMn(10nm)]x30

Instrument	Requested days	Allocated days	From	To
D17	6	5	09/09/2015	14/09/2015

Abstract:

We propose to investigate the magnetic depth profile of an exchange biased (EB) CoFe/IrMn multilayer using polarized neutron reflectometry (PNR). The York Model developed by us accounts for the general behaviour of the effect in polycrystalline thin films by the thermal stability of bulk AF grains. Nevertheless, details of coercivity and training effects are not understood and require further investigation to control the technologically highly important exchange bias. Our recent work suggests interfacial spin clusters to be responsible for the coercivity enhancement. These clusters can be seen as fine particle systems with an anisotropy transmitted by the antiferromagnet. The size of the clusters is determined by the AF grain size. Using PNR we will investigate the presence and field reversal of these interfacial spin clusters in dependence on AF grain size and anisotropy, which will determine the absolute magnitude of the contained magnetization. This study will therefore lead to a detailed understanding of the cause of coercivity enhancements and training effects.

Interfacial Spin Clusters in CoFe/$IrMn_3$ Exchange Bias multilayers

Polarized Neutron Reflectometry Beam Time on D17, September 2015, 5-54-196

Motivation:

Exchange bias (EB) belongs to the most important magnetic exchange coupling phenomena due to its technological importance in state-of-the-art storage and sensor devices. Still not all aspects of the effect are understood and investigations are ongoing to enhance the magnitude or tunability of the magnetic loop shift. CoFe/$IrMn_3$ bilayers belong to a technological important combination of ferromagnetic/antiferromagnetic (FM)/(AF) materials due to the high observed EB values, corrosion resistance and AF properties of $IrMn_3$. Our previous studies on CoFe/$IrMn_3$ exchange bias systems show that the EB magnitude, coercivity enhancement and training effect depend crucially on the AF grain size and anisotropy [1, 2, 3]. Interfacial spin clusters are identified as the main cause for the observed magnetic hysteresis loop changes. The size of these spin clusters depends on the AF grain size, while the anisotropy of the AF determines the irreversibility of the clusters and therefore the coercivity changes.

Using suitable seed layers, the crystalline texture of $IrMn_3$, and therefore the AF grain size, can be controlled. In experiment 5-54-196 we studied the influence of NiCr (=> high IrMn texture) and Cu (=> low IrMn texture) seed layers on the magnetic behavior of the AF. The goal was to determine if changes in the interfacial magnetization become observable in different external magnetic field and field cooling conditions by using polarized neutron reflectometry.

Experimental details:

Two samples were measured over the 5 day beamtime at different temperatures and external fields, following different field cooling procedures. In order to be most sensitive to small changes at the interface between CoFe and IrMn, we prepared multilayers with 20 repetitions of the sequence Seed/IrMn/CoFe, "Seed" being either NiCr or Cu, on Si substrates. All samples were capped with a Ta protective layer to prevent oxidation. The thickness of each layer was chosen to be equal (6 nm), which leads to a vanishing of the third order Bragg reflection. Roughness and interfacial magnetization break the periodicity of the structure and the third order Bragg peak becomes observable. Therefore, here the highest sensitivity to interfacial roughness and magnetism is achieved.

In order to set the AF spin orientation of each AF grain, the samples must be heated well above room temperature (RT) and field cooled in the setting field to the measurement temperature. At the time of the experiment, no magnetic field environment capable of a temperature range of 500 K – 3 K was available. Therefore, the samples were heated off-line in a custom build electromagnet setup and then transferred to the cryomagnet on the beamline. This involves a transfer through low and uncontrolled magnetic fields at RT.

Results:

Figure 1 shows the polarized reflectivity of **Si-substrate/[NiCr(6)/$IrMn_3$(6)/CoFe(6)]×20/Ta(6)** to a maximum Q-value of 0.27 $Å^{-1}$ at temperatures of 300 K and 100 K in an external field of +0.5 T. The two profiles show excellent agreement up to the 8^{th} order Bragg peak. The sample quality therefore appears sufficient to be sensitive to the interfaces, although significant intensity in the 3^{rd} and 6^{th} order Bragg peak is observed due to interfacial structural and magnetic roughness. A subsequent field reversal to -0.5 T did not reveal any differences in the magnetic profile. This sample was not heated externally and therefore there is a high possibility that the AF spin structure is not homogeneously set by field cooling from 300 K to 100 K.

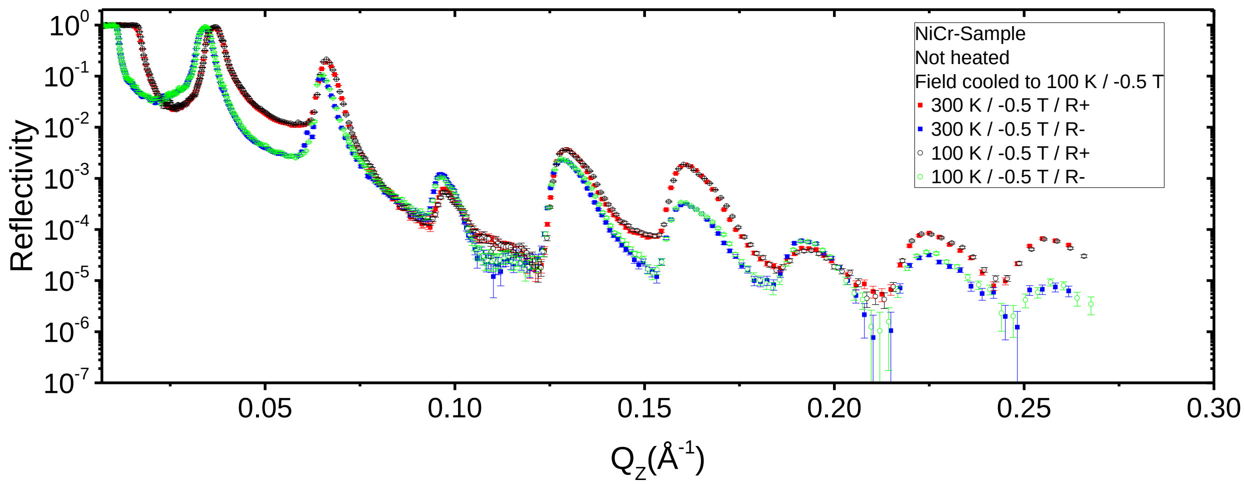

Figure 1: ***[NiCr/IrMn₃/CoFe]×20*** *comparison of* ***300 K*** *and* ***100 K*** *after FC in* ***-0.5 T****.*

The **Si-substrate/[Cu(6)/IrMn₃(6)/CoFe(6)]×20/Ta(6)** sample was heated externally in the electromagnet first to 180°C and subsequently to 200°C. After each annealing step, the sample was field cooled to 100 K and the reflectivity in positive (parallel to the field cooling direction) and negative (antiparallel to the field cooling direction) field recorded. Figure 2 shows a comparison of the two heating cycles at 100 K. Differences are observed at several Q-values of the reflectivity, indicated by the black arrows. These differences were confirmed by additional measurements showing the high reproducibility of Figure 1. Unfortunately, they do not coincide with locations where a change due to spin reorientation at the interface is expected and the changes might well be of structural origin.

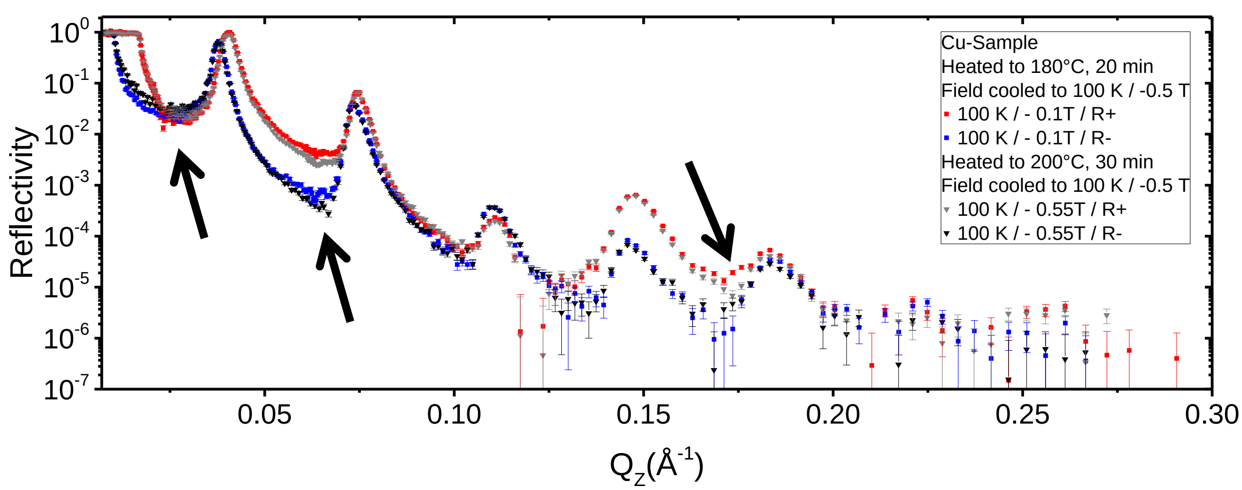

Figure 2: ***[Cu/IrMn₃/CoFe]×20*** *comparison of* ***-0.1 T FC from 180°C*** *and* ***+0.5 T FC from 200°C*** *at 100 K.*

Summary and conclusions:

In total, 11 R+ and R- reflectivities were recorded over the Q-range shown in Figure 1 and Figure 2. The analysis of all data is ongoing to determine the magnetic and structural effects of the annealing procedures and field reversals. For the moment, we did not observe a clear indication of interfacial magnetic differences after field reversal. This might well be connected to the additional complication of exposing the sample to zero field at room temperature while transferring to the instrument. Though the AF IrMn is thermally stable at 300 K, thin films, especially in multilayer form, with low anisotropy may break into domains, which would prevent the observation of interfacial spin clusters.

The multilayer samples prepared specifically for this experiment show sufficient quality to record the reflectivity over a large Q-range to 0.3 $Å^{-1}$. Changes in the reflectivity were observed upon annealing at 200°C. However, these were not at the Q-ranges were the highest interface sensitivity is expected. We are planning a follow-up experiment in which annealing on the beamline in a magnetic field will be imperative. Currently the steps for sample environment development are undertaken. The second part of the planned experiment looking at AF layers without the neighboring FM layer could not be performed due to time restrictions.

References:

[1] R. Carpenter, N.C. Cramp, G. Vallejo Fernandez, K.D. O'Grady, R. Carpenter, N.C. Cramp, G. Vallejo Fernandez and K.D. O'Grady, *Coercivity and interfacial spin clusters in exchange bias systems*, Journal of the Magnetics Society of Japan **38**, 61 (2014).

[2] R. Carpenter, G. Vallejo-Fernandez and K. O'Grady, *Interfacial spin cluster effects in exchange bias systems*, Journal of Applied Physics **115**, 17D715 (2014).

[3] N.P. Aley, G. Vallejo-Fernandez, R. Kroeger, B. Lafferty, J. Agnew, Y. Lu and K. O'Grady, *Texture Effects in IrMn/CoFe Exchange Bias Systems*, IEEE Transactions on Magnetics **44**, 2820 (2008).