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Depth-resolved studies of layered magnetic nanostructures using ⁵⁷Fe probe layers and Mössbauer spectroscopy

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ABSTRACT

An atomic-scale quantitative analysis of the structural and magnetic properties of surfaces, interfaces and complex nanostructures is of fundamental relevance for the development of new materials for spintronics. Studies of buried magnetic interfaces and depth-resolved measurements in layered magnetic nanostructures are particularly challenging, and the combination of conversion electron Mössbauer spectroscopy and/or nuclear resonant scattering of synchrotron radiation with isotopeenriched probe layers can be a powerful tool in this field.

The potential offered by the application of isotope-selective measurements for the study of Fe-based layered magnetic nanostructures is illustrated with our recent results on the investigation of depth-dependent spin structures and interfacial interdiffusion in exchange-biased ferromagnetic/antiferromagnetic bilayer systems and of an epitaxial magnetic system with perpendicular magnetic anisotropy, obtained from samples prepared with ultrathin ⁵⁷Fe probe layers placed at different depths during the growth processes, via molecular beam epitaxy or sputtering deposition.

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1. Introduction

For the development of new materials for spintronics, it is of fundamental relevance a detailed quantitative analysis of the structural and magnetic properties at surfaces, interfaces and along complex nanostructures, in an atomic scale [1]. In layered magnetic systems, there is a need for depth-dependent measurements, providing access to the study of buried magnetic interfaces. There are different experimental techniques that allow depthdependent characterization of both structural and magnetic properties, including neutron scattering and reflectometry, magnetic dichroism (XMCD), Mössbauer spectroscopy (MS), nuclear resonant scattering of synchrotron radiation (NRS), soft X-ray resonant Kerr rotation, soft X-ray resonant magnetic scattering. Among these techniques, isotope selective methods, like MS and NRS, offer a specific advantage, additional to chemical element selectivity, namely the possibility of separating out contributions coming from different crystallographic sites occupied by the same element. Depth-resolved measurements in layered magnetic nanostructures are particularly challenging, and the combination of conversion electron Mössbauer spectroscopy (CEMS) and/or NRS associated with the adequate growth of isotope-enriched probe layers allows a depth resolution of few Angstroms (monolayer (ML) regime), measurements on surfaces and on buried

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http://dx.doi.org/10.1016/j.jmmm.2014.04.035 0304-8853/© 2014 Elsevier B.V. All rights reserved. interfaces, the determination of the spin and local structure, degree of interdiffusion, and interface roughness on an atomic scale, being a very effective approach for the investigation of layered magnetic nanostructures, and, therefore, a powerful tool in this research field [2–7]. The recent developments in synchrotron-radiation-based MS in the energy domain add also new possibilities for the area [8].

In this paper, some of our recent results illustrating the potential offered by CEMS and NRS for the study of Fe-based layered magnetic nanostructures are reviewed. The focus is more specifically on depth-resolved studies using ⁵⁷Fe probe layers and isotope-selective measurements. The applications of these techniques is illustrated with our results on the investigation of depth-dependent spin structures, of interfacial interdiffusion in exchange-biased ferromagnetic (F)/antiferromagnetic (AF) bilayer systems, and of epitaxial magnetic systems with perpendicular magnetic anisotropy, obtained from samples prepared with ultrathin (a few Angstroms thick) ⁵⁷Fe probe layers placed at different depths during the growth processes, via molecular beam epitaxy or sputtering deposition.

2. Spin structure during magnetization reversal in Fe/MnF₂ exchange-coupled bilayers

We measured directly the depth-dependent Fe spin rotation upon magnetization reversal in exchange-coupled Fe/MnF₂ bilayers using nuclear resonant scattering of synchrotron radiation

from a ⁵⁷Fe-probe layer buried at different depths within the Fe film. Our results showed that the exchange-biased ferromagnetic layer develops a non-collinear spin structure along the film normal direction, reminiscent of a partial domain wall parallel to the Fe/MnF₂ interface [9].

The exchange bias (EB) effect, originating from the interface coupling between a F and an AF, gives rise to shifted hysteresis loop along the magnetic field (x-) axis [10–13]. This effect is used to set a reference magnetization direction in layered spin-valve devices and, therefore, is of great interest for many applications based on spintronics [1,14]. Despite the enormous technological impact and the intense research efforts, one of the main challenges to understand the microscopic mechanisms of EB is the investigation of the magnetic structure at the F/AF interface and its depth dependence perpendicular to the interface. Some EB models predict a spiraling AF spin structure perpendicular to the interface, and non-collinear spin structures in the F layer [12,15–18], but systematic experimental studies on that aspects are scarce [19,20].

Although different techniques allow the study of buried magnetic interfaces, experimentally, it is always difficult and challenging to obtain a detailed description of the depth dependent magnetic structure at the atomic scale. Previous conversion electron Mössbauer spectroscopy (CEMS) studies using ⁵⁷Fe probe layers at different depths of the F in exchange-biased Fe/MnF₂ did not indicate depth-dependent spin structure in Fe [21,22]. However, due to the inherent difficulties of detecting electrons while applying strong external magnetic fields, these CEMS measurements were conducted in remanence [21,22]

For Fe-containing nanostructures, the use of coherent nuclear resonant scattering (NRS) of synchrotron radiation technique with buried ultrathin (a few Angstroms thick) ⁵⁷Fe-rich probe layers in combination with wedge-type Mössbauer inactive ⁵⁶Fe layers, is a powerful approach to access depth-dependent properties in magnetic films and multilayers, with high sensitivity and high lateral resolution [23,24]. We applied NRS to conduct direct measurements of the depth-dependent Fe spin rotation in an exchange-coupled Fe/MnF₂ bilayer [9]. A SQUID magnetization loop at 10 K for such F/AF bilayer is shown in Fig. 1, where an exchange bias field (horizontal shift, H_E) of -80 Oe is observed.

The Fe/MnF₂ sample of 40 Å Cu cap/70 Å Fe (60 Å ⁵⁶Fe+10 Å ⁵⁷Fe wedge)/520 Å MnF₂(1 1 0)/160 Å ZnF₂(1 1 0) buffer layer was grown on MgO(1 0 0) [21]. The 10-Å thick ⁵⁷Fe probe layer (95.5% enrichment) was inserted between two ⁵⁶Fe wedges, as illustrated in Fig. 2(a), together with geometry details of the NRS experiments. The MnF₂ film grows as a quasiepitaxial layer with (1 1 0) orientation, and forms a compensated AF surface with the Mn spins in the interface plane, and the Fe layer is polycrystalline [21]. CEMS results (not shown) indicate a fully in-plane magnetized



Fig. 1. SQUID magnetization loop at 10 K for a 70 Å Fe+⁵⁷Fe/520 Å MnF₂ sample, after field cooling in a magnetic field of 2.0 kOe applied along the MgO[0 0 1] direction, from 150 K (adapted from Ref. [9]).



Fig. 2. (a) Schematic illustration of the wedge sample and NRS experimental geometry: the incident photon beam, reflected at an angle of 4 mrad relative to the surface, is oriented along the *x* direction (note that all the arrows are in the sample plane. (b) Typical 10 K NRS time spectra measured in decreasing magnetic fields with the 14.4 keV photons probing the ⁵⁷Fe center position. The red solid lines are least-squares fits to the experimental data. The +2000 Oe cooling field (CF) (applied from 150 to 10 K) and the sweeping field *H* were applied in plane along the MgO[1 0 0] (*x*) direction. *a* is the angle between the in-plane Fe spin direction and the +*x* direction (adapted from Ref. [9]).

⁵⁷Fe layer, in agreement with previous works [21,22]. Magnetic hysteresis loops below the MnF₂ Néel temperature (T_N =67 K) were measured using superconducting quantum interference device (SQUID) magnetometry. EB was always established by field cooling (FC) the sample from 150 K to 10 K in an external field (*H*) of 2.0 kOe applied in-plane along the MgO[0 0 1] direction (*x*-direction, Fig. 2(a)). Measurements were conducted between +2000 Oe and -2000 Oe along the same direction, and a H_E of -90 Oe was obtained at 10 K (Fig. 1).

The NRS experiments were performed at beamline ID18 of the European Synchrotron Radiation Facility (ESRF), with the 14.4 keV photon beam along the MgO[0 0 1] direction (*x*, Fig. 2(a)), and in grazing incidence of 4 mrad. A cryomagnet system allowed the fine adjustment and scanning of the sample in front of the 50 μ m (vertical) \times 300 μ m (horizontal)) photon beam, and, consequently, provided the depth selectivity of the measurements along the ⁵⁷Fe wedge, by probing regions at different distances from the F/AF

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