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The use of Lorentz microscopy for the determination of magnetic reversal mechanism of exchange-biased $Co_{30}Fe_{70}$/NiMn bilayer

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ABSTRACT

Lorentz transmission electron microscopy (LTEM) combined with *in-situ* magnetizing experiments is a powerful tool for the investigation of the magnetization of the reversal process at the micron scale. We have implemented this tool on a conventional transmission electron microscope (TEM) to study the exchange anisotropy of a polycrystalline $Co_{35}Fe_{65}$/NiMn bilayer. Semi-quantitative maps of the magnetic induction were obtained at different field values by the differential phase contrast (DPC) technique adapted for a TEM (SIDPC). The hysteresis loop of the bilayer has been calculated from the relative intensity of magnetic maps. The curve shows the appearance of an exchange-bias field reveals with two distinct reversal modes of the magnetization: the first path corresponds to a reversal by wall propagation when the applied field is parallel to the anisotropy direction whereas the second is a reversal by coherent rotation of magnetic moments when the field is applied antiparallel to unidirectional anisotropy direction.

1. Introduction

The observation with a nanometer of the spatial resolution of the magnetic configurations is nowadays of great interest for many engineering applications of magnetic materials. Lorentz transmission electron microscopy (LTEM) is one of the techniques which enable analysis of local magnetic properties. This technique allows *in-situ* observations of the domain structure of a magnetic material at different magnetic field values [1]. Classical LTEM relies on the fact that an electron beam passing through an area with a component in magnetic induction perpendicular to its trajectory will be deflected by the Lorentz force. The magnetic induction arises either from the magnetization in the sample itself or the nonzero divergence of the magnetization which leads to stray fields exterior to the sample. In the transmission electron microscope (TEM), the deflection of the electron beam results from the perpendicular component of magnetic induction averaged on the electron path (sample plus vacuum).

Two methods can be distinguished to image the magnetic-domain configuration in LTEM: the Fresnel mode and the Foucault mode. The Fresnel contrast appears when the Lorentz lens is defocused; the image intensity increases at the position of some domain walls and decreases at the position of others. In Foucault mode, a contrast aperture is introduced in the back focal plane of the Lorentz lens and positioned in order to intercept electrons which have passed through one set of domains magnetized in a given direction. The contrast corresponds to a dark-field image where only these set of domains appear dark. An extension of this technique is the acquisition of a series of Foucault images in order to get a magnetic map. This technique, so-called differential phase contrast (DPC) was initially developed [2] on a scanning transmission electron microscope (STEM) and latter adapted on a conventional transmission electron microscope [3]. This latter technique, so-called SIDPC (SI for Series of Images), records a series of images by moving the aperture or tilting the incident beam in $\pm X$ and perpendicular $\pm Y$ directions. It has been shown that adding the images in each direction produces two images linearly proportional to B_X and B_Y [3]. The magnetic components are then computed relative to the origin of the incident beam (no magnetic deflection) and the vector map can be easily displayed from the two images. Magnetic imaging of the remnant state is performed with the specimen in a field-free region in the TEM column by turning off the main objective lens and using another lens (so-called Lorentz lens) placed below the sample as imaging lens. In order to perform *in-situ* magnetization for reversal process analysis, the in-plane field applied on the sample can be created by tilting the specimen holder in the axial magnetic field of the objective lens (either residual field or field obtained by switching on the objective lens). These fields have been calibrated with a Hall-effect sensor. Major advances in the comprehension of

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magnetic/antiferromagnetic coupling response to an external field have thus been achieved using LTEM: temperature dependence of the exchange-bias field [4,5], implementation in spin valves [5], microstructure of the ferromagnetic layer [6,7], domains formation and domain walls propagation [4,8], or growth conditions [8,9].

The aim of this letter is to demonstrate the application of SIDPC for investigations at different magnetic field values of a system, following the complete hysteresis cycle of the film. More precisely, we use a technique to reconstruct the full hysteresis loop from the induction maps, giving access to magnetic properties as it was previously described by Daykin et al. in Ref. [6]. We focused here on the exchange anisotropy coupling between a ferromagnetic (F) layer and an antiferromagnetic (AF) layer and at the same time on the mechanism of magnetization reversal. The direct exchange coupling in AF/F bilayer has attracted great interest due to its importance in the spintronic devices as spin valves [10–12] or magnetic tunnel junctions [13]. This phenomenon discovered by Meiklejohn and Bean over 50 years ago [14,15] creates a bias field (H_B) corresponding to a shift of the hysteresis loop of the F layer and an increase of its coercive field (H_C). This behaviour is enhanced when the AF/F bilayer has been cooled under the application of a magnetic field through the ordering temperature of the AF, so-called Néel temperature [16].

2. Material and methods

The system studied in this work is a polycrystalline $Co_{35}Fe_{65}$ (70 nm)/NiMn (50 nm) bilayer. NiMn is widely used as an AF layer for its high crystalline anisotropy field and high blocking temperature [17,18]. Co_xFe_{1-x} is a promising candidate for future recording media [19] and RF applications [20–22] due to its high saturation magnetization. For the LTEM observation, the bilayer has been directly deposited by DC sputtering on carbon-coated thin film. Growth conditions and post-annealing treatment are described elsewhere [20,23].

The LTEM experiments were performed on a conventional TEM JEOL 3010 working at 300 kV with a LaB_6 gun and equipped with a gatan imaging filter (GIF). To reach the field-free conditions, the microscope was operated in low-mag mode using the objective minilens as the Lorentz lens, the main objective lens being switched off. The selective area aperture acts as contrast aperture. In order to place this aperture in a real back focal plane to get good Foucault images, each intermediate lens has been set in a free lens mode [24]. An acquisition script is used to drive the tilt series across the aperture [3] and to record 4 series of 20 Foucault images. The 512×512 pixel-size images are acquired with a CCD camera through a gatan image filter operated in a zero-loss mode with a 10 eV energy-selecting slit. In this case, the signal-to-noise ratio is improved compared to unfiltered images by removing the inelastic scattered electrons [25], and the magnification is increased by the GIF lenses. Due to long exposure times (10 min), sample drift may introduce artefacts. We have then developed a software using gatan digital micrograph to correct the drift on the series of images before computing B_X and B_Y components of magnetization. Finally the 2D-integrated induction map is processed.

The *in-situ* magnetization can be done in many ways using a specialized magnetization sample holder [6,26]. In our case, we have used the residual vertical magnetic field of the objective lens (measured to be of 305 Oe by a Hall probe). The specimen holder was tilted up to $|\alpha_{max}| \leq 21.5^\circ$ in order to apply a controlled in-plane magnetization between ± 110 Oe on the sample and so to achieve a magnetic reversal processes. The 0.1° angle uncertainty of the stage leads to an accuracy of the applied field of 0.5 Oe. In this method, a substantial out-of-plane magnetic field is applied to the sample but its effect on the magnetization is highly reduced by the shape anisotropy and can be neglected in our experiment.

3. Results

Fig. 1 presents a set of integrated magnetic-induction maps of the $Co_{35}Fe_{65}$/NiMn bilayer obtained by SIDPC at different field

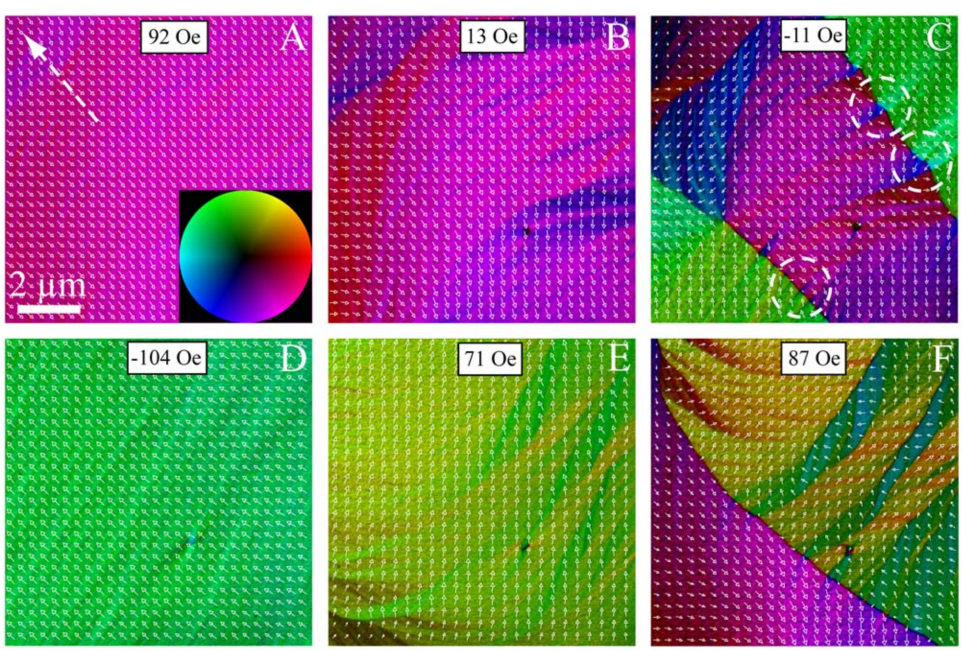

Fig. 1. Six magnetic maps obtained by DPC on the $Co_{35}Fe_{65}$(70 nm)/NiMn(50 nm) bilayer. On image (A) the unidirectional anisotropy direction is represented by the dashed white arrow. The inserted color wheel indicates the direction of magnetization by the color and the intensity corresponds to the strength of the magnetic signal. On image (C) some cross-tie walls are surrounded.