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Study of exchange bias effect in a patterned Fe/Pt multilayer with the thermal annealing

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A B S T R A C T

The present work reports the preparation of 5 μm wide and 10 μm grating periodicity patterned $[Fe(2.0nm)/Pt(2.5nm)]_{X10}$ multilayer and its thermal annealing behavior. Intermixing across the Fe and Pt layers is observed with vacuum annealing at 650 K resulting in the formation of disordered face centered cubic (FCC) and ordered $L1_0$ face centered tetragonal (FCT) FePt phases. The longitudinal magneto-optical Kerr effect (L-MOKE) hysteresis loop, measured from the single 5 μm FePt stripe shows double loop behavior indicating the presence of two magnetic phases, one with the low coercivity (H_C) and the second one with the higher H_C values. This could be due to the presence of disordered FCC and ordered $L1_0$ FCT phases of FePt, known to be soft and hard magnetic phases. Conversion electron Mössbauer spectroscopy of the annealed sample shows the presence of FCC and FCT FePt phases corroborating the x-ray diffraction and MOKE measurements. The hysteresis loop of the soft phase shows the exchange bias effect with the remnant state of the hard magnetic phase as initial condition akin to the exchange bias as observed in conventional ferromagnetic - antiferromagnetic bilayers.

1. Introduction

In order to overcome the thermal instability due to super-paramagnetic limit of the magnetic nano-particles in magnetic recording media the magnetic system should have huge magnetic anisotropy [1]. FePt is one such system which is being extensively explored in the literature as it exhibits huge perpendicular magnetic anisotropy (PMA) [1–3]. The PMA is due to the chemical ordering ($L1_0$ phase) of FePt along the out-of-plane c-axis in tetragonal structure (FCT) [2,3]. The easy axis is perpendicular to the iron/platinum layer stacking of the $L1_0$ structure and magnetic anisotropy depends strongly on the extent of the tetragonal $L1_0$ ordering. Many reports are published in literature dealing with issues such as reducing the $L1_0$ ordering temperature, increasing PMA etc [2–8]. Lowering of $L1_0$ ordering temperature, formation of hard and soft magnetic FePt phases etc., are observed with thermal annealing of Fe/Pt multilayers [5–8]. Composite films consisting $L1_0$ FePt as hard magnetic and other soft magnetic phases (like Fe, Co etc.,) are also being extensively studied in recent literature from the context of exchange spring phenomena [9,10]. Apart from this exchange spring phenomena, an exchange bias phenomena is also reported in such composite films [11,12]. In FePt/Fe system, the unidirectional anisotropy of the soft magnetic layer (Fe) is observed with the remnant state of the hard magnetic layer ($L1_0$ FePt) as initial condition [12].

On the other hand it is well known that magnetic properties hugely depend on the shape and size of the magnetic elements. For example magnetic properties like magnetic anisotropy, magnetization reversal process, switching fields etc., hugely depend on the dimension and geometry of the magnetic element [13–17]. Hence preparation of the magnetic films on the patterned substrates is one of the extensively adopted method to tune the magnetism due to shape anisotropy. In view of this, the present work reports the preparation and thermal annealing study of the patterned Fe/Pt multilayer. One of the main objective is to explore the possibility of exchange bias phenomena in such Fe/Pt multilayers with the thermal annealing. With the thermal annealing of the Fe/Pt multilayer, both the disordered and ordered FePt phases are

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expected to form and hence would act as a soft and hard magnetic phase respectively [8]. The second motivation is to study the effect of the shape anisotropy due to patterning on the growth of the FePt with large in-plane magnetic anisotropy i.e., with in-plane c-axis chemical ordering.

2. Experimental

Patterned silicon substrate with the 5 μm width and 10 μm periodicity is prepared with the standard UV lithography technique using AZ4903 photo-resist and in-house developed 405 nm UV broadband light setup. On this patterned substrate, $[Fe(2.0nm)/Pt(2.5nm)]_{\times 10}$ multilayer with the 10 nm Pt buffer layer is deposited using ion beam sputtering method at a base vacuum of 1.3×10^{-7} mbar. For deposition, a beam of argon ions are used to sputter ^{57}Fe enriched Fe, Pt targets using Kaufman-type hot-cathode ion source. After deposition, the photo-resist was etched out with the standard lift-off procedure. The as-deposited multilayers are annealed in a vacuum of 5.3×10^{-6} mbar for 1 h. X-ray reflectivity (XRR), rocking curve and grazing incidence x-ray diffraction (GIXRD) measurements are carried out using Bruker D8-Discover system, equipped with Cu source (0.154 nm) and LynxEye detector, for the estimation of thickness and structural information respectively. The incident x-rays are parallel to the length of the pattern for XRR and GIXRD measurements. For rocking curve measurements the incident x-ray are perpendicular to the length of the pattern. Conversion electron ^{57}Fe Mössbauer spectroscopy (CEMS) is recorded using the conventional PC-based ^{57}Fe Mössbauer spectrometer in the back scattering geometry with the homemade continuous gas flow detector using *He* and CH_4 gas mixture. The Mössbauer data is fitted by using NORMOS-DIST/SITE programs [18]. Magnetic domains were imaged by the high resolution magneto-optical Kerr microscope (M/s Evico Magnetics, Germany) and the magnetization loops were measured simultaneously by deriving the magnetization signal from the average domain image intensity [13]. One of the stripe (5 μm width) is selected as a region of interest (ROI) for measuring the local hysteresis loops. ROI is implemented by reducing the number of pixels in the CCD camera array of the Kerr microscope system. The magneto-optical Kerr effect measurements are carried out in longitudinal mode (L-MOKE). It is to be noted that the x-ray and ^{57}Fe Mössbauer measurements are from the entire sample averaged out over many patterned stripes.

3. Results and discussions

Optical image of the prepared $[Fe/Pt]_{x10}$ multilayer after lift-off process is shown in the inset of Fig. 1. One can clearly see the well defined periodic grating pattern formation. In fact, the grating periodicity is also confirmed from x-ray measurements as shown in Fig. 1. Fig. 1 shows the rocking curve measurements of the pristine and 650 K annealed patterned Fe/Pt multilayer sample and one can clearly see well defined grating peaks that confirms well defined periodic structure of the grating. The periodicity (d) of the grating obtained from rocking curve measurement using $\Delta q_x = \frac{2\pi}{d}$ is in good agreement with that of optical image data [19].

Fig. 2 shows the x-ray reflectivity (XRR) pattern of the as-deposited film and the data is fitted with the Parratt formalism [20]. The obtained thickness values of the Fe and Pt layers are about 2.0 ± 1 nm and 2.5 ± 1 nm respectively. Bottom inset of Fig. 2 shows the schematic diagram of the patterned multilayer for better visualization. From the best fit of the XRR data, electron density profile is obtained and is shown in the top inset of Fig. 2, which confirms a well defined periodic structure of the as-deposited Fe/Pt multilayer. Thus obtained sample is vacuum annealed at 650 K to

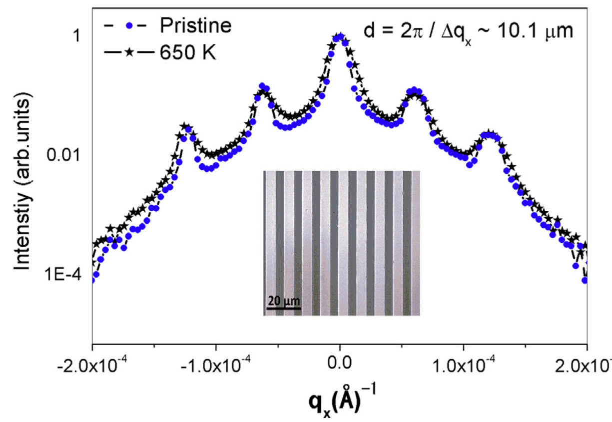

Fig. 1. X-ray rocking curve scans of pristine and 650 K annealed patterned Fe/Pt multilayer. Incident x-rays are perpendicular to the length of pattern stripes. From the separation of peaks (Δq_x) grating periodicity (d) is calculated. Inset shows the optical image of the patterned Fe/Pt multilayer after lift-off process.

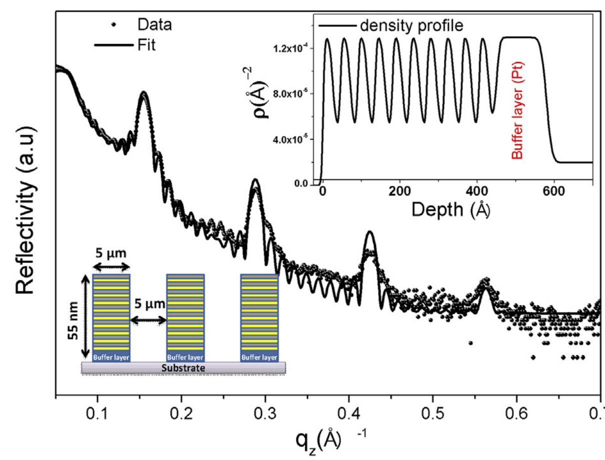

Fig. 2. X-ray reflectivity (XRR) data of pristine patterned Fe/Pt multilayer sample. Symbols are the experimental points and the solid line is the best fit to the data. Bottom inset shows the schematic of patterned Fe/Pt multilayer and the top inset shows the electron density profile along the depth of multilayer as obtained from the fitting of XRR pattern.

study the formation of $L1_0$ phase. The XRR measurements are carried out on the annealed sample also and Fig. 3 (a) shows the 1st Bragg peak of the as-deposited and 650 K annealed patterned Fe/Pt multilayer. The reduction in the intensity of the 1st Bragg peak can be understood as following. With the annealing there is an inter-diffusion across the Fe and Pt layers resulting in the FePt alloy formation at the interfaces and as a result the intensity of the first order Bragg peak reduces [20,21]. Finite intensity of Bragg peak indicates that complete intermixing is not observed with the 650 K annealing. It is to be noted that for complete intermixing annealing at temperatures higher than 773 K is required in such multilayers [5].

To understand the different phases that are formed with the annealing, GIXRD measurements are carried out and the data is shown in Fig. 3(b). With 650 K annealing, the signatures of $L1_0$ FePt phase is clearly seen in the GIXRD pattern. Peaks corresponding to FePt (001) is observed at about 24^o, while peaks corresponding to FePt (110) is observed at about 33^o. It is to be noted that FePt-(00l) represents the chemical ordering along the out-of-plane and FePt (110) represents the c-axis in the plane of the film. However, the intense peak at about 41^o indicates the strong (111) texture, which