

Annealing Effect on Exchange Bias in $\text{Ni}_{81}\text{Fe}_{19}/\text{Cr}_{50}\text{Mn}_{50}$ Bilayers

Haiwen Xi, Bo Bian, Zailong Zhuang, David E. Laughlin, and Robert M. White

Abstract—The effect of annealing on the exchange bias in $\text{Ni}_{81}\text{Fe}_{19}/\text{Cr}_{50}\text{Mn}_{50}$ bilayers was investigated. A large increase of the exchange field and coercivity was observed in the samples annealed at temperatures higher than 310°C . The annealing causes the Mn atoms in the $\text{Cr}_{50}\text{Mn}_{50}$ layer to diffuse into the $\text{Ni}_{81}\text{Fe}_{19}$ layer and form an antiferromagnetic NiFeMn phase, which coats on the $\text{Ni}_{81}\text{Fe}_{19}$ grain surface and contributes to the improvement of exchange bias. An increase of interface roughness due to the Mn diffusion also results in the exchange bias enhancement. The enhanced exchange field and coercivity were explained by the reversible and irreversible motions of NiFeMn moments of the inhomogeneous grains.

Index Terms—Exchange bias, antiferromagnet, magnetic film, thermal annealing.

I. INTRODUCTION

EXTENSIVE studies have been made on exchange biasing materials for their application in anisotropic magneto-resistive (AMR) and giant magnetoresistive (GMR) spin-valve sensors used in high-density recording [1], [2]. The exchange bias effect or exchange anisotropy, which arises from the interfacial exchange coupling between a ferromagnet (FM) and an antiferromagnet (AF), was discovered more than forty years ago. It is so named because the phenomenon manifests itself in a shifted hysteresis loop for the bilayer film. Exchange bias can be obtained in as-deposited samples for many kinds of AF/FM bilayers. However, for some AF/FM films such as NiFe/NiMn [3], [4] and NiFe/PtMn [5], thermal annealing is required. Recently, $\text{Cr}_{50}\text{Mn}_{50}$ based materials (e.g., CrMnPt) are under study as promising biasing materials with the advantages of high thermal stability and good corrosion resistance [6]. It has also been reported [7] that CrMnPt₁₀-based sensors are less sensitive to electrostatic discharge (ESD) than those based on other bias materials. However, the exchange anisotropy of CrMnPt-bilayers is small. It was found that the exchange bias of the CrMnPt/Co system could be improved by annealing [8]. We have carried out annealing on $\text{Ni}_{81}\text{Fe}_{19}/\text{Cr}_{50}\text{Mn}_{50}$ bilayers, and the results are presented in this article.

II. EXPERIMENTS

$\text{Ni}_{81}\text{Fe}_{19}$ (250 Å)/ $\text{Cr}_{50}\text{Mn}_{50}$ (600 Å) bilayers were sequentially deposited by RF magnetron sputtering onto glass substrates

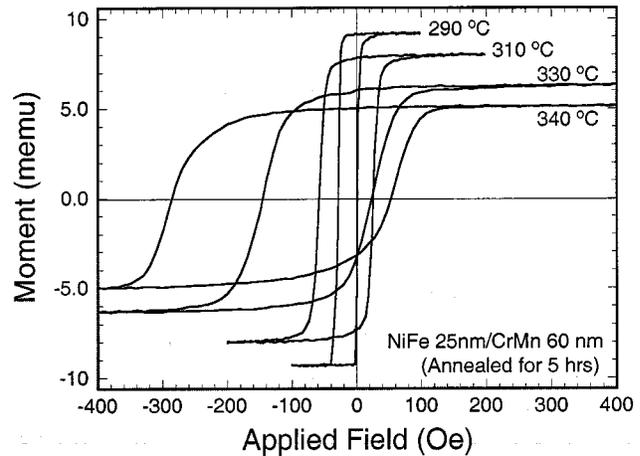


Fig. 1. Hysteresis loops of the annealed $\text{Ni}_{81}\text{Fe}_{19}/\text{Cr}_{50}\text{Mn}_{50}$ samples obtained by VSM measurements.

with a 50 Å amorphous Ta buffer layer in a home-made five target sputtering system. Another 50 Å Ta layer was deposited on top of the $\text{Cr}_{50}\text{Mn}_{50}$ layer to protect samples against oxidation in air. The base pressure of the system was typically 10^{-7} Torr. $\text{Ni}_{81}\text{Fe}_{19}$ was sputtered from a permalloy target at an Ar pressure of 4 mTorr and a supply power of 400 W. $\text{Cr}_{50}\text{Mn}_{50}$ was sputtered from a $\text{Cr}_{50}\text{Mn}_{50}$ alloy target at a high Ar pressure of 20 mTorr. The composition of $\text{Cr}_{50}\text{Mn}_{50}$ films was confirmed by energy dispersion X-ray fluorescence (EDXRF) measurements. The unidirectional exchange anisotropy and the uniaxial anisotropy of the exchange coupled $\text{Ni}_{81}\text{Fe}_{19}$ layer were induced by a magnetic field of about 10 Oe in the sputtering chamber during deposition.

Post deposition annealing was done in a vacuum below 10^{-6} Torr for 5 hours in a 600 Oe applied magnetic field in the direction of the easy axis of the films. The temperature ramp rate was 2°C per minute. The magnetic properties of the films were characterized by a vibrating sample magnetometer (VSM) at room temperature. The crystallographic structure and texture of the thin film samples were determined using X-ray diffraction (XRD). $\theta - 2\theta$ scanning was performed on a Rigaku diffractometer using $\text{Cu-K}\alpha$ radiation. Cross-section transmission electron microscopy (TEM) measurements were also carried out on the as-deposited and annealed samples.

III. RESULTS AND DISCUSSION

From hysteresis loop measurements, the exchange bias effect is characterized by an exchange field H_e and a coercivity H_c which are defined as the displacement and the half-width of the loop, respectively. Fig. 1 shows hysteresis loops for

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The authors are with Data Storage Systems Center (DSSC), Department of Electrical and Computer Engineering, Carnegie Mellon University, Pittsburgh, PA 15213 USA.

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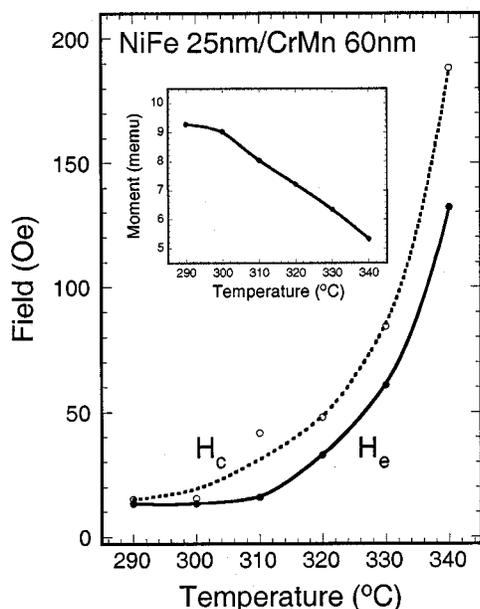


Fig. 2. The exchange field H_e and coercivity H_c for annealed $\text{Ni}_{81}\text{Fe}_{19}/\text{Cr}_{50}\text{Mn}_{50}$ samples. The moment of the exchange biased $\text{Ni}_{81}\text{Fe}_{19}$ after annealing is shown in the inset.

$\text{Ni}_{81}\text{Fe}_{19}/\text{Cr}_{50}\text{Mn}_{50}$ bilayer samples annealed at several different temperatures for 5 hours. The exchange field H_e and coercivity H_c of the as-deposited samples were 8 Oe and 4 Oe, respectively. No appreciable increase of H_e or H_c with annealing was observed until the annealing temperature reached 280°C . The exchange field and coercivity of samples annealed in the temperature range from 290°C to 340°C for 5 hours are shown in Fig. 2. A remarkable increase of the exchange field is obtained when the annealing temperature exceeds 310°C . In this temperature range, the coercivity is always greater than the exchange field. As shown in the inset, we also found that the moment of the exchange biased $\text{Ni}_{81}\text{Fe}_{19}$ film decreased with increasing annealing temperature.

The X-ray diffraction patterns for annealed $\text{Ni}_{81}\text{Fe}_{19}/\text{Cr}_{50}\text{Mn}_{50}$ samples are shown in Fig. 3. $\text{Cr}_{50}\text{Mn}_{50}$ films deposited on the underlying $\text{Ni}_{81}\text{Fe}_{19}$ film were found to have a disordered bcc structure, with the bcc (110) planes growing on a well (111)-textured $\text{Ni}_{81}\text{Fe}_{19}$ layer. Since the interplanar distances of $\text{Cr}_{50}\text{Mn}_{50}$ {110} and $\text{Ni}_{81}\text{Fe}_{19}$ {111} are very close, the XRD peaks of $\text{Cr}_{50}\text{Mn}_{50}$ (110) and $\text{Ni}_{81}\text{Fe}_{19}$ (111) are located at the same position. We did not find much texture improvement with annealing. However, another X-ray peak was found at 43° , suggesting a new phase resulting from the annealing.

Fig. 4 shows cross-section electron diffraction patterns of an as-deposited film and one annealed at 340°C for 5 hours on a Si (100) substrate. The patterns are composed of diffraction spots from the Si substrate and arcs from the $\text{Cr}_{50}\text{Mn}_{50}$ and $\text{Ni}_{81}\text{Fe}_{19}$ films. The reflections from $\text{Cr}_{50}\text{Mn}_{50}$ {110} and $\text{Ni}_{81}\text{Fe}_{19}$ {111} formed six arcs but not a continuous ring in both the as-deposited and annealed films. The arcs in the direction of the Si {002} spot from the center are very strong in intensity, implying the $\text{Cr}_{50}\text{Mn}_{50}$ and $\text{Ni}_{81}\text{Fe}_{19}$ films are (110)- and (111)-textured, respectively. This result was confirmed by

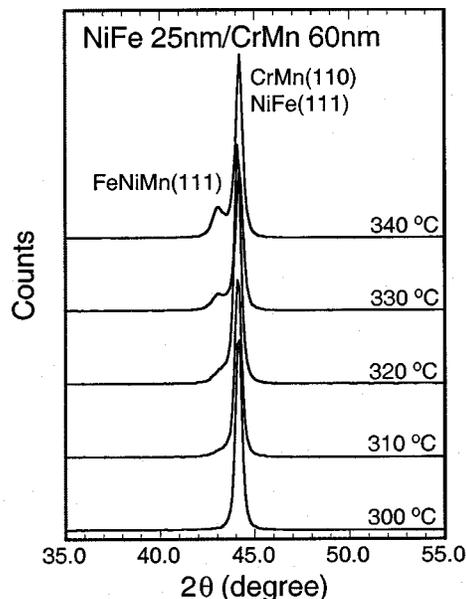


Fig. 3. X-ray diffraction patterns for annealed $\text{Ni}_{81}\text{Fe}_{19}/\text{Cr}_{50}\text{Mn}_{50}$ bilayer samples.

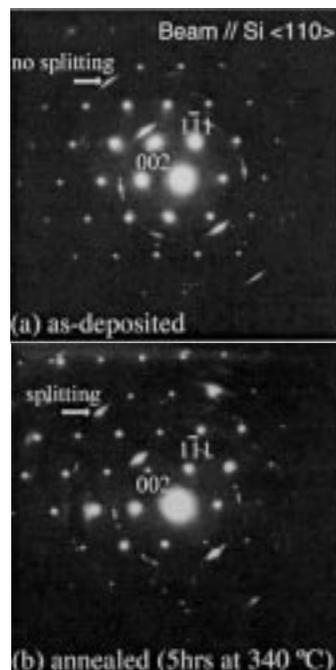


Fig. 4. Cross-section electron diffraction patterns for as-deposited and annealed $\text{Ta}/\text{Ni}_{81}\text{Fe}_{19}/\text{Cr}_{50}\text{Mn}_{50}/\text{Ta}$ films on Si (100) substrate.

micro-beam electron diffraction examination. Although the $\text{Cr}_{50}\text{Mn}_{50}$ (110) and $\text{Ni}_{81}\text{Fe}_{19}$ (111) arcs in the direction of Si {002} do not show any significant change upon annealing, the $\text{Cr}_{50}\text{Mn}_{50}$ (220) and $\text{Ni}_{81}\text{Fe}_{19}$ (222) arcs split after annealing as shown by the arrows in Fig. 4. This splitting is consistent with the new peak observed from X-ray diffraction in the annealed sample. Since no other crystal structure was found in the electron diffraction measurement, the new phase resulting from annealing is identified as a disordered fcc or ordered fct FeNiMn due to the diffusion of the Mn atoms into the $\text{Ni}_{81}\text{Fe}_{19}$ layer at an annealing temperature of 320°C . In the meantime,

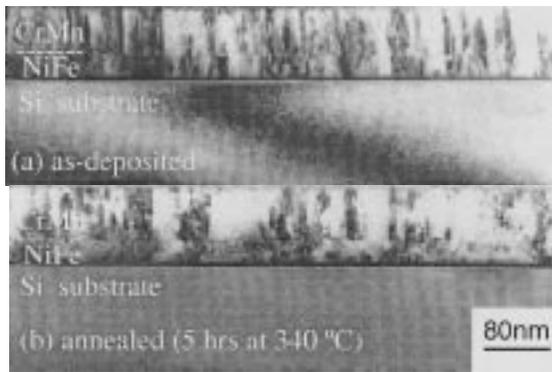


Fig. 5. Cross-section TEM images for the as-deposited and annealed $\text{Ni}_{81}\text{Fe}_{19}/\text{Cr}_{50}\text{Mn}_{50}$ bilayer samples.

since the atomic volumes of Mn and Cr are almost the same, the lattice parameter of CrMn is not changed with the diffusion of Mn atoms [9], and therefore the CrMn (110) peak remains unchanged after annealing. Recently, the diffusion of Mn atoms has been directly observed by mapping the Mn atoms in the annealed sample [10].

Bright-field cross-sectional TEM images of as-deposited and annealed samples, shown in Fig. 5, indicate the columnar growth of $\text{Cr}_{50}\text{Mn}_{50}$ and $\text{Ni}_{81}\text{Fe}_{19}$ grains. The morphologies of the $\text{Cr}_{50}\text{Mn}_{50}$ and $\text{Ni}_{81}\text{Fe}_{19}$ grains appear to be continuous across their interface. In an early study by Massenet *et al.* [11] of the exchange bias effect in annealed FeNi/Mn bilayers at 300°C , a transition layer was suggested to form at the interface and to give the exchange bias. However, the TEM image of our annealed sample indicates a sharp interface between $\text{Ni}_{81}\text{Fe}_{19}$ and $\text{Cr}_{50}\text{Mn}_{50}$ layers with no noticeable FeNiMn transition layer between the $\text{Ni}_{81}\text{Fe}_{19}$ and $\text{Cr}_{50}\text{Mn}_{50}$ layers.

In $\text{Ni}_{81}\text{Fe}_{19}/\text{NiMn}$ bilayers, thermal annealing results in the NiMn forming a face-centered-tetragonal θ phase, which is antiferromagnetic and responsible for the exchange bias [3]. The enhancement of the exchange anisotropy in annealed CrMnPt/Co films has been associated with a CrMnPt lattice distortion after annealing [8]. However, in the biased bilayers with the CrMnPt deposited on top of the $\text{Ni}_{81}\text{Fe}_{19}$, the lattice distortion of CrMnPt is negligibly small even after annealing. Consequently, the exchange bias in $\text{Ni}_{81}\text{Fe}_{19}/\text{CrMnPt}$ system does not increase [8], [12]. For our $\text{Ni}_{81}\text{Fe}_{19}/\text{Cr}_{50}\text{Mn}_{50}$ films, when the annealing temperature exceeds 310°C , the Mn atoms diffuse into the $\text{Ni}_{81}\text{Fe}_{19}$ layer and combine with $\text{Ni}_{81}\text{Fe}_{19}$ to form a new FeNiMn phase. The increased exchange biasing in the annealed samples is possibly associated with those grains of $\text{Ni}_{81}\text{Fe}_{19}$ exchange coupled with an antiferromagnetic NiFeMn "coating" on their surface. In addition, an increase of interface roughness due to Mn diffusion also contributes to the exchange bias enhancement. Based on our recent theoretical study [13],

the large enhancement of the coercivity with annealing is due to irreversible transitions of the NiFeMn moments for some grains, while the exchange biasing is associated with other grains for which the motion of the NiFeMn moments is reversible. For the annealed samples, the coercivity is always larger than the exchange field, suggesting that more grains show irreversible behavior.

IV. SUMMARY

We have found that annealing of $\text{Ni}_{81}\text{Fe}_{19}/\text{Cr}_{50}\text{Mn}_{50}$ bilayers results in the interdiffusion of Mn atoms into the $\text{Ni}_{81}\text{Fe}_{19}$ layer. This diffusion results in an antiferromagnetic NiFeMn phase, which is associated with the enhancement of the exchange field and the coercivity.

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