APPLIED PHYSICS LETTERS **87**, 222509 (2005)

Bidomain state in exchange biased FeF_2/Ni

O. Petracic,[a)] Zhi-Pan Li, Igor V. Roshchin, M. Viret, R. Morales,
X. Batlle, and Ivan K. Schuller
Department of Physics, University of California, San Diego, La Jolla, California 92093

(Received 27 May 2005; accepted 5 October 2005; published online 23 November 2005)

Independently exchange biased subsystems can coexist in ferromagnet/antiferromagnet bilayers after various field-cooling protocols. We find well separated double hysteresis loops in FeF_2/Ni bilayers for intermediate cooling fields, while for small or large cooling fields a negatively or positively shifted single loop, respectively, are encountered. The antiferromagnet breaks into a bidomain state with opposite signs but equal magnitude of bias acting on the ferromagnet. This idea is supported by micromagnetic simulations. Experiments are presented, where thermally activated motion of these antiferromagnetic domain boundaries can be achieved. © *2005 American Institute of Physics*. [DOI: 10.1063/1.2138357]

Antiferromagnetic/ferromagnetic (AF/FM) bilayers exhibit exchange bias (EB) due to the exchange interaction across the AF-FM interface.[1–4] It manifests as a negative or positive shift of the hysteresis loop along the field axis.[5] In several cases also double-hysteresis loops were reported. They are attributed to a coexistence of oppositely oriented interfacial AF moments in different mesoscopic regions. However, the way to prepare this state and, hence, the underlying mechanisms involved can be very different. In several cases this coexistence is found after zero field cooling a demagnetized sample.[6–9] Here the FM domain configuration is imprinted in the AF during cooling. Recently we succeeded to even control quantitatively the ratio of the two imprinted subsystems and map them by scanning magneto-optic Kerr-effect measurements.[9] In rare cases double loops are found after field cooling (FC) at intermediate fields.[9,10] Double hysteresis loops are also reported in measurements along the hard axis of the AF, which is attributed to an additional biquadratic AF-FM interaction.[11–13]

In this letter we present a systematic and tunable shift from negative to double, and finally to positive EB in an AF/FM bilayer, FeF_2/Ni, for various field cooling protocols. Due to its extremely small coercivity compared with the EB field, this system becomes ideal for studying this phenomenon. Micromagnetic simulations that set forth the criterion for the occurrence of double hysteresis loops are also presented.

$FeF_2/Ni/Al$ multilayers were grown on a single crystalline $MgF_2(110)$ substrate by electron-beam evaporation. The base pressure was lower than 2×10^{-7} Torr. The FeF_2 ($T_N=78$ K) was deposited at a temperature of 300 °C at a rate of 0.05 nm/s and the Ni layer at 150 °C at the same rate. As a protection against oxidation Al was deposited finally at 150 °C at a rate of 0.1 nm/s. From x-ray diffraction measurements one can identify that FeF_2 grows epitaxially in the (110) orientation, whereas the Ni is polycrystalline. $FeF_2(110)$ has a nominally compensated interface with the easy axis lying in-plane along the [001] direction.[2] This axis was found to be also the easy axis of the FM. The magnetization along the easy axis was measured using a superconducting quantum interference device magnetometer (Quantum Design).

Figure 1 shows the magnetization, M vs H, of FeF_2 (83 nm)/Ni (17 nm)/Al (6 nm) at $T=10$ K after FC from 150 K in $\mu_0H_{FC}=0.05$ T (curve number 1), 0.075 T (2), 0.1 T (3), 0.125 T (4), and 0.2 T (5). For intermediate fields ($0.075\text{ T}\leq\mu_0H_{FC}\leq0.125$ T) a double hysteresis loop is present with the EB and coercive fields of both subloops being almost identical, $|\mu_0H_E|\approx0.09$ T and $\mu_0H_c\approx0.006$ T, respectively. Upon application of a smaller ($\mu_0H_{FC}\leq0.05$ T) or higher cooling field ($\mu_0H_{FC}\geq0.2$ T) only a single loop with negative or positive EB shift, respectively, is found. The dependence of H_E as a function of the FC field is presented in the upper inset. A well-defined sharp crossover region with double hysteresis loops appears.

This shows that there are two spatially separated subsystems (bidomain) in the AF, each with a net frozen AF interfacial moment pointing parallel or antiparallel to the FC direction. Hence each AF subsystem gives rise to *local* EB on the FM,[9] exactly opposite to each other.[6–11,14,15] The origin of the net AF interfacial moment is possibly due to a fraction of locked interfacial Fe^{2+} moments of the FeF_2.[16,17] The crossover from negative to positive EB (for small or

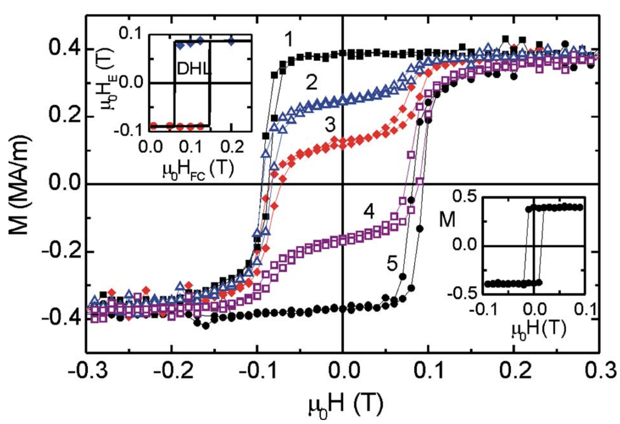

FIG. 1. (Color online) $M(H)$ at $T=10$ K after field cooling in $\mu_0H_{FC}=0.05$ T (curve number 1), 0.075 T (2), 0.1 T (3), 0.125 T (4) and 0.2 T (5). The upper inset shows a plot of the extracted EB field H_E vs FC field H_{FC}. The field region, where double hysteresis loops occur is denoted by two vertical lines. The lower inset shows $M(H)$ at $T=90\text{ K}>T_N$. Solid lines are guides to the eye.

[a)]Present address: Physics Department (EXSS), Imperial College London, London SW7 2AZ, UK; electronic mail: o.petracic@imperial.ac.uk

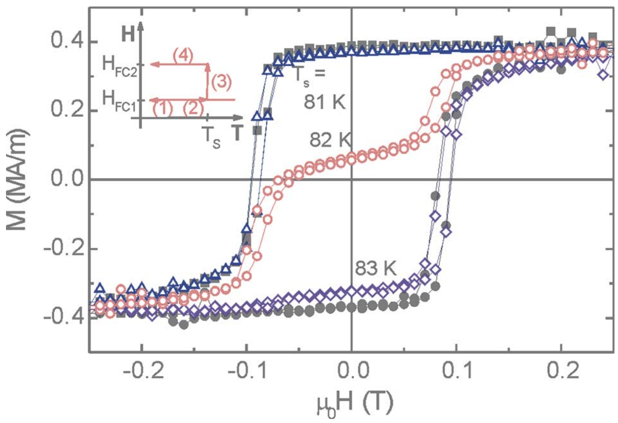

FIG. 2. (Color online) $M(H)$ at $T=10$ K after two different FC protocols. (i) FC in $\mu_0 H_{FC}=0.05$ T (solid squares) and 0.2 T (solid circles) and (ii) FC as the schematic in the inset shows, where a field step is applied at $T_S=81$ K (open triangles), 82 K (open circles), and 83 K (open diamonds), and $\mu_0 H_{FC1}=0.05$ T and $\mu_0 H_{FC2}=0.2$ T. Lines are guides to the eye.

large cooling fields, respectively) originates from the competition between the Zeeman and AF-FM exchange energy during FC as reported in Ref. 18. Previously a gradual shift from negative to positive EB is encountered,[5] whereas here positive and negative EB with similar absolute values coexist (see upper inset of Fig. 1). The difference must be due to the fact, that in our samples the FM does not experience an average AF moment, $\langle \mathbf{S}_{AF} \rangle$,[19] but rather two independent coexisting mesoscopic AF/FM subsystems.[8–10] The size of the AF domains has to be larger than the minimum domain size of the FM in order to avoid averaging as pointed out in Ref. 9. This coexistence of biasing directions can be explained either by a mesoscopic variation of the coupling strength, $J_{AF\text{-}FM}$, over the AF-FM interface or a third energy term being due to piezomagnetic or more generally magnetoelastic contributions.[15]

The lower inset of Fig. 1 shows a hysteresis loop measured at $T=90$ K (above T_N). It displays a coercive field of $\mu_0 H_c \approx 0.015$ T and saturation at 0.025 T. Hence, all cooling fields used in this study are larger than the saturation field at and above 90 K. This differs from several studies, where a double loop can only be found after demagnetizing the FM.[6–8] One should also note, that no perpendicular coupling[20] is observable, as evidenced from measurements along the hard axis (data not shown). Interestingly, the Ni layer shows an easy axis along [001] even above T_N at 150 K (data not shown), although the x-ray diffraction data indicate a polycrystalline Ni layer. This may be due to a growth-induced anisotropy in the FM or an induced anisotropy by the interaction with the FeF_2.[21,22]

The idea of two oppositely oriented AF domains is confirmed by another set of experiments, where the domain boundaries are moved by a field step. Figure 2 shows $M(H)$ curves of the same sample at $T=10$ K after two different FC procedures: (i) FC from 150 to 10 K in either $\mu_0 H_{FC1}=0.05$ T (single negatively shifted loop) or 0.2 T (single positively shifted loop), or (ii) first FC from 150 to 10 K in $\mu_0 H_{FC1}=0.05$ T (1), followed by field heating from 10 K to T_S in 0.05 T (2), then change the field to 0.2 T at T_S (3) and finally FC from T_S to 10 K in $\mu_0 H_{FC2}=0.2$ T (4) (inset of Fig. 2). The data obtained from the FC protocol (i) are shown as solid symbols. The curves measured after the FC protocol (ii) are shown with open symbols. For $T_S=81$ K no effect of the field change is observed. However, at a slightly higher $T_S=82$ K a double loop is found and, finally, for $T_S=83$ K virtually only a single positively shifted loop is encountered. Interestingly, a small signature of the negatively biased loop remains even at $T_S=120$ K (data not shown). Hence, the AF bidomain structure remains stable above the bulk Néel temperature of $T_N=78$ K, although no bulk long range order is present. This is either a consequence of a broad distribution of blocking temperatures[22,23] or a strain-induced enhancement of the AF/FM exchange coupling[24] and therefore an interfacial stabilization of the AF by the FM.

In order to investigate the origin of double hysteresis loops, micromagnetic simulations of a polycrystalline Ni layer of 20 nm thickness and lateral size 500 nm × 500 nm were performed using the OOMMF micromagnetic simulation package.[25] We assumed 10% of randomly distributed, rigid, uncompensated AF interfacial moments, $\mathbf{S}_{AF}$, being exchange coupled to the bottom layer of the Ni.[26,27] The interfacial coupling strength was taken to be twice the exchange constant in bulk FeF_2,[28,29] i.e., $J_{AF\text{-}FM}=-0.90$ meV. Apart from micromagnetic parameters for Ni (saturation magnetization $M_s=0.49$ MA/m, exchange constant $A=3.4$ pJ/m and anisotropy constant $K_1=-5$ kJ/m^3) we also included the demagnetizing effect by a shape anisotropy constant, $K_d=-(\mu_0/2)M_s^2=-148$ kJ/m^3, forcing the spins to be oriented in-plane. A uniaxial anisotropy constant, $K_u=-15$ kJ/m^3 introduces an in-plane easy axis, qualitatively similar to the experimental situation. During the simulation, no dipolar energy was taken into account. The bidomain state of the AF was modeled by laterally dividing the system in areas of size D_{AF} in the *x*-*y* plane with two opposite orientations of the rigid AF moments along the easy axis. The external magnetic field is applied parallel to the easy axis.

Figure 3(a) shows the calculated hysteresis loops, $M(H)$, for different AF subsystem sizes, $D_{AF}=30$, 60, 125, and 250 nm. A transition from one broad loop ($D_{AF}=30$ nm) to two separated subloops ($D_{AF}=250$ nm) is found. The curves for $D_{AF}=30$ and 60 nm exhibit a slight EB shift due to a statistical imbalance of the randomly chosen frozen AF moments. The value for D_{AF} necessary to produce double loops has to be compared to the domain size of Ni, whose lower limit is determined by the domain wall width, δ_B. This value reflects the length scale over which the FM averages the exchange interaction with the interfacial AF uncompensated moments.[9] Using $\delta_B=\pi\sqrt{A/(K_1+K_u)}$ and the values mentioned above for K_1 and K_u one arrives at $\delta_B=41$ nm. The simulation proves that double hysteresis loops are observed, when the criterion, $D_{AF} \gg \delta_B$, is fulfilled as in cases of $D_{AF}=125$ and 250 nm. In addition, spin structures of the top and the bottom layer at remanence ($H=0$) for $D_{AF}=30$ and 250 nm are shown in Fig. 3(b). One clearly observes an averaging effect for the 30 nm subsystem size. While the bottom layer still reflects the topology of opposite orientations of the subsystems, the top layer is virtually only positively magnetized. However, for $D_{AF}=250$ nm both the top and bottom layer show clearly separated regions with opposite magnetization directions.

In conclusion, we present an EB system, that exhibits a tunable double hysteresis loop and, hence, two oppositely biased subsystems (bidomain state). This case is found for intermediate cooling fields. Movement of the AF domain

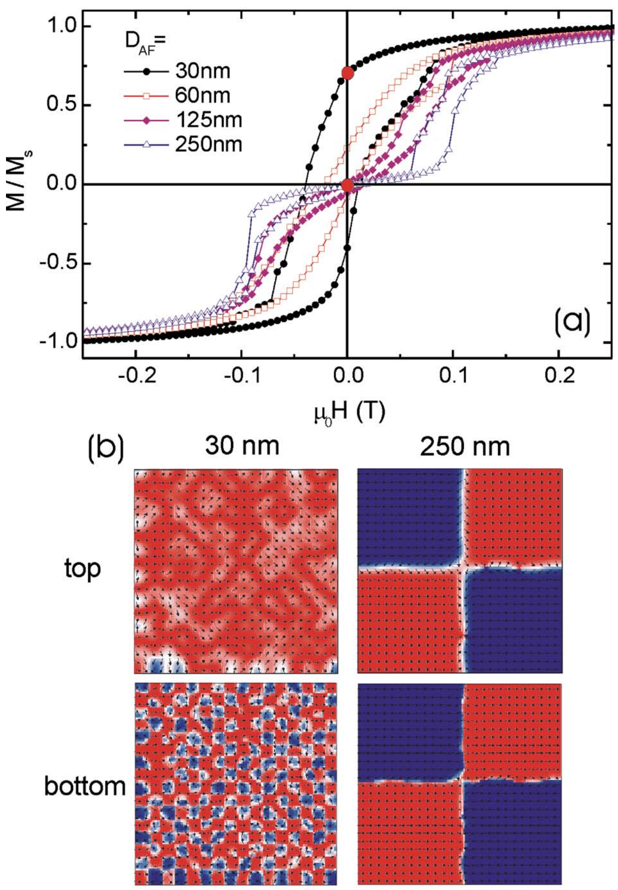

FIG. 3. (Color online) (a) $M(H)$ from simulations of a Ni layer, where a constant random-site field acts on the bottom layer. Two opposite orientations of the random-site field are used with the domain size varied: $D_{AF}=30$, 60, 125, and 250 nm as indicated in the legend. Lines are guides to the eye. (b) Spin structures of top and bottom layer at $H=0$ [red circles in (a)] for $D_{AF}=30$ and 250 nm. The color code "red-white-blue" ("light grey-white-dark grey") indicates "positive-zero-negative" magnetization along the easy axis.

walls can be induced by a field step through thermal activation. The criterion for obtaining independent subsystems is that the AF domain size must be larger than the minimum domain size (domain wall width) of the FM. This idea is confirmed by micromagnetic simulations of an EB Ni layer, where the AF is modeled by a fraction of randomly distributed, rigid, uncompensated interfacial moments being exchange coupled to the bottom layer of the FM.

The authors thank W. Kleemann and X. Chen for fruitful discussions. The work is funded by the U.S. Department of Energy and the AFOSR. Also, financial support from Alexander-von-Humboldt Foundation (O.P.), Cal(IT)^2 (Z.-P.L.), Spanish MECD (R.M., X.B.), Fulbright Commission (R.M.), and Catalan DURSI (X.B.) is acknowledged.

[1] W. H. Meiklejohn and C. P. Bean, Phys. Rev. **102**, 1413 (1956).
[2] J. Nogués and I. K. Schuller, J. Magn. Magn. Mater. **192**, 203 (1999).
[3] A. E. Berkowitz and K. Takano, J. Magn. Magn. Mater. **200**, 552 (1999).
[4] R. L. Stamps, J. Phys. D **33**, R247 (2000).
[5] J. Nogués, D. Lederman, T. J. Moran, and I. K. Schuller, Phys. Rev. Lett. **76**, 4624 (1996).
[6] P. Miltényi, M. Gierlings, M. Bamming, U. May, G. Güntherodt, J. Nogués, M. Gruyters, C. Leighton, and I. K. Schuller, Appl. Phys. Lett. **75**, 2304 (2005).
[7] N. J. Gökemeijer and C. L. Chien, J. Appl. Phys. **85**, 5516 (1999).
[8] C. L. Chien, V. S. Gornakov, V. I. Nikitenko, A. J. Shapiro, and R. D. Shull, Phys. Rev. B **68**, 014418 (2003).
[9] I. V. Roshchin, O. Petracic, R. Morales, Z.-P. Li, X. Batlle, and I. K. Schuller, Europhys. Lett. **71**, 297 (2005); J. Olamit, E. Arenholz, Z.-P. Li, O. Petracic, I. V. Roshchin, R. Morales, X. Batlle, I. K. Schuller, and K. Liu, Phys. Rev. B **72**, 012408 (2005).
[10] T. L. Kirk, O. Hellwig, and E. E. Fullerton, Phys. Rev. B **65**, 224426 (2002).
[11] C.-H. Lai, Y.-H. Wang, C.-R. Chang, J.-S. Yang, and Y. D. Yao, Phys. Rev. B **64**, 094420 (2001).
[12] T. Zhao, H. Fujiwara, K. Zhang, C. Hou, and T. Kai, Phys. Rev. B **65**, 014431 (2001).
[13] H. Shi and D. Lederman, Phys. Rev. B **66**, 094426 (2002).
[14] H.-W. Zhao, W. N. Wang, Y. J. Wang, and W. S. Zhan, J. Appl. Phys. **91**, 6893 (2002).
[15] Ch. Binek, Xi Chen, A. Hochstrat, and W. Kleemann, J. Magn. Magn. Mater. **240**, 257 (2002).
[16] H. Ohldag, A. Scholl, F. Nolting, E. Arenholz, S. Maat, A. T. Young, M. Carey, and J. Stöhr, Phys. Rev. Lett. **91**, 017203 (2003).
[17] P. Kappenberger, S. Martin, Y. Pellmont, H. J. Hug, J. B. Kortright, O. Hellwig, and E. E. Fullerton, Phys. Rev. Lett. **91**, 267202 (2003).
[18] C. Leighton, J. Nogués, H. Suhl, and I. K. Schuller, Phys. Rev. B **60**, 12837 (1999).
[19] L. Wee, R. L. Stamps, L. Malkinski, and Z. Celinski, Phys. Rev. B **69**, 134426 (2004).
[20] T. J. Moran, J. Nogués, D. Lederman, and I. K. Schuller, Appl. Phys. Lett. **72**, 617 (1998).
[21] C. Leighton, H. Suhl, M. J. Pechan, R. Compton, J. Nogues, and I. K. Schuller, J. Appl. Phys. **92**, 1483 (2002).
[22] M. Grimsditch, A. Hoffmann, P. Vavassori, H. Shi, and D. Lederman, Phys. Rev. Lett. **90**, 257201 (2003).
[23] X. Chen, Ch. Binek, A. Hochstrat, and W. Kleemann, Phys. Rev. B **65**, 012415 (2001).
[24] H. Shi, D. Lederman, K. V. O'Donovan, and J. A. Borchers, Phys. Rev. B **69**, 214416 (2004).
[25] OOMMF/OXSII micromagnetic simulation by M. Donahue and D. Porter, http://math.nist.gov/oommf.
[26] Z.-P. Li, O. Petracic, R. Morales, J. Olamit, X. Batlle, K. Liu, and I. K. Schuller (unpublished).
[27] Z.-P. Li, O. Petracic, J. Eisenmenger, and I. K. Schuller, Appl. Phys. Lett. **86**, 072501 (2005).
[28] M. T. Hutchings, B. D. Rainford, and H. J. Guggenheim, J. Phys. C **3**, 307 (1970).
[29] In bulk FeF_2 there are three exchange constants, where $J_2=-0.45$ meV is the dominant coupling constant with *eight* neighbors.