Variation in ferromagnetic domain density and domain asymmetry in Fe/FeF_2 exchange bias structures

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(Received 11 December 2007; published 30 May 2008)

The development of ferromagnetic domains in exchange-coupled ferromagnetic/antiferromagnetic Fe(2.6 nm)/FeF_2(50 nm) bilayers is studied by magneto-optical magnetometry and microscopy in a temperature range from 300 to 17 K. At room temperature, the cubic anisotropy of the single-crystal Fe can be clearly identified from the formation of characteristic domain patterns. With the onset of exchange bias, below the Néel temperature of FeF_2, the density of the nuclei of patchlike magnetic domains continuously increases with increase in exchange bias and the congruent increase in coercivity. The increase in nucleation density is interpreted as a local increase in the density of pinned interfacial moments. As changes in the bulk antiferromagnetic structure are excluded from field history dependent measurements, the temperature dependent exchange bias is shown to be directly connected to a lateral inhomogeneous increase in the interfacial exchange coupling.

DOI: 10.1103/PhysRevB.77.184433 PACS number(s): 75.70.Cn, 75.70.Ak, 75.50.Cc, 75.30.Et

I. INTRODUCTION

Since the discovery of the exchange bias effect,[1,2] structures with magnetic hysteresis loops centered about a nonzero magnetic field have been intensively investigated. However, despite the numerous experimental and theoretical studies,[3–8] the exchange bias effect is still not completely understood at the microscopic level. Recent experiments[9,10] provided direct experimental evidence for the existence of uncompensated interfacial spins being responsible for the magnetic loop shift. Among the variety of exchange biased systems, which usually consist of a ferromagnet (FM) coupled to an antiferromagnet (AFM), thin film structures have become the most relevant due to their use in industrial applications. For instance, exchange bias structures have become part of so-called spin-valve[11,12] devices, which are based on the giant magnetoresistance effect.

Here, we focus on the AFM/FM Fe_2F/Fe system, which has been extensively studied experimentally, enabling the creation of theoretical models on the exchange bias effect. The main motivation to investigate systems which consist of FeF_2 as the AFM is its simple spin structure[13] with a large uniaxial magnetic anisotropy ($K \approx 1.39$ erg/cm^3) along the c axis[14] below the magnetic phase transition temperature of $T_N = 79$ K. Because of the large anisotropy, FeF_2 behaves as an Ising system over a wide temperature range.[15] FeF_2 has a tetragonal structure with $a = 0.4697$ nm and $c = 0.3309$ nm at room temperature,[16] and it remains tetragonal down to 15 K with $a = 0.469\,33$ nm and $c = 0.330\,07$ nm.[17] The interface nature of the exchange bias effect leads to an exchange bias field H_E being strongly dependent on the AFM/FM interface structure, such as the crystalline orientation or the interface disorder. Nogués *et al.*[18] showed that the interface roughness in FeF_2/Fe bilayer films has a strong impact on the AFM/FM exchange. The exchange bias magnitude decreases with increasing roughness. It was also shown that the exchange bias field depends strongly on the spin structure at the interface and, in particular, on the angle between the FM and AFM spins. Models of the microscopic mechanisms determining the magnetic behavior of these systems have been put forward by, e.g., Kiwi *et al.*[19] and Mejia-Lopez *et al.*[20,21] An incomplete domain wall in the FM is suggested to arise as a direct consequence of an almost rigid canted spin configuration at the fully compensated AFM interface, which freezes into a metastable state close to the Néel temperature. According to these models, the exchange bias strongly depends on the value of the interfacial exchange constant, which is the only adjustable parameter used in the simulations. Further experimental studies presented by Fitzsimmons *et al.*[22–24] using polarized neutron reflectometry and magnetometry shed more light on the details of the interface spin configuration. Along with these, different theoretical models for H_E, including the presence of pinned and unpinned moments in the FM and AFM, have been recently proposed to explain the exchange bias effect.[24]

In this paper, the magnetization reversal of a *single-crystal* FM/*polycrystalline* AFM Fe/FeF_2 bilayer structure is analyzed in detail over a wide temperature range by conventional magneto-optical Kerr effect magnetometry and laterally resolved Kerr microscopy[25,26] images of the domain formations are presented. Different cooling field sequences are applied to distinguish between coupling and AFM anisotropy effects. Due to the use of an ultrathin single-crystal Fe layer with defined anisotropy, irregularities in the magnetization reversal process are suggested to originate directly from changes in the FM/AFM interfacial spin structure.

II. SAMPLE PREPARATION AND CHARACTERIZATION

A GaAs/Fe (0.6 nm)/Ag (75 nm)/Fe (2.5 nm)/FeF_2 (50 nm)/Au (2 nm) multilayer was deposited using a molecular beam epitaxy system[27] with 10^{-9} Torr base pressure (H_2 was the main component, 75% of all residual gases). During the deposition, the pressure was maintained at about 10^{-8} Torr

throughout the whole growth process. (001)-oriented GaAs was used as a substrate, which was carefully prepared by high-temperature annealing at $T \approx 840$ K followed by an argon ion sputtering process. The reconstruction 4×6 was expected (in the [010] and [100] directions, respectively) and checked by *in situ* reflection high energy electron diffraction (RHEED), confirming that the GaAs substrate had a Ga terminated surface.[28] A Fe seed layer was deposited directly onto the GaAs substrate and was followed by a 75 nm thick Ag buffer layer deposition. Both were grown at room temperature. The surface smoothness and crystallinity of the Ag layer was improved by annealing at $T \approx 550$ K for 12 h. The ferromagnetic layer of interest, 2.6 nm Fe, was grown at room temperature, at which the growth rate was always kept below 0.03 nm/min. The deposition rate and sample thickness were monitored by a quartz microbalance. The intensity of the RHEED signal displayed well developed oscillations during the Fe deposition upon the flat Ag buffer layer, proving that the Fe grows in layer by layer mode. *In situ* RHEED patterns clearly indicated that the Fe layer is a single crystal with fourfold symmetry. Thermal *K* cells were employed to grow all the metallic layers, whereas the 50 nm thick FeF_2 layer was grown by e-beam evaporation. A 6.5 kV e-beam source with a low emission current of 12 mA was used for the deposition. During the growth of the FeF_2 layer, the substrate temperature was kept around 320 K. The growth rate was 3 nm/min. *In situ* RHEED analysis on the completed FeF_2 layer displayed a ring pattern, clearly indicating a randomly oriented polycrystalline film. The film stack was capped with a Au layer in order to protect the sample for *ex situ* measurements after removing the sample from the UHV system.

Note that the studied exchange-coupled bilayer system of Fe/FeF_2 in the form of a single-crystal FM/polycrystalline AFM is different from the FeF_2/Fe structures presented by many authors before. In particular, in most studies[18] the Fe layer was polycrystalline, while the FeF_2 layer was single crystalline with twin defects.

III. EXPERIMENTAL RESULTS AND DISCUSSION

The Fe/FeF_2 bilayer was studied by longitudinal Kerr microscopy in the temperature range from 300 to 17 K. The in-plane cubic anisotropy of the thin Fe layer at room temperature could be clearly identified from the formation of characteristic fourfold domain patterns. For the investigated system, the AFM's Néel temperature $T_N = 79$ K is much smaller than the FM's Curie temperature $T_C = 1063$ K. Therefore, the magnetic properties of the FM are assumed as nearly temperature independent in the investigated temperature range. The observed temperature dependencies of the magnetic properties or the magnetic domain characteristics are therefore attributed to the presence of the AFM layer in direct contact with the FM layer.

In the experiments, the sample was cooled down in the presence of a static magnetic field (field cooled), $H_{cf} = 2.64$ kOe, along one of the FM's easy axis of magnetization. Magnetic hysteresis loop measurements performed in the direction parallel to the cooling field direction are displayed in Fig. 1. The FM layer hysteresis loop at 85 K, measured along one of the easy axis, exhibits the typical behavior of a thin single-crystal Fe layer, i.e., a rectangular shaped loop with a rapid change in magnetization around H_c. At the lowest attainable temperature of 17 K at the coercive field H_c, the loop displays a continuous change in magnetization with field. This reversal occurs gradually and spreads over a field range of approximately 150 Oe. This feature will be discussed later in more detail.

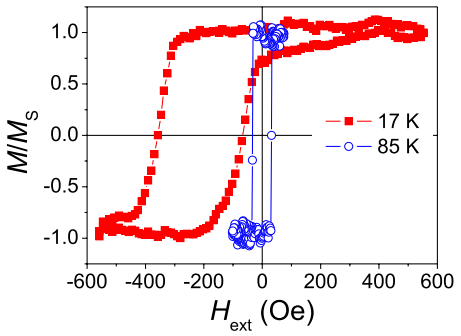

FIG. 1. (Color online) Typical hysteresis loops for the Fe/FeF_2 stack obtained at temperatures $T = 85$ K and $T = 17$ K after cooling in a positive magnetic field.

The temperature dependence of the exchange bias field H_E is presented in Fig. 2(a). It was derived from the hysteresis loop measurements along the cooling field direction based on the relation

$$\vec{H}_E = -\frac{1}{2}(\vec{H}_{c1} + \vec{H}_{c2}),$$

where H_{c1} and H_{c2} are the coercivity field values for the forward and backward loop branches, respectively. In the

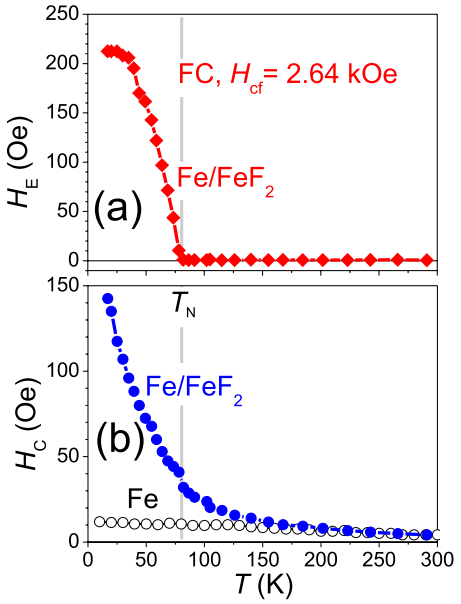

FIG. 2. (Color online) (a) Temperature dependence of the exchange bias field H_E. (b) Temperature dependence of the coercive field H_C. The lines are a guide to the eye. The position of $T/T_N = 1$, assuming an AFM's Néel temperature of $T_N = 79$ K is indicated. The corresponding data for a single Fe layer are shown.

temperature range between room temperature and T_N, no loop shift is observed; but the coercivity field [see Fig. 2(b)] slightly increases with decreasing temperature. The exchange bias H_E appears below T_N. It increases with the temperature decrease, reaching a saturation below T_N at $T \approx 30$ K. For the investigated system, the blocking temperature T_b, at which the exchange bias field H_E approaches zero, is equivalent to T_N. The results are similar to the previously published data by Nogués *et al.*[18] (see Fig. 7 therein) and Lund *et al.*[29] (see Fig. 2 therein), but the absolute values of H_E are slightly lower than those presented, for instance, in Refs. 18, 19, and 21. At this point it should be emphasized that in the FeF_2/Fe structures in above-mentioned Refs. 18, 19, and 21, the FM layer was polycrystalline; while in the present studies, the Fe layer was single crystalline, which can influence the value of H_E due to the different roughness of the prepared layers. In addition, an additional effect from the polycrystallinity of the FeF_2 layer to the observed differences cannot be excluded. Moreover, results from ferromagnetic resonance measurements made *ex situ* directly after sample growth and again 6 months later[30] indicate possible aging effects, which might also contribute to the observed differences in exchange bias as well. Last, a direct comparison of the data is valid only when assuming a homogeneous FM/AFM interface coupling and magnetization, which is not the case for the investigated ultrathin Fe layers, as will be shown later. Despite the polycrystalline AFM layer, the observations are in general agreement with earlier experimental investigations[29,31,32] for the FeF_2/Fe system.[33]

The coercive field H_c vs temperature curve does not exhibit any local extremes [Fig. 2(b)]. The monotonic dependence can be discussed separately for the two different temperature regimes: (i) for $T/T_N > 1$ and (ii) for $T/T_N < 1$. In the high-temperature range, the coercivity field slightly increases with decreasing temperature. Below the AFM ordering temperature, the enhancement of the coercivity field continues with an increased rate with decreasing temperature. Moreover, there is a visible jump in H_c, which takes place at T_N. Only a weak dependence of coercivity on temperature is found from a complementary Fe thin film structure, without the inclusion of the antiferromagnetic $FeFe_2$ layer.[34,35] The coercivity of the uncoupled FM layer is slightly increasing with decreasing temperature.

The observed coercivity enhancement is therefore a unique feature of the studied exchange-coupled system.[36,37] The existence of a pronounced coercivity enhancement even above the Néel temperature suggests that the exchange bias and the coercive field enhancement are not directly correlated with each other. Scholten *et al.*[35] interpreted the increase in coercivity field as caused by the coupling of the FM layer to an unpinned AFM interface layer magnetization, i.e., to the AFM magnetization, which follows the FM magnetization during a hysteresis cycle. A model was postulated in which the coercive field depends upon two contributions, one coming from the FM itself and one from the interaction with the AFM. The magnetization of the FM layer acts as an external parameter on the AFM layer similar to the applied magnetic field. Then, even above T_N, the AFM in its paramagnetic (PM) state experiences an interfacial exchange field coming from the FM. This becomes the origin of a magnetization within the PM or in the interfacial PM layer depending on the relation between the interface coupling energy FM-PM vs the paramagnetic thermal energy. The magnetization of the coupled PM follows the FM during field reversal and will yield zero exchange bias, but it contributes to an enhanced H_c. Moreover, chemical intermixing[24] effects at the FM/FeF_2 interface, as reported on the AFM/FM FeF_2/Co system, would complicate the situation. An analysis of the effects of a broad transition from the AFM to FM structure is beyond the scope of this paper; nevertheless, the interfacial layer might also contribute to the observed behavior.

The exchange bias field H_E saturates below 30 K, as shown in Fig. 2(a), which can be associated with the fully ordered magnetic structure within the AFM together with a well-established AFM interface layer. One interpretation is that the total anisotropy energy stored in the AFM is high enough to keep the orientation of the interfacial magnetic moments fixed.[38] Therefore, no magnetic training effect is also observed in our system. This is in contrast to low or higher order AFM anisotropy exchange-coupled thin film systems (see Ref. 39 for details). Following these arguments, assuming a fixed AFM magnetization structure, the change in H_E with T can be attributed to an increase in interfacial coupling. Lund *et al.*[29] successfully fitted the temperature dependence of the exchange bias field of Fe/FeF_2. However, the microscopic mechanism of this temperature dependence is still lacking. To have a better understanding on the temperature dependence of the magnetic reversal processes, especially on possible microscopic mechanisms involved, the magnetic domain formation during reversal has been studied by magneto-optical Kerr microscopy.

At room temperature, the fourfold anisotropy of iron is clearly identified from the formation of specific domain patterns, as shown in Fig. 3(a). The arrows represent the magnetization directions (easy axes), which are along the crystallographic directions ([100] and [010]) of the single-crystal iron film. Typical domain sizes at the coercivity field at room temperature are on the order of a few hundred micrometers. The FM domain patterns obtained for a simple iron reference at $T=77$ K did not display any temperature dependence regarding the size of magnetic domains at the coercive field. For the studied Fe/FeF_2 system, however, patchlike magnetic domains have been observed. Exemplary images for the backward loop branch below T_N are displayed in Figs. 3(b)–3(f). The reversal mechanism of the FM/AFM is distinctively different from that of the single Fe reference and for the Fe/FeF_2 film at room temperature, indicating that it originates directly from the coupling of the FM to the AFM.

A direct comparison of the ferromagnetic domain states obtained during magnetization reversal close to H_c is displayed in Fig. 4. Clearly a distinct difference in domain density occurs. A complete analysis of the domain density for the forward and backward loops over a wide temperature range is presented in Fig. 5. With the increase in H_E at lower temperatures, the density of nuclei of the patchlike magnetic domains continuously increases (see also Fig. 2). Below 30 K, the size of the domains reduces below the lateral resolution of the applied microscope technique, which manifests

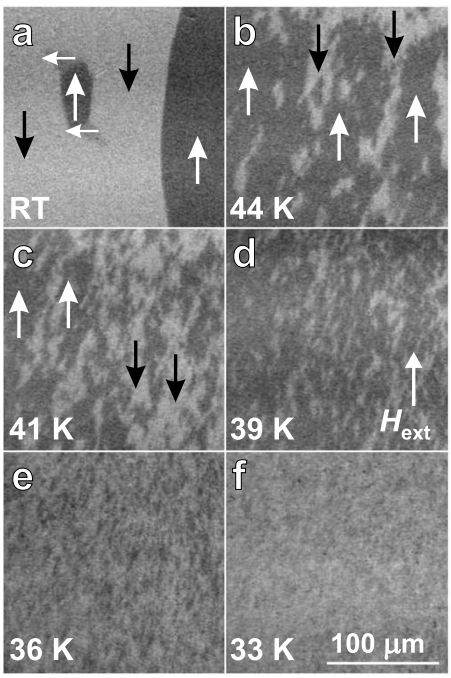

FIG. 3. Magnetic domain structures obtained at the coercivity field for the backward loop branch at different temperatures. The directions of magnetization are indicated.

itself in no observable domain wall activity and only a continuous change in magnetization is identified in the experiment. This behavior could, in principle, be also interpreted as a coherent rotation of magnetization; however, regarding the general trend of reduction in domain size with increase in H_E to lower temperatures, such behavior is unlikely. As mentioned before, below the temperature where no domain wall activity is found, H_E also saturates. The increase in nucleation density is obviously related to an increase in the density of pinned interfacial states acting as domain nucleation sites.

Another relevant observation, which is important for the interpretation of our data, is visible in Fig. 5. The reversal displays signs of asymmetry[40,41] not visible from the hysteresis loops. The densities of nuclei for the increasing and decreasing loop branches differ, being larger for the forward loop branch. Note that the ultrathin Fe layer is expected to mirror the lateral FM/AFM interface properties.[42] Assuming partially strongly coupled and weakly coupled interfacial regions, the magnetic inhomogeneity at the FM/AFM interface

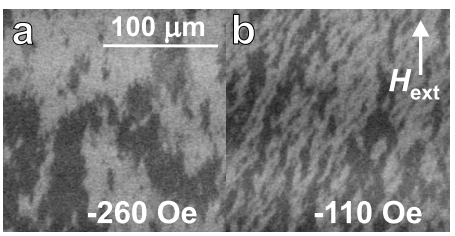

FIG. 4. Magnetic domain structures obtained at the coercivity field for the backward (a) and forward (b) loop branches at $T=44$ K. Magnetic field values are indicated.

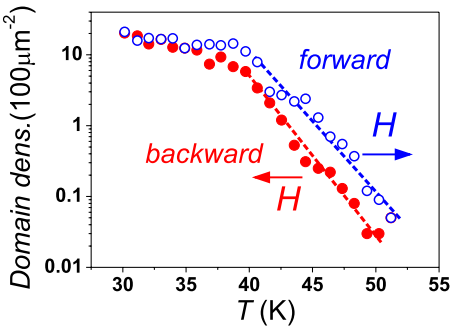

FIG. 5. (Color online) Temperature dependence of the magnetic domain density for the forward and backward loop branches.

is expected to be lower for the downward reversal, wherein the pinned and unpinned states initially are aligned with the FM layer direction. After FM reversal, however, the pinned and unpinned moments are aligned antiparallel to each other and the resulting additional magnetic inhomogeneity leads to a lower density of nuclei for the backward reversal. This suggests that the increase in effective exchange coupling with decreasing temperature is an inhomogeneous process and that it is (at least partially) attributed to an increase in the density of the pinned sites and not to a general and homogeneously distributed increase in interfacial coupling. An inhomogeneous FM state as a consequence of an inhomogeneous FM/AFM state for a similar system was also suggested and modeled before[43] but have not been observed until now. The existence of a complicated magnetic FM/AFM interfacial structure was also found by Fitzsimmons *et al.*,[24] who showed that a interfacial region of pinned magnetization exists not only in the AFM but also in the FM layer. Assuming a similar behavior for our ultrathin FM film, the observed domain formation directly correlates with the magnetic distribution of the interfacial moments. The existence of such pinned moments in the FM entails the presence of incomplete domain walls, which separate domains of pinned and unpinned magnetization.

Similar effects from unpinned and pinned regions, as described above, could also result from instability effects in the "bulk" AFM itself.[7,44] To correlate the presence of magnetic inhomogenities at the AFM/FM interface with the behavior of exchange bias field, we therefore carried out a series of measurements. Experiments with different field cooling sequences were performed, and the results are shown in Fig. 6. First, the sample was cooled down ($H_{cf}^{(0)}=2.65$ kOe) as before to 17 K and H_E was measured for increasing temperatures up to $T=50$ K, at which a significant decrease in H_E had already been observed. Starting from the condition of reduced loop shift, the sample was then again cooled down but in a static magnetic field of the opposite direction ($H_{cf}^{(1)}=-2.65$ kOe). Neither with decreasing temperature nor with increasing temperature was a change in H_E relative to the initial condition observed (see Fig. 5). A second attempt to influence the exchange bias was carried out during cooling from 65 K back to 17 K in a static magnetic field ($H_{cf}^{(2)}=H_{cf}^{(1)}=-2.6$ kOe). The same temperature dependence of the exchange bias $H_E^{(2)}$ was observed again. This proves that the AFM's magnetic structure and the sign of exchange bias is

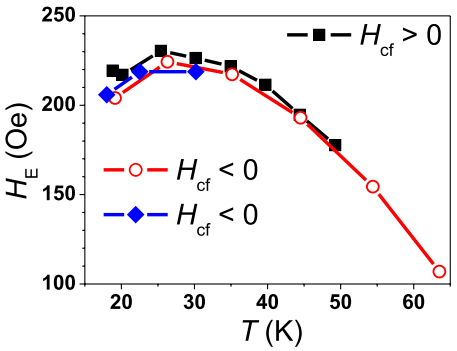

FIG. 6. (Color online) Change in H_E with magnetothermal history.

solely set at T_N. The observed change in H_E with decreasing temperature is due to an increase in coupling strength to the fixed AFM structure. Below T_N, the magnetization state of the FM layer has no influence on the sign or strength of the exchange bias. The interpretation made above on the change of the FM/AFM interface is valid.

IV. SUMMARY

The results of our study of exchange-coupled ultrathin single-crystal Fe/polycrystalline FeF_2 bilayers are in qualitative agreement with previously reported data, in which the FM has been polycrystalline. From that we conclude that the nature of exchange bias in these systems is the same as in the system presented. Our results strongly support the idea presented by Fitzsimmons *et al.*[24] of the presence of interface regions, which are magnetically distinctively different from either AFM or FM magnetic arrangements. Yet, such interface regions seem to be well defined for a given material system and our results indicate that the crystallographic arrangement plays only a minor role; i.e., it influences the value of the exchange bias but not the nature of the temperature dependence.

A significant increase in the FM domain density below T_N with decreasing temperature has been observed. We associate this observation with a locally increasing number of pinned moments.[35] Only the FM domain structure was imaged in our studies and our observations are in agreement with a recently proposed exchange bias model,[23] in which the pinned moments not only exist in the AFM interface layer but are also present in the FM layer. The occurrence of local pinning sites also manifests itself in an asymmetry visible from the magnetic domain formation during the magnetization reversal.

The measurements of the temperature dependence of domain density in conjunction with coercivity field enhancement and exchange bias field clearly suggest that both effects, exchange bias and coercivity, are different in origin. The experimental findings support recent theoretical predictions by Scholten *et al.*[35] The unpinned magnetic moments at the AFM/FM interface are responsible for the enhancement of the coercivity field. The temperature dependence of the coercive field can be associated with a change in coupling strength across the interface.

The observed behavior of exchange bias indicates that the magnetic arrangement in general, together with the interfacial magnetic structure in particular, has been set very close to T_N. It is immediately stabilized due to the large anisotropy of FeF_2 and increases with the interface exchange coupling. Despite the polycrystalline nature of the AFM, only local changes in the strength of interface exchange coupling occur below T_N.

ACKNOWLEDGMENTS

S.W. acknowledges funding through the Deutscher Akademischer Austausch Dienst (DAAD) for the stay at the IFW Dresden. The work at UCCS was supported by the National Science Foundation (Grant No. DMR 0605629).

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