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Coercivity engineering of exchange biased magnetic multilayer samples for digital encoding applications

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The dependence of the exchange bias field and coercivity enhancement on ferromagnetic (FM) and antiferromagnetic (AF) layer thickness in exchange biased bilayers has been systematically investigated in CoFe/FeMn and CoFe/PdMn bilayers for digital encoding applications in biotechnology. A magnetic multilayer structure can be used as a digitally encoded tag if each (bi)layer has two magnetic states, positive and negative saturation, available at remanence and if each layer can be uniquely identified by its coercivity. We will demonstrate that by adjusting the AF and FM layer thickness in an AF/FM bilayer, both the bias field and the coercivity of the bilayer can be controlled. By contrasting CoFe/FeMn bilayers with CoFe/PdMn bilayers, it becomes apparent that the relative magnitudes of the coercivity enhancement and bias field depend on the particular AF material, although the qualitative behavior remains unchanged. In order to create a multilayer that can retain one of many magnetic states at remanence, a large coercivity enhancement but absent or small bias field are preferred. Analysis of the bilayers suggest that PdMn is a better choice of AF layer for this purpose and results on some multilayer films are shown which validate this claim.
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I. INTRODUCTION

Magnetic media, already widespread in data storage technology, are currently being intensely considered in biotechnology for a variety of applications. One challenging potential application is the use of magnetically encoded tags in order to track, separate, and manipulate biological entities. Currently, optical encoding is most widely used in biotechnology. Although a powerful tool, optical encoding has inherent limitations due to the finite optical bandwidth and consequently the number of optical codes available for parallel processing is limited. Magnetism offers unique advantages due to the advanced development of magnetic decoding at high speed, which is the process underlying all present-day computer technology.

Magnetic beads are already used in a variety of biological applications, e.g, Refs. 1–10, but there are only a couple of types of magnetic beads available commercially, and their only distinguishing features are their size and magnetic moment. It is feasible to develop magnetic tags that are magnetically encoded in a more advanced way, e.g., by having a type of magnetic tag that can be put into one of many magnetic states. The ultimate outcome of this process would be the development of magnetic tags that consist of many magnetic layers that each can be brought into one of two magnetic uniform states (positive or negative saturation) and made to retain this magnetic state at remanence. Associating negative saturation of a magnetic element within such a tag with a "1" and positive saturation with a "0," a structure of many magnetic layers can thus represent a binary code. In this way a large library of uniquely identifiable tags could be generated which could be used for a large range of biotechnology applications. Although the focus of the current research is on the application of this kind of structure for biotechnological applications, it should be noted that similar structures are also being investigated as a method to increase the capacity of storage media by effectively extending the number of accessible (remanent) states per memory element.[11]

Much research has been devoted to the development and optimization of magnetic read heads and storage media, a process commonly referred to as spin engineering. Although the same basic magnetic principles underlie the development of magnetic tags, the constraints involved are fundamentally different and impose different requirements on the design. First of all, each magnetic layer should have a unique coercivity so that each layer can be manipulated separately and identified uniquely. A large range of coercivities is desirable, since the number of magnetic codes of a tag scales with the number of uniquely identifiable layers. Furthermore, the magnetic layers should have only two (positive and negative saturation) uniform magnetization states available with a sharp transition between these states (i.e., uniaxial anisotropy) and each of these states should be preserved at remanence. Finally, if the change in magnetization direction of one of the (bi)layers is to be detected by a change in total magnetic moment of the multilayer tag, then the magnetic moments of each layer need to be of the same order of magnitude in order to facilitate sensitive detection.

This set of requirements implies that optimal control

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over the switching behavior of each magnetic layer is required and therefore that just selecting different magnetic materials will not be sufficient. The coercive field of magnetic layers in a multilayer can be manipulated by tailoring the interlayer coupling with the nonmagnetic layer thickness, e.g, Ref. 12, but this requires epitaxy of the layers and is difficult to replicate exactly between different ultrahigh vacuum systems. For magnetic storage media, a bilayer of a ferromagnetic and antiferromagnetic material is often used in order to make one layer insensitive to a small applied field. Although this particular feature would not be useful for developing magnetic tags, the manipulation of the behavior of a magnetic (bi)layer under an applied field in this way is a desirable property if it can be extended to serve our purpose of additional control of the coercivity of a magnetic (bi)layer.

Exchange biasing is the effect where the close contact of two magnetic layers increases the coercivity of the ferromagnetic layer and shifts the hysteresis loop both horizontally and vertically, where the horizontal shift of the hysteresis loop is called the exchange bias field. This effect was discovered for the first time by Meiklejohn in 1956.[13] In the last decade, the potential uses of the exchange bias phenomenon for magnetic memory and hard-disk read heads has sparked a strong interest in exchange bias research.

Most commonly the exchange bias effect is observed in bilayers composed of a ferromagnetic and antiferromagnetic material,[14,16–19] or between a magnetic material and its native oxide,[20,21] and a very detailed review on research done in this area has recently appeared.[22] The effect is not confined to antiferromagnetic (AF)/ferromagnetic (FM) bilayers, however, recently there have been reports of bilayers with two FM layers exhibiting exchange bias as well.[23] In addition and in common with other forms of magnetic interactions, the two layers do not have to be in physical contact: exchange bias effects persist over thin metallic interlayers.[24–28]

Despite intensive research, the theoretical explanation of the phenomenon is still incomplete. Most models assume the exchange bias effect is due to stabilized domain structures due to the AF/FM interaction and theoretical calculations support the idea that this is a mainly antiferromagnetic parallel domain wall (Ref. 29, and references therein). There is much debate whether this is an interface effect[30] or a bulk-defect effect.[31,32] A detailed review of all theories focusing on interfacial effects by Kiwi[30] leads him to conclude that although the experimental values for bias field and coercivity can be theoretically derived for certain predefined systems, the dependence of the effect on interface roughness remains unsolved.

In general, the effect of the AF layer thickness on the coercivity is better documented than the effect of FM layer thickness. For PtMn (Ref. 22) and FeMn (Ref. 14) AF layers on NiFe, the coercivity was reported to exhibit the same behavior. To be more specific, there is a small coercivity for very thin AF layers, a steep increase, and a peak at a certain AF thickness that strongly depends on the material, and a more gradual decrease and leveling off of the coercivity after that. However, for chromium AF films on NiFe only an irregular increase in the coercivity was reported.[19] For Pd_{70}/Mn_{30} layers on 10 nm Co, the peak is also reported, but is followed by an almost linear increase in coercivity for AF layer thicknesses over 15 nm.[33] There are some results showing the dependence of the coercivity on the thickness of the FM layer, which report a $1/t$ dependence for the coercivity in Fe/MnPd bilayers[34] and the coercivity of IrMn/CoFe bilayers.[35]

The bias field dependence on the FM layer thickness has been shown to follow $1/t$ for NiFe/FeMn bilayers[17] and Fe/MnPd bilayers,[34] whereas as a function of the AF layer thickness it seems to steeply increase for a particular AF layer thickness and stay relatively constant for AF layers above that thickness.[14]

Both the bias field and coercivity enhancement have a strong temperature dependence, which in turn depends on the FM layer thickness. The importance of both the AF and FM layer thickness in the exchange bias phenomenon points to the crucial role of the anisotropies in both layers.[15]

Although coercivity is easily influenced by growth methods, surface and interface roughness as well as defects, the dependence of the coercivity on the AF layer means that exchange biasing is a possible way to control the coercivities of magnetic (bi)layers in a multilayer structure.

We will demonstrate that by adjusting the AF and FM layer thickness in an AF/FM bilayer, both the bias field and the coercivity of the bilayer can be controlled. By contrasting CoFe/FeMn bilayers with CoFe/PdMn bilayers, it becomes apparent that the relative magnitudes of the coercivity enhancement and bias field depend on the particular AF material, although the qualitative behavior remains unchanged. In order to create a multilayer that can retain one of many magnetic states at remanence, a large coercivity enhancement but absent or small bias field are preferred. Analysis of the bilayers suggests that PdMn is a better choice of AF layer for this purpose and results on some multilayer films are shown which validate this claim.

II. METHODS

The samples were fabricated by direct current-magnetron sputtering, in a CEVP sputtering chamber with a base pressure of 3×10^{-9} Torr and an argon processing pressure of 3×10^{-3} Torr. During growth, a magnetic field of 200 Oe was applied along the (110) direction of the silicon wafer and the sample was not rotated. The growth was computer controlled and all the growth rates were in the range of 1–2 nm per minute. The samples were grown at room temperature on *p*-type silicon pieces of 1 cm^2, where 5 nm tantalum was used as a buffer and capping layer, and in the case of the double bilayer also as a spacer layer. It is known that the Ta seed layer promotes a ⟨111⟩ directional axis growth of the subsequent CoFe layer.[36]

For the samples with FeMn as the AF layer, a $Fe_{50}Mn_{50}$ (at. %) target was used, while for the samples with PdMn as the AF layer, a $Pd_{50}Mn_{50}$ (at. %) target was used. The CoFe target had a $Co_{50}Fe_{50}$ (at. %) composition. All targets have a 99.9% purity. The Ta, as-deposited, grows in the body-centered-cubic (bcc) structure with a lattice parameter of 3.301 Å. $Co_{50}Fe_{50}$ is a ferromagnetic material also known as Permendur, with a Curie temperature of 800 °C,[37] which

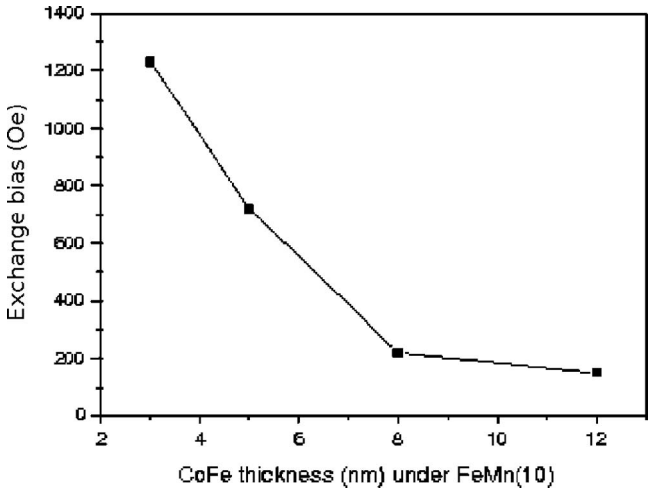

FIG. 1. Bias field as a function of CoFe layer thickness for CoFe/FeMn bilayers.

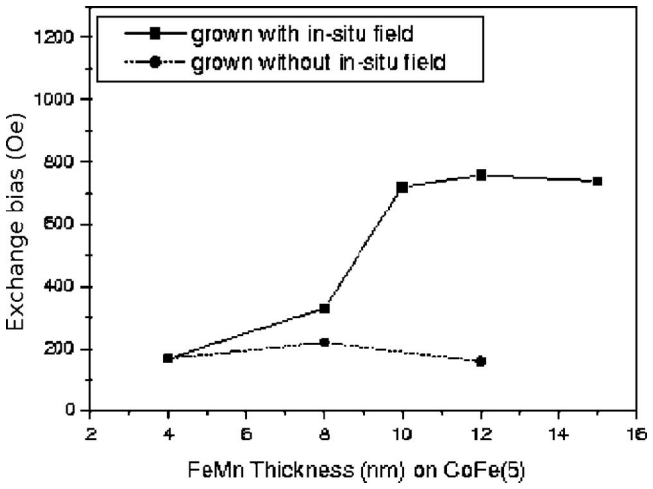

FIG. 2. Bias field as a function of FeMn layer thickness for CoFe/FeMn bilayers.

grows in the bcc structure with a lattice parameter of 2.855 Å.[38,39] FeMn is a face-centered-cubic antiferromagnet with a lattice parameter of $a=3.692$ Å for the disordered γ-FeMn alloy and a Néel temperature of 500 °C. PdMn has a Néel temperature of 540 °C and grows in the chemically ordered CuAu I phase ($L1_0$). The lattice parameters are 3.58 and 4.07 Å for the c and a axis, respectively, in bulk PdMn,[37] and it has been reported previously to grow epitaxially on Fe.[40,41]

The structure of all samples with thicknesses given in nanometers were as follows: Si/Ta(5)/CoFe(x)/PdMn or FeMn(y)/Ta(5), where both the CoFe thickness and PdMn or FeMn thickness were varied. A comparison of the lattice parameters of the materials in the samples prepared indicates that all the multilayers are expected to be polycrystalline. After deposition, magnetic measurements were performed using a high sensitivity magneto-optical Kerr-effect (MOKE) setup. The focused-spot size of this setup is 3 μm, although the spot was deliberately defocused during the experiments so as to avoid detecting domain-size effects.

III. RESULTS AND DISCUSSION

After growth, the loop width (as a measure of coercivity) and the exchange bias field of the as grown samples were measured as a function of both AF and FM thickness. For clarity the results for FeMn as the AF layer and PdMn as the AF layer are discussed separately.

A. FeMn as AF layer

The samples show an approximate $1/t_{\mathrm{CoFe}}$ decrease of the bias field as a function of the CoFe thickness, going from almost 1200 Oe for 4 nm CoFe under 10 nm FeMn to 180 Oe for 12 nm CoFe under 10 nm FeMn, Fig. 1. The bias field of the CoFe/FeMn samples grown under a field increases with FeMn thickness from 200 Oe for 4 nm FeMn on 5 nm CoFe to 800 Oe for 16 nm FeMn on 5 nm CoFe, Fig. 2. It can be seen that even for thin AF layers there is a significant bias field.

In Fig. 3, the loop width can be observed to decrease with increasing FeMn thickness from around 350 Oe with 4 nm FeMn on top of 5 nm CoFe to around 100 Oe with 15 nm FeMn on top of 5 nm CoFe. A similar decrease is seen for the dependence of the loop width on the CoFe thickness, Fig. 4, where the loop width of 470 Oe for 3 nm CoFe under 10 nm FeMn decreases to around 100 Oe for 12 nm CoFe under 10 nm FeMn. Previous reports[22] would make one expect to see a peak in the coercivity dependence on the AF thickness, rather than an exponential decay. It is possible, however, that the FeMn biased samples show a peak in coercivity for a certain FeMn thickness (like will be observed later for the PdMn biased samples) at a thickness less than 4 nm FeMn, which thus does not show up in our measurements.

For reference, three samples have been grown without a field *in situ*, the results of which are represented by the second line in Figs. 2 and 3. Without an *in situ* field grown samples do not show the increase in bias field as a function of FeMn thickness. For thicker FeMn layers, however, the loop width tends to the same values as for the field grown samples, but for the thin FeMn layers the loop width is much

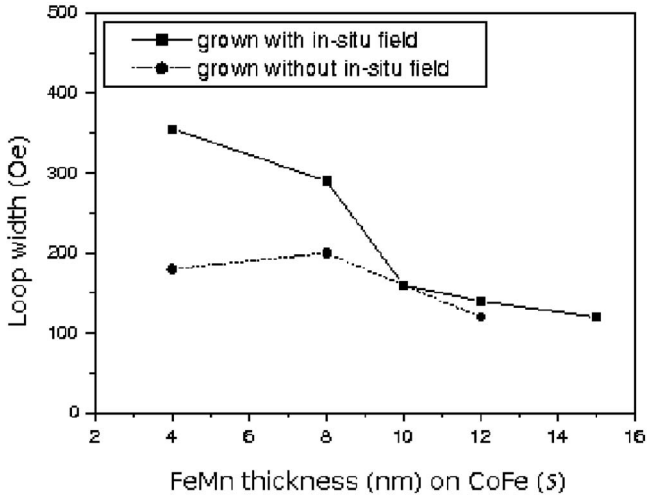

FIG. 3. Loop width as a function of CoFe layer thickness for CoFe/FeMn bilayers.

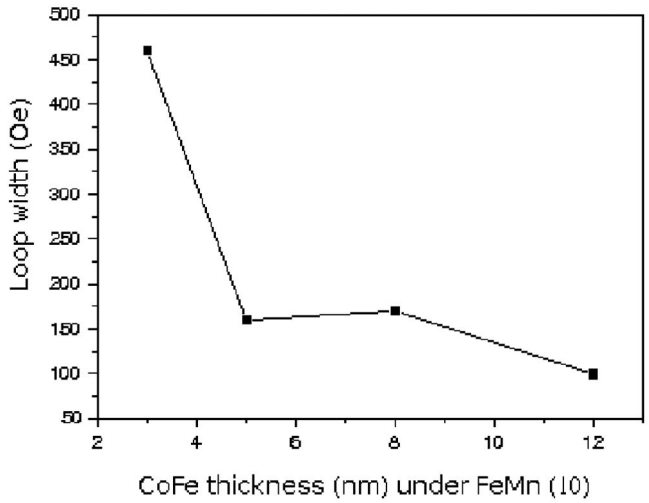

FIG. 4. Loop width as a function of FeMn layer thickness for CoFe/FeMn bilayers.

smaller, only 175 Oe as compared to the 370 Oe for the field grown samples.

Combining similar bilayers in a multilayer structure gives independently switching bilayers. In Fig. 5 the magnetic behavior of a sample with the following composition can be seen (where the numbers between brackets are in nm): Si/Ta(5)/Co(5)/Ta(10)/Co(2)/FeMn(10)/Ta(10)/Co(5)/FeMn (20)/Ta(5). Due to the limited penetration depth of the laser in a MOKE setup, these samples are measured in a superconducting quantum interference device, at 110 K. It can be seen that the single cobalt layer (labeled 1) is not biased and switches with a loop width of about 20 Oe symmetrically around the origin. From the two exchange biased layers, the Co(5)FeMn(20) layer is biased less than the Co(2)FeMn(10) and thus is the switch visible around (−)500 Oe (labeled 2) with a coercivity of approximately 200 Oe. The Co(2)FeMn(10) layer is most biased and has a switch around

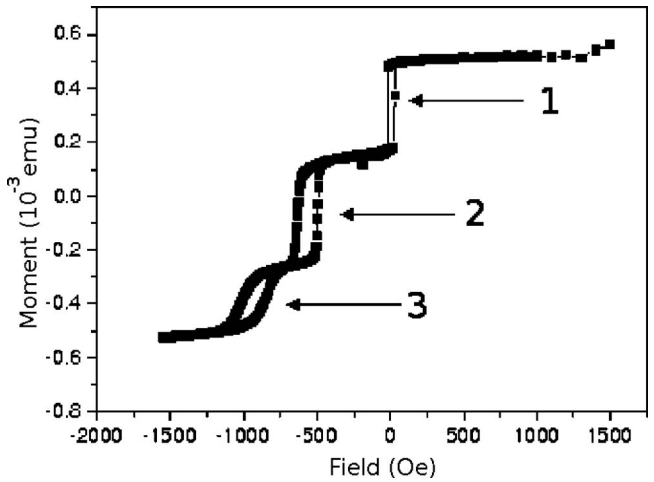

FIG. 5. Hysteresis loop of Si/Ta(5)/Co(5)/Ta(10)/Co(2)/FeMn(10)/Ta(10)/Co(5)/FeMn(20)/Ta(5), where the numbers in brackets are thicknesses in nanometers. The switch of the single cobalt layer is not biased and labeled "1." The Co(5)FeMn(20) bilayer switch is labeled "2" and the Co(2)FeMn(10) layer switch is labeled "3." It can be seen that both biased layers lack a choice of state at remanence; they will always be saturated along the positive field direction.

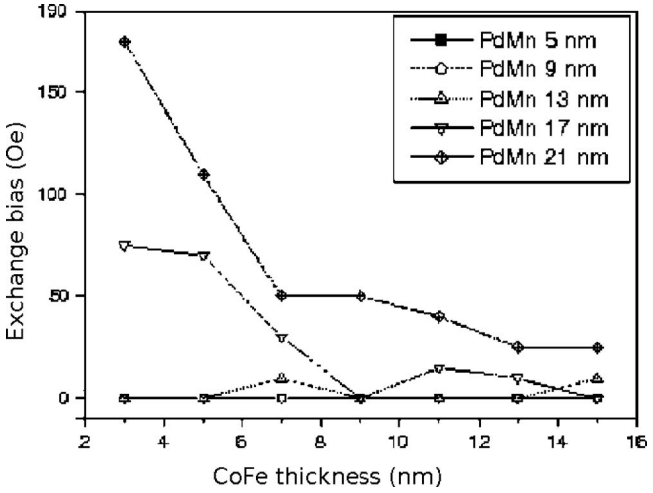

FIG. 6. Bias field as a function of CoFe layer thickness for CoFe/PdMn bilayers.

(−)1000 Oe (labeled 3) with a similar loop width as the other bilayer, around 200 Oe. Although these samples show independent switching of each magnetic (bi)layer, the very large bias of the bilayers means that the loops of the magnetic layers are so far separated that it is not possible to put the sample in a state that is preserved at remanence. In order to make samples that fulfill both requirements of independent switching as well as the possibility of a preserved state at remanence, it is necessary to look for an AF layer that does have a similar enhancing effect on the coercivity but does not show such a large bias field. This is the reason for using a less commonly used AF material in the form of PdMn.

B. PdMn as AF layer

In order to examine the magnetic behavior of CoFe/PdMn bilayers, a large number of samples of the form Ta(5)/CoFe(x)/PdMn(y)/Ta(5) were grown in identical circumstances. The CoFe thickness was varied systematically under a range of PdMn thicknesses. For each PdMn thickness, the value of the exchange bias with varying CoFe thickness has been plotted and represented by a separate line in Fig. 6. It can be seen that in general the bias field decreases almost as $1/t_{\mathrm{CoFe}}$, similar to the FeMn case. However, this effect is only pronounced in the samples with 17 and 21 nm of PdMn, where the bias field decreases from 175 and 75 Oe, respectively, with 3 nm CoFe to 0 Oe for larger CoFe thicknesses. For thinner layers of PdMn the bias field seems negligible. In Fig. 7 it can be seen that for the studied thickness range of CoFe films the bias field increases with PdMn thickness and that the effect is more pronounced for the thinner CoFe layers. All these trends are similar to the results of the FeMn biased samples, however, the value of the exchange biased field observed is smaller.

In Figs. 8 and 9 the loop-width characteristics of the same CoFe/PdMn samples are presented. All samples show a greatly enhanced loop-width, up to as much as 550 Oe for 3 nm CoFe under 13 nm PdMn, which decays with increasing CoFe thickness, Fig. 8. Although all samples exhibit a decreasing loop width upon increasing the CoFe thickness, it is important to note that the samples with a very high initial

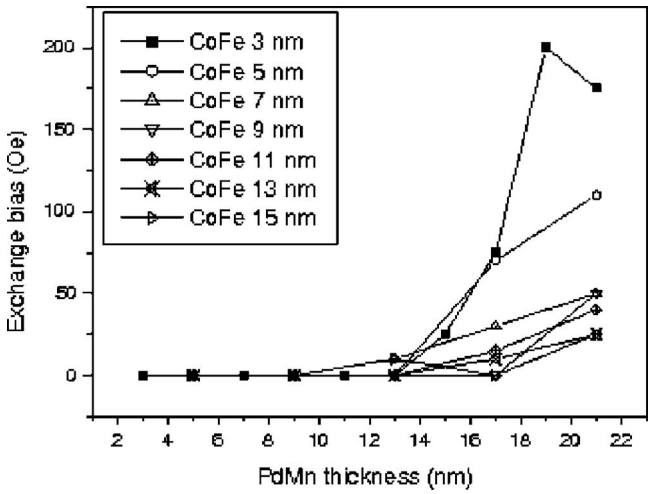

FIG. 7. Bias field as a function of PdMn layer thickness for CoFe/PdMn bilayers.

loop width still retain it even for large CoFe thicknesses, e.g., the before mentioned sample of 3 nm CoFe under 13 nm PdMn still has a loop width of 200 Oe when the CoFe film thickness is increased to 15 nm. Furthermore, the coercivity enhancement has a distinct peak around 12 nm of PdMn for the whole thickness range of CoFe films studied, as can be seen in Fig. 9. We do not observe an increase in coercivity for larger AF film thicknesses as reported by Thuy *et al.*[33] for Pd_{70}/Mn_{30} layers on 10 nm Co.

For molecular beam epitaxy grown Fe/MnPd bilayers, the absolute value of both the coercivity and the bias field have been shown to decrease as $1/t_{Fe}$.[34] The samples prepared for this research show a similar decay for the coercivity and the exchange bias as a function of the FM layer thickness.

The small exchange bias combined with the high coercivity makes the CoFe/PdMn system a more suitable magnetic system for the purpose of creating independently switching bilayers in a multilayer system that retain their magnetic configuration at remanence because there is not

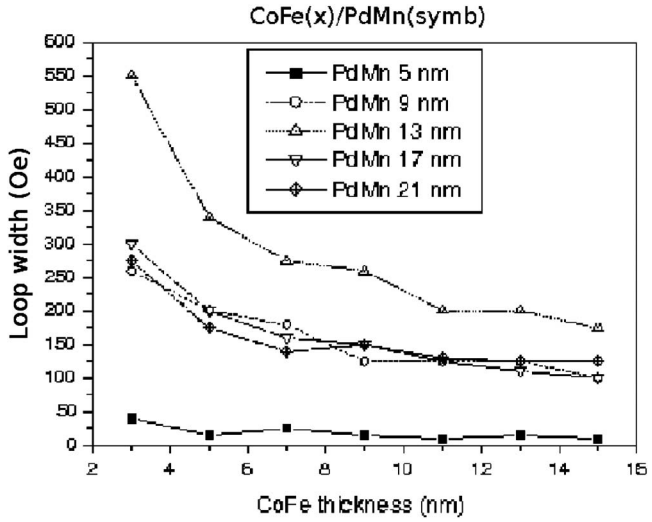

FIG. 8. Loop width as a function of CoFe layer thickness for CoFe/PdMn bilayers.

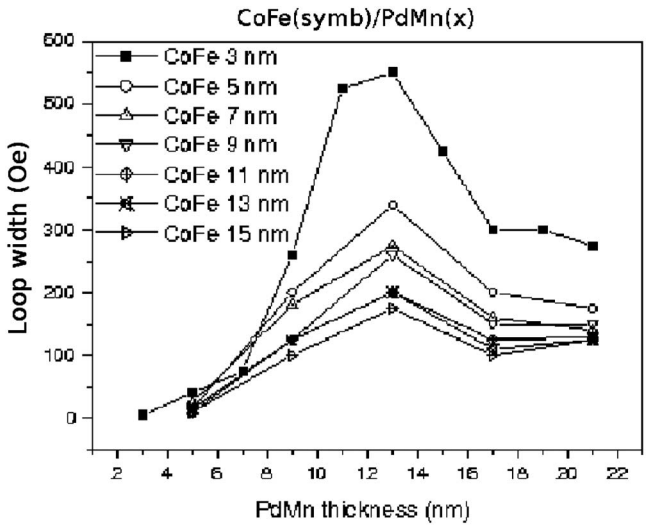

FIG. 9. Loop width as a function of PdMn layer thickness for CoFe/PdMn bilayers.

enough exchange bias to separate the different hysteresis loops. This principle is demonstrated in Fig. 10, where a PdMn(5)/CoFe(9) (top) and a PdMn(13)/CoFe(3) (bottom) are separated by 5 nm of Ta. The bilayer with 13 nm PdMn has the largest coercivity and thus forms the outside loop, indicated with the number 2. The PdMn(5)/CoFe(9) layer has a small loop width and the switch is indicated with the number 1. The fields are well resolved and by selecting a minor hysteresis loop a particular configuration of magnetization directions in the bilayers can be selected at remanence. For example, applying a large (over 300 Oe) field in the negative direction will saturate both bilayers in the same direction, and up on removal of the field the multilayer stack will remain in state "*C*." If, however, the field is brought down from positive saturation to −100 Oe, only the top layer will reverse direction and upon removal of the state the

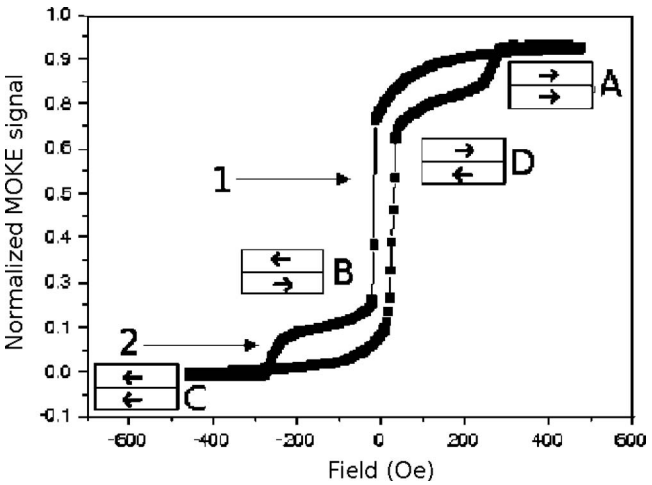

FIG. 10. Hysteresis loop of PdMn(5)/CoFe(9) (top layer) and a PdMn(13)/CoFe(3) (bottom layer) separated by 5 nm of Ta. Schematics indicate the magnetization direction of each bilayer. The PdMn(5)/CoFe(9) layer has the smallest coercivity and reverses magnetization at 1. The PdMn(13)/CoFe(3) layer has the largest coercivity and reverses magnetization at 2. It can be seen that both (bi)layers switch independently at well-resolved fields and have four magnetic configurations that can be preserved at remanence.

multilayer will remain in state "*B*." Similarly starting from negative saturation the multilayer can be brought to and left in state "*D*" or "*A*" dependent on the magnitude of the positive field applied. The principle thus demonstrated could be extended to more layers, if each layer has a well-defined unique coercivity. This can be achieved by careful selection of the FM and AF layer thicknesses.

A comparison of the lattice parameters of the materials in the samples prepared indicates that all the multilayers are expected to be polycrystalline. This possibly explains why the switch labeled with the number (2) looks harder than the first switch: it is the top layer and thus is expected to have a larger roughness, enhancing dipole coupling between the two layers and making the layer harder to switch. This could also explain why the loop width of the PdMn(13)/CoFe(3) layer of almost 600 Oe is slightly larger than the 530 Oe one would expect from Fig. 8.

The benefit of using PdMn rather than FeMn as an AF layer can immediately be seen. FeMn has a large bias field and comparatively low coercivity enhancement compared to PdMn. When using FeMn in a multilayer stack, the hysteresis loop of each bilayer thus moves far away from around the origin, as can be seen in Fig. 5, which means that bilayer will always have the same magnetic configuration at remanence. This is in contrast to the PdMn biased multilayers, where the coercivity enhancement is large but the bias field small, allowing for a design of stack that has a hysteresis loop centered around zero and with each layer switch showing as a distinct step in the hysteresis loop. A possible explanation for the different effect observed for PdMn and FeMn could be differences in the direction and magnitude of the anisotropy present in each due to their different crystal structure. The key role of anisotropy in models of exchange bias has been outlined by Binek and co-workers.[42] In addition, it should be noted that CuAu-I type AF materials, like PdMn, generally need postannealing to achieve the fully ordered state[33,43] and that omitting this annealing procedure could lead to a frustrated interface resulting in a higher coercivity and smaller bias field.[44] The reproducibility of the bias and coercivity when incorporated in the multilayer system suggest this is not the case, but further research is planned to investigate the effect of annealing.

In this work the as-deposited structures have been measured. This has been done because it is anticipated that with the intended eventual application in biotechnology the ease and cost of fabrication will be an important factor. In addition, the effect of cooling procedures becomes rather more complicated in a multilayer structure which will potentially have up to several tens of bilayers. However, it is recognized that the coercivity can be tuned depending on the particular cooling procedure[44,45] and that the bias field can be removed by cooling in an alternating current field[46,47] and future research is anticipated to investigate the effect of these procedures on the systems investigated.

IV. CONCLUSION

The coercivity enhancement and exchange bias field in CoFe/FeMn samples have been systematically compared to those in CoFe/PdMn samples. A contrasting behavior is observed, where the samples with FeMn as the AF layer show a much larger exchange bias field compared to the samples with PdMn as the AF layer, but a much smaller coercivity enhancement. The possibilities of using the exchange bias phenomenon in order to design magnetic multilayer stacks have been investigated and it has been shown that PdMn is a more suitable AF material for this purpose, due to its large coercivity enhancement combined with a small horizontal displacement of the hysteresis loop. These magnetic multilayer stacks could potentially contain many tens of magnetic (bi)layers, that with an appropriate choice of materials and thicknesses could all be designed to reverse their magnetization at well resolved field values either side of the origin, thus allowing a selection of the layer magnetization state at remanence. Using AF/FM bilayers has the additional advantage that all layers have magnetic moments in the same order of magnitude, which facilitates high-resolution detection. Magnetic multilayer stacks designed in this manner could have many potential applications ranging from a three-dimensional extension of memory arrays to applications as magnetic labels in biotechnology.

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