Systematic layer-by-layer characterization of multilayers for three-dimensional data storage and logic

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Abstract
Magnetic kink solitons are used as a probe to experimentally measure the layer-by-layer coercivity and interlayer coupling strength of an antiferromagnetically coupled perpendicularly magnetized Co multilayer. The magnetic response is well described by a nearest neighbor Ising macrospin model. By controlling the position of one, two or three solitons in the stack using globally applied magnetic fields, we successfully probe the switching of individual buried layers under different neighboring configurations, allowing us to access individual layer’s characteristic parameters. We found the coercivity to increase dramatically up the multilayer, while the interlayer coupling strength decreased slightly. We corroborate these findings with scanning transmission electron microscopy images where a degrading quality of the multilayer is observed. This method provides a very powerful tool to characterize the quality of individual layers in complex multilayers, without the need for depth-sensitive magnetic characterization equipment.

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(Some figures may appear in colour only in the online journal)
(RKKY) interaction [13–15]. For an 11 magnetic layers system, CoFeB-based MLs showed coercivities and interlayer coupling values independent of the layer number. On the contrary, similar Co based MLs showed a large spread in their magnetic properties [11]. This points to a possible degradation of the magnetic properties with increasing layer number. Achieving both spatial and magnetic resolution in the depth of such an ultrathin ML sample is a challenging problem. Some very powerful techniques exist, such as polarized neutron reflectometry [16, 17] or x-ray magnetic dichroism [18, 19]. However, these techniques involve heavy instrumentation and for the latter, depth sensitivity is achieved due to the element specific sensitivity of the technique, which renders it useless for a ML composed from repeating material sequences as used here. Magneto optical Kerr effect (MOKE) measurements also provide depth resolution [20], but in addition to the difficulty of the method, it is also fundamentally limited to how deep the light penetrates into the ML, ~25 nm corresponding to roughly 13 magnetic layers for metallic ML as used here. In this paper we show that by placing solitons at different heights in a soliton ratchet ML stack we can use a simple bulk magnetometry technique (vibrating sample magnetometry—VSM) to probe every layer in the ML individually, effectively using solitons as local probes at the scale of the ultrathin magnetic layers. This is achieved by using solitons to prepare independent switching configurations for each layer. In a near the nearest neighbor Ising macrospin model (nnIMM), which describes our ML, the ability to measure the switching of a given layer with different neighboring configurations allows us to systematically determine the magnetic properties of each magnetic layer individually, i.e. its coercivity and RKKY coupling strength to its nearest neighbors. In other words this process allows us to compose a magnetic map of the full ML. This gives us insight into changing magnetic properties as a function of layer number while keeping growth conditions and nominal ratchet properties constant. This work is fundamental to optimizing soliton ratchet MLs. This map of magnetic properties is compared to structural measurements performed using scanning transmission electron microscopy (STEM).

The layout of this paper is the following. In section 2, we present the sample fabrication conditions as well as the magnetic and structural measurements performed. We explain the principle of the nnIMM and use the simple case of a bilayer to illustrate how to determine the coercivity and coupling parameters for each layer. Then we present our ML sample and briefly review the soliton ratchet mechanism. In section 3, we start by showing how, when nominally identical layers are not identical, the transitions present in the major hysteresis loop cannot be unequivocally attributed to their corresponding layer. We then present two detailed examples of measurements which allow for controlled layer-by-layer switching of the stack. Using the as-determined switching fields of all layers in various configurations we subsequently extract the coercivity and interlayer coupling strength as a function of layer number. We find that although the interlayer coupling decreases only slightly, the coercivity of the magnetic layers increases substantially as the layer number increases before dropping abruptly for the topmost layer. Finally, we show that the results are consistent with the degradation of the structural properties of the ML as observed using a STEM micrograph of a lamella of the ML.

2. Methods

2.1. Sample fabrication

The magnetic layers are made of Co, the coupling layers are 0.9 nm Ru. In order to tune the strength of the AF coupling as well as ensure strong perpendicular anisotropy, different thicknesses of Pt are inserted on both sides of the Ru layer [21]. The sample studied here is the same as the one studied in [11]. The samples are fabricated by DC magnetron sputtering using an Ar pressure of 7.5 × 10⁻³ mbar in a vacuum system with a base pressure of 3 × 10⁻⁵ mbar. All samples are prepared on precut Si substrates (~1 × 1 cm²) with a native oxide layer. The ML is grown on a buffer layer of Ta (4 nm)/Pt(20 nm) and capped with a 2 nm Pt layer. For Ta, Co and Ru DC magnetron powers of 50, 60, 100 W were used, respectively. For Pt we used 100 W for the buffer and capping layers and 30 W for the interlayers. During deposition the substrates were rotating (20 rot min⁻¹).

2.2. Measurements

Easy axis (perpendicular to the film plane) VSM measurements were taken at room temperature. All VSM data presented are obtained by subtracting the diamagnetic background of the Si substrate measured at applied fields >10 kOe. The switching fields were estimated as the peak of the derivative of the magnetization with respect to the applied field (see the supplementary material section). To investigate the microstructure of the samples we have performed high resolution STEM on cross-section lamellas prepared by focused ion beam milling. STEM was carried out using a FEI80-300 Titan Supertwinlens TEM operating at 300 kV equipped with a Fischione HAADFSTEM detector and Fischione tomography holders.

2.3. Nearest neighbor macrospin model, bilayer case and beyond

Here we explain how we determine the coercivity and coupling parameters from hysteresis loops using the nnIMM [6] and we illustrate the procedure in the particular case of a bilayer. In the nnIMM, the switching field $H_{SW}$ of each layer $i$ is characterized by its coercivity $H_c(i)$ and the interlayer coupling strength $J_{i-1}$ and $J_i$ with layers $i - 1$ and $i + 1$. It further depends on the magnetic configuration $\mu_{i±1}$ (=±1) of neighboring layers in the following manner:

$$H_{SW}(i) = -\mu_i H_c(i) + \mu_{i-1} \frac{J_{i-1}}{t_i} + \mu_{i+1} \frac{J_i}{t_i},$$

where $t_i$ is the thickness of layer $i$. In order to determine the layer-by-layer values of $H_c(i)$, $J_{i-1}$ and $J_i$, it is necessary to experimentally access switching events where each layer reverses under the influence of different neighboring
configurations $\mu_{i-1}$ and $\mu_{i+1}$. Since each interlayer coupling is shared between two magnetic layers, only two non-equivalent switching configurations are required for each magnetic layer.

In the following we illustrate this in the simple case of a bilayer. In a bilayer system with thicknesses $t_1$ and $t_2 > t_1$ and AF interlayer coupling $J$ (see figure 1(a)), a typical minor loop (solid line) and major loop (dotted line) might look like the ones represented figure 1(b). Here and in the rest of the paper, the subscript for the switching field $H$ is of the form $X \pm \rightarrow Y \pm$, where $X$ and $Y$ describe the initial and final configurations of the layer considered relative to its neighbor(s), either parallel P or AP, and ‘+’ and ‘−’ describe the direction of the magnetization in the layer considered, both up (+) or down (−). According to equation (1), the minor loop shows two transitions, at $H_{P \rightarrow AP-1}(1) = H_{c}(1) - J/t_1$ and $H_{AP-\rightarrow P-1}(1) = -H_{c}(1) - J/t_1$. $H_{P \rightarrow AP}$ and $H_{AP-\rightarrow P}$ are two independent linear combinations of $H_{c}(1)$ and $J$. Upon inversion, these two equations yield: $H_{c}(1) = \frac{1}{2}(H_{P \rightarrow AP-1}(1) - H_{AP-\rightarrow P-1}(1))$ and $J = -\frac{1}{2}(H_{AP-\rightarrow P-1}(1) + H_{P \rightarrow AP-1}(1))$. In other words, layer 1 was fully characterized by measuring its switching in two independent configurations (AP to P and P to AP). Because $J$ is shared between layers 1 and 2, only one equation involving the switching of layer 2 is needed in order to determine $H_{c}(2)$. This is provided by the second switching of the major hysteresis branch: $H_{AP-\rightarrow P-2}(2) = H_{c}(2) + J/t_2$. Using the previously determined value for $J$, $H_{c}(2)$ can now be estimated and the whole ML is characterized. It should be noted that $H_{P \rightarrow AP-2}(2)$ is not accessible experimentally, as from the P configuration, the thinner (and therefore more highly coupled) layer 1 will always switch into the AP configuration before layer 2.

This simple method cannot be extended to a non soliton-carrying ML stack with three or more layers. By soliton-carrying ML we refer to a ML engineered to sustain the controlled propagation of a soliton, for instance the one demonstrated in [6]. In the case of a non soliton-carrying ML, it is not generally possible to prepare the required magnetic configuration to isolate the switching of a single layer and a depth-insensitive bulk magnetometry technique will not allow unique identification of each individual transition with its corresponding layer. In a soliton carrying ML however, soliton positions can be tuned such that single layer switching can be realized at any position in the ML. This allows the switching field of every layer to be extracted, similarly to the bilayer case. In the following we demonstrate how this analysis was successfully performed on an 11 magnetic layer ML, allowing us to extract each individual coercivity and interlayer coupling strength. In the ideal case where the magnetic properties of the soliton ratchet are homogenous throughout the ML (from now on called nominal identical) a single set of $H_{c}$, $J$ and $t_1$ determines all switching fields.

2.4. Principle of soliton propagation in soliton ratchet MLs

Here we review the basic operation of the soliton ratchet ML in the framework of the nnIMM model as described in [6]. Our 11 layers ML sample is schematized in figure 2(a). In the propagation region (layers 4–11), alternating the magnetic thicknesses ($t_1$ and $t_2 > t_1$), and the interlayer AF coupling strengths ($J_1$ and $J_2 < J_1$) ensures upward propagation of solitons as long as $H_{c} > (t_2 - t_1)(J_1 - J_2)/(2J_1t_2)$ [6]. Solitons are defined as the two layers pointing in the same direction present at the boundary between two domains of opposite AP phase (layers 5 and 6 in figure 2(a)). In devices demonstrated experimentally so far, the bottom three ‘injector’ layers are made of thin ($t_1$ and $t_0 < t_1$) and highly coupled ($J_0 > J_1$) layers and are used to inject new solitons at the bottom of the ML. The behavior of the injector, although not described by the nnIMM, is however fully characterized and reproducible [11]. The upward propagation of a soliton occurs in two steps. A negative soliton (by convention defined as pointing down when straddling a $J_2$ coupling at remanence—see figure 2(a)) propagates one layer up upon increasing the external perpendicular $z$ field (see figure 2(d)) as the top $t_2$ layer forming the soliton switches at propagation field step 1:

$$H_{P- \rightarrow P-1} = H_{c} + (J_1 - J_2)/t_2.$$  

(2)

The negative soliton is now in an intermediate position where it points upwards and straddles a $J_1$ coupling—see figure 2(b). It propagates one more layer up upon decreasing the field as the $t_1$ layer, now at the top of the soliton, switches...
In a homogeneous ML, the switching of odd identical layers 5, 7 and 9 from the P to AP configuration should happen at once (T2, expected height 0.47), followed by the switching of layer L11 from P to AP (T3+0.15). The experimental height between C4 and C2 is about 0.62, very close to the expected value of 4 × 0.15. However, what should happen over two distinct transitions in a nominally identical ratchet ML happens over three transitions (a, b and c in figure 3(c)). Similarly, the simultaneous switching of all even layers 6, 8 and 10 from AP to P (T6) happens over three distinct transitions in the experimental loop (d, e and f), which all have a height close to the expected 0.23. This clearly shows that a single value of $H_c$, $J_1$ and $J_2$ cannot describe the whole of the propagation region. However, no further quantitative analysis is possible using the major loop only.

3.2. Single soliton propagation loop

We will now show how we can use solitons to prepare the magnetic configuration of the ML in order to observe the switching of each layer individually. Our sole assumption, which will be verified later as a self-consistency check, is that individual layer parameters locally fulfill the ratchet conditions so that a single soliton can still propagate through the ML. See the supplementary material section for more details and the self-consistency check stacks.iop.org/nano/27/155203/mmedia.
So far, the loop shown in figure 4 has allowed filling of the dark gray entries in table 1. Each entry has three numbers: $A \pm B (C)$; $A$ is the transition field averaged over $C$ different measurements, $B$ is the standard deviation of the measurement set.

### 3.3. Preparing the ML into different switching configurations: soliton annihilation

Now that all the propagation transitions are determined, we need to prepare the ML into configurations which allow each layer to be isolated as it switches from AP to P or from P to AP. Figure 5 shows such a process. This measurement was already presented in [11]. Starting from the C4 configuration with a negatively polarized soliton straddling layers 3 and 4, the field increases to above $H_{P1-}(4)$ (black), then decreases to below $H_{P2-}(5)$ (red) and finally increases to above $H_{P1-}(6)$ (dark green), so that the soliton is now between layers 6 and 7. Then, instead of decreasing the field in order to further propagate the soliton, the field keeps increasing until it reaches the injection field $H_{Inj}$ at which the bottom three layers flip and a new positively polarized soliton is injected between layers 3 and 4 (blue). The field subsequently decreases to below $H_{P2-}(7)$ (orange), and further to below $H_{P1-}(4)$ (black). The switching fields appearing in this sequence so far were already measured in figure 4. The new values measured here participate into the final averaging already mentioned. We are now in the configuration marked with a black star, where two solitons are present in the stack, straddling layers 4 and 5 and layers 7 and 8 (marked with a black oval). More importantly, only layers 6, 9 and 11 are left to switch from AP+ to P− if the field further decreases. These three layers are nominally different. Since it is stabilized by an AP layer on one side only, the first one to switch is layer 11 at $H_{AP+p−}(11)$ (light green). Then the injection field $H_{Inj}$ is reached and the bottom three layers reverse (blue), followed by layer 6, which is thicker than layer 9 and therefore has the smaller $H_{AP+p−}(9)$ (pink), and finally by layer 9 at $H_{AP+p−}(9)$ (gray). The light gray entries in table 1 are now determined.

In this example, two solitons were placed in the stack so that the layer in between the solitons (layer 6) could be isolated from the other layers and prepared to switch from the AP+ to the P− configurations. In the supplementary material is shown another example where the same method is used for layer 8.

### 3.4. From switching values to layer-by layer characteristic properties: coercivity and coupling versus layer number

Seven more sequences are required to generate all the necessary switching configurations and fill the table, these are shown in the supplementary material section. This selection of nine sequences which we present is by no means exhaustive. Two more independent measurements are shown in the supplementary material section, and another 12 (not shown) were performed, which confirmed the previous measurements.
and allowed building up the number of measurements for each transition which we used in the final averaging of table 1. The distribution of switching fields for a given transition is very narrow compared to the values of the switching fields, showing the reproducibility and the robustness of the switching processes.

Once the table is full, we have more data than necessary in order to determine the layer-by-layer coercivities and interlayer couplings. The details of the algebra for the inversion of the table can be found in the supplementary material section. The final results are shown in figure 6(a) for the coercivity and (b) for the interlayer coupling as a function of layer number. Apart from the top layer 11, the coercivity shows a clear and almost steady increase up the stack, from 500 Oe for layer 4 up to 2000 Oe for layer 10, i.e. +400%, before dropping back to 500 Oe at layer 11. In parallel, both $J_1$ and $J_2$ interlayer couplings are seen to decrease up the stack, although the relative amplitude of the change in coupling, $-18\%$ (for $J_2$, from 340 to 280 Oe nm) and $-15\%$ (for

| Layer | $H_{P1}^-$ | $H_{P2}^+$ | $P$- to $AP^+$ | $AP^-$ to $P^+$ |
|-------|------------|------------|---------------|-----------------|
| L_4   | 1560 ± 25  | -855 ± 15  | 2320 ± 30     | 3260 ± 40       |
| L_5   | 1980 ± 30  | -265 ± 30  | 3510 ± 35     | 3830 ± 40       |
| L_6   | 2565 ± 10  | 30 ± 25    | 2730 ± 35     | 3260 ± 40       |
| L_7   | 2975 ± 110 |            | 3860 ± 50     | 3475 ± 25       |
| L_8   |            |            | 4300 ± 60     | 2335 ± 40       |
| L_9   |            |            |                |                 |
| L_10  |            |            |                |                 |
| L_11  |            |            |                |                 |

Table 1. Table summarizing the values of the different possible switching fields for each layer in the ML. Each entry has three numbers: $A \pm B$ ($C$); $A$ is the transition field averaged over $C$ different measurements, $B$ is the standard deviation of the measurement set. In order to represent a negative to positive transition, $H_{P1}^-$ is reported for a negative soliton (−) and $H_{P2}^+$ is for a positive soliton (+).
$J_1$, from 1300 to 1100 Oe nm) is a lot smaller than the relative amplitude of the change in coercivity.

In order to correlate our findings on the change in properties with layer number with some possible structural variations in the ML, we have performed a cross-sectional STEM of the ML. This is shown in figure 7. In figure 7(a) we show a large area image. A columnar growth mode is observed where the columns/grains are schematically indicated by the vertical dashed white lines. The horizontal size of the columns/grains (~8–25 nm) seems to be set by the roughness at the bottom of the thick Pt buffer layer: defects at the bottom of the Pt buffer layer propagate upwards and define the grain size in the Pt buffer layer. Whether this originates from the Si substrate or from the Ta underlayer is not clear. In figure 7(b) a zoom-in (green-box) is shown of an area where a column/grain is showing a high individual layer contrast. The Pt layers appear bright and the Co layers appear dark, with Ru at an intermediate brightness. From this zoom-in we can see that the top surface is very rough, where the roughness is correlated with the columnar structure underneath. This roughness makes it very difficult to identify the top Co layer which is visible in some areas and absent in others, indicating that the top Co layer might not be continuous. In figure 7(c) we have labeled all the FM layers, where the first $t_1$ layer (layer 1) and all thicker $t_2$ layers are highlighted by white dashed lines. We observe an increasing bending of the layers higher up in the ML. This is typically observed for all grains/columns (see figure 7(a)). This indicates an increasing strain in the layers higher up in the ML.

4. Discussion

Our finding that $H_c$ increases from layer 4 upwards, up to a factor of four for layer 10 (see figure 6), points to stronger domain wall pinning or delayed domain wall nucleation [22] in layers higher in the stack, which indicates a change in microstructure. Interestingly, the increase in $H_c$ correlates with the observed increased curvature of the layers (see figure 7(c)) and the related increasing strain might be at its origin [23]. The reason for the low $H_c$ of the top Co layer (layer 11) and why it is so different from the layer immediately below is not clear. It could be attributed to its non-continuous nature and/or to a relaxed strain at the extremity of the ML [24], perhaps enhanced by an incomplete Pt capping due to the roughness. This is speculative and further quantitative study is needed to unravel the exact mechanisms but a correlation is evident. The interplay between coercivity, surface roughness and film thickness is complex [25–27] and beyond the scope of the present paper. The observed decrease of the interlayer coupling strength up the ML can clearly be related to the overall degradation of the interface quality and an increasing number of pinholes [28]. In ultrathin perpendicular layers the effect of orange peel coupling could also lead to a decreased interlayer coupling [29]. Overall, the STEM data show that further improvements in microstructure could be obtained by, for instance, tuning the growth conditions and/or buffer layers. However, there may be limited scope to improve the microstructure in Co and alternative, for instance amorphous compounds, may provide a better materials set [6].

5. Conclusion

In conclusion, we have shown experimentally that we can use solitons in a ratchet ML to measure the coercivity of each individual magnetic layer and the strength of their exchange coupling to neighboring layers. Our ML was made of 11 perpendicularly magnetized Pt/Co/Pt layers of alternating Co thicknesses, AF coupled via Pt/Ru/Pt with two alternating coupling strengths. The behavior of the soliton-propagating part of the ML was well described by a nIMM, where the switching of each layer only depends on its coercivity, the coupling strengths to neighboring layers and their magnetic configuration. We have demonstrated that we can use solitons to prepare different magnetic configurations and hereby measure the switching of individual layers independently. By preparing enough independent switching configurations for each layer, we could determine the individual coercivity and
interlayer coupling strengths. We found that the coercivity increases steadily up the stack, up to a factor of four for the penultimate layer, before dropping back down at the last layer. The interlayer coupling was seen to decrease in strength up the ML, by about 16%. We presented STEM images of our ML, which showed a columnar growth and a clear degradation of the structural quality of the layers up the ML, which directly correlates with the higher coercivity and lower interlayer coupling strength. This work is fundamental to the optimization of magnetic ML designed for three-dimensional data storage and logic.

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