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# Investigation of magnetic hysteresis in biased Ta/Pt/Co/FeMn/Ta antidots: Influence of structural dimensions

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## **13 ABSTRACT**

Q1

There exists a controversy in the literature concerning the values of coercive and bias fields in antidots magnetic structures formed by a hex-14 agonal network of nanoholes. The coercive fields ( $H_C$ ) and the exchange bias fields ( $|H_{EXC}|$ ) for antidots (deposited on ultrathin anodic alu-15 16 minum oxide, namely, AAO) are either increased or diminished by comparison with the same magnetic nanostructures grown on continuous substrates (namely, CML). We propose to elucidate these debates by showing the importance of the easy axis of the magnetiza-17 tion, the direction of the applied magnetic field, the thicknesses of the layers, and the 3D-topology of nanoholes, as well as the magnetic 18 and thermal history of the magnetic measurements. Here, biased Ta(5 nm)/Pt(5 nm)/Co(0.6 nm)/Fe<sub>50</sub>Mn<sub>50</sub>(X)/Ta(5 nm) antidots are inves-19 tigated by extraordinary Hall effect measurements at 5 K, where X varies in the (0-5.5) nm range. The substrate consists in a hexagonal 20 array of holes, described by the pair of (p,d) values, respectively, the period as the distance from center to center of two consecutive holes 21 and the hole diameter. The dimensions of antidots are ( $p \approx 100$  and  $d \approx 40$  nm) for X = (2-5.5) nm, ( $p \approx 150$  and  $d \approx 60$  nm) for 22 X = 3.5 nm, and ( $p \approx 100$  and  $d \approx 60$  nm) for X = 0. A continuous stack using Si/SiO<sub>2</sub>(100 nm) is used for comparison. H<sub>C</sub> and |H<sub>EXC</sub>| 23 gradually increase when X is enhanced for both substrates, with nevertheless a weak decrease at high X for the continuous system. 24 Perpendicular magnetic anisotropy is only observed for both unbiased samples, the X = 2 nm continuous sample, and both X = 5 nm25 26 samples that have undergone field cooling treatment from 500 to 5 K under -2 T. Usually,  $H_C(AAO) > H_C(CML)$ ,  $|H_{EXC}(AAO)| > |H_{EXC}(AAO)| > |H_{EXC}($ (CML)|, and  $|H_A(AAO)| \leq |H_A(CML)|$  ( $H_A$  designating the anisotropy field). However, for certain conditions, as, for instance, for FC-pro-27 cedures starting from high temperatures and/or strong magnetic field, other situations might be observed. A discussion pertaining to the 28 amplitudes of H<sub>C</sub>, |H<sub>EXC</sub> | and the anisotropy field (|H<sub>A</sub> |) of continuous and discontinuous samples is given for our experimental results as 29 30 well as for published data in the literature, in the light of structural characteristics (wedge-to-wedge distance, porosity, or coverage ratio). Such biased perpendicular antidots might be particularly used in specific nanomaterials devoted to spintronics. 31

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#### 33 I. INTRODUCTION

34 By reducing the size of magnetic materials, interesting physical 35 properties that are potentially useful for applications might be induced.<sup>1</sup> For instance, a marked enhancement of the coercivity  $H_c^2$ 36 and the exchange bias  $H_{EXC}$ ,<sup>3</sup> when ferromagnets and antiferromag-37 nets are combined leading to an antiferromagnetic coupling, is 38 obtained in the magnetic hysteresis. In these two previous references, 39 a lithographic process is used resulting in a discontinuous magnetic 40 layer containing nonmagnetic holes called antidots. The two relevant 41 42 physical parameters are the pore size, namely, d, and the period or the spacing between two consecutive holes from center to center, 43

namely, *p*. For instance, *d* and *p* are sensibly higher than 100 nm in the two previously mentioned studies.<sup>2,3</sup> As new potential interest of antidots, magnetic studies have established the generation of magnetic skyrmions in an antidot multilayered  $[Pt/Co/Ta]_{12}$  with perpendicular magnetic anisotropy and Dzyaloshinskii–Moriya interactions (DMIs),<sup>4</sup> or a continuous soft ferromagnetic film coupled to a hard magnetic antidot matrix with exchange and dipolar interactions, without any DMI, from micromagnetic simulations.<sup>5–7</sup> 51

In the aim to reduce these lengths, Masuda and Fukuda<sup>8</sup> have 52 pioneered the fabrication of d lower than 100 nm by using chemical 53 methods based on a two-step anodization method. For biased 54

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planar antidots<sup>9-14</sup> using such nano-templates, the authors claim 55 that the magnetic properties of antidots elaborated by this chemical 56 57 process might be understood by considering the pinning effects 58 around the nanoholes. As a result, the hysteresis loop is systematically more slanted for an applied magnetic field in the planar easy 59 direction;  $H_C^{9-11,13,14}$  and  $H_A^{10}$  are, respectively, increased and 60 lowered for the antidot, by comparison with the continuous film. 61 Concerning  $H_{EXC}$ , there seems to be contradictory results. The bias field might be weaker,<sup>9,12</sup> or higher,<sup>10,11,13,14</sup> or have a complex dependence on the ferromagnetic layer,<sup>12</sup> for the network structure 62 63 64 comparatively with the unholed sample. 65

In the case of unbiased perpendicular antidots,<sup>15-20</sup> the 66 reduction of the perpendicular magnetic anisotropy is also 67 obtained, when the holed sample is used. As for planar systems, an 68 increase in  $H_C$  is also reported, for  $[Co(0.5 \text{ nm})/Pt(2 \text{ nm})]_5$ ,<sup>12</sup> 69 CoCrPt[(5-20) nm],<sup>16</sup> TbCo(30 nm),<sup>17</sup> DyFe<sup>18,19</sup> ( $\approx 25-30 nm$ ), 70 and  $[Co(0.5 \text{ nm})/Pd(1 \text{ nm})]_5^{20}$  antidots. Finally, perpendicular 71 biased antidots are seldom studied in the literature, as, for instance, 72 in some isolated Refs. 21-24, where Co(0.5 nm)/Pd(0.5 nm) multi-73 layers exchange-coupled with an IrMn layer<sup>21-23</sup> and a Co(0.5 nm)/ 74 Pt(1 nm)] multilayer coupled to IrMn<sup>24</sup> are studied. An enhance-75 ment of the bias field is reported in both references, whereas the 76 77 coercivity is either diminished<sup>24</sup> or enhanced<sup>21-23</sup> by using the 78 antidot structure.

Unfortunately, in these mentioned references, the value of  $H_A$ 79 80 (or  $K_{eff}$  the effective anisotropy constant) is not systematically evaluated based on magnetic measurements (for instance, any values 81 are indicated in Refs. 9, 11, 12, 13, 15, 16, 21, 22, 23, and 24). 82 Moreover, contradictory results appear for  $H_C$  and  $H_{EXC}$  when the 83 patterned and the continuous structures are compared. In the 84 present study, we address these contradictions, thanks to a system-85 atic approach, and highlight the role of the magnetic atomic frac-86 tions in the valley and on the top of the nanopores, on the 87 magnetic characteristics (coercive, exchange fields, effective anisot-88 ropy constant) for a series of exchange-coupled Ta/Pt/Co/FeMn/Ta 89 antidots by intensive extraordinary Hall effect (EHE) measure-90 91 ments at 5 K. And we also give possible explanations to the contra-92 dictory results published in the literature in the light of our own findings by discussing the influence of structural characteristics 93 (such as wedge-to-wedge distance or porosity, for instance). 94

#### 95 II. EXPERIMENTAL DETAILS AND STRUCTURAL 96 CHARACTERIZATIONS OF ANTIDOTS

The fabrication method of antidot arrays, detailed in Ref. 25, 97 uses a particular procedure as largely employed in the literature. In 98 order to prepare antidot arrays, an anodic alumina membrane was 99 100 prepared by the so-called two-step anodization process.<sup>8</sup> After 101 mechanically polishing a mirror-like aspect and electropolishing at 30 V for 2 min in a perchloric acid/methanol mixture (1/4 in 102 volume) at 0 °C, high-purity (99.999%) aluminum foils were anod-103 ized in a 0.5M oxalic acid with a constant potential of 40 V and 104 temperature around 16 °C. The first and second anodization were 105 performed during 17 and 7 h, respectively, which led to templates 106 with a thickness of  $60\,\mu\text{m}$ , and hole diameter and interpore dis-107 tance around 40 and 100 m, respectively. The obtained free-108 standing nanoporous membranes are subject to a pore opening/ 109

widening treatment in a phosphoric acid solution at 35 °C was 110 applied during an etching time (namely, tetching) that was varied 111 between typically 30 and 90 min. This procedure allows the 112 removal of the barrier layer closing the bottom of the pores and 113 permitted us to obtain a pore diameter close to the native one of 114 d = 40 nm for  $t_{etching} = 30 \text{ min}$ . For a longer  $t_{etching}$  of 60 min, a 115 higher d is achieved ( $\approx 60$  nm). As a consequence, two holed sub- 116 strates of the studied series in this paper are chosen ( $d \approx 40$  and 117  $d \approx 60$  nm where  $p \approx 100$  nm). Finally, a third type of holed sub- 118 strate is investigated where the period is higher, typically 119 p = 150 nm and for comparable d. This latter substrate was 120 obtained in the same oxalic acid solution but with a two-step anod- 121 izing process of 2 h, but at a higher voltage of V = 60 V. At this 122 voltage, the alumina growth rate is increased by a factor almost 3 123 compared to what is obtained at 40 V (due to the linearity of p 124 with V as  $p = 2.5 \times V$  as detailed, for example, in Ref. 26). At the 125 same time, the hole diameter is also increased from 40 to 60 nm 126 [the relation between d and V as  $d = 1.3 \times V^{26}$  is also checked]. 127 With these conditions, a less well self-organized array with dis- 128 torted pore openings is obtained. For instance, a poor regularity of 129 a pore arrangement is seen after a second anodization step in the 130 0.3M oxalic acid solution at 2 °C under anodizing voltages of 60 V 131 in Ref. 27. Three kinds of substrates are used in the following of the 132 study:  $(d \approx 40 \text{ nm}, p \approx 100 \text{ nm})$ ,  $(d \approx 60 \text{ nm}, p \approx 100 \text{ nm})$ , and 133  $(d \approx 60 \text{ nm}, p \approx 150 \text{ nm}).$ 134

Then, each holed substrate is placed with a continuous sub- 135 strate Si/SiO<sub>2</sub>(100 nm) on the sample holder during the sputtering 136process, which features a base pressure of about  $6.0 \times 10^{-8}$  mbar, 137 an Ar pressure of  $3.0 \times 10^{-3}$  mbar during deposition, deposition 138 temperature  $\approx 20$  °C, and 0.05, 0.07, 0.09, and 0.1 nm/s as deposi-139 tion rates for Co, FeMn, Pt, and Ta, respectively. As published in 140 Ref. 15, a Co thickness of 0.5 nm induces a perpendicular magnetic 141 anisotropy in  $[Co(0.5)/Pt(2)]_5$  multilayers [the thicknesses in 142] parentheses being expressed in nm]. Moreover, an optimization of 143 the bias field and the coercivity at RT by varying the Co thickness 144 (X) has been observed in Ta(5)/Pt(2)/Co(X)/FeMn(4)/[Ta(3) or Pt 145 (3)] multilayers for  $X \approx 0.4$  nm.<sup>28</sup> With this in mind, we focus on 146 systems with a close enough Co thickness value of 0.6 nm in that 147 present study. We have employed Ta as seed layer due to its (111) 148 texture, inducing a similar texture for the (Pt, Co, FeMn) subse- 149 quent layers, which favors perpendicular exchange bias.<sup>29</sup> Unbiased 150 Ta(5)/Pt(5)/Co(0.6)/Ta(5)and biased Ta(5)/Pt(5)/Co(0.6)/ 151 Fe<sub>50</sub>Mn<sub>50</sub>(X)/Ta(5) were sputtered on continuous and holed sub- 152 strates, where X = (0-2-3.5-5.5) nm. Here, the error in the deter- 153 mination of the thickness is estimated to be  $\pm 10\%$ . The nominal 154 dimensions of antidots are ( $p \approx 100$  and  $d \approx 40$  nm) for X = (2.0-155)5.5) nm, ( $p \approx 150$  and  $d \approx 60$  nm) for X = 3.5 nm, and ( $p \approx 100$  156 and  $d \approx 60$  nm) for X = 0 nm. In order to ensure better visibility 157 of results, the magnetic data for antidots and continuous multi- 158 layers are plotted in black lines and red lines, respectively. 159

Typical SEM view of samples are given in Figs. 1(a)-1(d). The 160 presence of nanopore arrays arranged in a hexagonal way is con-161 firmed, and no significant reduction in the pore diameter is 162 observed after completion of the film. Some dislocations are visible 163 in the network as appeared in these SEM figures, as expected by 164 using such nanosubstrates. In addition, circular shapes and ellipti-165 cal ones are detected in Figs. 1(a), (b), and (d) and 1(c), 166

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**FIG. 1.** (a) SEM views of Ta(5 nm)/Pt(5 nm)/Co(0.6 nm)/Fe<sub>50</sub>Mn<sub>50</sub>(X)/Ta(5 nm) antidots (thicknesss in nm) for different FeMn thicknesses X ranging from 0 to 5.5 nm. The mean period p as the distance from center to center of two consecutive holes and the mean hole diameter d are the dimensions of antidots are ( $p \approx 100$  and  $d \approx 40$  nm) for X = (2-5.5) nm, ( $p \approx 150$  and  $d \approx 60$  nm) for X = 3.5 nm, and ( $p \approx 100$  and  $d \approx 60$  nm) for X = 0 nm. Note the elliptical shape of holes in (c) and circular ones in (a, b, and d).

167 respectively. This particular shape of ellipses has been reported in 168 Ref. 30 where authors have used the porous alumina as masks for 169 the deposition of ordered ferromagnetic nanodot arrays. For our 170 sample where  $p \approx 150$  and  $d \approx 60$  nm, the distortion of the hexag-171 onal arrangement of spherical pores is explained by the use of high 172 anodizing voltage (60 V), as in the published studies.<sup>27,31</sup>

Coming back to the pore networks, the size distribution of 173 both p and d is given in Fig. 2 for these antidots by taking into 174 account the holes appearing in insets of Fig. 1. To do this, a con-175 version of grayscale SEM images to binary black-and-white images 176 is performed. The pixels with grayscale values exceeding a thresh-177 old are set to black, all others are set to white. The threshold values 178 are manually chosen so that the black areas in the black-and-white 179 images reproduce the pores in the original grayscale images. NIH 180 IMAGEJ software package is used to measure p and d, as well as 181 their standard deviations. The values of mean diameter  $d_{mean}$ , 182 mean period  $p_{mean}$ , mean spacer  $s_{mean}$  between two consecutive 183 184 holes, and density of holes  $\delta$  are gathered in Table I. Periodicity of  $p\approx 100$  and  $p\approx 150\,\rm nm$  is calculated starting from the distributions, as well as holes density reaching  $1.1\,10^{10}$  and  $0.5\,10^{10}\,\rm holes/$ 185 186 cm<sup>2</sup>, in Figs. 1(a), (b), and (d) and 1(c), respectively. The size dis-187 188 tribution of pores is peaked around  $d \approx 40$  and  $d \approx 55$  nm, in 189 Figs. 1(b)-1(d) and Figs. 1(a)-1(c), respectively. Since the shape of hole is elliptical in Fig. 1(c), a size distribution of ellipses is 190 assumed and the average length ratio (defined as minor axis/major 191 axis =  $b_{mean}/a_{mean}$ ) reaches 0.67. As expected, we retrieve that the 192 area of the mean circle { $(\pi/4) \times d_{mean}^2 = 2.445 \, 10^3 \, \text{nm}^2$ } is close to 193 the one of a mean ellipse { $\pi \times (a_{mean} \times b_{mean})/4 = 2.435 \, 10^3 \, \text{nm}^2$ }. 194 The size distribution is broader for the 3.5 nm multilayer. 195 Moreover, this 3.5 nm multilayer contains spacing between two 196 consecutive nanoholes more important than ones of the other 197

multilayers, indicating that more ferromagnetic material is depos-198 ited on the top of multilayers compared to the ones elaborated on 199 the vicinity of nanoholes. This finding is important for magnetic 200 results, as developed in the following of the paper. 201

The extraordinary (also called anomalous) Hall effects (EHEs) 202 are currently described by a phenomenological equation expressing a 203 resistance as a function of different terms as  $EHE = R_o \times H$  204  $+R_e \times M_Z$ , where H designates the external magnetic field,  $M_Z$  the 205 perpendicular magnetization, Ro the ordinary or normal coefficient, 206 and Re the extraordinary Hall one. In case of materials with marked 207 spin-orbit scattering such as the case of our materials (Pt/Co), the 208 second term ( $R_e \times M_Z$ ) dominates the first one ( $R_o \times H$ ). As a con- 209 sequence, this kind of measurement is a direct signature of the 210 perpendicular magnetization as a function of applied magnetic 211 field, a magnetic loop being obtained. Experimental and 212 theoretical approaches are found in a review paper devoted to 213 EHE.<sup>32</sup> For the practical measurements, a cross Hall is used with a 214 line of current (typically a current of I =  $10 \mu A$ ) perpendicular to a 215 line of voltage (leading to a measurement of variation of voltage 216  $\Delta V$ ). EHE is simply defined by EHE =  $\Delta V/I$  (typically around 0.1  $\Omega$ ). 217 In this study, we have normalized the EHE response as 218  $M_Z^{norm}(H_Z) = [2EHE(H_Z) - (EHE_{max} + EHE_{min})]/\Delta R_{EHE}$ , where  $H_Z$  219 is the perpendicular magnetic field,  $\Delta R_{EHE} = EHE_{max} - EHE_{min}$ , 220  $\text{EHE}_{\text{max}}$  and  $\text{EHE}_{\text{min}}$  being the maximum and minimum of resis- 221 tance, respectively.  $M^{norm}$  varies between -1 and +1 as for the case 222 of a classical normalized hysteresis loop.  $\Delta R_{EHE}$  designates the total 223 amplitude of the Hall signal. In the following,  $M_Z^{norm}(H_Z)$  is recorded 224 at 5 K and for H<sub>Z</sub> up to 3 T after zero field cooling (ZFC), or after 225 Field Cooling (FC) H<sub>FC</sub>-FC(T<sub>max</sub>). Here, H<sub>FC</sub> and T<sub>max</sub> designate the 226 magnetic field applied during the field cooling procedure and the 227 maximum of temperature before cooling. The blocking temperature 228  $T_b$  for Fe<sub>50</sub>Mn<sub>50</sub>, temperature at which  $H_{EXC} = 0$ , was about 229 (390-480) K for relatively high or moderate FeMn thicknesses (>a 230 few nm),<sup>33</sup> or lower for thinner FeMn ones (< 1 nm).<sup>34,35</sup> Thus, we 231 have fixed T<sub>max</sub> at 500 K, and 300 K is also employed for compari- 232 son. These measurements allow us to derive  $H_C$  and  $H_{EXC}$ . Then, 233 after the out-of-plane measurement, without extracting the sample 234 from the EHE setup, a second measurement is immediately per- 235 formed with field  $H_X$  up to 3 T, and applied parallel to the sample 236 plane with a very small misorientation ( $\approx 2^{\circ}$ ) in order to ensure a 237 coherent rotation of the Co moments. The normalized in-plane com- 238 ponent of the magnetization  $M_X^{norm}$  is  $(1-[M_Z^{norm}]^2)^{0.5}$ . The anisotropy 239 field  $H_A$  is determined from the area between perpendicular and 240 planar EHE measurements, and the latter is defined as  $2 \int_{0}^{31} dH \cdot 241$  $[M_Z^{norm}(H_Z^{mean})-M_X^{norm}(H_X)]$ , where  $M_Z^{norm}$  and  $M_X^{norm}$  are previously 242 recorded,  $H_Z^{mean}$  being the mean magnetic field defined by taking 243 into account the descending and ascending branches of the hysteresis 244 loop. Here, the maximum magnetic field 3 T is efficient for saturat- 245 ing magnetic hysteresis. Concerning the effective anisotropy constant 246  $K_{eff}$ , its value is calculated as  $H_A \times (M_S/2)$  where  $M_S$  designates the 247 saturation magnetization of the sample measured at 5 K by SQUID. 248

## III. INVESTIGATIONS OF MAGNETIC LOOPS AT 5 K

## A. Unbiased stacks

After a ZFC procedure from RT to 5 K,  $M_Z$  vs  $H_Z$  (left part) 251 and  $M_X$  vs  $H_X$  (right part) at T = 5 K of 2 Ta/Pt/Co/Ta unbiased 252

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FIG. 2. (a) Distributions of periods p (distance from center to center of two consecutive holes), and hole diameter d deduced from Fig. 1 for Ta(5)/Pt(5)/Co(0.6)/Fe<sub>50</sub>Mn<sub>50</sub> (X)/Ta(5) antidots (thickness in nm) with FeMn thicknesses X = (0-2-3.5-5.5) nm. In inset of (c) is plotted the distribution size of the minor and major axes of elliptical holes.

samples (antidot in black lines and continuous multilayer in red 253 lines) are displayed in Figs. 3(a) and 3(b), respectively. Here, the 254 dimension of the antidot is ( $p \approx 100$  and  $d \approx 60$  nm). A 3 kOe is 255 needed for saturating the magnetization in bots directions of mag-256 netic field. A clear square hysteresis for perpendicular magnetic 257 fields, as well as a hard axis for the planar H, are obtained for both 258 samples. In Fig. 3(a), the remanence  $M_R^{\perp}$  reaches a high value 259  $(\approx 97\%)$  for both data, and the saturation field (namely,  $H_{sat}^{\perp}$ ), 260 defined as the field necessary for reaching 98.5% of the saturation 261 magnetization, is  $H_{sat}^{\perp} = (2.346 - 2.780)$  kOe for the continuous 262

**TABLE I.** Structural characteristics of Ta(5)/Pt(5)/Co(0.6)/FeMn(X)/Ta(5) (thickness in nm) antidots where X = (0-2-3.5-5.5) nm: nominal FeMn thickness X, mean diameter  $d_{mean}$ , mean period  $p_{mean}$ , mean distance  $s_{mean}$  between two consecutive holes, and density of holes  $\delta$ . The standard deviations are indicated in parentheses.

X(nm)	d <sub>mean</sub> (nm)	$p_{mean}$ (nm)	s <sub>mean</sub> (nm)	$\delta$ (10 <sup>10</sup> holes/cm <sup>2</sup> )
0	56.4(1.1)	101.7(1.3)	45.3(1.5)	1.044(0.027)
2	40.9(1.3)	98.4(3.1)	57.5(3.6)	1.086(0.135)
3.5	55.8(3.3)	148.3(10.9)	92.5(12.3)	0.490(0.065)
5.5	42.7(0.8)	99.9(2.5)	57.2(2.5)	1.097(0.060)

multilayer and antidot, respectively. The different characteristics of 263 magnetic hysteresis (remanence, coercivity, bias field, maximum of 264 susceptibility, saturation field, anisotropy field, effective anisotropy 265 constant) are gathered in Table II. From the planar measurement 266 in Fig. 3(b), it appears that the saturation field ( $H_{sat}^{//}$ , see Table II) is 267 reached for lower magnetic field compared to the discontinuous 268 multilayer. In addition, by defining the susceptibility  $\chi$  as  $\frac{dM}{dH}$  (the 269 derivative of M following H), the maximum susceptibility for 270  $H \approx H_C$ , namely,  $\chi_{max}$ , is calculated for the stacks. We find 271  $\chi_{max}^{//} \approx 1.2 \, 10^{-4} \, \text{Oe}^{-1}$  and  $\chi_{max}^{\perp} \approx 3 \, 10^{-2} \, \text{Oe}^{-1}$  for these samples. 272 These features indicate that  $H_A(AAO) < H_A(CML)$ . Concerning the 273 anisotropy field, we first apply the approximate method of linear 274 extrapolation of the EHE signal (in the high field regime) as was 275 done, e.g., in Ref. 36, and we find  $H_A(AAO) \approx 1.714$  kOe and  $H_A$  276  $(CML) \approx 2.005 \text{ kOe}$  [see Fig. 3(a)]. More precisely, from the 277 formula using the integration of EHE data [and by using 278 the averaged signal as plotted in dotted lines in Fig. 3(a)], H<sub>A</sub> 279 (AAO) = 1.485 Oe and  $H_A(C) = 2.165$  kOe are derived as indicated 280 in Table I. The effective anisotropy constants  $(K_{eff})$  are calculated 281 from  $H_A$  and the saturation magnetization measured at 5 K by 282 SQUID (1250 emu/cm<sup>3</sup>) and are listed in Table I. This method of 283 integration is more accurate than the one using the slope of 284 the magnetization M(H) for a hard magnetic field as shown in 285



FIG. 3. Normalized extraordinary Hall effects  $M_Z(H_Z)$  (a) and  $M_X(H_X)$  (b) at 5 K after ZFC from RT to 5 K for the unbiased continuous multilayer (red lines) and the unbiased antidot (black lines). The stacking is Ta(5)/Pt(5)/Co(0.6)/Ta(5) (thickness in nm), and the dimension of the antidot is ( $p \approx 100$  and  $d \approx 55$  nm). The maximum magnetic field is 3 T and only 0.3 T is the maximum field of the graph for clarity of presentation. The dotted lines in (a) are the mean responses of data (decreasing and increasing brunches), and the dashed linear extrapolation in (b) leads to a rough estimation of the anisotropy field (lower for the antidot structure).

Fig. 3(b). The reasons are that M is rarely linear in the complete 286 range of hard before saturation, and M(easy H) is not systemati-287 288 cally a perfect square loop, as the case for our samples. Other methods exist, such as the generalized Sucksmith-Thompson 289 method,<sup>37</sup> which is less approximate than the method of the linear-290 291 ity of M(hard H), but less accurate than the method of integration, 292 could be employed. Concerning the coercive field  $H_C^{\perp}$ , its value is enhanced by using the antidot (1.418 kOe) compared to the contin-293 uous multilayer (1.194 kOe) as seen in Table II. As a result, the 294 coercivity for Heasy (H aligned with the easy axis, namely, perpen-295 dicular to the layers) and the anisotropy field are, respectively, 296 enhanced and reduced at 5 K when the antidot is used, as found in 297 the literature<sup>15-17</sup> for perpendicular systems. The probable reasons 298 of the reduction of  $H_A$  (or the effective constant) and the enhance-299 ment of  $H_{L}^{\perp}$  are (i) the in-curved magnetic moments around nanopores.<sup>12,13,15,16,21-24</sup> orientated almost in the plane of layers 300 orientated almost in the plane of layers 301 nanopores,<sup>1</sup> and perpendicularly to the out-of-plane applied magnetic field, 302 provoking a reduction of the magnetic anisotropy; (ii) the pinning 303 effects<sup>11-</sup> <sup>13,15,17</sup> induced by the nanoholes leading to an increase in 304 the coercive field. As expected, the magnetic data do not change 305 when a FC procedure is applied for unbiased samples, and no bias 306 fields  $H_{\text{EXC}}^{\perp}$  are recorded since no antiferromagnetic layer is grown 307 on the sample (curves not shown). 308

#### B. Biased stacks with $T_{\text{max}}=300\,\text{K}$ 309

After a ZFC procedure from RT to 5 K,  $M_Z$  vs  $H_Z$  (top part) 310 and EHE<sub>X</sub> vs H<sub>X</sub> (bottom part) at T = 5 K for Ta/Pt/Co/FeMn(X)/ 311 Ta biased samples are displayed in Fig. 4, where X = 2.0 nm for 312 [(a)-(d)], X = 3.5 nm for [(b)-(e)], and X = 5.5 nm for [(c)-(f)].313

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$K_{eff}$ (10 <sup>6</sup> )	1.353	0.928	0.256	-1.337	-3.230	-1.329	No	No	-3.210	-3.019	No	No	2.61	1.206
$H_A (10^3)$	2.165	1.485	0.410	-2.139	-5.167	-2.127	No	No	-5.136	-4.830	No	No	4.179	1.930
${ m H}^{//}_{sat}$ (10 <sup>3</sup> )	2.300	1.993	7.516	3.352	12.010	5.520	No	No	9.894	10.941	No	No	15.008	18.871
$(10^3)$	346	780	532	130	89	619	324	184	126	181	540	96	80	801

stacking is Ta(5)/Pt(5)/Co(0.6)/FeMn(X)/Ta(5) where > and ( $p = 150$ and $d = 55$ nm) for X = 3.5 nm. The m: (+1, + 2, - 2) TJ. M <sub>R</sub> ( $\pm 3\%$ ), H <sub>C</sub> (Oe, $\pm 7\%$ ), H <sub>EXC</sub> maximum of susceptibility, saturation field, anisotropy = an samples	$\langle \epsilon = (0-2-3.5-5.5]$ agnetic and thei $\epsilon^{\circ}$ (Oe, $\pm 7\%$ ), $\chi_{\rm m}$ field, and effectiv $M_R^{\perp}$ (%)	) nm, the antidots mal procedure c ax ( $Oe^{-1}$ , $\pm 10\%$ ax ( $e^{-1}$ , $\pm 10\%$ H $^{\rm ax}_{\rm C}$ ( $10^3$ )	the period of the period the period of the measurement of the period $(0, H_{sat} (0e, \pm h_{start}, the period the perio$	ed with dimensions ts is either ZFC ( $5\%$ ), H <sub>A</sub> (Oe, $\pm^{4}$ ely. Here, "No" me $\mathcal{X}_{max}^{\perp}$	s of ( $p = 100$ and d (zero field cooling 4%), and K <sub>eff</sub> (erg/ tans the absence o $\mathcal{X}_{max}^{//}$	= 55 nm) for $X = ($ from RT to 5 K) cm <sup>3</sup> , $\pm 10\%$ des f measurements. H <sup>LL</sup> (10 <sup>3</sup> )	0 nm, ( $p = 100$ an or FC [field cool ignate the remant $H_{sat}^{//}$ (10 <sup>3</sup> )	I $d = 40$ nm) for X ing from RT or 5 ince, coercivity, e ince, coercivity, e H <sub>A</sub> $(10^3)$	$(= (2-5.5) \text{ nm}, 00-5 \text{ K under xchange bias}, \text{Keff} (10^6)$
X = 0 nm, continuous, ZFC	97.4	1.194	1.7	$3.6 \times 10^{-2}$	$1.2 imes 10^{-4}$	2.346	2.300	2.165	1.353
X = 0 nm, antidot, ZFC	96.5	1.418	2.5	$2.2  imes 10^{-2}$	$1.3  imes 10^{-4}$	2.780	1.993	1.485	0.928
X = 2 nm, continuous , ZFC	75.4	2.820	-24.2	$3.4 \times 10^{-4}$	$4.0 imes 10^{-4}$	10.532	7.516	0.410	0.256
X = 2 nm, antidot, ZFC	63.6	2.517	-127.0	$3.5  imes 10^{-4}$	$8.1 imes 10^{-4}$	12.030	3.352	-2.139	-1.337
X = 3.5 nm, continuous, ZFC	41.7	3.226	-57.6	$1.4  imes 10^{-4}$	$4.4 \times 10^{-4}$	17.889	12.010	-5.167	-3.230
X = 3.5 nm, antidot, ZFC	61.6	2.625	-196.7	$3.4 \times 10^{-4}$	$3.1  imes 10^{-4}$	13.519	5.520	-2.127	-1.329
X = 3.5 nm, continuous, 1T-FC(RT)	59.3	5.034	-3239.3	$1.9  imes 10^{-4}$	No	18.324	No	No	No
X = 3.5 nm, antidot, 1T-FC(RT)	61.5	2.614	-299.3	$3.4 \times 10^{-4}$	No	14.084	No	No	No
X = 5.5 nm, continuous, ZFC	39.2	2.501	-38.7	$1.9 imes10^{-4}$	$2.8  imes 10^{-4}$	19.426	9.894	-5.136	-3.210
X = 5.5 nm, antidot, ZFC	48.6	3.338	-495.7	$2.0  imes 10^{-4}$	$1.4  imes 10^{-4}$	24.381	10.941	-4.830	-3.019
X = 5.5 nm, continuous, 1T-FC(RT)	55.4	3.967	-2879.3	$2.4 \times 10^{-4}$	No	16.540	No	No	No
X = 5.5 nm, antidot, 2T-FC(500 K)	50.8	2.966	-1165.6	$2.3 \times 10^{-4}$	No	21.196	No	No	No
X = 5.5 nm, continuous, $-2$ T-FC(500 K)	62.0	4.400	+2156.9	$2.4 \times 10^{-4}$	$2.4 \times 10^{-4}$	23.180	15.008	4.179	2.61
X = 5.5  nm,  antidot, -2T-FC(500  K)	52.3	3.335	+778.6	$1.9 \times 10^{-4}$	$0.4  imes 10^{-4}$	20.801	18.871	1.930	1.206





**FIG. 4.** Normalized extraordinary Hall effects  $M_Z(H_Z)$  [(a)–(c), top panel] and  $M_X(H_X)$  [(d)–(f), bottom panel] at 5 K for biased continuous multilayers (red lines) and biased antidots (black lines), after ZFC procedures. The stacking is Ta(5)/Pt(5)/Co(0.6)/FeMn(X)/Ta(5) (thickness in nm) where X = 2 nm for [(a)–(d)], X = 3.5 nm for [(b)–(e)], and X = 5.5 nm for [(c)–(f)]. The maximum magnetic field is 3 T and the dimensions of antidots are (p = 100 and d = 40 nm) for X = (2-5.5) nm and (p = 150 and d = 55 nm) for X = 3.5 nm.  $H_{FC}$ -FC(T<sub>max</sub>) data are added in (b) and (c) for comparison, where  $H_{FC} = 1$  T and  $T_{max} = 300$  K designate the magnetic field applied during the Field Cooling procedure and the maximum of temperature before cooling.

314 The data for antidots and continuous multilayers are plotted in 315 black lines and red lines, respectively.

We first concentrate on the lowest FeMn thickness 316 [X = 2 nm, Figs. 4(a)-4(d)]. The magnetic field necessary for satu-317 rating the magnetic hysteresis is 1 T, meaning that a higher H is 318 necessary when a biased sample is measured [0.3 T was necessary 319 for the unbiased samples as plotted in Fig. 4(a)]. For this lowest 320 FeMn thickness, slanted M(H) magnetic loops are recorded, the 321 susceptibility and the remanence reach  $\chi^{\perp}_{max}\approx 3.4\,10^{-4} \mathrm{Oe}^{-1}$  and 322  $M_R^{\perp} \approx 70\%$ , respectively, for both samples (as merged in Table II). 323 As a consequence, the remanence and the maximum susceptibility 324 are reduced when a FeMn layer is added to the sample, and weak 325 differences seem to appear for the two biased samples. Concerning 326 the saturation field for perpendicular magnetic data,  $H_{sat}^{\perp}$  is 327 increased for the antidot (12.030 kOe) compared to the continuous 328 329 structure (10.532 kOe). In the same way, for planar magnetic H [see Fig. 4(d)], M(H) saturates at larger applied fields for the 330 X = 2 nm continuous sample ( $H_{sat}^{//} = 7.516$  kOe), accompanied by a weak slope of the hysteresis ( $\chi_{max}^{//} = 4.0 \ 10^{-4} \text{Oe}^{-1}$ ), indicating that, by combining the results from configurations of magnetic 331 332 333 fields,  $H_A$  seems to be higher for the continuous multilayer than 334 the antidot as expected. Surprisingly, from the integration method, 335  $H_A = -2.139$  kOe and  $H_A = +0.410$  kOe for the antidot and the 336 continuous multilayer, respectively. This result means that a spin 337 reorientation transition driven by the substrate geometry takes 338

place for the X = 2 nm biased sample, from a planar magnetic 339 anisotropy for the antidot to a perpendicular one for the continu- 340 ous multilayer. Here, there are probably different contributions to 341 the magnetic switching mechanism for this sample. By comparing 342 magnetization curves calculated using an appropriate switching 343 model with measured ones, it will be possible to determine these 344 contributions (another study). 345

The reverse situation has been reported in the literature for 346 films deposited on nanoporous alumina membranes, as the form 347  $Dy_{13}Fe_{87}(30 \text{ nm})$  with d = 45 nm and p = 105 nm,<sup>18,19</sup> where the 348 easy axis of the magnetization is laying along the planar direction for 349 the continuous stack whereas it is out-of-plane for the antidot array. 350 A similar result was also observed for samples by Gräfe et al. in Fe 351 (200 nm) antidot arrays (d = 100 and p = 200 nm) fabricated by 352 nanosphere lithography.<sup>38</sup> At our knowledge, it is the first time that 353PMA is suppressed for an exchange-coupled continuous system 354 (with, in particular, a weak anisotropy field) by using an antidot as 355 substrate. The last striking characteristic of Fig. 4(a) is that 356  $H_C^{\perp} = (2.820/2.517)$  kOe and  $H_{EXC}^{\perp} = (-24.2/-127.0)$  Oe for the 357 (CML/AAO) samples pair. Thus, the coercive field is lowered when 358 the antidot is used, similarly to Ref. 24 but in contrast to Refs. 21-23, 359 an increase in the bias field with antidots is confirmed.<sup>2</sup> 360

Now, we come to discuss the EHE results for the higher FeMn 361 thicknesses. For X = 3.5 nm, the magnetic loop for the ZFC data 362 [see Fig. 4(b)] is more slanted for the continuous structure with a 363

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reduction of the remanence ( $M_R^{\perp}$ , 61.6– 41.7%) and the susceptibility ( $\chi_{max}^{\perp}$ , 3.4 × 10<sup>-4</sup> to 1.4 × 10<sup>-4</sup>Oe<sup>-1</sup>) accompanied by an enhance-365 ment of the saturation field ( $H_{sat}^{\perp}$ , 13.519–17.889 kOe), from the 366 367 discontinuous to the continuous system. Similarly, the planar susceptibility and the planar saturation field increase from the antidot to 368 the unholed substrate, respectively,  $\chi_{\text{max}}^{//} = (3.1 \, 10^{-4} / 4.4 \, 10^{-4}) \text{Oe}^{-1}$ 369 and  $H_{sat}^{//} = (5.520/12.010)$  kOe. At a first sight, this is a an intrigu-370 ing discrepancy between our results and some published in the litera-371 ture where M(H) are more slanted for antidots rather than 372 373 continuous substrates. In fact, as indicated by the values of remanence and saturation field for both configuration of magnetic fields, 374 the easy axis of the magnetization seems to be in plane. Indeed, the 375 anisotropy fields are evaluated from the method of integration: H<sub>A</sub> 376 377 (AAO) = -2.127 kOe and  $H_A(CML) = -5.167 \text{ kOe}$ . As a conse-378 quence, for both X = 3.5 nm samples, the magnetic anisotropy is planar due to the FeMn layer which forces the Co pinned spins to be 379 in the plane of layers. Nevertheless, for perpendicular magnetic fields 380 (H), the Co spins around holes are parallel to H and force in part 381 the magnetization to be out-of-plane. For the continuous sample, 382 majority planar Co spins exchange-coupled with antiferromagnetic 383 FeMn spins induce the easy axis of the magnetization to be more 384 planar. This is the reason why planar magnetic anisotropy is 385 observed for both samples and  $|H_A(AAO)| < |H_A(CML)|$ . Then, for 386 387 a hard axis of the magnetic field, the M(H) magnetic loops are more in-curved for the continuous structure. This feature has been 388 observed in the literature for perpendicular antidot arrays, as 389  $[Co(0.5 \text{ nm})/Pt(20 \text{ nm})]_5^{15}$  over a wide range of diameters from 7 to 46 nm, CoCrPt(10 nm),<sup>16</sup> and DyFe[(30 nm)],<sup>18,19</sup> where the M(H) 390 391 392 magnetic loop is more slanted for the continuous film when the magnetic field is applied in the plane of layers (hard axis of the mag-393 netization). Concerning the coercivity and exchange bias, its values 394 are, respectively, lowered and enhanced (in accordance with 395 396 Refs. 21–24) when the antidot is used as indicated in Table II, simi-397 larly to the X = 20 nm sample. This result indicates that both X = 2 nm and X = 3.5 nm stacks have similar behaviors for coerciv-398 ity and bias field due to the fact that probably the antiferromagnetic 399 thickness is too weak to efficiently induce exchange coupling, at least 400 for ZFC measurements. At first glance, the behaviors of coercivity 401 and exchange fields with the antiferromagnetic thickness for our 402 samples are consistent with the results published in the literature.<sup>12</sup> 403 Nevertheless, by comparing both coercivity and bias fields for the 404 405 two types of substrates, the results are contrast to same Refs. 21–23. We will come back to the possible reasons for explaining these pecu-406 407 liar results about  $H_C^{\perp}$ ,  $H_{EXC}^{\perp}$ , as well as  $H_A$  in the discussion part (Sec. IV). 408

The effect of a FC procedure on the M(H) cycle for both 409 biased antidots and continuous multilayer is scarce in the literature, 410 411 such as in Refs. 22 and 23 for [Co(0.5 nm)/Pd(1 nm)]<sub>5</sub>/IrMn(6 nm) 412 (with the appearance of a weak bias value, 270 Oe). For the X = 3.5 nm samples (antidot and continuous), a +1T-FC proce-413 414 dure has been performed from RT to 5 K as shown in dashed lines Fig. 4(b), since noticeable exchange bias effects have been mea-415 in sured for this multilayer. As the blocking temperatures for these 416 samples might be comparable with the room temperature<sup>34,35</sup> 417 due to the weak thickness of FeMn, we can suspect eventual modifica-418 tions of M(H) loops between ZFC and FC data. Indeed, for the 419 continuous structure, the magnetic hysteresis is more squared with 420

an increase of  $M_R^{\perp}$  and  $\chi_{max}^{\perp}$ . The bias  $|H_{EXC}^{\perp}|$  and coercive  $H_C^{\perp}$  421 fields have clearly increased from 57.6 to 3239.3 Oe and from 3.226 422 to 5.034 kOe, respectively (see Table II), a saturation field seems to 423 be relatively constant ( $H_{sat}^{\perp} \approx 18$  kOe). By contrast, very weak 424 changes between ZFC and FC data are observed for the antidot 425 sample with close values of coercivity, bias field, remanence, sus- 426 ceptibility, and saturation fields. As a result, during the FC proce- 427 dure from RT to 5 K under +1 T, magnetic spins of Co of the 428 unholed sample are partially tilted in the perpendicular axis along 429 the applied magnetic field. At the opposite, for the holed sample, 430 the spins are relatively pinned and/or the blocking temperature 431 could be higher than RT, and the applied magnetic has a weak 432 influence on the reorientation of the magnetization. As a conse- 433 quence, the bias is enhanced for the continuous structure compared 434 to the holed one, the pair of ratios  $[H_C^{\perp}(CML)/H_C^{\perp}(AAO), H_{EXC}^{\perp}]$  435 (CML)/  $H_{EXC}^{\perp}$ (AAO)] enhancing from [1.2,0.3] for ZFC measure- 436 ments to [1.9,10.8] for FC ones. 437

Now, to reach saturation of the magnetic hysteresis of the 438 5.5 nm samples, it is necessary to apply 3 T as maximum fields. 439 Across the series of samples and focusing on the ZFC hysteresis 440 loops, we notice (i) a reduction of  $M_R^{\perp}$  and  $\chi_{max}^{\perp}$  and (ii) a rounding 441 of the M(H) curves as X increases. The largest saturation fields 442  $\mathrm{H}_{\mathrm{SAT}}^{\perp}$  [ $\approx$ (19.4 – 24.4) kOe] are obtained for the X = 5.5 nm 443 samples. In the same way, the bias field  $-H_{EXC}^{\perp}$  and the coercive field 444  $H_C^{\perp}$  increase with X for the antidots. For the continuous structures, 445  $(-H_{EXC}^{\perp}/H_{C}^{\perp})$  seems to decrease between X = 3.5 and X = 5.5 nm: 446 (57.6/3226) and (38.7/2501) Oe, respectively. These behaviors of 447 coercive and bias fields are coherent with published results in the lit- 448 erature at least for continuous structures,<sup>28,29,34</sup> where FeMn is used 449 as a antiferromagnetic layer, and for holed systems<sup>13</sup> (NiO as anti- 450 ferromagnetic layer). To our knowledge, no comparison between 451 both kind of substrates has been reported in the literature for biased 452 FM/FeMn systems with FM as a ferromagnetic layer and as a func- 453 tion of antiferromagnetic thicknesses. Moreover, by gathering per- 454 pendicular [Fig. 4(c)] and planar [Fig. 4(f)] ZFC EHE data, the 455 anisotropy field  $-H_A$  is maximized, reaching 4.830 and 5.136 kOe 456 for the antidot and continuous 5.5 nm samples, respectively. This 457 result indicates planar magnetic anisotropy for the X = 5.5 nm 458samples. As for the X = 3.5 nm sample, by applying a +1 T-FC 459 procedure for the X = 5.5 nm continuous sample, the M(H) mag- 460 netic loop [red dashed line in Fig. 4(c)] is more squared, with 461 higher  $\chi^{\perp}_{max}$  and  $M^{\perp}_R$ , coupled to lower  $H^{\perp}_{SAT}$  (see Table II). The 462 +1 T-FC procedure partially reinforces the out-of-plane magnetic 463 anisotropy for the continuous sample, accompanied by an enhance- 464 ment of couples of ratio [H<sup> $\perp$ </sup><sub>C</sub>(CML)/ H<sup> $\perp$ </sup><sub>C</sub>(AAO),H<sup> $\perp$ </sup><sub>EXC</sub>(CML)/ H<sup> $\perp$ </sup><sub>EXC</sub> 465 (AAO)] from [0.7,0.08] for ZFC measurements to [1.3,2.5] for FC 466 ones. Since the FC procedure from 300 to 5 K has a strong influence 467 on only continuous structures with persisting in-plane magnetic 468 anisotropy for both substrates, the effect of a higher  $T_{\text{max}}$  (500 K) is 469 studied in the next part of the paper. 470

## C. Biased stacks with $T_{max} = 500 \text{ K}$

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Finally, a  $-2\,\text{T-FC}$  procedure with  $T_{max}=500\,\text{K}$  has been 472 performed on both samples (see Fig. 5 in dotted lines). Once again, 473 some weak changes appear for the antidot by comparing with 474 -2 T-FC(500K) and ZFC data [see Fig. 4(c)], with, in particular, a 475

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marked positive exchange bias (778.6 Oe) due to the negative value 476 of the applied magnetic field during cooling. For the 5.5 nm contin-477 478 uous sample, an opening of the M(H) loop appears with high rem-479 anence (62.0%), strong coercivity (4.4 kOe), and bias field (2.157 kOe). The remarkable result is the appearance of perpendic-480 ular magnetic anisotropy for both samples, meaning that a spin 481 reorientation from planar to perpendicular takes place by choosing 482 the two types of substrate. For the 5.5 nm continuous sample, the 483 highest anisotropy field is extracted from magnetic data and 484 reaches 4.179 kOe. For the X = 5.5 nm holed structure, a +2 T-FC 485 procedure has been also tested with  $T_{max} = 500 \text{ K}$  above  $T_b$  [black 486 dashed line in Fig. 5(a)]. Weak changes are detected for this sample 487 between ZFC and FC data, except a marked enhancement of  $-H_{FVC}^{\perp}$ 488 from 495.7 to 1165.6 Oe, respectively. As a consequence, for a 489 maximum temperature higher than  $T_b$ , the field cooling from 500 490 to 5 K re-orients the magnetization in a perpendicular configura-491 tion due to the blocking temperature of the antidot lower than 492 500 K, with pronounced modifications of magnetic hysteresis (for 493 perpendicular magnetic H) on the continuous sample rather than 494 the antidot. The positive magnetic anisotropy is higher for the con-495 tinuous stack by a factor of 2. 496

In addition, we believe that uncompensated spins at interfaces between Co and FeMn are key parameters for the understanding of underlying mechanism of antiferromagnetic domain formation. Then, a x-ray magnetic circular dichroism study, as in Ref. 39 for Ni/FeF<sub>2</sub>, on the role of uncompensated spins to exchange bias for a series of antidots when a unique structural parameter is varied (as, for instance, *d*) could be useful in that sense.

#### 504 D. Amplitude of the Hall resistance

The amplitudes of  $\Delta R_{EHE}$  of antidots and continuous Ta/Pt/ 506 Co/FeMn(X)/Ta as a function of X for ZFC data are displayed in



**FIG. 5.** Normalized extraordinary Hall effects  $M_Z(H_Z)$  (a) and  $M_X(H_X)$  (b) at 5 K after -2 T/FC from 500 to 5 K for the biased continuous multilayer (red lines) and the biased antidot (black lines). The stacking is Ta(5)/Pt(5)/Co(0.6)/FeMn (5.5)/Ta(5) (thickness in nm), and the dimension of the antidot is (p = 100 and d = 40 nm). The maximum magnetic field is 3 T. The dotted lines in (a) are additional perpendicular data for +2 T/FC(500 K for the antidot.

Fig. 6. The strongest value is recorded for the membrane, more  $X_{507}$ and more  $\Delta R_{EHE}$  is recorded for both substrates, except the 508 X = 3.5 nm antidot. An increased Hall effect resistance in Pd/Co 509 antidots<sup>40</sup> and Co/Pd multilayered nanodomes<sup>41</sup> has been attrib- 510 uted to the reduced number of available paths for the electric 511 current to flow through in the discontinuous sample. Such an 512 explanation of the increase in the total amplitude of the Hall signal 513 could be applied to our antidots. After +1 T/300 K procedure for 514 both 3.5 nm samples,  $\Delta R_{EHE}$  is almost constant as appeared in 515 Fig. 6, revealing that the saturation magnetization is almost the 516 same after ZFC and FC procedures where  $T_{\text{max}} = 300$  K, since 517  $\Delta R_{EHE}$  is in part proportional to the  $M_z$  component of the magneti- 518 zation. For the 5.5 nm samples,  $EHE_{max}$  is lowered and increased 519 after FC procedures for the antidot and continuous structure. 520 respectively. Here, for the highest FeMn thickness (5.5 nm) and 521 continuous substrate, more out-of-plane magnetic domains due to 522 the FC procedure participate to the increase of the Hall signal. 523 More  $T_{\text{max}}$  and/or  $H_{FC}$  are, more the amplitude of  $\Delta R_{EHE}$  is 524 recorded. And for the membrane, even for high  $T_{\text{max}}$ ,  $\Delta R_{EHE}$  is 525 weakly lowered. The magnetic field (+2 or -2 T) during the 526 FC-procedure do not reorientate the magnetic spins in the direc- 527 tion perpendicularly to the layers due to the pinning centers 528 around the edges of antidots, which force it in the circular direction 529 of the membrane. 530

#### IV. DISCUSSION

For a ZFC procedure, a sharp transition in the perpendicular 532 magnetic loop M(H) at 5 K have been only observed for the 533



**FIG. 6.**  $\Delta R_{EHE}$  (=EHE<sub>max</sub>-EHE<sub>min</sub>) vs FeMn thickness X at 5 K of Ta(5)/Pt(5)/ Co(0.6)/FeMn(X)/Ta(5) antidots (AAO) and continuous multilayers (CMLs) for X in the (0 - 2 - 3.5 - 5.5) nm range. The dimensions of antidots are (p = 100and d = 55 nm) for X = 0 nm, (p = 100 and d = 40 nm) for X = (2 - 5.5) nm, and (p = 150 and d = 55 nm) for X = 3.5 nm. The data (empty symbols and cross symbols) are obtained after ZFC and FC procedures, respectively.

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unbiased samples (continuous and antidot) and for the 534 X = 2 nm-continuous sample. This result suggests a switching by 535 536 domain wall motion only for these stacks where the easy axis is 537 perpendicular to the plane of layers. For the other biased samples, more rounded shapes of M(H) are detected for perpendicular mag-538 539 netic fields due to the magnetization rotation process. Now, the axis perpendicular to the layers planes becomes the hard axis for 540 the magnetization for both substrates. A simple Pt/Co/FeMn 541 system with perpendicular magnetic anisotropy is difficult to 542 obtain, as for the isolated Ref. 28 for Pt(2/Co(0.6)/FeMn 543 (X > 4 nm). By using a multilayer with a period N > 1 in a 544 (Pt/Co)<sub>N</sub>/FeMn, an easy perpendicular axis is induced, as, for 545 instance, in Ref. 42 for  $[Co(0.6)/Pt(2)]_{N=5}/FeMn(X > 4 nm)$ . For 546 the series of multilayers where X = (0-3.5-5.5) nm, |  $H_A$ (anti-547 dots) $| < | H_A(CML) |$  is obtained, whereas for X = 2 nm,  $| H_A(anti-$ 548 dots) $|>| H_A(CML)|$ . For this particular sample (2 nm), a spin 549 re-orientation of the magnetic anisotropy from planar (for the 550 antidot sample where  $H_A < 0$  to perpendicular (for the continu-551 ous one where  $H_A > 0$ ) is observed. Concerning exchange bias, we 552 have  $|H_{EXC}(antidots)| > |H_{EXC}(CML)|$  for the whole series of struc-553 tures. About the coercivity,  $H_C(\text{antidots}) > H_C(C)$  for no FeMn and 554 large FeMn thickness (5.5 nm), whereas  $H_C(\text{antidots}) < H_C(\text{ML})$ 555 for intermediate FeMn thickness (2 and 3.5 nm). 556

557 The remarkable result is when a FC procedure is employed from high temperature (500 K) to 5 K with  $H_{FC} = -2$  T, a new 558 spin reorientation appears for both 5.5 nm samples with the occur-559 rence of PMA. The anisotropy field is stronger for the continuous 560 multilayer, similarly to the ZFC data. Now, the coercivity is the 561 highest for the continuous structure (as for the ZFC data for X = 2562 and 3.5 nm) for an applied magnetic aligned with the easy perpen-563 dicular axis. The magnetic field during the FC procedure favors the 564 appearance of PMA, as well as exchange bias and coercivity fields 565 (only for the continuous multilayer). Nevertheless, for the antidot, 566 567 the coercivity is almost constant between ZFC and FC data, reaching 3.338 and 3.335 kOe, respectively. 568

In fact, films deposited on top of antidots are known to have a 569 crescent shape profile.<sup>43</sup> For instance, as developed in a previous 570 study in Cu/Co/Co antidots,<sup>44</sup> a two-step reversal process in the 571 magnetic loop is explained by a three-dimensional profile of the Co 572 magnetization due to both magnetic moments between nanopores 573 aligned within the film plane and magnetic moments along the 574 walls of the nanopores aligned perpendicular to the surface. As a 575 result, the surface morphology of the alumina membrane is a key 576 577 for understanding the magnetic properties. In Ref. 45, the authors have succeeded in characterizing quantitatively both the reversible 578 579 and the irreversible magnetization processes in FePd antidots with planar magnetic anisotropy. In these holed nanostructures, d = 35580 581 and p = 105 nm, from experimental and simulated first-order 582 reversal curve measurements, the natural surface roughness of antidots is at the origin of a parallel interaction field. These features 583 could be also present in our antidots. 584

An additional etching operation at the surface of the alumina membranes might contribute to the reduction of the corrugated profile of antidots. For instance, an appropriate ion-milling procedure of antidots has led to a smoothing of a AAO surface.<sup>46</sup> As a result, we could differentiate what is coming from the geometrical roughness of antidots and what is coming from the spin orientation at the edges of the nanoholes. The next study will be focused on 591 this particular point. 592

As detailed in a previous study,<sup>25</sup> the relative proportions of 593 magnetic atoms deposited inside the nanopores vs those on the top 594 of membranes, namely,  $c_{Walls}$  and  $c_{Top}$ , respectively, are evaluated as 595 the form 596

 $c_{Top} = \frac{1 - (\pi/4)(d/p)^2}{S_{Total}},$  (1)

$$S_{Walls} = \frac{\pi dL/p^2}{S_{Total}},$$
 (2)

where  $S_{Total} = 1 - \pi (d/2p)^2 + \pi dL/(p)^2$  and *L* is the penetration 598 length of the deposited multilayer along the inner pore wall. These 599 equations can be applied to calculate proportions of deposited 600 material inside the pores and on top for our antidots where *d* and 601 period *p* are known. 602

In the following, we discuss the  $c_{walls}$  and  $c_{top}$  values, as well as 603 *p*, *d*, and the ratios of coercivity, exchange bias and anisotropy 604 between antidots (AAO) and continuous multilayer (CML) 605 obtained from our present study and those reported in literature 606 (Table III). We have selected planar and perpendicular systems 607 with unbiased and biased magnetic layers. For the calculation, a 608 penetration depth of L = 30 nm was assumed since this value has 609 been suggested for sputtered multilayers by Sousa *et al.*<sup>1</sup> and also 610 reported by Ref. 47, as well as found in our work from an analysis 611 performed by polarized x-ray absorption spectroscopy.<sup>25</sup> In addi-612 tion, the magnetic field during magnetic measurements is applied 613 following the easy axis except Refs. 18 and 19 and in our study, and 614 the operating temperature is also indicated in Table III.

In the references listed in Table III, a well-defined hexagonal 616 network of nanoholes is observed as in the case of our antidots, 617 and the values of *d* and *p* are relatively close to ones presented in 618 that study. FC procedures from 300 K have been only performed 619 for Refs. 12 and 13 for low temperature measurements (10 K). For 620 Ref. 11, annealing has been performed at a temperature higher than 621 RT (230 °C) for 30 min in a magnetic field of 1.5 kOe to promote 622 the exchange bias effects. And a magnetic field of 250 Oe was 623 applied perpendicularly to the film plane during depositing to 624 induce the exchange bias effects in Ref. 24. Concerning Ref. 12, two 625 different continuous substrates have been used, Si(001) and Al<sub>2</sub>O<sub>3</sub> 626 (0001); only the results for the latter substrate are presented here. 627 For the case of Si(001), similar tendency was observed. For the 628 other cited references, Si, <sup>9,13,15,16,24</sup> SiO<sub>2</sub>, <sup>11,21</sup> and glass<sup>17–19</sup> contin-029 uous substrates were used. 630

For the whole of references cited in Table III, anodic alumina 631 membrane templates were prepared by a two-step anodization 632 process by using  $oxalic^{9,10,16-19,48,49}$  or sulfuric<sup>11-13,15</sup> acid, expect 633 for Ref. 21 where nanoporous anodized titania (TiO<sub>2</sub>) templates 634 were fabricated by two-stage anodization of Ti film in ammonium 635 fluoride solution in ethylene glycol. For Ref. 9, at very low tempera-636 ture (2 K), the exchange bias is lowered for the continuous substrate 637 in the (2–250) K range of operating temperature as listed in 638 Table III; nevertheless, the result is the reverse for higher tempera-639 tures. Here, the values are weak for both substrates 640

<b>Indext III.</b> Comparison or surveyor and address the monitored biased (B) and unbiased (UB) systems mm/, pore diameter <i>d</i> and period <i>p</i> (in mm), calculatemperature, coercive field, exchange bias and amis applied in the easy direction except Refs. 18 and 11 this value from the area between perpendicular and the increased (/decreased) for antidots. These latter	are substruct properties or a are separated in planar ( $\rightarrow$ ) and proportions of intraterial ( $\rightarrow$ ) otropy field of the antidot, ar our study. The sign I planar published M(H) loop cases are discussed in the	and perpendicular (1) mile and perpendicular (1) mile aposited on the walles (c <sub>ue</sub> d the associated values c "-" means a planar mag s. In colors (blue, red, an paper. Here, "No" means	well as colling agnetic aniso agnetic aniso $I_{8}$ , ±2) and the continuent of the continuent of the continuent of the absence the absence the absence the continuent of the absence the absence agnetic the	trouce stack (information stack) (information to the substrate surfacter sufficiency and a sign "+" a py and a sign "+" a corresponding to ca of measurements.	when, reported in the interval tions of antidots are as (w <sub>10</sub> , +2) in % from the are indicated in parenth PMA, the * in the anisol ses where absolute valu	follows: the multilayer system follows: the multilayer system model developed in the R eses. The direction of the ropy field meaning that we les of bias and coercivity (r	is present survy. am (thickness in ef. 25, operating magnetic field is have calculated anisotropy field)
Systems	$d \pmod{p(nm)}$	$c_{walls}$ (%)/ $c_{top}$ (%)	T (K)	$H_C$ (Oe)	$H_{EXC}$ (Oe)	$H_A$ (kOe)	Reference
$UB \rightarrow$ , NiFe(18)	54/105	36.8/63.2	300	120(2)	:	$\approx^* - 0.11(-4.90)$	48
$UB \rightarrow$ , NiFe(80)	55/105	37.5/62.5	300	60(14)	:	No	49
$UB \rightarrow$ , Ni(20)	30/103	22.2/77.8	300	180(130)	:	$^{*}-0.61(-0.94)$	50
$\text{UB} \rightarrow, \text{Co}(20)$	30/103	22.2/77.8	300	280(38)	:	* - 1.26(-1.44)	50
$B \rightarrow$ , FeMn(15)/Co(20)/FeMn(15)	50/80	51.5/48.5	2	514(98)	-97(-92)	No	6
$B \rightarrow$ , (NiFe(20)/FeMn(15)) <sub>10</sub>	60/120	32.8/67.2	300	30(11)	-114(-65)	No	10
$B \rightarrow$ , IrMn(8)/CoFe(10)	45/60	67.9/32.1	300	502(29)	-227(-148)	No	11
$B \rightarrow$ , Co(1.5)/NiO(5) FC	40/90	35.5/65.5	10	320(4084)	-150(-1977)	No	12
$B \rightarrow$ , Co(8)/NiO(5) FC	40/90	35.5/65.5	10	2418(2275)	-2081(-1539)	* - 4.62(-7.95)	12
$B \rightarrow$ , Co(8)/NiO(1) FC	50/110	31.7/68.3	10	3977(1717)	-3386(-999)	No	13
$B \rightarrow$ , Co(8)/NiO(20) FC	50/110	31.7/68.3	10	2506(1409)	-2042(-1293)	No	13
$B \rightarrow$ , (CoO(1.9)/Co(1.4)) <sub>7</sub> FC	64/107	41.7.7/58.3	60	441(70)	-146(-41)	No	14
$B \rightarrow$ , Pt(5)/Co(0.6)/FeMn(2)	40.9/98.4	31.5/68.5	5	2517(2820)	3(2)	-2.14(+0.41)	Our study
$B \rightarrow$ , Pt(5)/Co(0.6)/FeMn(3.5)	55.8/148.3	21.2/78.8	5	2625(3226)	-127(-58)	-2.13(-5.17)	Our study
$B \rightarrow$ , Pt(5)/Co(0.6)/FeMn(3.5) FC	55.8/148.3	21.2/78.8	5	2614(5034)	-299(-3239)	No	Our study
$B \rightarrow$ , Pt(5)/Co(0.6)/FeMn(5.5)	42.7/99.9	32.0/68.0	5	3338(2501)	-496(-39)	-4.83(-5.14)	Our study
UB $\uparrow$ , (Co(0.5)/Pt(2)) <sub>5</sub>	30/60	49.4/50.6	300	1350(140)		$\approx^{*} + 3.00(+5.53)$	15
UB ↑, CoCrPt(10)	55.7/105	37.9/62.1	300	361(151)		$\approx^{*} + 0.63/(+5.47)$	16
UB ↑, TbCo(30)	75/105	51.7/48.3	300	5000(2000)		* + 3.04(+12.43)	17
UB ↑, DyFe(30)	45/105	31.0/69.0	300	$\approx$ 926(545)	:	* + 1.54(-11.18)	18,19
UB $\uparrow$ , (Co(0.5)/Pd(1)) <sub>5</sub>	35/140	15.0/85.0	5	3500(2000)	:	No	20
UB 1, Pt(5)/Co(0.5)	56.4/101.7	40.4/59.6	5	1418(1194)	:	+1.48(+2.16)	Our study
B $\uparrow$ , (Co(0.5)/Pt(0.5)) <sub>3</sub> /IrMn(15)	40/100	30.1/69.9	300	50(200)	-566(-333)	No	24
B $\uparrow$ , (Co(0.5)/Pd(1)) <sub>5</sub> /CoFe(0.8)/IrMn(6)	50/100	37.2/62.8	300	1306(562)	-241(-194)	No	21
B ↑, IrMn(6)/CoFe(0.8)/(Pd(1)/Co(0.5)) <sub>5</sub>	50/100	37.2/62.8	300	1525(966)	-18(-13)	No	21
B 1, IrMn(6)/CoFe(0.8)/(Pd(1)/Co(0.5))5	30/100	23.3/76.7	300 7	1263(505)	-190(-179)	No 1 02( 1 10)	- - - -
Ut (c.c)Utervin(c.u)/Co(U.c) the second termination of the second s	42.//99.9	0.80/0.26	n	(UU44)0000	(/CI7+)6//+	+1.93(+4.18)	Our study

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 $(-H_{EXC} < 100 \text{ Oe})$  and the trilayer defined by FeMn/Co/FeMn is a 641 structure more complex than a simple bilayer one with Co and 642 643 FeMn. For Refs. 10 and 11, other results are published with similar 644 tendency (both coercivity and bias fields higher for antidots) merged in Table III. In these previous works,  $(NiFe_X/Co_{150})_{10}$ 645 where X varies in the (40-60-80-100) nm range and for different 646 hole diameters [(2-5.5) nm] and pore diameter in the (7-45) nm 647 range<sup>11</sup> have been studied. 648

Now we discuss the behaviors of coercivity, exchange bias, and anisotropy as a function of employed substrate (antidots vs continuous) for a given easy axis (planar or perpendicular) and for (biased or unbiased) structures.

(1) Magnetic anisotropy. By using the formula for the anisotropy 653 field  $(H_A = 2 \int_0^{H_{max}} dH \cdot [M_Z(H_Z^{mean}) - M_X(H_X)])$ , for planar and perpendicular magnetic fields  $(H_{max}$  designating the field where 654 655 the magnetization is saturated),  $H_A$  is calculated from the mag-656 657 netic loops published in Refs. 12, 15, 16, 17, 18, 19, 48, and 50, as indicated by "\*" in the anisotropy field column in Table III. For 658 Refs. 15, 16, and 48, even if the applied magnetic field is not effi-659 cient for getting the saturation, an extrapolation of the magnetic 660 hysteresis is applied for the calculation of  $H_A$ . We systematically 661 662 found  $|H_A(\text{antidots})| \leq |H_A(\text{CML})|$  for the different published 663 references as well as for our results, except for the X = 2 nm multilayer (as highlighted in green color in Table III). For a given 664 easy axis (planar for Refs. 12,48,50 and our results or perpendicu-665 lar for Refs. 15-19 and our results) and for a continuous stack, by 666 667 introducing a perforated network, additional opposite (perpendicular/planar) magnetic moments are induced in the valley of anti-668 dots such that the total anisotropy field is reduced for the 669 discontinuous sample. For instance, for the case of Ni antidot 670 arrays,<sup>51</sup> the continuous structure, as well as the antidot ones for 671 d in the (35-80) nm range, behave as a planar magnetic anisot-672 ropy ( $H_A < 0$ ). More d is less  $-H_A$  for antidot is recorded due to 673 the increase in the out-of-plane magnetic moments in the valley 674 of nanoholes. A PMA is only favored for the largest hole diame-675 ters (d > 80 nm), and the anisotropy sign changes (from negative 676 677 to positive value inducing out-of-plane magnetic anisotropy). The same tendency is observed in Refs. 18, 19, and 51 for DyFe(30) 678 and Ni, respectively. Concerning the X = 2 nm stack, the situa-679 tion is the reverse for the substrates: a weak PMA (+0.41 kOe) is 680 obtained for the continuous sample antidot whereas a moderate 681 in-plane magnetic anisotropy (-2.14 kOe) is achieved for the 682 683 antidot. Indeed, the anisotropy field changes sign from positive to negative by using an antidot substrate. This particular feature 684 explains the strongest absolute magnetic anisotropy field for the 685 antidot larger than the one of the continuous stacks. Moreover, 686 687 the field cooling procedure re-orientates the magnetization in the 688 perpendicular direction for the X = 5.5 nm multilayer for both substrates, leading to positive  $H_A$  first reported for biased perpen-689 dicular antidots in the literature. 690

(2) Coercive fields. Concerning unbiased antidots systems, the coercivity is systematically enhanced by employing the antidot as a substrate. The applied magnetic field is in the direction of the easy magnetization (planar for Refs 48–50 and perpendicular magnetic anisotropy for Refs. 15–20), and holes act as pinning centers of domain wall displacement, specially at the edges of the pores

during magnetization reversal. This feature is largely admitted in 697 the literature.<sup>11-17,20,21,23,40,43,48,50</sup> For the biased systems, results 698 seem more contrasted at a first sight.  $H_C(AAO) > H_C(CML)$  is 699 measured for most of Refs. 9, 10, 11, 12, 13, 14, 21, and 22. Here, 700 the applied magnetic field is applied in the easy direction of the 701 magnetization. Nevertheless, for our samples with X = (2 - 3.5) 702 nm measured after a ZFC procedure, and possibly the 3.5 nm 703 samples after a FC one, the perpendicular applied magnetic field 704 being now a hard axis for the antidots. In the same way, FC mea- 705 surements show similar features for systems with low ferromag- 706 netic thicknesses [(1.5-3) nm Co for Ref. 12] with, in particular, 707 an asymmetric hysteresis loop for the sheet samples and a 708 two-step magnetization reversal process in the antidot arrays for 3 709 nm Co, similarly to our study.44 Concerning Ref. 24, 710  $H_C(AAO) < H_C(CML)$  is also observed. For both samples in the 711 previous reference, a perpendicular magnetic field has been used 712 during the deposition. Consequently, the nature of the easy axis 713 and the role of the magnetic field (for a FC-procedure or during 714 the disposition) have drastic influences on the amplitude of 715 the coercivity. A possible explanation is the three-dimensional 716 magnetization effect induced by the valley of nanoholes and the 717 magnetic field reinforcing the coercivity for the sheet sample 718 rather than for the discontinuous one. This might be the case for 719 our X = 5.5 nm samples after a FC procedure [ $H_C(AAO) = 3335$  720  $Oe < H_C(CML) = 4400 Oe$ ]. Finally, for the antidot deposited 721 with the high antiferromagnetic thickness (X = 5.5 nm) and for 722 the ZFC procedure, even if the magnetic is planar for both 723 samples as indicated in Table III, coercivity is larger for the 724 antidot. Here, the antiferromagnetic coupling for both samples is 725 high such that the pinning effects around valleys of nanoholes 726 lead to larger  $H_C$  for the antidot compared to the continuous 727 structure. 728

(3) Exchange bias fields. The exchange bias is lowered for the 729 sheet sample compared to the antidot, expected the group in 730 Ref. 12 for Co<sub>15</sub>/NiO<sub>50</sub> as seen in red color in Table III. Similar 731 features are checked for our multilayers (X = 3.5 and 5.5 nm). 732 All these measurements include a FC-procedure, indicating once 733 again the key role of the applied magnetic field on the arrange-734 ment of the magnetic moments on the 3D-profile of antidots. 735 For Ref. 24,  $H_C(AAO) < H_C(CML)$  but  $|H_{EXC}(AAO)| > |H_{EXC}$  736 (CML)| whereas a magnetic field has been employed during the 737 deposition. This particular result seems to establish the predomi-738 nant role of the magnetic field during the FC procedure on the 739 one during the deposition. 740

To our knowledge, there are no published results combining 741 variations of coercivity, bias as well as anisotropy as a function of 742 structural dimensions (as hole diameter). A noteworthy investiga-743 tion, showing variations of  $H_C$  and  $K_{eff}$  as a function of d at RT in 744 20 nm-Ni antidot arrays thin film (where p = 103 nm), is found in 745 Ref. 51. For a magnetic field orientated in the easy direction of the 746 magnetization,  $H_C(AAO) > H_C(CML)$  is also found. We can also 747 note the behaviors of  $H_C$  and  $H_{EXC}$  as a function of d in CoO/Co 748 multilayer thin films (where p = 107 nm) in Ref. 14.

A discussion about the variation of the coercivity, exchange 750 bias, and the magnetic anisotropy for the whole of data published 751 in the literature (see Table III) is difficult due to the different 752

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nature of systems, the different thicknesses of magnetic layers, and 753 the nature of the magnetic measurements (ZFC or FC, or magnetic 754 755 field applied during the deposition or post-deposition). An effort 756 could be done for a series of samples with, for instance, a fixed stack (unique composition and same thicknesses) and by varying 757 only the hole diameter. From our part, a forthcoming paper will 758 detail magnetic properties of exchange-coupled Cu/Co/Cu trilayers 759 for operating temperature in the (5-400) K range and for different 760 d. Nevertheless, variations of the coercivity for biased planar (left-761 bottom part) and perpendicular (top-right part) antidots as a func-762 tion of the walls content  $c_{Walls}$  are plotted in Fig. 7. The data are 763 extracted from the literature and from our own results, as merged 764 in Table III. Here, the hole diameter d is comprised between 40 765 and 60 nm, even if p is varied between 60 and 150 nm. For these 766 studies, the magnetic field is applied in the easy direction of magne-767 tization. A general tendency indicates an increase in the coercivity 768 when the fraction of atoms on the valley of antidots enhances. The 769 770 density of pinning sites imposed by the antidots pores leads to strong coercivity at least for c<sub>Walls</sub> in the (20-70)% range for biased 771 planar systems and the (30-40)% range for biased perpendicular 772 systems. A second reason at the origin of the increase of  $H_C$  is the 773 magnetization reversal more favorable via domain wall movement 774 775 (low wedge-to-wedge s value), as claimed by other studies.<sup>2</sup> 776 And for highest  $c_{Walls}$  values (i.e., large hole diameter), it is observed that the coercivity for antidots is drastically lowered when 777 d is enhanced, as in Refs. 15 and 51 for unbiased perpendicular 778 and planar multilayers, respectively, c<sub>Walls</sub> reach 69% and 66% for 779 780 Refs. 15 and 51, respectively. The loss of the magnetic anisotropy and the magnetization reversal coherent rotation<sup>50</sup> 781 constitute two factors explaining the decrease in coercivity with cwalls. 782

A second way to estimate the importance of wall fraction is to take account the wedge-to-wedge distance between two consecutive



**FIG. 7.**  $H_c^{AAO}/H_c^{CML}$  ratio vs calculated proportions of material deposited on the walls ( $c_{Walls}$ ) for biased antidots systems with planar (left-bottom part) and perpendicular (top-right part) magnetic anisotropy. The data are extracted from the literature and are also corresponding to our present study (see Table III for additional information about the sample and the operating temperature).

holes (s = p - d as introduced in Table I). By using this previous 785 relation and replacing d = p - s in Eq. (2), it arrives 786

$$c_{Walls} = \frac{\pi(p-s)L/p^2}{1 - \pi \left(\frac{p-s}{2p}\right)^2 + \pi \frac{(p-s)L}{p^2}}.$$
(3)

Here, p = 100 nm (as largely used in the literature), 788 L = 30 nm, and *s* in varied in the (0–100) nm. The *s* dependence of 789  $c_{Walls}$  is given in Fig. 8. As expected,  $c_{Walls}$  decreases with *s*, a linear 790 fit leads to  $c_{Walls} \approx 0.764 - 0.007640.007 \, 64 \times s$  (where *s* is 791 expressed in nm). Here, the good quality of linear fitting is 792 observed for *s* in the (10–100) nm range, where typical dimensions 793 are used for the fabrication of antidots. As example, s = 35 nm (or 794 d = 65 nm currently used for antidots in the literature) leads to 795  $c_{Walls} = 49\%$  (see the vertical and horizontal lines in Fig. 8). This 796 model [Eq. (3)] can be easily used for a unique stack where only *s* 797 (or *d*) is varied (*p* being fixed), in order to quantify  $c_{Walls}$ .

(or *d*) is varied (*p* being fixed), in order to quantify  $c_{Walls}$ . 798 In many reports, <sup>2,49–51,53–55</sup> coercivity is assumed to vary as 799  $H_C \propto 1/(p-d) = 1/s$ . Therefore, an increase in *d* (or a decrease 800 of *s*) leads to an enhancement of the coercivity. This corresponds to a 801 regime where the nanoholes are more or less disconnected from each 802 other, i.e., for high *s* or low  $c_{Walls}$ . In fact, the pinning effects become 803 more prominent when the magnetic atoms are mainly deposited 804 around the nanoholes. Consequently, a strong magnetic field is necessary to overcome the pinning coupling. Then, from the linearity of *s* 806 and  $c_{Walls}$ ,  $H_C$  can be rewriting as  $H_C \approx 1/(100 - 130.89 \times c_{Walls})$ . 807 In Fig. 7,  $H_C^{AAO}/H_C^{CML} = 221.4/(100 - 130.89 \times c_{Walls}) - 2.4$  is 808 derived from a adjustment of data via our model, revealing its 809 validity. 810



**FIG. 8.** Calculated proportions of material deposited on the walls ( $c_{Walls}$ ) vs the wedge-to-wedge distance between two consecutive holes (*s*) from Eq. (3). Here, p = 100 nm, L = 30 nm, and a linear adjustment is added for easily calculating  $c_{Walls}$  starting from *s*.

811 A third way for analyzing the magnetic features is to consider 812 the filling factor, namely, the porosity (Po) defined by<sup>1,48,56</sup>

$$Po = \frac{\pi (d/p)^2}{2\sqrt{3}}.$$
(4)

By replacing *d* with *Po* [from Eq. (4)], and using Eq. (2),  $c_{Walls}$  reads

$$c_{Walls} = \frac{\pi L/p}{\left[\frac{2\sqrt{3}\mathbf{Po}}{\pi}\right]^{-1/2} - \left[\frac{2\sqrt{3}\mathbf{Po}}{\pi}\right]^{1/2} + \frac{\pi L}{p}}.$$
 (5)

The Po dependence of  $c_{Walls}$  is plotted in Fig. 9 in the left-816 bottom part where p = 100 and L = 30 nm.  $c_{Walls}$  is enhanced 818 when the porosity is increased as expected. In the (15-58)% range 819 820 of porosity, a linear fitting as  $c_{Walls} \approx 0.2214 + 0.6588 \times Po$  might be used for an accurate estimation of the atomic proportion in the 821 periphery of nanopores. As example, Po = 40% leads to 822  $c_{Walls} = 49\%$ , corresponding to s = 35 nm (see the vertical line in 823 Fig. 9). Equation (5) can be easily used, for a unique stack where 824 only *Po* is varied (p fixed), in the aim to calculate  $c_{Walls}$  from *Po*. 825 Finally, the coverage ratio C is defined as C = 1 - Po,<sup>14,52</sup> i.e., 826

$$C = 1 - \frac{\pi (d/p)^2}{2\sqrt{3}}.$$
 (6)

As a consequence, Eq. (7) permits to express  $c_{Walls}$  as a function of C,



**FIG. 9.** Calculated proportions of material deposited on the walls ( $c_{Walls}$ ) vs the porosity *Po* from Eq. (5) in the bottom-left part, and vs the coverage ratio *C* (=1 - *Po*) in the top-right-part. Here, p = 100 nm, L = 30 nm, and linear adjustments are added for easily calculating  $c_{Walls}$  starting from *Po* or *C*.

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The top-right part of Fig. 9 gives the variation of  $c_{Walls}$  with *C*. 831  $c_{Walls}$  decreases when *C* is enhanced, similarly to  $c_{Walls}(s)$  (see 832 Fig. 8). Higher values of *C* correspond to less material deposited 833 around the holes. The value of 49% for  $c_{Walls}$  from the previous 834 example corresponds to a coverage ratio C = 60% (or Po = 40%, 835 see the vertical line in Fig. 9). In the (42–85)% range of *C*, a linear 836 fit as  $c_{Walls} \approx 0.8767 - 0.6540 \times C$  might be used for an accurate 837 estimation of the atomic proportion in the periphery of nanopores. 838 Here, we check that the slopes of linear fittings appearing in Fig. 9 839 are opposite due to the simple relation C = 1 - Po. 840

By merging Eqs. (2), (3), (5), and (7), and considering an 841 antidot network where *p* is fixed, and L = 30 nm, we are be able to 842 express *c*<sub>*Walls*</sub> as a function of *d*, *s*, *Po*, and *C*, respectively. The mag- 843 netic properties of the sample (coercivity, exchange bias, anisot- 844 ropy, etc.) might be discussed in the light of variation of these 845 structural parameters. It would be interesting to possibly use these 846 expressions for magnetic studies devoted to antidots. 847

#### **V. CONCLUSION**

From intensive extraordinary Hall effects performed at 5 K, 849 biased Ta(5 nm)/Pt(5 nm)/Co(0.6 nm)/FeMn(X)/Ta(5 nm) stacks 850 employing two types of substrates (antidots vs  $Si/SiO_2$ ) for X in the 851 (0-5.5) nm range reveal either planar magnetic anisotropy for ZFC 852 measurements and  $X \neq 0$ , or perpendicular magnetic anisotropy for 853 ZFC data for X = 0 samples (both multilayer sheet and antidot) 854 and for X = 2 nm (only sheet sample), and by using a 855 FC-procedure (with a maximum temperature around Néel temper- 856 ature of FeMn) for X = 5.5 nm. From perpendicular magnetic 857 fields data, more X is more both  $|H_{EXC}|$  and  $H_C$  enhanced for the 858 series of antidots and sheet samples (accompanied by a weak 859 decrease for the largest X thickness for the large regime of X), as 860 expected for ferromagnetic/antiferromagnetic exchange-coupled 861 nanostructures. As discussed from our results as well as in pub- 862 lished data in the literature, we believe that  $H_C(AAO) > H_C(CML)$ , 863  $|H_{EXC}(AAO)| > |H_{EXC}(CML)|$ , and  $|H_A(AAO)| < |H_A(CML)|$  for 864 ZFC measurements with magnetic fields in the easy direction of 865 the magnetization performed on continuous and antidots samples 866 behaving similar signs of the magnetic anisotropy. For magnetic 867 fields in the hard direction of the magnetization and/or FC proce- 868 dures with efficient maximum temperature ( $\approx T_h$  and/or  $\approx$ Néel 869 temperature) performed on the discontinuous sample containing 870 moderate thicknesses (X < 5 nm), these inequalities of coercivity 871 and exchange bias might be in the opposite way due to the 872 3D-profile of nanopores. In addition, we have expressed the struc- 873 tural parameters, such as the wedge-to-wedge distance (s), the 874 porosity (Po), and the coverage ratio (C) as a function of propor- 875 tion of atoms deposited in the nanopores. The geometry of anti- 876 dots, as well as the magnetic and thermal history of the 877 nanodevice, tunes the magnetic properties of magnetic antidots. 878 This can lead to antidots which is an interesting possibility for syn- 879 thesizing and controlling skyrmions. 880

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881

848

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#### **884 AUTHOR DECLARATIONS**

#### 885 Conflict of Interest

886 The authors have no conflicts to disclose.

#### 887 Author Contributions

888 F. Fettar: Conceptualization (lead); Data curation (lead); Formal analysis (lead); Investigation (lead); Methodology (lead); Resources 889 (equal); Software (equal); Supervision (lead); Validation (lead); 890 Writing - original draft (lead); Writing - review & editing (sup-891 porting). L. Cagnon: Resources (equal); Visualization (equal). 892 D. Barral: Investigation (equal); Methodology (equal); Resources 893 (equal). P. David: Investigation (equal); Methodology (equal); 894 Resources (equal). L. Naudin: Writing - review & editing (equal). 895 Blondelle: Investigation (equal); Methodology (equal); 896 F. 897 Resources (equal). F. Gay: Investigation (equal); Methodology (equal); Resources (equal). 898

#### 899 DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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