

In situ monitoring of electric field effect on domain wall motion in Co ultrathin films in direct contact with an electrolyte

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ABSTRACT

AQ1

15 We present experimental data on the electric field effect on the magnetic domain wall dynamics in Co ultrathin films in direct contact with

16 an aqueous electrolyte and in the absence of any oxide layer. We use a three electrode electrochemical setup to apply a large and uniform

¹⁷ electric field and to precisely separate chemical effects induced by hydrogen from pure electric field effects. We show that in the case of the

¹⁸ pure electric field effect, the domain wall velocity varies exponentially with the electric field and that these variations are larger than those

19 observed previously on similar systems due to a larger magnetoelectric coefficient in our case.

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20 Domain wall (DW) motion in perpendicularly magnetized ferromagnets is a field of intensive research.¹⁻³ DWs may be displaced by 21 22 applying an external magnetic field⁴ or by injecting an electric cur-23 In spite of the large current densities required and the associrent.⁵⁻⁷ 24 ated heat generation and power consumption, the latter process is of 25 the greatest technological relevance since it allows controlling the 26 motion of a larger number of neighboring DWs in racetrack memo-27 ries³ and addressing individual magnetic nanodevices as memristors.⁸ An elegant approach to reduce the current density and the device 28 power consumption is to lower the magnetic anisotropy energy 29 (MAE) that controls the DW depinning and the motion process. A 30 promising means to achieve this goal is to apply a voltage across an 31 32 insulating layer between a gate electrode and the ferromagnetic layer to modify the layer MAE.⁹ This method, usually called voltage control 33 of magnetization (VCM), has been widely investigated experimentally 34 and theoretically⁹⁻¹¹ since the early publication of Weisheit et al.¹² 35 36 More recent studies have demonstrated that magnetic DW velocity in 37 ultrathin films can be modified by the application of a voltage.4,13

When considering metallic ferromagnets in contact with an insulating layer, VCM is related to several effects. The first effect is the potential induced accumulation of charges at the ferromagnet surface,^{12,18} which is commonly called the electric field effect (EFE). The second effect is related to the modification of the ferromagnet surface chemistry either by oxidation at the ferromagnet/oxide interface¹⁹ or at the solid/electrolyte contact²⁰ or by molecular adsorption at the ferromagnet surface in electrolytes.¹⁸ This effect is usually called the 45 magnetoionic effect. The latter process induces large MAE modification reaching several 100 fJ V^{-1} m⁻¹, while MAE induced by charge 47 accumulation is generally small (a few 10 fJ V^{-1} m⁻¹).⁹ 48

The large majority of the experimental studies has been per-49 formed on solid state devices using an insulating layer which may 50 induce chemical modification of the magnetic layer in the presence of 51 an external electric field. Gating directly through an electrolyte, i.e., 52 putting the ferromagnetic layer in direct contact with an electrolyte, is 53 54 appealing as it prevents possible artifacts induced by the insulating 55 layer and allows the application of larger electric fields.¹² However, the major drawback of this approach is the sample oxidation during its 56 transfer in air between the sample preparation chamber and the cell 57 where magnetic measurements are performed. One alternative 58 approach used by us and others combines in situ electrodeposition 59 and magnetic characterization in a single electrochemical cell which 60 avoids transfer of the sample through air.¹⁸ 61

In this work, we investigate the voltage control of MAE and the 62 DW velocity in Co layers in direct contact with an electrolyte and in 63 the absence of any insulating layer using in situ magneto-optical Kerr 64 effect (MOKE) imaging of magnetic domains and complementary in 65 situ MOKE characterization. We used a specific experimental proce-66 dure for contacting Co ultrathin epitaxial layers with an electrolyte in 67 situ avoiding any transfer in air and surface oxide formation. In this 68 procedure which was successfully used in recent studies,^{18,21} an 69

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ultrathin epitaxial Co layer is grown *in situ* by electrodeposition in the cell used for MOKE measurements. Using this approach, we clearly separate EFE from the magnetoionic effect in an electric field range comparable to that in solid state device studies. We also show that in the case of EFE, the DW velocity varies exponentially with the electric field but with a larger variation coefficient.

76 The Co films (~0.8 nm thick) were electrodeposited on a Pd/Au/ Si(111) substrate in situ in a custom three electrode electrochemical 77 flow cell,^{18,24,25} which was inserted either in the MOKE setup or in the 78 MOKE microscope and connected to a potentiostat. The sample struc-79 ture and the cell are sketched in the supplementary material (Fig. S1). 80 81 The atomically flat Au(111) buffer layers (8 nm thick) were prepared by epitaxial electrodeposition on Si(111).²⁶ The Co layers were covered 82 by a monolayer of carbon monoxide to increase the layer out-of-plane 83 anisotropy and to widen the Co film potential stability range.¹⁸ All 84 magnetic characterization presented below is conducted under a con-85 tinuous flow of 0.1 M K₂SO₄ + 1 mM H₂SO₄ + 1 mM KCl saturated 86 with CO. MOKE results on CO covered Co layers deposited on 87 Au/Si(111) samples without a Pd underlayer are given in the supple-88 mentary material for comparison. The polar MOKE setups are home-89 90 built and described in previous works and in the supplementary material.^{18,20,21} The cell is installed vertically between the two poles of 91 92 an electromagnet, which applies a magnetic field perpendicular to the 93 sample surface. DW velocity v was determined from the traveling dis-94 tance of the DW during the propagation magnetic pulse averaged over 95 the entire domain.

The CO covered Co/Pd/Au/Si(111) samples in contact with the 96 97 electrolyte are perpendicularly magnetized as demonstrated by square M-H curves with a high coercive field ($H_{\rm C}$) measured at different 98 potentials in the range of -0.8 V to -1.3 V [see insets of Fig. 1(a)]. 99 100 The variation of $H_{\rm C}$ during a potential sweep from -0.8 V to -1.3 V and backward is presented in Fig. 1(a) (open symbols) together with 101 the electrochemical current (solid line). One notices two distinct 102 regimes: for potentials U > -1 V, H_C varies quasilinearly with a nega-103 tive slope, and for U < -1 V, H_C decreases, while a significant electro-104 chemical current starts to flow. This decrease is concomitant with the 105 106 onset of the electrochemical reaction where H⁺ cations in the electro-107 lyte are transformed into H₂ gas through the hydrogen evolution reaction (HER) $2H^+ + 2e^- \rightarrow H_2$ ²⁷ Since the observed H_C behavior in 108 the HER regime is quasiabsent in the absence of the Pd underlayer 109 (see the supplementary material, Fig. S3), the prominent decrease in 110 $H_{\rm C}$ below -1 V observed in Fig. 1(a) suggests that the effect related to 111 112 HER is specific to the presence of the Pd underlayer.

In Fig. 1(b), we present the ratio $\Delta H_C/H_C^{0}$, the relative variation 113 of $H_{\rm C}$ with respect to $H_{\rm C}^{0}$, the value of $H_{\rm C}$ for U = -0.8 V. $\Delta H_{\rm C}/H_{\rm C}^{0}$ 114 is plotted as a function of potential measured at different time delays t 115 after the potential step [see the inset in Fig. 1(b)]. The $\Delta H_C/H_C^0$ time 116 transient is composed of an instantaneous jump followed by a slow 117 118 decay with a time constant τ , before a steady state value is reached (see also Fig. S2). If measured at $t > \tau$ after a potential step (red open sym-119 bols and red line are guides to the eye), $\Delta H_C/H_C^0$ follows a trend simi-120 lar to that measured during a potential sweep [Fig. 1(b) black curve], 121 with a maximum around -1.05 V. The results change considerably 122 123 when considering the values of $\Delta H_C/H_C^0$ acquired immediately after 124 the potential jump [Fig. 1(b) black stars and black line are guides to 125 the eye]. In this case, the variation is almost linear with time over the entire potential range. By comparison, $\Delta H_{\rm C}/H_{\rm C}^{0}$ is a linear and 126



FIG. 1. (a) Potential induced variations of coercive field $H_{\rm C}$ and electrochemical current upon sweeping the potential at a rate of 20 mV/s. Insets: left, normalized M-H curve of the sample and right, a selection of M-H curves during a potential sweep with a zoom on one edge of the M-H curve. The different colors correspond to various potentials between -0.8 V and -1.3 V. (b) Potential dependence of $\Delta H_C/H_C^0$ measured in steady state conditions (red open squares) or immediately after the application of the potential step (black stars). The black curve corresponds to the potential sweep experiment shown in (a). The black and red lines are guides to the eye. The inset shows $\Delta H_C/H_C^0$ as a function of time during a potential step. The black star and the red square indicate which values are used in the main plots.

reversible function of the applied potential over the same potential 127 range for CO-covered Co/Au/Si(111) samples in both potential step 128 and potential sweep experiments (see Fig. S3). Consequently, the effect 129 induced by the HER may be completely avoided by shortening the residence time, i.e., the time spent by the sample in this potential range. 131

We now focus on voltage control of DW velocity (ν). Figure 2(a) 132 presents a typical image of the magnetic domain in Co/Pd/Au/Si(111) 133 films obtained by polar MOKE microscopy, by subtracting two images 134 taken after applying the H_{nucl} nucleation pulse and H_{prop} propagation 135 pulse. The magnetic domain propagates within the dark crown from the 136 center to the edge (see white arrows). The circular shape of the crown 137 suggests that the DW propagates isotropically, indicating a rather 138 homogeneous MAE landscape. The dependence of ν as a function of 139 the propagation field H_{prop} is shown in Fig. 2(b). These measurements 140

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100 µm

200

-1.0 V -0.9 V

-0.8 V

1.5

H_{prop}(Oe)

250

300

10⁻³

10-

10⁻⁵

10⁻⁶

(b)

1.3

DW velocity (m/s)

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(a)

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200 06

-0.8

b

Steady State Potential steps

-0.8

-0.9

-0.9



FIG. 2. (a) Example of a "differential" MOKE image of a Co(0.8 nm)/Pd/Au/Si(111) sample after the application of a magnetic field pulse (nucleation 220 Oe during 5 ms, propagation 160 Oe during 2 s) at U = -0.85 V to expand an initially formed inverted magnetic domain. (b) Plots of DW velocity as a function of H_{prop} kOe^{-1/4}) measured at different potentials. For clarity, the top x-axis displays the values of H_{prop} in Oe.

1.4

 $\mathsf{H}_{\mathsf{prop}}$

-^{-1/4} (kOe ^{-1/4})

are performed in conditions where the potential is constant during the 141 142 entire MOKE image acquisition (few seconds). This procedure will be 143 called hereafter steady state conditions. The linear dependence of the logarithm of v as a function of $H_{\text{prop}}^{-1/4}$ indicates that the DW propaga-144 tion is in the creep regime, i.e., the propagation of the wall is dominated 145 146 by thermal activation over the energy barrier of pinning sites, and fol-147 lows the phenomenological equation,

$$\nu = \nu_0 \exp\left[-(U_C/kT) (H_{prop}/H_{dep})^{-1/4}\right].$$
 (1)

In this equation, v_0 is a numerical prefactor, T is the temperature, k is 148 149 the Boltzmann factor, H_{dep} is the depinning magnetic field, and U_C is the disorder-induced energy barrier arising from the collective pinning 150 151 of small DW sections. Figure 3(a) presents the dependence of v (on 152 a logarithmic scale) as a function of potential for different values of 153 H_{prop} . For U > -1 V, the plots are linear with a slope quasiindependent of H_{prop} . Conversely, for U < -1 V, the DW velocity 154

FIG. 3. (a) DW velocity as a function of the potential at different magnetic fields in steady state conditions (where the potential is constant during the entire MOKE image acquisition). A break is introduced along the y-axis to compensate for the rapid increase in v with H_{prop} between 220 and 330 Oe. (b) DW velocity as a function of the potential pulse amplitude at 330 Oe. In this case, all images are recorded at a potential of -0.8 V and the pulses of the field and potential are synchronized as shown in the drawing in the inset. Note the exponential law (red line).

becomes quasi-independent of the potential, a phenomenon which is 155 concomitant with the regime change of $H_{\rm C}$ variations observed in Fig. 156 1(b). These results suggest that $H_{\rm C}$ and ν variations as a function of 157 potential are due to the potential dependence of the Co layer MAE. In 158 order to study v without the influence of the HER, we performed 159 experiments where the residence time at a potential U is short and all 160 MOKE images are acquired at a rest potential of -0.8 V, i.e., outside 161 the HER region. For these experiments, potential and magnetic field 162 pulses are synchronized and last typically 0.2 s. Figure 3(b) presents 163 data measured using this procedure (red squares) together with the 164 steady state measurements (black stars) for a magnetic field of 330 Oe. 165 One clearly observes that using this second procedure ν follows an 166 exponential dependence over the full potential range. The slope is 167 slightly different from the one measured in steady state conditions (for 168 U > -1 V), and the two plots are slightly shifted. The origin of these 169 differences may probably be a reorganization of the Co surface in con- 170 tact with the electrolyte, after long steady state measurements over sev- 171 eral hours. 172

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The polar MOKE and polar MOKE microscopy results clearly 173 174 show that the more positive the electrochemical potential, the larger 175 the DW velocity and the lower the coercive field. In agreement with our previous studies,¹⁸ this is consistent with a decrease in the MAE at 176 more negative potentials induced by the electric field at the 177 electrolyte/Co interface. In addition to this trend, three main outcomes 178 179 of this work can be highlighted: (i) in situ grown Co layers in contact 180 with an electrolyte present large propagating magnetic domains which can be observed by in situ MOKE microscopy, and their DW propaga-181 182 tion velocity v follows the creep regime equation in the explored magnetic field range; (ii) v could be measured at high electric fields (up to 183 3 V/nm) in the absence of an intermediate oxide layer and in the 184 absence of any chemical effect; (iii) v varies exponentially with the 185 186 applied potential in the absence of chemical effect and becomes quasi-187 independent of the potential in the HER range. These variations are entirely reversible and take place with a short time constant, corre-188 sponding to the one of the electrochemical cell we used in this study 189 190 (20 ms).

191 The first outcome suggests that these electrochemically grown Co 192 films in direct contact with the electrolyte present a low and homogeneous density of defects as shown in previous in situ scanning tunnel-193 ing microscopy and in situ X ray diffraction studies of the Co layer 194 grown on Au(111).^{28,29} These flat Co layers yield a DW propagation 195 196 regime similar to what is observed in solid state devices prepared by 197 sputtering deposition. It is interesting to note that we obtain ν values similar to those for solid state samples with a similar Co thickness and 198 at a $H_{\rm prop}$ value of 250 Oe as AlOx/Co/Pt¹³ and HfO₂/MgO/Co/Pt 199 200 (after extrapolation to higher magnetic fields).¹⁷

201 The second outcome is directly related to our approach: (i) we 202 grow the ferromagnetic layer in situ in an electrochemical environ-203 ment with the potential control over the sample ensuring that no Co 204 oxide is formed and (ii) the direct contact of the Co layer with the elec-205 trolyte allows applying large electric fields which are spatially homogeneous using low applied voltages. This ferromagnetic metal/electrolyte 206 207 contact with potential control ensures the separation of the pure charge accumulation effect from other effects. In solid state devices, 208 209 the dielectric layer often interferes in the EFE signal because of electric field induced ion migration and charge trapping in the dielectric layer 210 211 and surface oxidation of the ferromagnetic layer, leading to slow and often irreversible modifications of the MAE. In our case, potential 212 induced chemical modification of the sample which takes place in the 213 HER regime is well characterized and can be avoided if the potential is 214 215 applied during short periods.

Regarding the last outcome, the exponential dependence of DW 216 velocity v and the linear variations of $\Delta H_C/H_C$ with applied potential 217 are consistent with a linear MAE potential dependence and a pure 218 charge accumulation effect at the electrolyte/Co interface. Indeed, in 219 our previous papers,^{18,21} we demonstrated that the linear behavior of $\Delta H_{\rm C}/H_{\rm C}$ is related to a linear change of the electrolyte/Co interface 221 Electrolyte/Co. We also showed that whenever electrochemical 222 MAE $K_S^{L\mu}$ effects are involved, nonlinear variations of $\Delta H_{\rm C}/H_{\rm C}$ are observed as a 223 function of potential.¹⁸ The exponential dependence of ν with applied 224 potential linked to a linear modification of the interface anisotropy 225 226 energy is also consistent with the interpretation given in Ref. 13.

We now compare the potential dependence of the velocity for electrolyte/Co/Pd/Au/Si(111) with other studies on solid state devices. In the study of Ref. 13, the influence of the potential on ν was measured at 220 Oe (as in our study), and the range of v as a function 230 of the propagation field is very similar to that measured in our study. 231 This suggests that the parameters in the expression of v are also similar 232 in both cases. To compare quantitatively the potential dependence of 233 v, we rewrite the expression governing v in the creep regime, 234

$$\nu(\mathbf{E}) = \nu_0 \exp\left[-\alpha(\mathbf{E})(H_{prop})^{-1/4}\right],\tag{2}$$

where E is the electric field and $\alpha = (U_C/kT)(H_{dep})^{1/4}$. Following the 235 derivation done in Ref. 13, α is proportional to the MAE, 236

$$\alpha(\mathbf{E}) = \alpha_0 \Big[K_V + \Big(K_S^{Total} + \beta \mathbf{E} \Big) / d \Big], \tag{3}$$

where α_0 contains different micromagnetic parameters of the magnetic 237 layer, K_V is the Co bulk MAE, $K_S^{Total} = K_S^{Co/Pd} + K_S^{Electrolyte/Co}$, 238 with $K_S^{Co/Pd}$ being the Co/Pd interface MAE and $K_S^{Electrolyte/Co}$ the 239 electrolyte/Co interface MAE, *d* is the layer thickness, and *β* is the EFE 240 coefficient, i.e., the variation coefficient per V m⁻¹ of $K_S^{Electrolyte/Co}$. We 241 assumed that $K_S^{Electrolyte/Co}$ is linear with the electric field. It is conve-242 nient to estimate the electric field difference $\Delta E = E_1 - E_2$ necessary 243 to increase ν by one order of magnitude, the smaller the ΔE , the larger 244 the EFE. Using Eqs. (2) and (3), we can write 245

$$\nu(\mathbf{E}_{1})/\nu(\mathbf{E}_{2}) = \exp\left[-\alpha_{0}(\beta/d)(H_{prop})^{-1/4}\Delta\mathbf{E}\right] = 10.$$
 (4)

The fit of the data in Fig. 2(b) does not allow obtaining a consis- 246 tent trend in the potential dependence of the parameters governing v. 247 This is due to the large uncertainty on the fitted slope and offset most 248 probably because of the small range of available magnetic field. It is 249 therefore more accurate to use the potential dependence of v measured 250 at a fixed magnetic field. From the fit of the data in the exponential 251 regime in Fig. 3(a) and considering that the distance between the 252 charged planes responsible for the EFE is \sim 0.14 nm (see Fig. S1),²¹ we 253 obtain $\Delta E \sim 1.5$ V/nm. In the case of AlOx/Co/Pt, the AlOx thickness 254 is 3.8 nm and the data reported in Ref. 13 yield $\Delta E \sim 4 \text{ V/nm}$. 255 Consequently, the EFE is 2.7 times larger in our case as compared to 256 Ref. 13. This is related to one of the parameters inside the exponential 257 term. The cobalt thickness d and $H_{\rm prop}$ are similar in both studies. The 258 parameter α_0 should be also similar for both systems since the mea- 259 sured DW velocity is similar for similar H_{prop} . Consequently, the dif-260 ference should come from the β coefficient. In our case, β equals 261 $34 \text{ fJ V}^{-1} \text{ m}^{-1}$, ¹⁸ whereas it amounts to 14 fJ V⁻¹ m⁻¹ for AlOx/Co/ 262 Pt,¹³ i.e., 2.4 times smaller, a value very close to that obtained above, 263 2.7. This indicates that the higher EFE measured in our case is essen- 264 tially due to the higher value of the parameter β in our work. The dif- 265 ference between the two systems may originate from the orbital shape 266 of the Co atoms which are bonded to the carbon atoms of the CO 267 overlayen in our case and to oxygen in the case of the AlOx/Co inter- 268 face. The comparison with HfO2/MgO/Co/Pt is less straightforward 269 because v at 220 Oe is two orders of magnitude larger than in our 270 case.¹⁷ In addition, the value of β in this study is very large (~150 fJ 271 V^{-1} m⁻¹), well above the range of 10–50 fJ V^{-1} m⁻¹ of β values usu- 272 ally measured for Co and CoFeB layers,³⁰⁻³⁴ which suggests that 273 chemical processes might be involved. 274

In steady-state conditions and in the HER region, i.e., at poten-275 tials between -1 V and -1.3 V, the DW velocity levels off and deviates 276 from the exponential law, suggesting the presence of a second effect on 277

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MAE with the opposite sign compensating the EFE. In the CO satu-278 279 rated electrolyte, this second regime exists only in the presence of the 280 Pd underlayer and is concomitant with significant changes of the sam-281 ple reflectivity (see Fig. S4). Since it was previously observed that Pd 282 reflectivity changes upon H-insertion/release,³⁵ we infer that this sec-283 ond effect on MAE is connected with the insertion and removal of 284 atomic hydrogen in and out of the Pd layer. Magnetic studies of 285 Co/Au(111) layers capped with Pd clearly show that H loading into Pd modifies the Co MAE.³⁵ A similar phenomenon can be expected in 286 287 our case, provided that the H can reach the Pd underlayer, either by 288 diffusion through the Co layer or directly in the Pd layer through pin-289 holes in the Co layer.

The magnetic domains of electrodeposited perpendicularly 290 291 magnetized Co epitaxial layers were imaged by MOKE microscopy, while the sample surface is oxide-free and in contact with an elec-292 293 trolyte. The domain walls propagate in the creep regime, and the 294 velocity varies exponentially with the potential (slope ~2.5 deca-295 des/V). The exponential variation originates from the linear varia-296 tions of the surface anisotropy energy with potential (i.e., charge 297 accumulation at the surface), and a quantitative comparison with 298 literature suggests that the slope scales with the parameter β used to 299 characterize the efficiency of the electric field effect on MAE, which 300 is \sim 2.5 larger at the electrolyte/Co than at the AlOx/Co interface. 301 In the steady state condition and at potential where HER settles in, the DW velocity levels off, probably due to hydrogen incorporating 302 303 in the Pd underlayer.

304

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305 See the supplementary material for complete experimental details 306 and additional magnetic and reflectivity results of Co/Pd/Au/Si(111) and Co/Au/Si(111) samples. 307

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