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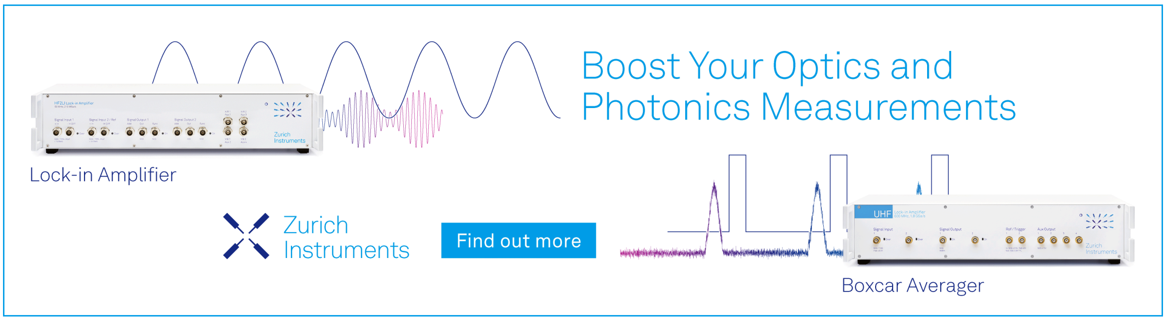

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ABSTRACT

This study investigates the out-of-plane magnetization component of electrodeposited $Ni_{90}Fe_{10}$ thin films grown under different applied magnetic field conditions. The formation of stripe domains is gradual, as there is a thickness range in which the transcritical shape appears in the hysteresis loops, while only magnetic ripples are measured in the magnetic force microscopy images. For instance, samples deposited under the residual magnetic field generated by the switched-off magnetic stirrer exhibit the transcritical shape in the in-plane hysteresis loops at a thickness of 400 nm, even though corresponding magnetic force microscopy images do not reveal the presence of stripe domains. When a perpendicular magnetic field of 100 Oe is applied during growth, stripe domains become visible in microscopy images, along with the transcritical shape in the hysteresis loop at 400 nm. This implies that the critical thickness for stripe formation can be reduced by applying a perpendicular magnetic field during electrodeposition. We have experimentally confirmed that the larger the magnetic field applied in the out-of-plane direction during growth, the smaller the critical thickness. These results underscore the importance of controlling the external magnetic field during electrodeposition for more effective tuning of the magnetic textures in electrodeposited $Ni_{90}Fe_{10}$ films.

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I. INTRODUCTION

Spin textures are gaining increasing interest in the field of spintronics due to their reconfigurability and stability.[1–3] The periodic modulation of a ferromagnet can be achieved through artificial arrangements of dots and stripes, or ion implantation.[4] In materials with moderate perpendicular magnetic anisotropy (PMA), the periodic magnetic configuration known as stripe domains appear due to competing interactions, arising from the alternating up and down out-of-plane (OOP) orientation of the magnetization.[5,6] These stripe domains can be utilized, for example, to generate spin waves.[7–10] In ferromagnets with moderate or low PMA, stripe domains appear above a critical thickness (t_{cr}),

$$t_{cr} = 2\pi\sqrt{A_{ex}/K_{OOP}}, \tag{1}$$

with A_{ex} being the exchange energy per unit length and K_{OOP} the OOP magnetic anisotropy energy constant. When PMA is not sufficiently large to induce the formation of stripe domains, a magnetic contrast known as ripple, characterized by corrugation, becomes observable.[11–15] The origin of magnetic ripple lies in the inhomogeneous magnetization caused by magnetic anisotropy fluctuations. Therefore, reducing these fluctuations through the enhancement of the PMA will promote the formation of stripe domains.

Ni–Fe alloys have been extensively studied due to their low coercivity and large saturation magnetization (M_{sat}).[16] $Ni_{90}Fe_{10}$ is particularly interesting because of its magnetoelastic properties,[17–19] exhibiting a magnetostriction constant (λ) of approximately −20 ppm. Evaporated $Ni_{90}Fe_{10}$ thin layers deposited on Cu can show a significant OOP magnetic anisotropy contribution due to mechanical strain.[18] In our investigation of electrodeposited $Ni_{90}Fe_{10}$ thin films, we have successfully induced the formation of stripe domains. However, this phenomenon occurs for thicknesses considerably larger (800 nm) than the theoretically inferred t_{cr} (105 nm) based on expression (1), and only when the electrolyte is not magnetically stirred during growth.[17] Typically, in-plane (IP) hysteresis loops exhibit the characteristic shape denoted as "transcritical" when stripe domains are present.[5,20] However, our observations show that electrodeposited $Ni_{90}Fe_{10}$ layers display the transcritical shape for smaller thicknesses, where stripe domains are visible in magnetic force microscopy (MFM) images.[17] One of the aims of this study is to gain deeper insights into this thickness range where mixed features are observed in the experimental measurements. Such insights can provide valuable hints about the process of stripe domain formation.

Here, we have systematically investigated the evolution of the out-of-plane magnetic textures in electrodeposited $Ni_{90}Fe_{10}$ layers with thickness ranging from 250 to 800 nm, where the formation of stripe domains has not been observed previously, but rather a magnetic ripple. This study focusses on examining the impact of various applied magnetic fields during the growth process on the magnetic behavior of these layers to gain a better understanding of stripe domain formation. To achieve this, we have not magnetically stirred during growth and compared layers deposited under three conditions: (i) zero applied field, (ii) the residual magnetic field generated by a switched-off magnetic stirrer, and (iii) a magnetic field of 100 Oe perpendicular to the sample plane (see Fig. 1). These represent completely new growth conditions in comparison to our previous work.[17] Our experimental findings reveal that the higher the perpendicular magnetic field applied during growth, the lower the critical thickness for the formation of stripe domains. Due to the gradual evolution, the observed features in hysteresis loops can vary depending on growth conditions and thickness; transcritical shape may be observed in hysteresis loops, while magnetic ripple appears in MFM images.

II. EXPERIMENTAL SECTION

$Ni_{90}Fe_{10}$ layers were grown by electrodeposition on glass substrates covered with a gold layer to increase the electrical conductivity. Samples were deposited at room temperature and not stirred during growth. We have used a three-electrode cell with a platinum mesh as counter electrode and an Ag/AgCl (3M NaCl) reference electrode. A PalmSens EmStat3 + Blue potentiostat was used to perform the depositions at room temperature in non-rotating substrates in the horizontal position at different growth conditions: (i) at zero applied magnetic field, (ii) under the residual magnetic field (H_{res}) that has an IP component of 30 Oe and OOP of 24 Oe, respectively, and (iii) under an applied external magnetic field of 100 Oe perpendicular to the sample plane (H_{per}) produced by a Helmholtz coil (Fig. 1),

The growth time of NiFe was adjusted to reach the expected thicknesses (t) by means of the Faraday's law:

$$C = \frac{nFd}{M_{atom}} St, \quad (2)$$

where C is the electric charge measured in the cathode, n is the number of electrons involved in the reduction reaction, F is Faraday's constant (96 485.34 C/mol), d is the density of the electrodeposited material, S is the area of the sample, and M_{atom} is the molecular mass. First, a set of calibration samples was deposited in which the layer thickness was measured by atomic force microscopy (AFM). These samples were used to adjust the formula of Faraday's law to take into account hydrogen evolution.

The electrolyte for $Ni_{90}Fe_{10}$ was only slightly modified taken into account previous literature.[21,22] It is water-based with a fixed pH of 2.2, and a composition of 0.4M H_3BO_3, 0.017M saccharine, 0.7M $NiSO_4$, and 0.02M $FeSO_4$. It was applied a growth potential of −1.2 V in all cases.

We have used x-ray diffractometry (XRD) in the Bragg–Brentano configuration to study the structural properties. Measurements were performed in a D8 Brucker equipment using the Cu K_{α} wavelength

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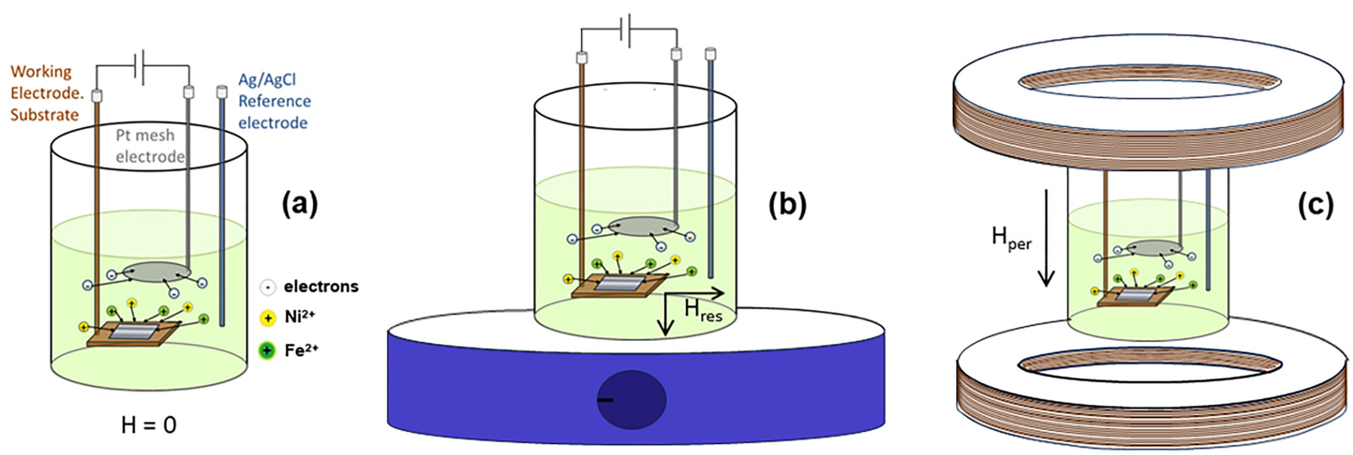

FIG. 1. Scheme of the different growth conditions studied: (a) no external applied magnetic field, (b) residual magnetic field generated by the stirrer when it is switched off, and (c) under an applied field of 100 Oe.

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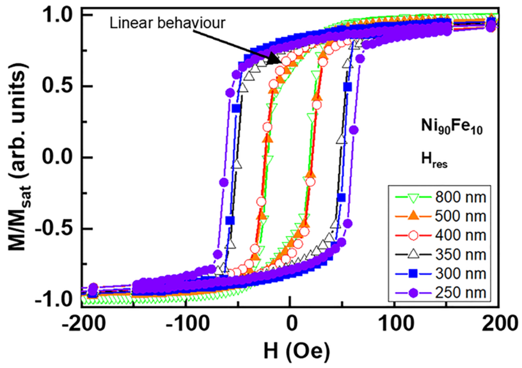

FIG. 2. IP hysteresis loops for $Ni_{90}Fe_{10}$ layers with different thicknesses deposited under the magnetic field generated by the magnetic stirrer when it is switched off (H_{res}) (●) 250 nm, (■) 300 nm, (Δ) 350 nm, (○) 400 nm, (▲) 500 nm, and (∇) 800 nm.

(1.540 56 Å). IP and OOP hysteresis loops were performed in a vibrating sample magnetometer (VSM) at room temperature. AFM and MFM measurements were performed under standard conditions using a customized and modified system from Nanotec Electrónica S.L. The data analysis process was carried out using WSxM software.[23] The amplitude modulation (AM) method was employed keeping the cantilever oscillation amplitude at about 20 nm for MFM imaging. The resulting magnetic signal was recorded in the phase shift channel acquired at a typical distance of 60 nm. Commercial Budget Sensors MagneticMulti75 probes with CoCr coating were used for the measurements. The magnetic signal of the probes was always checked before and after each experiment. For the roughness study, highly doped silicon probes with a typical tip radius of curvature of less than 10 nm from Nanosensors (PPP-FMR) have been used.

III. RESULTS AND DISCUSSION

First, we measured the roughness of the studied samples using AFM. Regardless of the growth conditions, a root mean square of approximately 7 nm was inferred. In Fig. 2, we present the IP hysteresis loops for layers with thicknesses ranging from 250 to 800 nm deposited under H_{res} generated by the magnetic stirrer when it is switched off. It is important to note that the magnetic bar is not placed inside the electrolyte cell for this growth condition [Fig. 1(b)]. Although none of the studied layers were magnetically stirred during growth, it is important to investigate this growth condition because switching off the magnetic stirrer does not imply that the layers are deposited under a zero magnetic field. This is due to the existence of a residual magnetic field when the stirrer is switched off.

The first noteworthy point is the decrease in the coercive field (H_C) from 61 Oe for a thickness of 250–23 Oe when the thickness is equal to or above 400 nm (Fig. 2). This reduction in H_C is accompanied by the emergence of a somewhat linear behavior in the hysteresis loop, which can be associated with the onset of the transcritical shape in the IP hysteresis loop for a thickness of 400 nm. From XRD diffraction patterns (Fig. 3), we determined the grain size (D) using Scherrer's formula. The observed changes in both H_C and hysteresis loop shape cannot be attributed to variations in the grain size, as D remains between 20.0 and 22.6 nm in all these samples. While the transcritical shape is somewhat present in the hysteresis loop for a thickness of 400 nm but not for 300 nm (Fig. 2), MFM images reveal only an enhancement of the magnetic ripple when comparing 300–400 nm, with no evidence of magnetic stripes (Fig. 4). According to the Stoner–Wohlfarth model, hard magnetic axes are known to exhibit a smaller coercivity compared to easy ones.[16,24] Therefore, the decrease in H_C together with the presence of a linear behavior in the IP hysteresis loop can be associated with an enhancement of the PMA for a thickness of 400 nm. However, the PMA for this thickness (400 nm) is not significant

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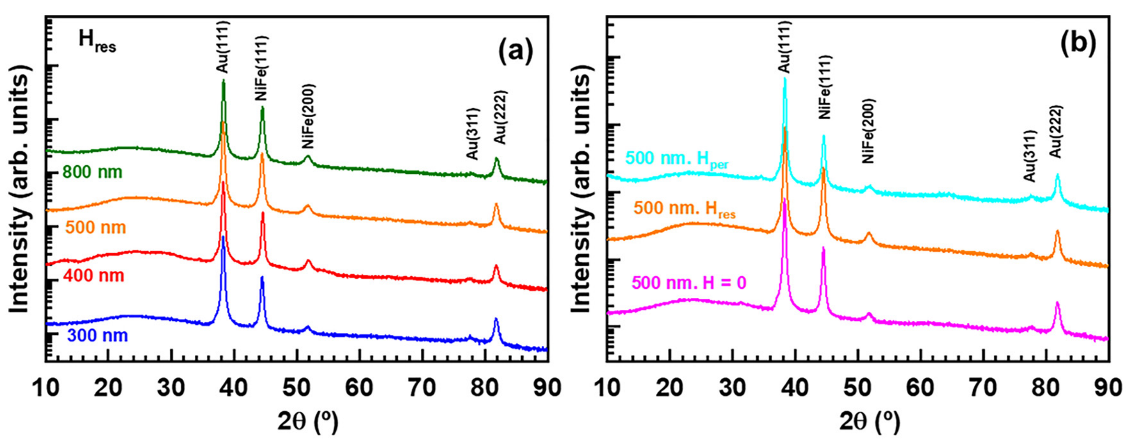

FIG. 3. (a) XRD diffraction patterns for $Ni_{90}Fe_{10}$ layers deposited under the magnetic field generated by the magnetic stirrer when it is switched off (H_{res}) with different thicknesses ranging from 300 to 800 nm. (b) XRD diffraction patterns for 500 nm thick $Ni_{90}Fe_{10}$ layers deposited under the different magnetic field conditions studied in this work: H = 0 Oe, H_{res}, and H_{per}. Curves in (a) and (b) have been shifted for clarity.

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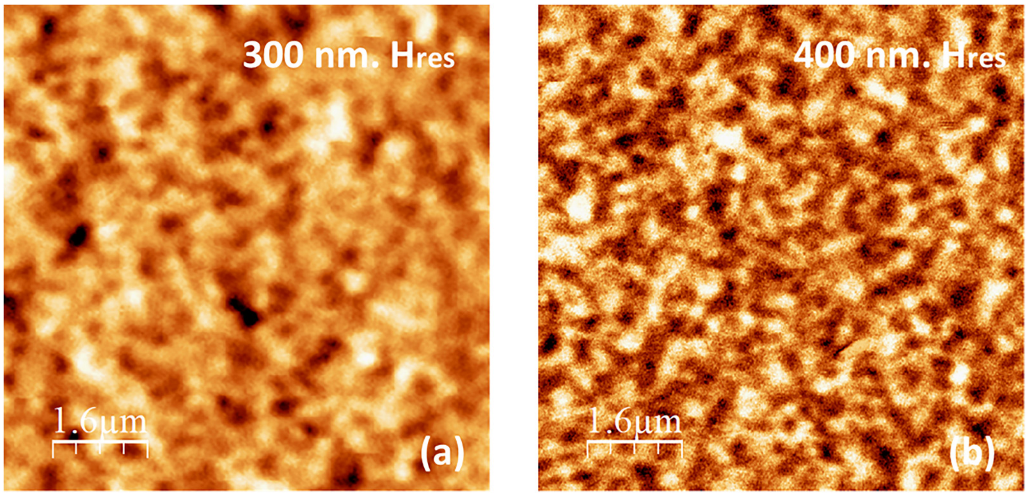

FIG. 4. MFM images for $Ni_{90}Fe_{10}$ samples deposited under the magnetic field generated by the magnetic stirrer when it is switched off (H_{res}) with a thickness of (a) 300 nm and (b) 400 nm. Images size: $5 \times 5\,\mu m^2$.

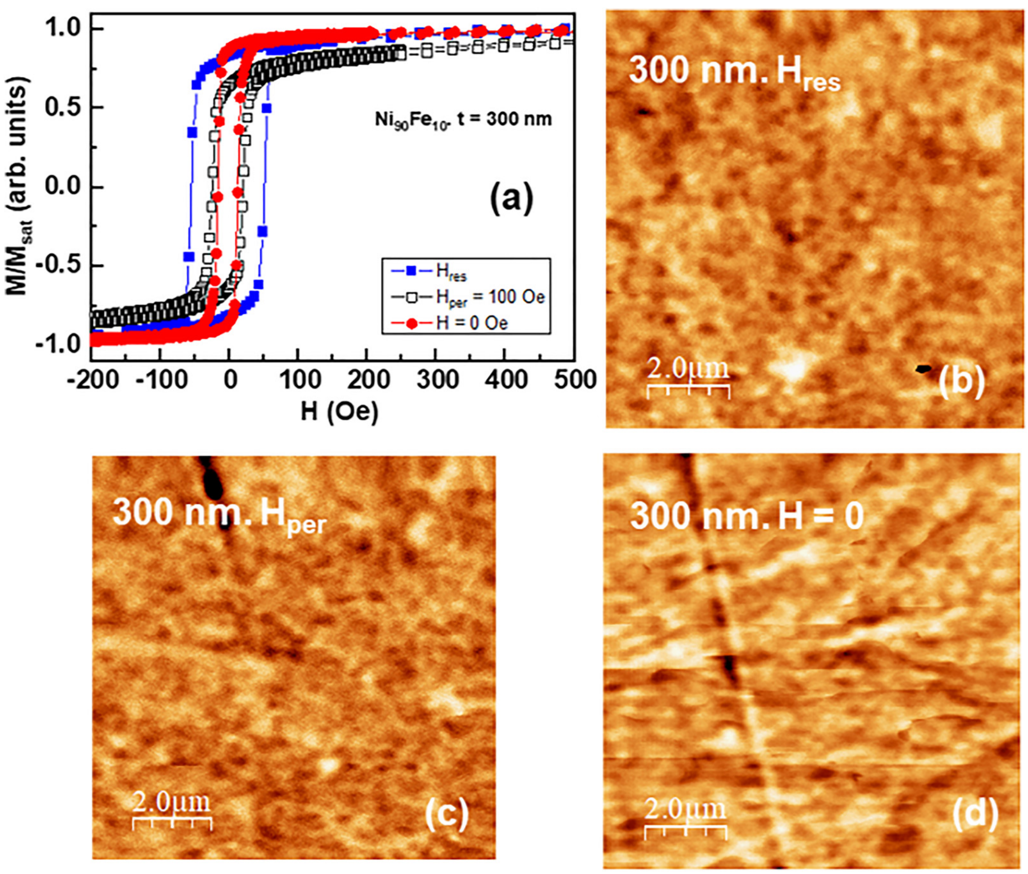

FIG. 5. (a) IP hysteresis loops for $Ni_{90}Fe_{10}$ layers with a thickness of 300 nm deposited under different growth conditions: (■) H_{res}, (□) H_{per} = 100 Oe, and (●) zero field. MFM images for samples with 300 nm deposited under different growth conditions: (b) H_{res}, (c) H_{per} = 100 Oe, and (d) zero field. Images size: $10 \times 10\,\mu m^2$.

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TABLE I. Summary of the OOP magnetic anisotropy constant energy for layers deposited under different growth conditions in terms of applied magnetic field: H_{res} refers to the residual magnetic field generated by the magnetic stirrer when it is switched off, and H_{per} is the perpendicular to the sample plane of 100 Oe generated by the Helmholtz coil.

	H_{res}	H_{per} =100 Oe	$H = 0$ Oe
Thickness (nm)	K_{OOP} (kerg/cm^3)		
300	17	27	…
400	18	28	…
500	18	28	…

enough to promote the formation of stripe domains. Instead, an increase in the magnetic ripple is observed in the MFM image compared to 300 nm (Fig. 4), despite the transcritical shape being somewhat present in the IP hysteresis loop for 400 nm (Fig. 2).

In our investigation into the potential effect of magnetic fields during growth, we conducted electrodeposition of layers both under an applied perpendicular magnetic field of 100 Oe and without any magnetic field. The XRD diffraction patterns show no apparent differences regardless of the magnetic field condition during growth [Fig. 3(b)]. Lattice parameter and grain size are neither significantly modified. As an example, a comparison is presented among 500 nm thick layers deposited under zero field, H_{res}, and H_{per}.

In the hysteresis loops we observed that when the layer is deposited under H_{per}, there is a notable decrease in coercivity from 53 to 22 Oe for a smaller thickness of 300 nm, compared to the 400 nm thickness required to observe the same behavior when depositing under the residual field H_{res} [as depicted in Fig. 5(a)]. In contrast, layers deposited under zero magnetic field exhibit an IP hysteresis loop characteristic of a soft magnetic thin film. This observation further supports the notion that the perpendicular magnetic field applied during growth enhances the PMA. This conclusion is supported by the calculation of K_{OOP} (refer to Table I), which was quantitatively inferred from both IP and OOP hysteresis loops, following the same procedure as outlined in prior studies.[17,25] Specifically, for the 300 nm thickness, K_{OOP} is 17 kerg/cm^3 for the layer deposited under H_{res}, while it increases to 27 kerg/cm^3 for the layer deposited under H_{per} (as shown in Table I).

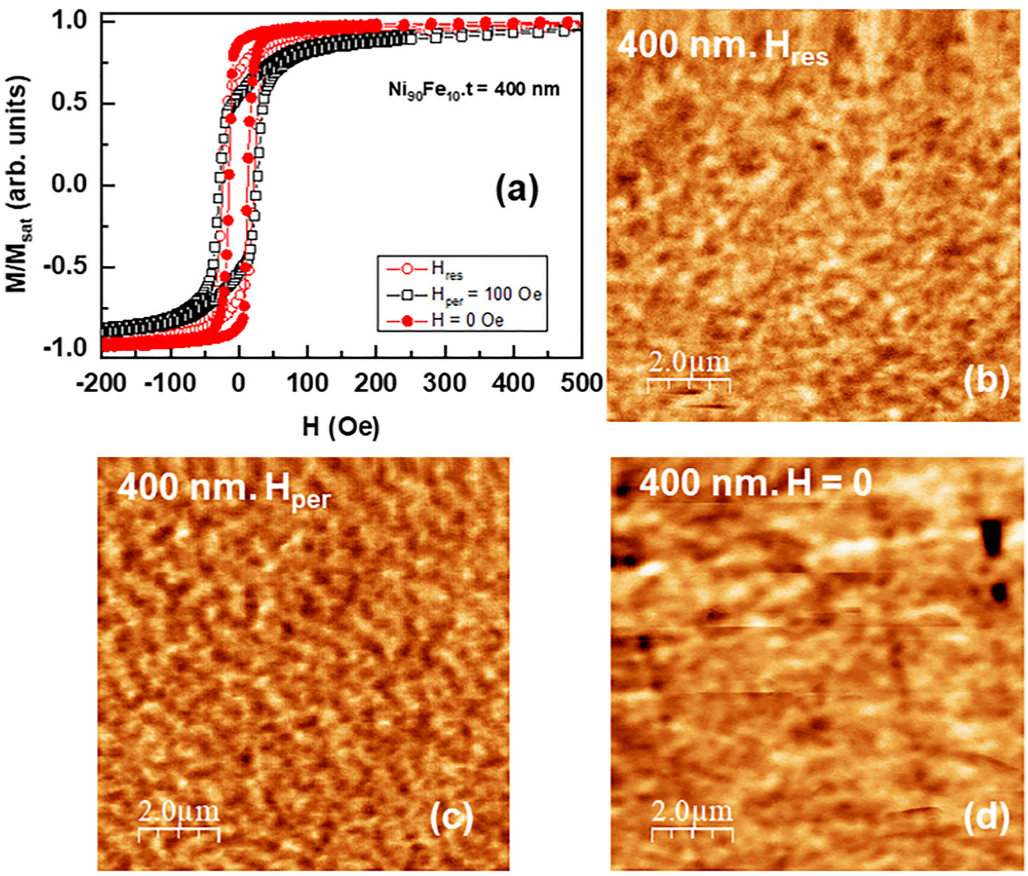

FIG. 6. (a) IP hysteresis loops for $Ni_{90}Fe_{10}$ layers with a thickness of 400 nm deposited under different growth conditions: (○) H_{res}, (□) H_{per} = 100 Oe, and (●) zero field. MFM images for samples with 400 nm deposited under different growth conditions: (b) H_{res}, (c) H_{per} = 100 Oe, and (d) zero field. Images size: 10 × 10 μm^2.

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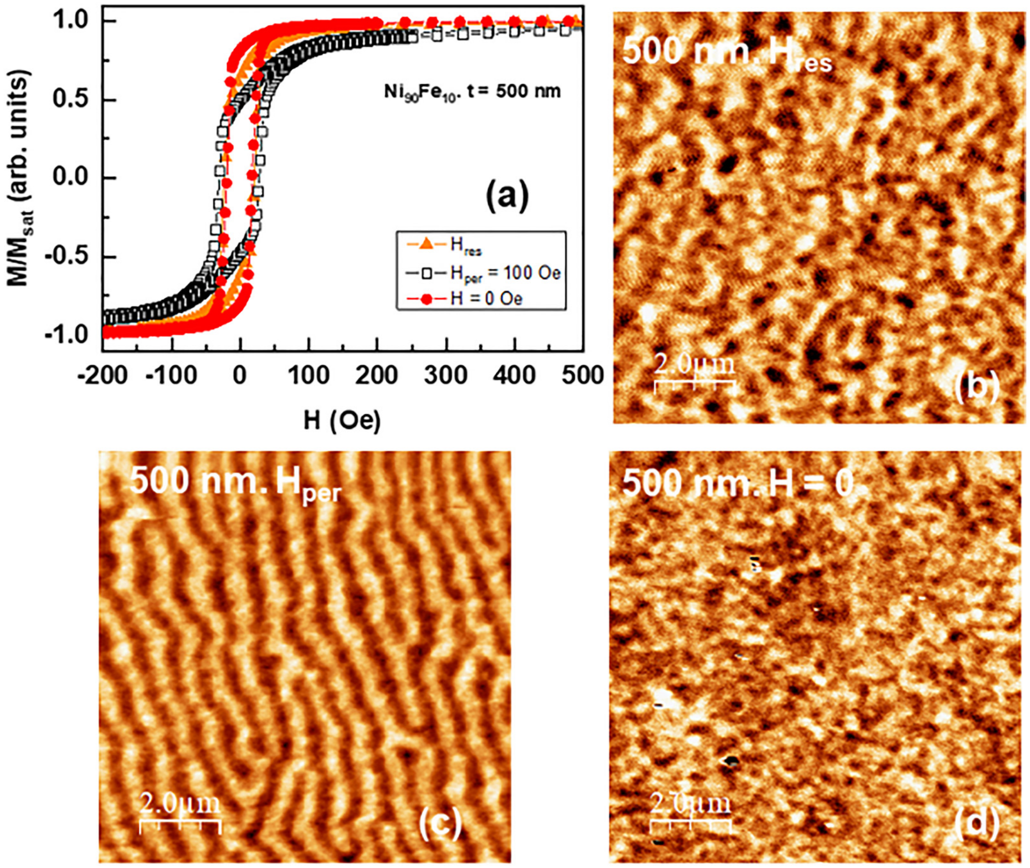

FIG. 7. (a) IP hysteresis loops for $Ni_{90}Fe_{10}$ layers with a thickness of 500 nm deposited under different growth conditions: (▲) H_{res}, (□) H_{per} = 100 Oe, and (●) zero field. MFM images for samples with 500 nm deposited under different growth conditions: (b) H_{res}, (c) H_{per} = 100 Oe, and (d) zero field. Images size: 10 × 10 μm^2.

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Notably, it is not feasible to calculate K_{OOP} when no magnetic field is applied during growth. Furthermore, all 300 nm-thick layers exhibit a similar MFM magnetic contrast, suggesting that the PMA, even in the sample deposited under H_{per}, is not significant enough to promote stripe domains [as depicted in Figs. 5(b)–5(d)].

If we compare the hysteresis loops of the layers deposited under H_{per} with thicknesses of 300 and 400 nm [Figs. 5(a) and 6(a)], a more distinct transcritical shape is observed in the layer of 400 nm. For both H_{per} and H_{res} conditions, K_{OOP} only slightly increases when the thickness is increased from 300 to 400 nm (Table I). Nevertheless, there is a much greater enhancement of K_{OOP} when evaluating samples with the same thickness but deposited under H_{per} compared to H_{res} (Table I). Therefore, applying a perpendicular magnetic field during growth is more efficient than increasing the thickness to enhance the PMA. Layers deposited under zero field with 300 and 400 nm do not exhibit any evolution [Figs. 5(a) and 6(a)]; coercivity remains at 14 Oe, and the shape of the hysteresis remains consistent. When comparing MFM images for 400 nm layers [Figs. 6(b)–6(d)], stripe domains can be observed in the layer deposited under H_{per}, whereas the layer deposited under H_{res} only exhibits a magnetic ripple that is somewhat more intense than in the layer deposited under zero field.

The impact of the applied magnetic field in $Ni_{90}Fe_{10}$ layers is further confirmed for a thickness of 500 nm (Fig. 7). The comparison between IP and OOP hysteresis loops is shown in Fig. 8. When the transcritical shape is present in the IP loops, the remanence in the OOP magnetization curves is almost zero due to the presence of stripe domains, in agreement with previously reported works.[5,26] The layer deposited under H_{per} clearly displays both the transcritical shape in the hysteresis loop and the presence of stripe domains in the MFM image. In contrast, the sample deposited under H_{res} exhibits a hysteresis loop quite similar to that of 400 nm, although the magnetic ripple is more pronounced in the MFM image. The layer deposited under zero fields continues to exhibit a magnetic ripple in the MFM, and the IP hysteresis loop still lacks features associated with the transcritical shape.

The formation of stripe domains is attributed to the presence of significant PMA, which can counterbalance demagnetizing energy.[5,6,25] While the transcritical shape in IP hysteresis loops is typically considered an indication of stripe domain formation, we have

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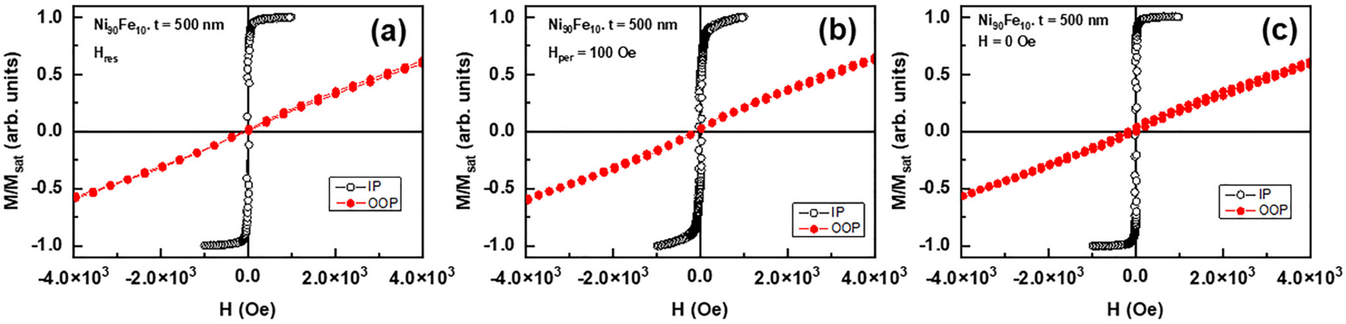

FIG. 8. IP (○) and OOP (●) hysteresis loops for $Ni_{90}Fe_{10}$ layers with a thickness of 500 nm deposited under (a) the residual field generated by the stirrer when it is switched off, (b) a perpendicular field of 100 Oe, and (c) zero field.

experimentally observed a progressive evolution of hysteresis loops shape toward the transcritical shape without clear evidence of stripes in the MFM images. In the study of Cao and co-workers on electrodeposited $Ni_{50}Fe_{50}$ layers,[27] a thickness range was identified where it was challenging to infer the presence of stripe domains from hysteresis loops alone. Similar observations were reported for FePt, where the transition is gradual, and layers close to the critical thickness can show mixed features.[25] These findings align with our results, as layers can exhibit the transcritical shape in IP hysteresis loops without clear evidence of stripe domains in MFM. Moreover, in this transition regime, the magnetic contrast is characterized by a ripple that is due to magnetic fluctuations, as also observed in FeGa films.[14,17]

Typically, the quality of factor (Q) is used to quantitatively classify materials with PMA, defined as[6]

$$Q = K_{OOP}/2\pi M_{sat}^2, \tag{3}$$

where M_{sat} is the saturation magnetization. $Q < 1$ indicates materials with moderate or low PMA. With $M_{sat} = 590\ \text{emu/cm}^3$ for $Ni_{90}Fe_{10}$,[17] Q ranges from 0.008 for the layers deposited under the residual field to 0.012–0.013 for those deposited under H_{per}. These quality factors are smaller compared to systems with higher PMA, where stripe domains are visible in the MFM images and Q values are around 0.3. For example, Fe–N reports values of 0.28–0.38,[5] and the heterostructure Cr_2O_3/FeGa obtains 0.3.[28] However, Q values for $Ni_{90}Fe_{10}$ are comparable to works on other soft magnetic alloys where stripes are present, with $Q \approx 0.02 - \approx 0.005$, even lower than in our study.[29]

The critical thickness for the formation of stripe domains in $Ni_{50}Fe_{50}$ layers[27] closely aligns with previously published data, where columnar growth also appears to play a pivotal role in enhancing PMA.[30] However, various parameters such as substrate temperature or deposition pressure can alter t_{cr} in sputtered layers,[25,31] underscoring the sensitivity of the PMA to growth conditions. In our electrodeposited $Ni_{90}Fe_{10}$ layers, we have observed that t_{cr} for the formation of stripe domains depends on the applied magnetic field during growth. Higher perpendicular field applied during electrodeposition leads to a larger PMA, enabling to obtain stripe domains at a smaller thickness than previously reported.[17] Indeed, applying a perpendicular magnetic field during growth proves to be more effective than increasing the thickness to enhance the PMA.

IV. CONCLUSIONS

The combined use of magnetic magnetometry and MFM characterization has provided valuable insights into the formation of stripe domains in electrodeposited $Ni_{90}Fe_{10}$ thin films. When no magnetic field is applied during growth, neither stripe domains nor the transcritical shape are observed, even for a thickness exceeding 500 nm. However, applying a perpendicular magnetic field of 100 Oe during growth results in the presence of stripes for a thickness of 400 nm. Layers deposited under a magnetic field with IP and OOP components (H_{res}) exhibit a mixed behavior. The transcritical shape is observed in the hysteresis loop for a thickness of 400 nm, but MFM only detects magnetic ripple, even a thickness of 500 nm. In summary, our experimental findings on $Ni_{90}Fe_{10}$ electrodeposited layers demonstrate that the enhancement of the OOP magnetic texture is more efficiently promoted by an external perpendicular magnetic field applied during growth than by the increase in thickness. Therefore, this work opens new routes to tailor magnetic textures in $Ni_{90}Fe_{10}$ films.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

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Author Contributions

N. Cotón: Data curation (equal); Investigation (lead); Methodology (equal); Writing – review & editing (equal). **J. P. Andrés:** Funding acquisition (equal); Investigation (equal); Methodology (equal); Writing – review & editing (equal). **M. Jaafar:** Funding acquisition (equal); Investigation (equal); Methodology (equal); Writing – review & editing (equal). **A. Begué:** Investigation (equal); Methodology (equal); Writing – review & editing (equal). **R. Ranchal:** Conceptualization (lead); Funding acquisition (equal); Supervision (lead); Writing – original draft (lead); Writing – review & editing (equal).

DATA AVAILABILITY

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

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