Magnetic force microscopy study of dense stripe domains in Fe-B/Co-Si-B multilayers and the evolution under an external applied field

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The effect of the permanent compression acting on multilayered Fe-B/Co-Si-B thin films has been studied by using magnetic force microscopy (MFM). Since both materials exhibit positive and negative saturation magnetostriction, respectively, the magnetic-moment distribution and thus the domain structure of these multilayers are strongly dependent on the thickness of each layer. Different MFM contrast levels and domain sizes have been measured in stressed and unstressed multilayers with various Fe-B and Co-Si-B layer thickness. Both kinds of samples, stressed and unstressed thin films, present a weak perpendicular anisotropy. For identical composition of the multilayers, an increase of the magnetization component perpendicular to the sample plane has been observed in stressed samples compared to their unstressed counterparts. In addition, the effect of the stress on the magnetization process has been studied by analyzing the evolution of the domain structure with an externally applied magnetic field in MFM imaging.

INTRODUCTION

The study of the magnetic domain structure allows us to extract direct information about magnetization and anisotropy distributions as well as further details about the magnetization processes. In thin-films research, it is especially important to determine the domain structure on the sample surface. In this kind of samples, the orientation of the magnetization mainly results from the competition among magnetocrystalline, shape, and induced anisotropies. One case of particular interest is when a sample exhibits weak perpendicular anisotropy. The out of the plane anisotropy is then not strong enough to overcome the shape anisotropy and consequently the magnetization lies mainly within the plane of the film. However, a perpendicular component of the magnetization exists and oscillates periodically with a consequent reduction of the magnetostatic energy of the sample. This type of domain structure has been called dense stripe domains.[1,2] Initially, the dense stripe domains could only be visualized by Lorentz microscopy. Nowadays, magnetic force microscope (MFM)[3,4] has become a useful and convenient alternative for visualizing these kinds of patterns. Furthermore, the magnetization process of these samples can also be studied by imaging the evolution of their domain structure under an applied field.

The aim of this work has been to investigate multilayered thin films exhibiting an induced anisotropy that gives rise to an easy magnetization axis oriented out of the plane. Although a number of routes for inducing anisotropies in thin films is described in the literature,[5,6] a method has been recently developed, the so-called bowed substrate sputtering technique.[7,8] This method consists basically of growing the films onto arched substrates. A permanent mechanical compression is applied on the thin film when the bowed substrate recovers its planar shape. The sign of the magnetostriction constant of the particular magnetic material deposited onto these substrates determines its induced easy magnetization direction. Whenever the thin film is composed of alternate layers of opposite magnetostriction materials, the magnetoelastic anisotropy develops easy magnetization axes perpendicular to each other from layer to layer. The magnetic moment configuration of this kind of multilayered films is then determined by the relative strengths of the induced anisotropies and by the exchange interaction between the different layers.[8]

In this work, MFM imaging has been performed to disclose the existence of domains in stressed and unstressed multilayers composed of positive magnetostriction Fe-B and negative magnetostriction Co-Si-B alloys exhibiting certain out of the plane anisotropy. Their well-defined domain structures and their evolution with an externally applied field have been analyzed. Notice the importance of the MFM not only because of its capability for imaging the domain structure, but also for its usefulness as a powerful tool to understand the magnetization process in the submicrometer scale.

EXPERIMENT

The films were deposited using a magnetron rf-sputtering system with a chamber base pressure better than 10^{-6} mbars. During the deposition, a flow of argon was introduced and the pressure was kept at 5×10^{-3} mbars. The deposition power was 300 W and the target-to-sample distance was 100 mm. The nominal compositions of the targets were $Fe_{80}B_{20}$ and $Co_{75}Si_{15}B_{10}$. Deeper details about samples preparation are given elsewhere.[8] Multilayered films were grown simultaneously onto planar and bowed glass slides in order to establish the effect of the stress on the sample properties. After removing the arched glass from the sputtering chamber, the substrate recovered its initial planar shape producing a permanent compressive stress on the magnetic films. The samples analyzed were selected by cutting a rectangle of 4×22 mm from the central region of the substrate. In this way, a similar compressive strain of around 3.5×10^{-4} along its short axis is expected for all the stressed samples. In the following, the shortest edge of the sample will be ascribed to transverse direction, and the longest one to longitudinal direction. These directions correspond to the induced easy magnetization axis of the Co-Si-B and Fe-B layers, respectively. Notice that Co-Si-B presents a negative magnetostriction constant (about -3×10^{-6}) while Fe-B exhibits positive magnetostriction (about $+3\times10^{-5}$).

In the multilayers series under study ($\text{Fe-B}_{25\,\text{nm}}/\text{Co-Si-B}_{x})n$, n stands for the number of Fe-B and Co-Si-B layers. The Fe-B layer thickness was kept constant at 25 nm and the Co-Si-B layer thickness (x) was varied between 25 and 100 nm. Samples with $x=25$, 50, and 100 nm Co-Si-B layer thickness were prepared. The number of layers n was chosen in each case in order to keep the total thickness at 1000 nm. The samples will be named using their Co-Si-B layer thickness and an ''*s*'' will label those films prepared by the bowed substrate sputtering technique.

In addition to the multilayers, a Fe-B single layer was grown onto a planar substrate with the same thickness (25 nm) as the Fe-B layers in the multilayer series. This sample has been prepared in order to study the roughness of the Fe-B single layer.

High angle x-ray-diffraction patterns reveal the amorphous structure of the samples. The roughness of the surface was determined by atomic force microscopy (AFM). In-plane hysteresis loops (along longitudinal and transverse directions) were measured at room temperature by using a vibrating sample magnetometer (VSM). The domain structure was studied using a MFM equipment from Nanotec™. Such a system, working in a noncontact mode, allows us to acquire simultaneously the topography of the surface and the magnetic force gradient map, which is proportional to the frequency shift of the vibrating cantilever. The NT-MDT™ tips coated with a 100 nm Co-Cr film were magnetized along the tip axis. The force constant and the resonant frequency of the cantilevers are 6 N/m and 180 kHz, respectively. We have implemented Helmholtz coils to the commercial MFM system in order to allow the imaging under variable applied magnetic fields. The average strength of the external field was calibrated against the input current by using a Hall probe. A maximum applied field of 7.2 kA/m can be reached with the described system.

RESULTS AND DISCUSSION

VSM measurements

Figure 1 shows the longitudinal and transverse hysteresis loops of some samples grown onto bowed (right column) and planar substrates (left column). The series of unstressed films exhibits an increase of the coercivity and the in-plane anisotropy field with the Co-Si-B layer thickness. It is worth mentioning that the in-plane anisotropy fields of the longitudinal and transverse hysteresis loops are rather identical. A similar trend has been found in the films deposited onto the bowed glass slides, although in this case the increment of the anisotropy field with the Co-Si-B layer thickness is comparatively larger. Notice the shift of the longitudinal remanence with respect to the transverse one in the stressed samples due to the effect of the compressive stress in the Fe-B layers. This enhancement of the longitudinal remanence is expected since the magnetostriction of the Fe-B alloy is around ten times

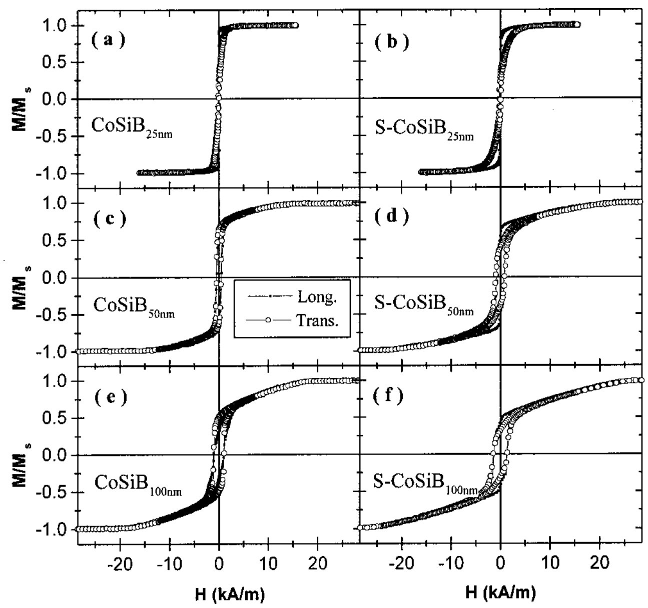

FIG. 1. Hysteresis loops measured by VSM corresponding to the stressed (labeled with *s*-) and unstressed multilayer samples.

higher than that of the Co-Si-B. In both series (deposited onto bowed and planar substrates), the samples with a Co-Si-B layer thickness above 25 nm present a rotational-like magnetization process for large longitudinal and transverse magnetic fields. Such a behavior is interpreted as arising from the presence of an out of the plane easy magnetization direction.[9] This perpendicular component, which appears in both stressed and unstressed samples, could be induced by the columnar growth of the films. This statement is supported by the AFM topographic image shown in Fig. 2 which was obtained in noncontact mode. This topography corresponds to a Fe-B single layer 25 nm thick deposited onto an unbowed substrate. The topography of the sample reveals that the Fe-B grows developing grains rather than continuously. The rms roughness of the sample deduced from the AFM image is 7 Å.

The increase of the in-plane anisotropy field with the Co-Si-B layer thickness indicates the reinforcement of the perpendicular anisotropy when the Co-Si-B layers become thicker. This evolution with the Co-Si-B thickness can be explained as follows: the ratio between the length and the diameter of the columnar structures in the films is larger for samples with less number of layers, i.e., for thicker Co-Si-B layer. The change of composition during the fabrication of the samples implies a discontinuity in the columnar growth and hence a decrease of their length-diameter ratio. Similar behavior has been observed in different multilayerlike structures obtained by electrodeposition.[10]

MFM imaging at the remanent state

MFM imaging confirms the existence of a perpendicular magnetic component on the samples with thicker Co-Si-B layers. In the stressed and unstressed 25-nm-thick Co-Si-B layer samples no magnetic contrast has been detected. However, when the Co-Si-B layer thickness is higher than 25 nm, the domain structure obtained in the remanence state consists of parallel dense stripe domains. The MFM images in Fig.

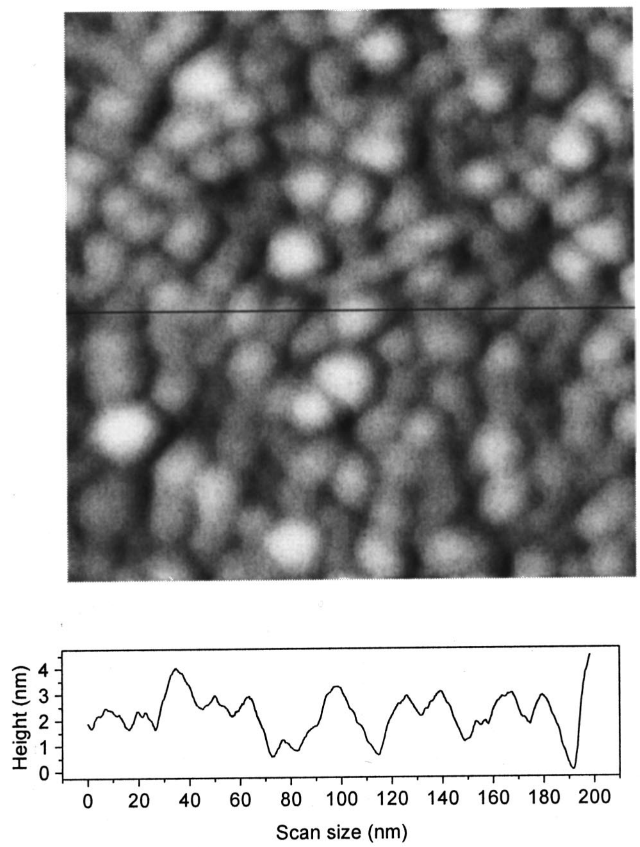

FIG. 2. AFM image of a 25-nm-thick film of Fe-B grown onto a planar glass. Image size: 200×200 nm. Height difference (gray scale): 8 nm.

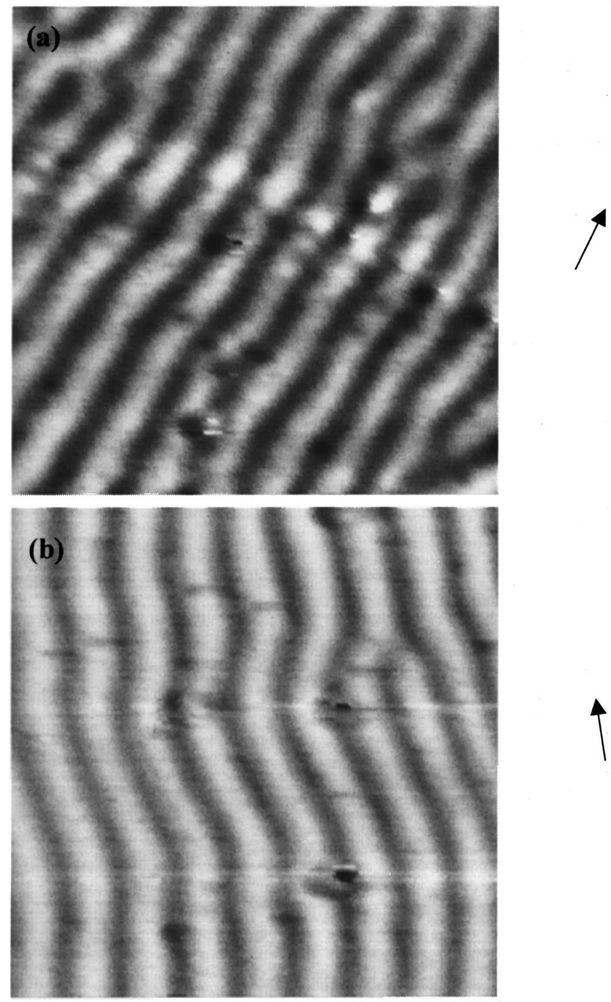

FIG. 3. Remanence MFM images of the samples (a) Co-Si-B_{100} grown onto planar and (b) s-Co-Si-B_{100} grown onto bowed substrates. Image sizes: 10.8×10.8 μm. The contrast of the image is (a) 0.35 mN/m and (b) 0.42 mN/m. The orientation of the arrows corresponds to the transverse direction.

3(a) and (b) correspond to the Co-Si-$B_{100\,\mathrm{nm}}$ unstressed and stressed samples, respectively. In both cases, the stripes are parallel to the transverse direction of the samples which coincide with the previous saturation direction. The alignment of the wall magnetization with the applied field is expected in order to reduce the Zeeman energy[11] giving rise to a parallel stripe domain structure. The observed pattern corresponds to domains with perpendicular magnetization in the middle of the stripe whereas the magnetic moments within the wall lie in the plane contributing to the in-plane remanent magnetization[12] (see the hysteresis loops performed in longitudinal and transverse directions in Fig. 1).

The dense stripe domains are expected for thin films with a perpendicular anisotropy not strong enough to overcome the shape anisotropy.[9] In such a case, beyond a critical thickness, the perpendicular component of the magnetization is oriented periodically up and down out of the surface[9] as schematically shown in Figs. 4(a) and (b). This periodicity must be comparable to the thickness of the film.[13] In the very weak perpendicular anisotropy case, the oscillation closes the magnetic flux whereas for higher anisotropy the oscillation is one-dimensional [see Figs. 4(b) and (a)]. Thus an increment of the magnetic contrast measured by MFM is expected for the samples with higher perpendicular anisotropy since the pole density increases.

The MFM observations are in agreement with the previous predictions since the average period of the stripe domains in Figs. 4(a) and (b) 800 nm is comparable to the sample thickness and the MFM image contrast increases for the samples with thicker Co-Si-B layer (see Table I).

The analyses of the MFM inspections performed in the remanent state are summarized in Table I: the domain width decreases and the image contrast increases for the samples with thicker Co-Si-B layer. The anisotropy field values H_k, deduced from the in-plane hysteresis loops, are also collected. The H_k values as well as the MFM contrast show the increment of the out of the plane magnetization component in the stressed samples regarding to the unstressed ones. This behavior arises from the easy magnetization plane created in the Fe-B layers by the compression, which includes the normal to the surface direction. Hence the domain pattern of stressed samples could fit better to the scheme showed in Fig. 4(a), where the oscillation of the magnetic moments is one dimensional rather than to the arrangement shown in Fig. 4(b) where the magnetic flux closes by the turning of the magnetic moments within the film plane.

MFM imaging under an externally applied field

In addition, the evolution of the domain structure with longitudinally applied field has been measured for both se-

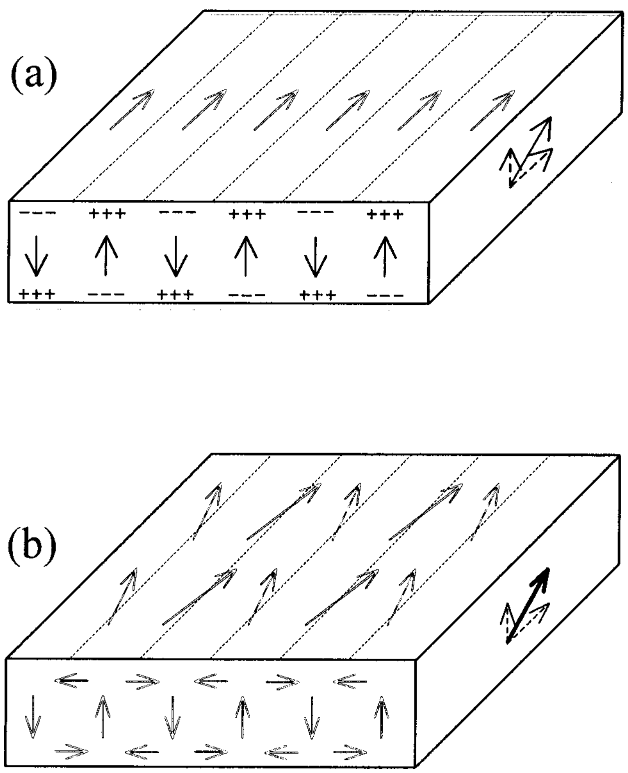

FIG. 4. Dense stripe domains models for weak out of the plane anisotropy thin films. The domain distribution in (a) corresponds to a sample with stronger out of the plane anisotropy than in (b). Perpendicular and in-plane magnetization components are shown separately.

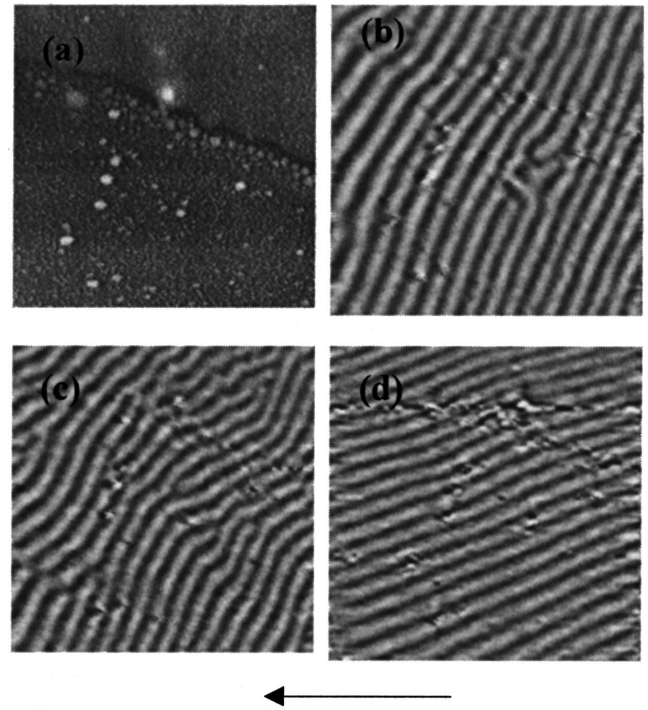

FIG. 5. (a) AFM image and (b)–(d) MFM images of the Co-Si-B_{100} sample grown onto a planar substrate. The magnetic images have been recorded under an applied field of (b) 2.1 kA/m, (c) 3.8 kA/m, (d) 7.2 kA/m. Image size: 17.5×17.5 μm. The arrow indicates the applied field direction which corresponds to the longitudinal direction.

ries. In the Co-Si-$B_{100\,nm}$ sample deposited onto a planar substrate, the domains size is reduced and the wall direction rotates continuously with the applied field as shown in Fig. 5. The domain width decreases about 40% when the applied field varies between 0 and 7.2 kA/m. Simultaneously, the orientation of the walls evolves to lower energy states by decreasing the angle between the applied field direction and the stripe domains. Most of the walls remain parallel to each other during the magnetization rotation due to the absence of any preferential direction within the film plane. Figure 6 shows the dependence with the applied field of the domain width and the angle between the stripes direction and the longitudinal axis for the Co-Si-$B_{50\,nm}$ and Co-Si-$B_{100\,nm}$ unstressed samples. Notice the existence of certain field threshold for the domain width decrease and for the collective rotation. In the Co-Si-$B_{100\,nm}$ sample, the reduction of the domain width starts about 1.7 kA/m later than in the Co-Si-$B_{50\,nm}$ sample. In the same way, the alignment of the walls with the field is delayed in the case of the Co-Si-$B_{100\,nm}$. That is in agreement with the hysteresis loops measurements since the Co-Si-$B_{100\,nm}$ sample exhibits smaller relative magnetization than the Co-Si-$B_{50\,nm}$ sample for a given applied field.

The domain pattern evolution with the longitudinal applied field has been also observed in the case of the stressed samples. After transverse saturation, the samples present the same periodic domain structure with the stripes aligned to the field direction. When the field is applied along the longitudinal direction, a change in the domain width as well as in the domain pattern is found (see Fig. 7). The behavior of the stressed samples with the field is totally different to that observed for unstressed films. Here, the domain width evolves continuously but the domain walls do not remain parallel to each other throughout the sample. Certain angular positions seem to be energetically favorable for the alignment of the

TABLE I. Magnetic contrast and domain width measured by MFM, coercivity measured in perpendicular, longitudinal and transverse direction and in-plane anisotropy field. Results are collected for stressed and unstressed samples.

	Contrast (mN/m)	Domain width (nm)	$Hc\perp$ (kA/m)	Hc_{lo} (kA/m)	Hc_{tr} (kA/m)	H_k (kA/m)
Co-Si-B_{50}	0.12	1000	3.52	0.50	0.46	13.87
Co-Si-B_{100}	0.35	570	12.81	0.99	1.39	17.45
s-Co-Si-B_{50}	0.21	920	7.48	0.71	0.89	22.94
s-Co-Si-B_{100}	0.42	640	14.88	1.27	1.40	25.90

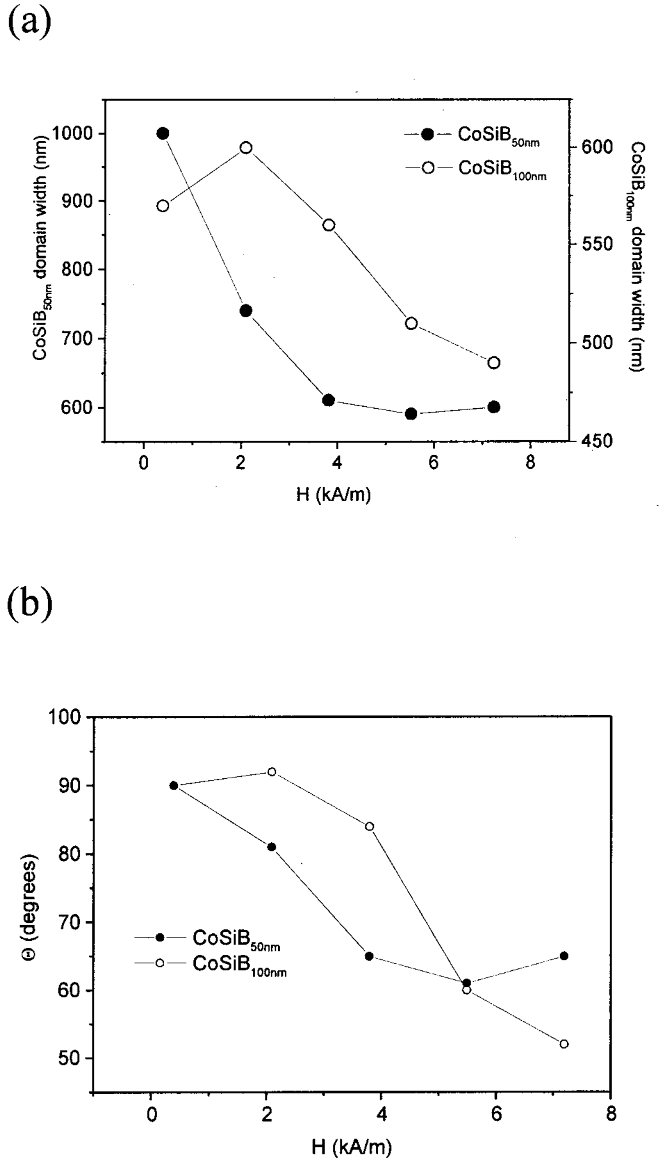

FIG. 6. Evolution of the domain width (a) and the angle between the stripes and the longitudinal direction (b).

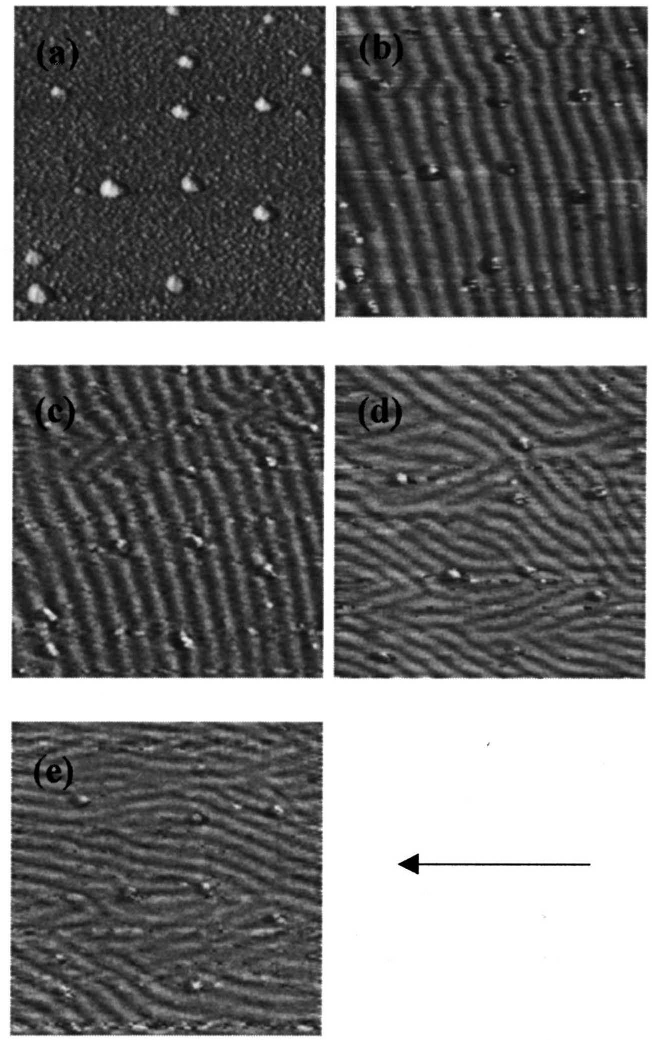

FIG. 7. (a) AFM image and (b)–(e) MFM images of the s-Co-Si-B_{100} sample grown onto bowed substrate obtained at (b) 0.4 kA/m, (c) 3.8 kA/m, (d) 5.5 kA/m, (e) 7.2 kA/m. Image size: 17.5×17.5 μm. The arrow indicates the applied field direction which corresponds to the longitudinal direction.

magnetic walls (see Fig. 8). This behavior reflects the existence of some in-plane anisotropy due to the compressive stress applied in the transverse direction. The directions where the domain walls stabilize form a certain angle with the longitudinal direction of the stressed samples. Figure 8(b) shows the angular distribution of the stripe domains in the s-Co-Si-$B_{50\,\mathrm{nm}}$ sample. The angles of these ''stabilization directions'' are lower for the s-Co-Si-$B_{50\,\mathrm{nm}}$ sample (about $\pm16°$ with respect to the longitudinal direction) than for the s-Co-Si-$B_{100\,\mathrm{nm}}$ sample shown in Fig. 8(c) (about $\pm$ 30°, average value calculated from the MFM data at 2.1, 3.8, and 7.2 kA/m). These preferential orientations of the stripe domains can be interpreted considering the crossed easy magnetization axes of the layers and an exchange interaction in the interfaces. Evidences of such an exchange interaction are discussed by the authors elsewhere.[8] In the case of the Fe-B single layer, the preferential orientation of the in-plane magnetization was the longitudinal one, that is, 0° in our notation. When the Fe-B layers are surrounded by Co-Si-B layers with a transverse easy magnetization axis, the magnetic moments tend to move apart from the longitudinal direction to minimize the anisotropy energy stored in the Co-Si-B layers. In this frame, the values of the preferential orientations of the stripes domains can be considered as the in-plane directions for which the anisotropy energy stored by the magnetic moments is minimum. Accordingly, the ''stabilization directions'' of the stripes are closer to the longitudinal direction than to the transverse one, due to the higher strength of the Fe-B anisotropy. Furthermore, the angles of the ''stabilization directions'' of the s-Co-Si-$B_{100\,\mathrm{nm}}$ sample are higher than the ones of the s-Co-Si-$B_{50\,\mathrm{nm}}$ sample. This is due to the relatively higher influence of the Co-Si-B anisotropy in the former multilayer. The coexistence of domains oriented along positive and negative angles could be attributed to the existence, in the remanent state, of regions with opposite direction of the in-plane component of the magnetization. Since the MFM contrast is mainly originated by the perpendicular component of the magnetization, these regions would not be identified in the remanent state.

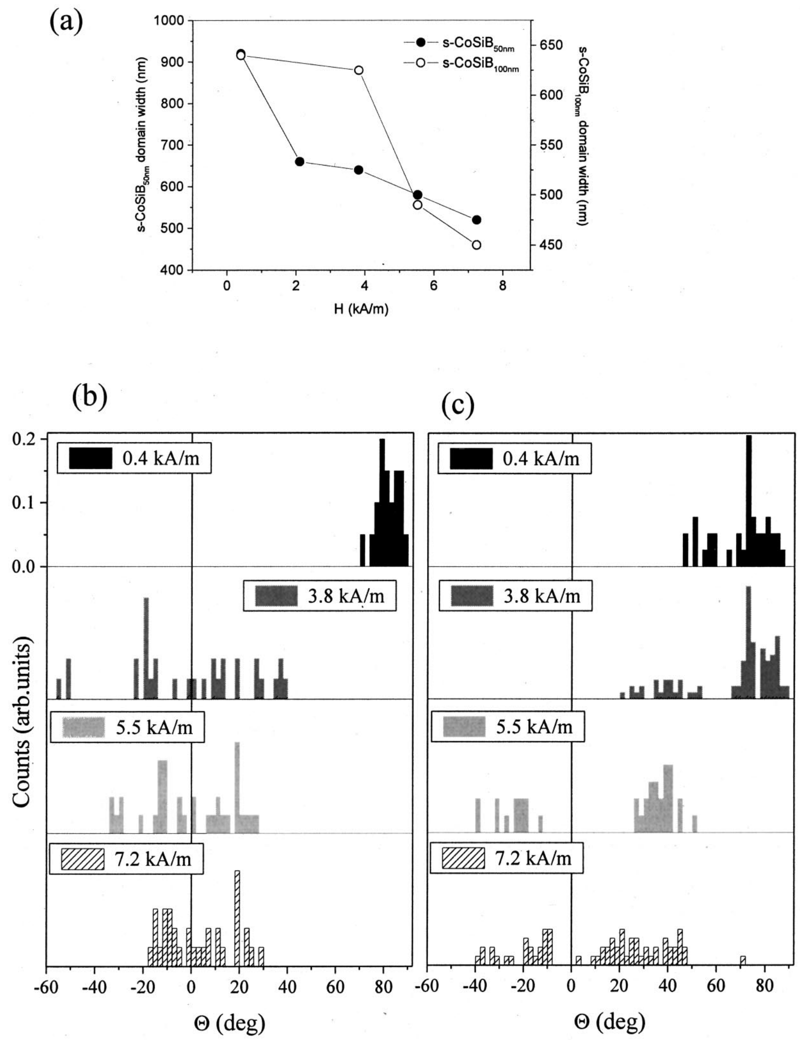

FIG. 8. (a) Evolution of the domain width and the angle between the stripes and the longitudinal direction for the samples (b) s-Co-Si-B_{50} and (c) s-Co-Si-B_{100}.

CONCLUSIONS

Two series of samples have been fabricated: samples grown onto planar and bowed substrates. Onto both kinds of substrates, three different multilayers have been deposited varying the thickness of the Co-Si-B layers. As shown in the longitudinal and transverse hysteresis loops, a larger rotational regime in the magnetization process is observed for thicker Co-Si-B layer samples.

Dense stripe domains have been observed in Fe-B/Co-Si-B multilayers grown onto planar and bowed substrates. The existence of this type of domain pattern is due to the presence of an out of the plane anisotropy arising from the fabrication process. In particular, the perpendicular anisotropy can be ascribed to the columnar growth as observed by AFM. This out of the plane anisotropy increases with the Co-Si-B thickness. MFM imaging performed in the remanent state after saturating the samples in the transverse direction shows parallel stripe domains aligned along the transverse direction. As expected, the width of the stripes is close to the thickness of the sample.

In addition to the MFM study of the samples in the remanent state, the domain evolution with the field applied in the longitudinal direction has been analyzed. In the samples grown onto planar substrates, the parallel stripes direction changes continuously to align with the applied field direction in order to decrease the Zeeman energy. However, in the case of samples grown onto bowed substrates, when the field is applied, the parallel stripe domains break into stripes which are oriented in different angles due to the presence of the induced anisotropy (arising from the negative magnetostriction of the Co-Si-B layers).

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