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Research articles

Silicon substrate orientation influence on structural and magnetic properties of $BaFe_{12}O_{19}$ thin films obtained by RF magneton sputtering

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ABSTRACT

In this work, the fabrication of $BaFe_{12}O_{19}$ (BaM) thin films by radio-frequency sputtering and the influence of substrate orientation on structural, microstructure and magnetic properties were carried out. Measurements pointed out the deposition time and power have no significant effect over stoichiometry in the studied condition, producing films with satisfying Fe:Ba rate confirmed by EDX analysis. The orientation of the Si substrate showed to be essentially correlated with in-plane orientation of BaM phase. Both Si (1 0 0) and (1 1 1) substrates privileged in-plane orientation of the c-axis of BaM and atomic force microscopy found needle-like grains spread randomly onto substrates, although Si (1 1 1) substrate exhibited higher magnetic anisotropy.

1. Introduction

Widely studied since the 1950's, ferrites are technologically interesting materials, due to their chemical stability, easy synthetization, electrical conductivity and ferromagnetic properties. Particularly barium hexaferrite ($BaFe_{12}O_{19}$ or BaM) is an important M-type hexaferrite with high magnetic anisotropy and saturation magnetization [1], which make it a vastly applicable material for fabrication of permanent magnets, phase shifters, microwave and millimeter filters [2–5] and for development of new magnetoelectric materials [6–8]. The high orientation of the c-axis along a specific direction is desired to enhance magnetic anisotropy in case of BaM thin films. In this sense, studies have been developed based on the elucidation of the deposition methods and substrate types on the effective crystallographic and magnetic anisotropy and orientation. For physical deposition methods, characteristics such as sputtering pressure [9], temperature of the substrate [10], oxygen pressure [11], power and annealing methods [12,13] or material used as substrate [13] have already been studied. There are, however, few works concerning to BaM deposition on Si substrates. Most of them consist of Si/Pt or Si/SiO_2 substrates leading to strong out-of-plane orientation of the c-axis [14–16]. In-plane c-axis films has been obtained mainly to sapphire substrates [17,18], and a recent study reported out-of-plane oriented films over Si (1 0 0) substrates [19]. In this work, we present an investigation on the fabrication of magnetron sputtered BaM films on different Si substrate orientations (Si (1 0 0) and Si (1 1 1)) and the correlation between the substrate orientation and magnetic properties of in plane c-axis oriented BaM thin films.

2. Experimental procedures

$BaFe_{12}O_{19}$ (BaM) film depositions were carried out in a radio frequency magnetron sputtering (RF Sputtering) system using a fabricated stoichiometric BaM target with diameter of 50 mm. Si substrates at (1 1 1) and (1 0 0) directions were used. Deposition time (from 30 min to 90 min) and the rf power (from 30 W to 90 W) were varied to obtain stoichiometric BaM thin films. After evacuating the chamber to a base pressure below $4x10^{-4}$ Pa, Ar gas was introduced as the working gas. The Ar pressure remained 1.6 Pa during the depositions. After depositions, as-deposited films were annealed in air atmosphere at 800 °C for 60 min, by conventional method. Phase composition and crystal orientation analysis were performed by X-Ray Difraction using CuK_α radiation (Shimadzu XRD-6100) and stoichiometric characterization was performed by Energy Dispersive X-ray spectroscopy (Shimadzu EDX-800HS). Films thickness was evaluated using a surface perfilometer. Microstructural analyses were carried out using Scanning Electron Microscopy (SEM Jeol JSM-5800LV) and Atomic Force Microscopy images (Shimadzu SPM). Topography and Magnetic Force Microscopy images were obtained using silicon cantilevers coated with a hard magnetic layer of cobalt alloy (MFMR – Nanoworld). The measurements were realized in two steps: a first a topography image in the tapping mode is obtained, then the tip is lifted about 10–30 nm from the

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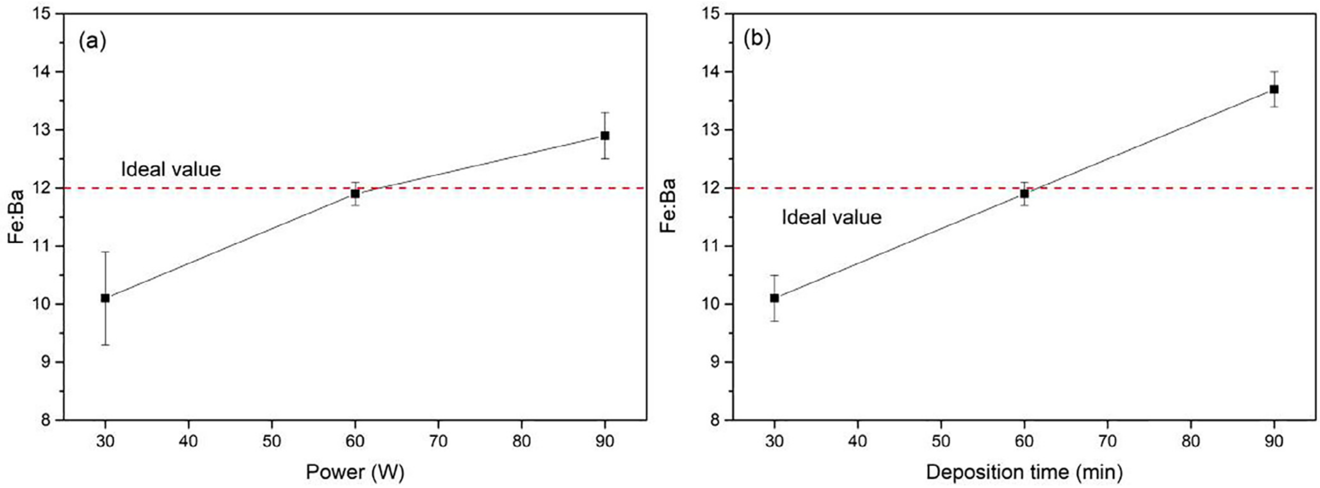

Fig. 1. (a) Fe:Ba atomic rate versus rf power; (b) Fe:Ba atomic rate versus deposition time.

sample and a second image of the phase shift of the tip vibration is obtained. Magnetic properties measurements were performed using a vibrating sample magnetometer (VSM-SQUID MPMS Quantum Design). Magnetic hysteresis loops were measured along both edges of substrates at room temperature.

3. Results and discussion

Fig. 1 shows the Fe:Ba ratio obtained from the EDX analysis as a function of rf power (Fig. 1 (a)) and deposition time (Fig. 1 (b)) for the thin films grown onto Si (1 0 0) substrate. The Fe:Ba ratio increased with both deposition time and sputtering power. It is clear to see a good accordance between ideal Fe:Ba ratio and the stoichiometric ratio obtained for all fabricated thin films, which indicate a stoichiometric presence of elements onto substrate, independently of the deposition parameter investigated. Also, the thickness of films increases from 50 to 150 nm as the deposition time increases from 30 min to 90 min resulting in a deposition rate of ~ 1,7 nm/min (or ~ 100 nm/h). The slow deposition rates obtained is in accordance to the reported by different hexaferrite materials fabricated from RF-sputtering [17]. Based on these fabrication parameters, BaM thin films were growth on both Si (1 0 0) and Si(1 1 1) substrates at optimized condition of 60 W for 60 min at argon atmosphere.

The phases of BaM thin films annealed, in air atmosphere at 800 °C for 60 min by conventional method, have been checked by XRD. The results are shown in Fig. 2 for samples deposited on different silicon substrates orientation. All the samples showed only peaks corresponding to the BaM hexagonal structure. Two main peaks corresponding to (1 1 0) and (2 2 0) planes of BaM hexaferrite phase can be seen, a small reflection of the (1 0 8) plane for the silicon (1 1 1) is also present, suggesting an intense in-plane orientation for both films. The presence of additional minor BaM (1 0 7) and (1 1 4) peaks in the film grown onto Si(1 0 0) substrate, indicate random nucleation compared to the film obtained onto Si(1 1 1) substrate. Quantitative analysis of degree of preferential orientation at in-plane direction using the Lotgering factor (f_L) [20,21] given by eq. (1), is showed in Table 1. It is clear to see a significant increment of crystallographic orientation to BaM film obtained on Si(1 1 1) compared to BaM films on Si(1 0 0) substrates, which is evidenced by the higher f_L factor. In this equation, $I(hk0)$ is the intensity of (hk0) diffracted plane in the oriented film, and $I(hkl)$ corresponds to (hkl) planes in the same film. The intensities I_0 correspond to the same pattern, but in a random oriented sample. In this case, we used the diffraction pattern of powder as reference.

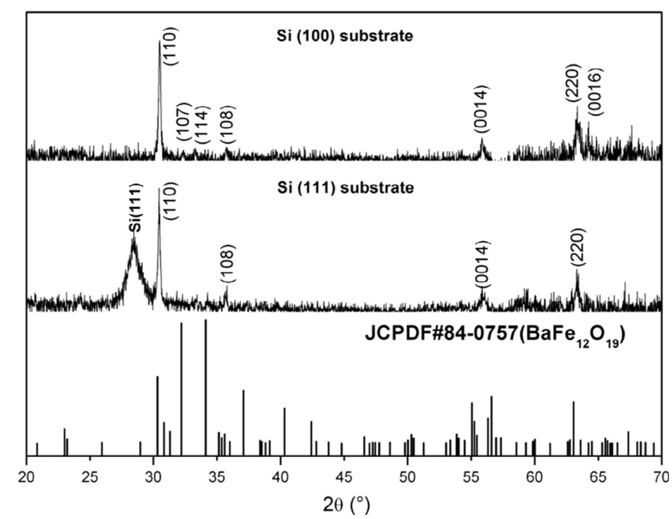

Fig. 2. XRD patterns for films grown onto Si(1 0 0) and Si(1 1 1) substrates.

Table 1
Lotgering factor (f_L) for BaM haxaferrites fabricated on Si(1 0 0) and Si(1 1 1) substrates.

Substrate	F_L
Si(1 0 0)	0.40
Si(1 1 1)	0.68

$$f_L = \frac{\sum I(hk0)/\sum I(hkl) - \sum I_0(hk0)/\sum I_0(hkl)}{1 - \sum I_0(hk0)/\sum I_0(hkl)} \quad (1)$$

Based on these evidences it is possible to conclude the existence of preferential in-plane c-axis orientation, which can be enhanced using Si (1 1 1) substrates. Usually, high degree of out of plane c-axis orientation is achieved in BaM by use of the Pt/SiO substrate [14]. On the other hand, the c-axis in-plane orientation (IPCA) in BaM is obtained on sapphire substrates as reported by Zhang et al. [17]. In this case, the IPCA orientation was related to the formation of α-Fe_2O_3 intermediate phase, which was responsible to release the lattice mismatch between film and substrate, similarly to a buffer layer. Meng et al. [18] shows an enhancement of IPCA orientation for samples deposited on Al_2O_3(1 1 0) in presence of O_2, since the presence of oxygen during the deposition processes contributes to the α-Fe_2O_3 intermediary phase formation. In

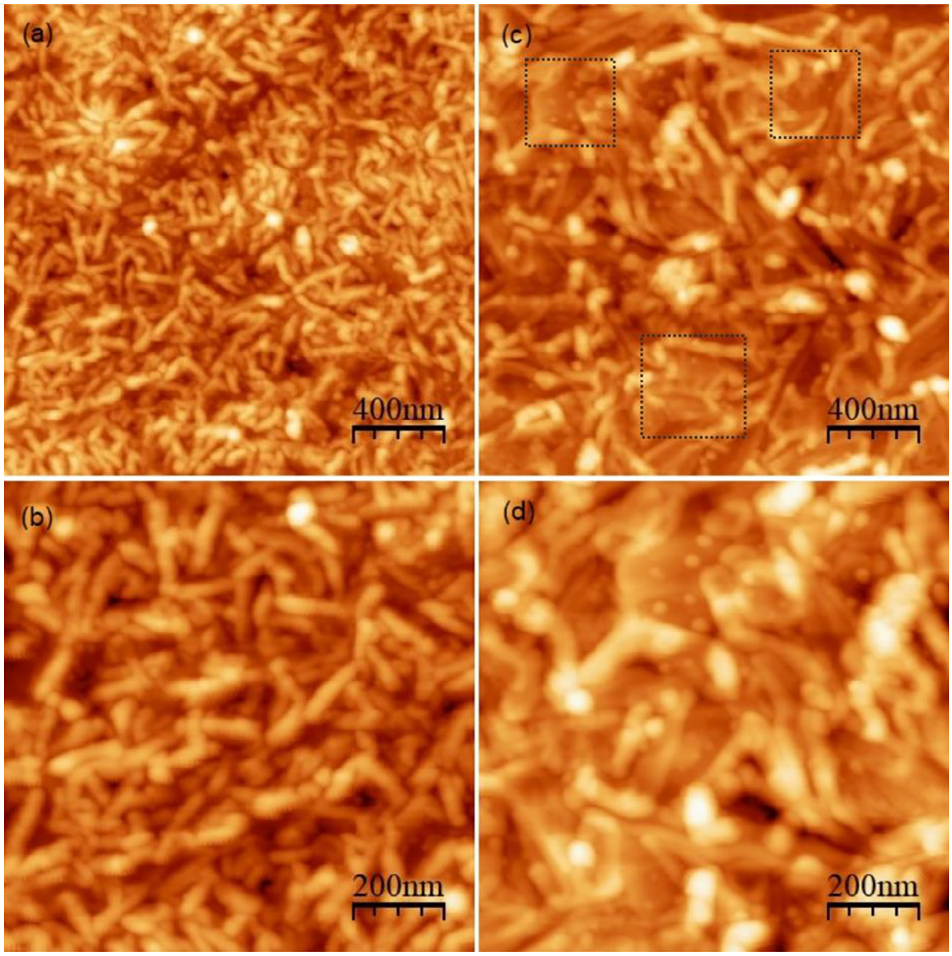

Fig. 3. AFM images for films grown on Si(1 1 1) ((a) 2x2 µm and (b) 1x1 µm) and Si(1 0 0) ((c) 2x2 µm and (d) 1x1 µm) substrates.

both case, the existence of intermediary hematite phase plays a crucial role to the IPCA orientation in BaM. In the present work, both films were fabricated in the same deposition conditions. Han el al. [22] report an intermediary hematite phase formation, more evidenced using Si(1 1 1) substrate, for $Ba(CoZn)_2Fe_{16}O_{27}$ thin films. In agreement, Irene et al. [23] show the oxidation rate is larger in to Si(1 1 1) than Si (1 0 0) substrate. Based on these evidences, the formation of enhanced SiO_2 interlayer may improve the α-Fe_2O_3 intermediate phase that contributes to higher IPCA orientation in the Si(1 1 1) substrate compared to the Si(1 0 0) orientation, since both thin films were annealed at 800 °C for 60 min in air atmosphere.

Fig. 3 represent the microstructure for annealed BaM hexaferrite growth at Si(1 1 1) and Si(1 0 0) substrates. The analyses of Fig. 3a indicate the existence predominantly of needle-shaped grains with average grain size of the aprox.50x150 nm (cylindrical shape) distributed mainly in the in-plane direction of substrate for BaM grown on Si(1 1 1) substrate. On the other hand, the BaM grown on BaM Si(1 0 0) substrate has distribution of grain with large placed shape, detached by dashed rectangles in the Fig. 3c, and large needle-shaped grains. It can be seen that both films show needle-shaped grains, which is an indicative of IPCA orientation [16,17]. BaM films fabricated on Si(1 0 0) show larger needle-shaped grains, however it can be seen the occurrence of plated grains, which are typical of out of plane c-axis (OCA) orientation [9], as indicated in Fig. 3 (c) and (d). On the other hand, BaM films growth on Si(1 1 1) substrate showed an uniform distribution with needle-shaped grains, as indicated in Fig. 3 (a) and (b). These microstructures corroborate with XRD results indicating stronger IPCA in the film grown at Si(1 1 1) compared with BaM film fabricated on Si (1 0 0) substrates.

Textures and morphologies of films greatly influence magnetic properties. Magnetic hysteresis loops for the films were measured in both out-of-plane and the in-plane geometry by VSM-SQUID, at room temperature, with a maximum available applied magnetic field of 50kOe. The hysteresis loop squareness ratio S (defined by the ratio between the remanent magnetization and the saturation magnetization referred to the magnetization in the highest available field) were calculated. The magnetic hysteresis loops are illustrated in the Fig. 4. Corresponding squareness ratio and coercivities for the hysteresis loops taken in the out-of-plane ($S\perp$ and $H_C\perp$) and in plane ($S//$ and $H_C//$) are listed in Table 2. The magnetic properties of the deposited BaM thin films are strongly dependent on the different substrate orientation. Hysteresis loops referring to in-plane applied field are larger than to out-of-plane in both films. However, it can be seen a weak magnetic anisotropy in the film growth on Si(1 0 0) substrate, compared to the observed on Si(1 1 1) BaM thin films. For the sample Si(1 0 0), the difference between the saturation magnetization for the in-plane and out-plane measurement is about 12.2 emu/g, while for the Si(1 1 1) sample, this difference is about 32 emu/g. It can be explained by the direction of the easy magnetization axes. As shown in the AFM images, the Si(1 1 1) sample has a stronger IPCA in which changes the easy axes magnetization, changing the saturation magnetization values. Therefore, it is expected that a larger difference will be observed in sample Si (1 1 1), since the Si (1 0 0) sample behaves closer to an isotropic sample. The magnetic uniaxial anisotropy was calculated, given by [24]

$$K = \int_0^{M_S} (H_{eff}^{out} - H_{eff}^{in})dM, \tag{2}$$

leading to a K = 0.9×10^6 erg/cm^3 for the films grown over Si (1 0 0)

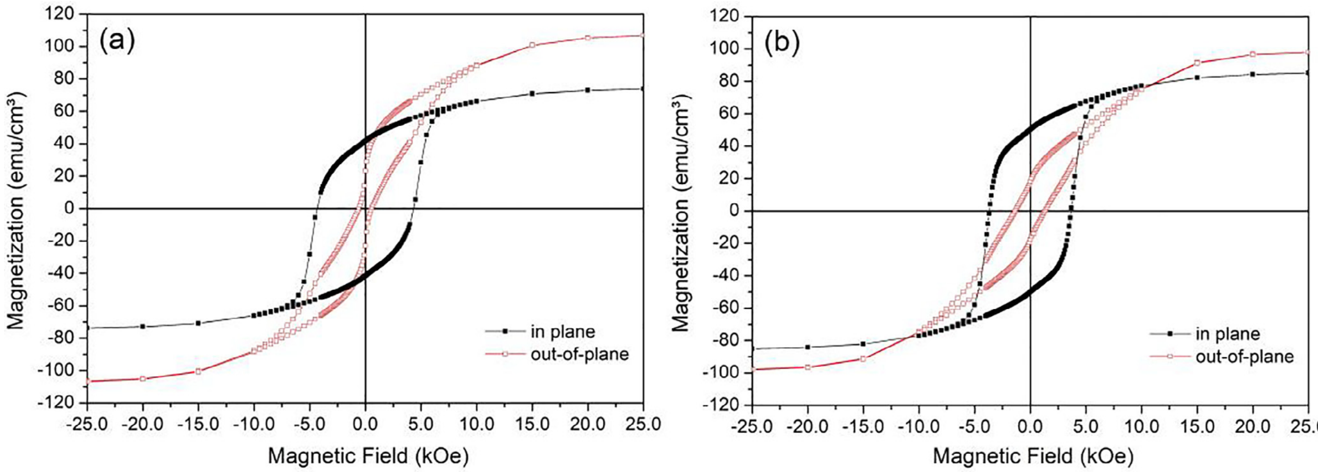

Fig. 4. Magnetic loops for films grown onto (a) Si(1 1 1) and (b) Si(1 0 0).

substrate, and K = 2.2×10^6 erg/cm^3 for the Si (1 1 1) one. Based on structural characteristics of BaM phase, it is expected values for magnetic uniaxial anisotropy constant K close to 3.3×10^6 erg/cm^3 for BaM single-crystals [25]. Hylton et al. [26] reports values close to 2.4×10^6 erg/cm^3 for epitaxial BaM thin film grown on sapphire substrate with in-plane orientation by RF sputtering. On the other hand, for BaM thin films on Si based substrates, the level of the magnetic anisotropy has been discussed based on hysteresis loop squareness ratio differences between in-plane and out of-plane directions, which is dependent of the parameters such as thickness, pressure, temperature and atmosphere of deposition [9,13,14,22]. Han et al. [22] and Capraro et al. [13] have deposited BaM based hexaferrites on Si(1 0 0) substrate. The thin films were found isotropic with a random distribution of easy axis direction. In our work, the significant difference between the out-of-plane and in-plane loops evidenced by changes in the magnetic uniaxial anisotropy factor (K), confirms that the both film possesses strong magnetic anisotropy and has its easy magnetic anisotropy axis (c-axis) parallel to the film plane. These results are in good agreement with the XRD and morphology measurements, which indicate Si(1 1 1) substrate enhances IPCA orientation compared to the Si(1 0 0) substrate resulting in an increase of anisotropy factor K. In addition, it can be seen from the Fig. 4 and table 2, a hinder of the rotation process of magnetic dipoles in the in-plane measurements, as evidenced by changes in the $Ms_{//}/Ms_{\perp}$ ratio for different substrates and enhanced for Si(1 1 1) substrate. In fact, for magnetic thin films the microstructural, structural and shape anisotropy play critical parameters to magnetization of samples. Dhakal et al. [24] report similar behavior for CoFe2O4 thin film deposited on STO and MgO, which the higher $Ms_{//}/Ms_{\perp}$ ratio obtained for CFO/STO thin film compared to the CFO/MgO was related to compressive strain generated by lattice mismatch between substrate and magnetic phase. Pullar et al. [27] discusses the dependence of saturation magnetization with alignment in BaM fibers, which have demonstrated an enhanced of the magnetization along the axis of alignment of fibers with respect to perpendicular to the axis. On the other hand, the effect of strain on magnetization in BaM thin films grown on Si substrate was reported by Suzuki et al. [28] which differences in Ms compared to $Ms_{\perp}$ is evident. The authors report the presence of a tensile strain due to thermal expansion coefficients differences between the BaM and Si phases. In another work, an enhancement of out-of-plane magnetization was achieved with a biaxial tensile strain on CoFe2O4 thin films [29]. In our work, in spite of thermal expansion is similar in both substrates, the Young modulus and lattice characteristics are dependent of Si orientation [30] and both parameters play a crucial role to strain conditions of the BaM on Si substrate [31,32]. An important evidence of the strain difference between BaM/Si(1 0 0) and BaM/Si (1 1 1) thin films is related to changes in the average grain size and morphology of grains, as can be seen in the Fig. 3. Thus, the increase of the $Ms_{//}/Ms_{\perp}$ ratio for BaM/Si(1 1 1) compared to the BaM/Si(1 0 0) thin films can be related to the strain environment generated by Si orientation on BaM phase. Since the Young modulus and lattice parameters are larger for Si(1 1 1) substrate (188 GPa) compared to the Si (1 0 0) substrate (130 GPa) [33], a larger tensile strain is expected for Si (1 1 1) substrate. In this case, the magnetic domains switching on thin film plane are reduced compared to the out of plane domains switching.

Fig. 5 (a) and (b) shows the topography and MFM images obtained in the BaM films grown on Si(1 1 1) and Si(1 0 0) substrates, respectively. It can be observed from the magnetic response of both BaM thin films that magnetic domains are not restricted by grain boundaries, as indicated by dashed regions in Fig. 5. The correspondence between the MFM image and the domain structure in such system is not trivial. The MFM senses variation of vibration of the cantilever due to the gradient of the magnetic force in the Z-direction. However, the source of the magnetic force can be domains with an out-of-plane component of the magnetization or in-plane domain walls, as example. Considering the magnetic properties, magnetization lies preferentially in-plane for both cases, although is more evident for the BaM/Si(1 1 1). Thus, it is likely that the grains are domain with magnetization mostly lying in the plane of the substrate, but some out-of-plane orientation might be present, due to tilt of the grains, or misalignment of the magnetization from the easy axis. Comparing the green and white dashed regions of panels in the Fig. 5 (as prepared samples and after magnetizing in the out-of-plane direction applying ± 20 kOe) it is clearly observed the switching of magnetic domains in both samples. By application of the + 20 kOe, it is possible to observe the formation of new magnetic domains due to

Table 2
Coercive magnetic fields towards in plane ($H_{C//}$) and out-of-plane ($H_{C\perp}$) direction; in plane ($Mr_{//}$) and out-of-plane ($Mr_{\perp}$) remanent magnetizations; in plane and out-of-plane squarenesses ($S_{//}$ and $S_{\perp}$); magnetic stored energy *(K)* and saturation magnetization ratio ($Ms_{//}/Ms_{\perp}$) for BaM/Si(1 0 0) and BaM/Si(1 1 1) thin films.

Substrate	$H_{C//}$ (kOe)	$H_{C\perp}$ (kOe)	$Mr_{//}$ (emu/cm^3)	$Mr_{\perp}$ (emu/cm^3)	$S_{//}$	$S_{\perp}$	K (erg/cm^2)	$Ms_{//}/Ms_{\perp}$
Si(1 0 0)	3.7	1.3	50.15	17.68	0.58	0.20	$0.9\text{x}10^6$	0.88
Si(1 1 1)	4.3	0.6	41.61	14.25	0.55	0.21	$2.2\text{x}10^6$	0.69

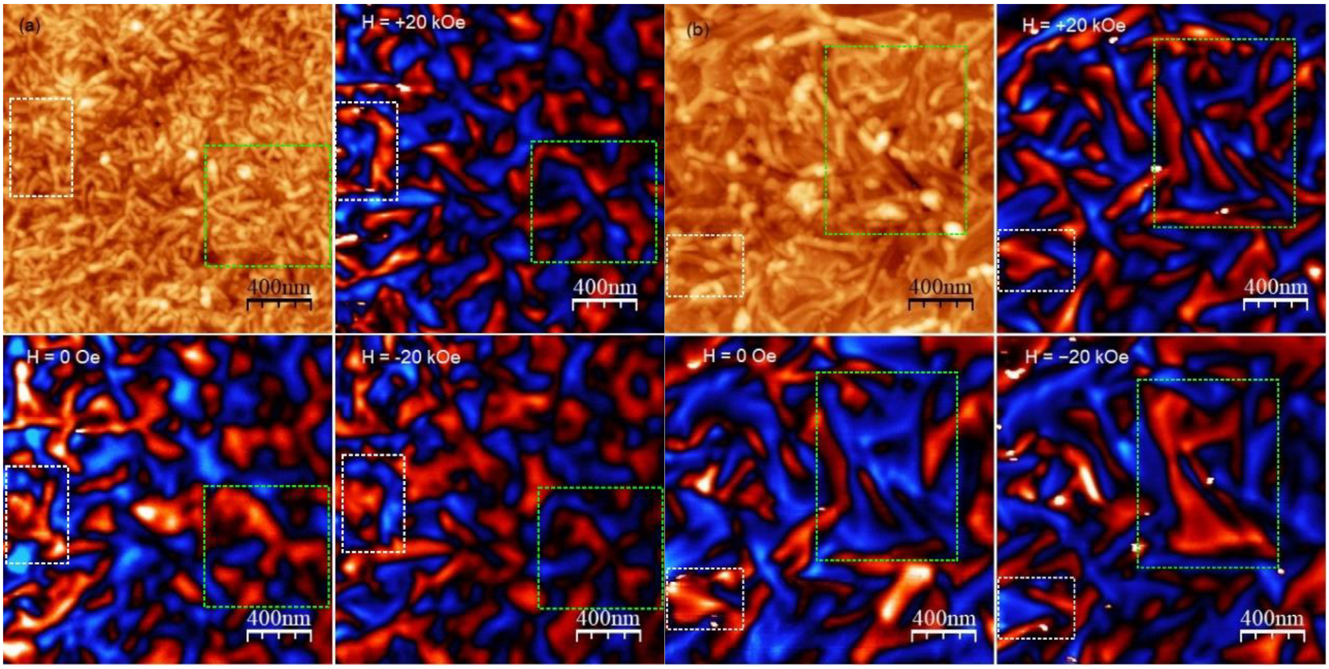

Fig. 5. Topography and MFM images, of the BaM thin films grown on (a) Si(1 1 1) and (b) Si(1 0 0) substrates and the images after applying, respectively, +20 kOe and −20 kOe, in the out-of-plane direction.

their reorientation. By applying −20kOe these regions invert the contrast, highlighting the switching of the magnetic domains. The size of magnetic domain structure may be related to the average grain size and stress of ferromagnetic system. The width of the stripe magnetic domain, obtained from Fig. 5 (a) and (b), is aprox. 75 nm and 68 nm for BaM/Si(1 1 1) and BaM(1 0 0), respectively. Comparing with expected value for uniaxial anisotropic crystals, the width of the stripe domain is expressed as [15]

$$d = \sqrt{\frac{4L\sqrt{AK}}{0.85(4\pi M_s)^2}}$$

where A = 0.5x10^{-6} erg/cm is exchange constant [15], L = 100 nm is film thickness, the width of stripe magnetic domain for BaM/Si(1 1 1) is aprox. 75 nm and for BaM/Si(1 0 0) is close to 60 nm, which match closely to the observed values from MFM. A slightly increment on the width of the magnetic domains for barium hexaferrite grown on Si (1 1 1) compared to the BaM/Si(1 0 0) is related to increment of the magneto-crystalline anisotropy observed by magnetic characterization. Also, the magneto-crystalline anisotropy observed by magnetic characterization indicate a larger stress condition for BaM grown onto to Si (1 1 1) compared to the Si(1 0 0) thin film.

4. Conclusions

The fabrication of $BaFe_{12}O_{19}$ hexagonal ferrites thin films have been prepared by the rf-magnetron sputtering method on Si(1 0 0) and Si (1 1 1) substrates. The results confirm that in-plane oriented BaM thin films can be obtained using silicon substrates. In this sense, orientation of the substrate plays an important role, due to the mismatching between Si and BaM structures. Concerning to its morphology, uniform needle-shaped grains are desired to IPCA oriented BaM thin films, and Si(1 1 1) showed to be a preferable silicon cut for this purpose, leading to higher magnetic anisotropy and magnetic energy.

Author contributions

F. L. Zabotto conceived and planned the experiments. V. P. Rossi, R. P. Bonini, A. M. Gonçalves and A. J. Gualdi carried out the experiments. All authors contributed to the interpretation of the results. R.P.Bonini took the lead in writing the manuscript. All authors provided critical feedback and helped shape the research, analysis and manuscript.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jmmm.2020.166705.

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