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Structural properties of cobalt ferrite thin films deposited by pulsed laser deposition: Effect of the reactive atmosphere

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Abstract

Cobalt ferrite thin films have been elaborated by pulsed laser ablation of a $CoFe_2$ metallic target on Si (100) substrates. The films were deposited at low temperature (300 °C) in various pressures of two different reactive atmospheres (O_2/N_2, 20:80 and O_2). We present the influence of the nature of the reactive gas and of the deposition pressure on the crystallisation. It has been shown that a strong (111) preferential orientation is obtained for intermediate pressures of the O_2/N_2 reactive gas. The degree of orientation is higher for the O_2/N_2 mixture than for pure O_2. This behaviour is explained in terms of kinetic energy of the deposited species.

Keywords: Cobalt ferrite; Pulsed laser deposition; Reactive atmosphere; X-ray diffraction

1. Introduction

Numerous studies are devoted to magnetoresistive devices for their uses in various applications such as magnetic recording. The relative control of the magnetizations of the layers in these devices necessitates to pin the magnetization of one of the layers. This is possible by magnetically coupling this layer to a hard one. Cobalt ferrite thin films are receiving increasing interest for their potential use as hard pinning layer thanks to their high coercivity, high Curie temperature around 800 K [1], high corrosion resistance and good mechanical stability [2,3]. Some works address the problem of exchange coupling between cobalt ferrite and other magnetic layers [4–8]. However, elaboration processes at low temperatures have to be developed to involve these films in electronic devices and avoid chemical reactions with the other components.

Thin cobalt ferrite films have been elaborated in the past by various methods, the most used being pulsed laser deposition (PLD) from a $CoFe_2O_4$ target. The films obtained by Guyot et al. [9] on glass, quartz or MgO at 450 °C were amorphous. The crystallization occurred by annealing at 600 °C. Epitaxially crystallised cobalt ferrite thin films have been obtained at low elaboration temperatures only when deposited on costly single crystal substrates like MgO [10,11] (the authors then worked at temperatures between 200 and 800 °C) or $CoCr_2O_4$ buffered $SrTiO_3$ and $MgAl_2O_4$ substrates [12–14] (the authors then worked at 400 °C).

However, mass-produced spin electronics devices require inexpensive substrates. Silicon substrates fulfil these conditions and, furthermore, have a good flatness. But the large lattice mismatch (29%) with $CoFe_2O_4$ makes it difficult to grow epitaxial films. Kennedy et al. [15] obtained (111) textured Fe_3O_4 films by PLD of a metallic target (Fe) on Si (100) substrates at 450 °C in a molecular oxygen atmosphere. However, magnetization values of 650–800 kA/m (650–800 emu/cm^3) were measured, which are too high to be attributed to ferrite, and lead the authors to consider the formation of metallic Fe-rich regions. Matsushita et al. [16] obtained a strong (111) orientation of $CoFe_2O_4$ on silicon substrates buffered with ZnO underlayers at low temperature

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(80 °C) but they, as well as Ding et al. [17], obtained polycrystalline cobalt ferrite when depositing directly on Si substrates at high temperature (600 °C). Until now, highly (111) oriented cobalt ferrite thin films by PLD on Si substrates have only been obtained by Terzzoli et al. [18] at high temperature (700 °C) using a $CoFe_2O_4$ oxide target, but the surface roughness was important (around 8 nm).

We have recently shown that it was possible to get polycrystalline $CoFe_2O_4$ films by pulsed laser deposition between 200 °C and 400 °C from a metallic target in a reactive atmosphere and on silicon substrates [19]. This paper is devoted to the determination of the optimal deposition conditions to obtain highly (111) oriented films on Si (100) substrates at low temperature (300 °C). The effect of the nature of the reactive atmosphere on the structural properties of the films is discussed.

2. Experimental procedure

The $CoFe_2O_4$ films were grown by PLD technique using a nanosecond KrF excimer laser (wavelength 248 nm, pulse length 20 ns, repetition rate 10 Hz and maximum energy 32 mJ/pulse). The laser fluence was 3 J/cm^2. The laser beam moved in two directions of the fixed $CoFe_2$ target plane in order to homogeneously ablate it. The metallic $CoFe_2$ target was obtained from the fusion and solidification in the shape of a disk of a stoichiometric mixture of metallic Co and Fe. Its density was 8060 kg/m^3. The films were deposited onto 10×10 mm^2 (100) Si substrates in-situ heated at 300 °C. Before deposition, the substrates were cleaned with an HF (40%)–HNO_3 (60%) solution. The distance between the target and the substrate was kept constant at a value of 4 cm during all experiments. The growth chamber was evacuated down to a base pressure of around 10^{-6} Pa prior to the film deposition. Two series of films were deposited, differing in the nature of the reactive gas. The first series was elaborated in (O_2/N_2/20:80) at various pressures comprised between 0.5 and 10 Pa. The second was elaborated in pure O_2 pressures varying between 0.06 and 10 Pa. The use of pure O_2 instead of O_2/N_2 was expected to allow oxidation at lower deposition pressures and, therefore, a lower roughness of the deposited layers (the roughness is observed to increase with the deposition pressure [19]).

The thickness of the different samples was measured with a profilometer (Talystep) Hobson Taylor. It was uniform over the whole substrate surface and varied between 150 and 350 nm from one sample to another.

A first crystallographic characterisation of the films was operated using X-ray diffraction (XRD) at room temperature in θ–2θ mode (λ_{Co}=0.1789 nm). These first observations revealed a strong degree of crystallite alignment. The heteroepitaxy was then studied using a Huber 4-circle X-ray diffractometer (using the monochromatised λ_{Co}=0.15406 nm radiation) equipped with a curved position sensitive detector [20]. Cross-sections and plane views of the films were observed by high-resolution transmission electron microscopy (HR-TEM) using a TOPCON EM-002B microscope operating at 200 kV with a point-to-point resolution of 0.18 nm. The Co/Fe ratio was checked by energy-dispersive X-rays (EDX) spectroscopy in a scanning electron microscope (SEM) (JEOL JSM-6700F, FEG). It always matched that of the metallic target (0.5).

The oxygen content of the cobalt ferrite films was measured using nuclear reaction analysis (NRA) with a 900 keV $^2H^+$ beam and a scattering angle of 165°. NRA spectra were simulated with the SAM (Simulation for Analysis of Material) software [21]. The surface of the films was checked to be free of droplets by SEM and then scanned by atomic force microscopy (AFM) using a Digital Instruments Nanoscope III. The measurements were performed at a scan rate of 0.5 Hz with a 125-μm-long tapping mode Nanosensors Si cantilever having a force constant of 42 N/m and a resonance frequency of 300 kHz. Magnetic measurements were performed using a MicroMag 2900 Alternating Gradient Field Magnetometer operating at room temperature with a maximum applied field of 1040 kA/m (ca.13 kOe).

3. Results

The XRD patterns measured in the θ–2θ mode for cobalt ferrite films deposited at various (O_2/N_2, 20:80) and O_2 pressures are displayed in Figs. 1 and 2, respectively.

The intense (111), (222) and (333) peaks of the spinel structure in Fig. 1 indicate a strong preferential orientation with the ⟨111⟩ directions of the crystallites perpendicular to the film plane, for the films deposited in O_2/N_2 at pressures lower than 10 Pa. This preferential ⟨111⟩ orientation had not been observed on $CoFe_2O_4$ layers deposited by our group in similar conditions (gas and temperature) with another laser (XeCl excimer laser, λ=308 nm, with a fluence of 2 J/cm^2) [19]. A semi-quantitative measure of the degree of orientation of the layers may be given by the (111) to (311) integral peak ratio. This ratio displayed in Fig. 3 first increases with pressure, reaching a maximum around 5 Pa and then decreases for higher pressures. The FWHM of the rocking curve around the (111) reflection is about 4° for all pressures.

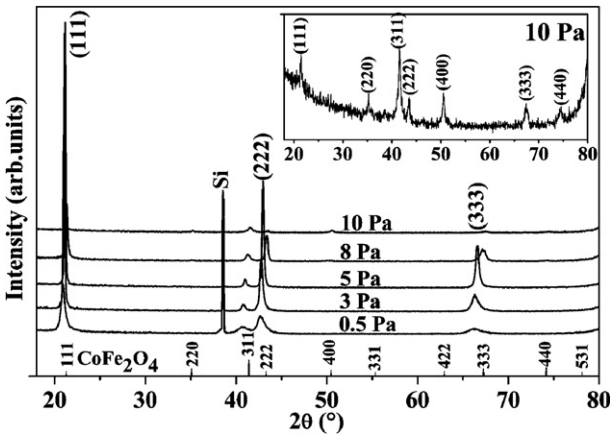

Fig. 1. X-ray θ–2θ scans for $CoFe_2O_4$ films elaborated in O_2/N_2.

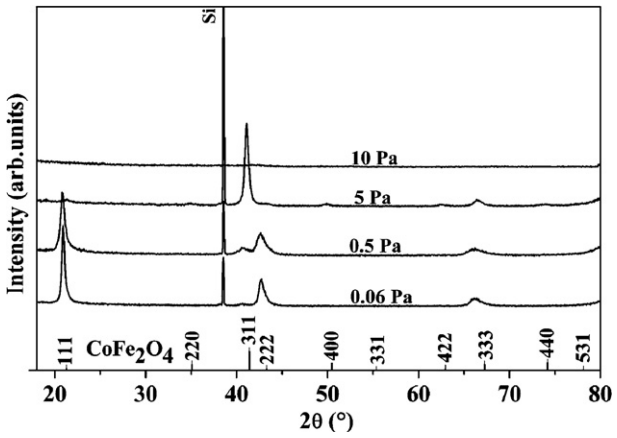

Fig. 2. X-ray θ–2θ scans for $CoFe_2O_4$ films elaborated in O_2.

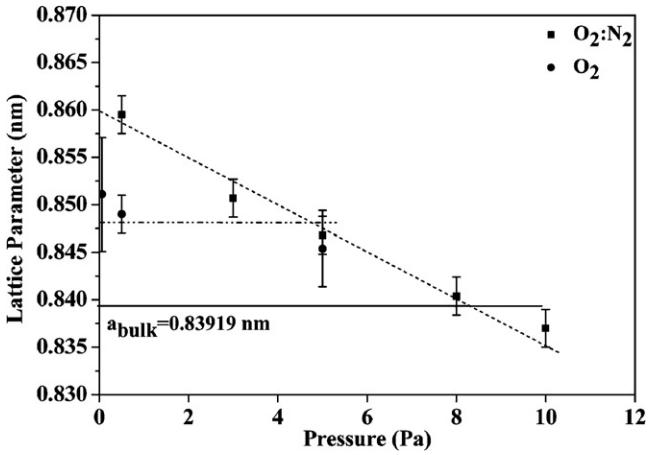

Fig. 4. Lattice parameters measured for the films deposited in O_2/N_2 (squares) and O_2 (circles) reactive atmosphere; dotted lines are only guides for eyes.

In Fig. 2, the diffraction patterns obtained for the films deposited in pure O_2 show three pressure domains:

(1) For low deposition pressures (lower than 5 Pa), a ⟨111⟩ preferred orientation is observed, similar to that obtained for deposition in the O_2/N_2 atmosphere.
(2) For a 5 Pa deposition pressure, the diffraction pattern is characteristic of a randomly oriented $CoFe_2O_4$ polycrystalline spinel phase film.
(3) For high deposition pressures (higher than 5 Pa), diffraction peaks are no longer visible, the films are either amorphous or composed of very small crystallites (<10 nm), too small to diffract significantly in our measuring conditions. Electron diffraction patterns of plane view recorded on the layer confirm these assumptions.

The variation of the lattice parameter with the deposition pressure of the two reactive gases is presented in Fig. 4. The lattice parameter of the films, as deduced from all peak positions deposited in O_2/N_2 atmosphere decreases with increasing pressures and reaches the bulk value $a=0.83919$ nm [22] for a pressure of 8 Pa. For the films deposited in a pure O_2 reactive atmosphere and which are crystalline, it has an approximately constant value of 0.848 nm, larger than the bulk value.

The mean coherence length along the normal to the film plane was estimated from Scherrer's formula:

$$\Phi = 0.9\lambda/\beta\cos\theta$$

where λ is the wavelength of the used radiation ($\lambda_{Co}=0.1789$ nm), β (radian) represents the full width at half maximum of the (111) peak in 2θ, and θ is the Bragg angle.

The coherence lengths as well as the crystallite sizes observed on TEM plane views for the films deposited in O_2/N_2 vary from 20 nm to 48 nm, the maximum being obtained for a deposition pressure of 5 Pa. The variation of the degree of orientation of the layers follows a similar trend and also shows a maximum for the 5 Pa deposition pressure. For the films deposited at low pressures of O_2 ($P<5$ Pa), comparable mean coherence lengths are observed, they are, however, smaller (around 20 nm) for the films deposited under 5 Pa pressure.

θ–2θ measurements alone insufficiently characterize the degree of orientation of the films. Pole figures are necessary to determine in particular the quality of the in-plane orientation. We measured pole figures using a 5°×5° grid in tilt (χ) and azimuthal (φ) angles, in the ranges 0–60° and 0–360°, respectively, as is usually operated.

The X-ray pole figure measured for the $CoFe_2O_4$ (311) reflection ($2\theta=35.43°$) for the sample elaborated in 5 Pa O_2/N_2 pressure, which shows the highest intensity on the θ–2θ scan, is presented in Fig. 5. The continuous rings, typical of fibre textures, at $\chi=29.50°$ and 58.52° are due to the {311} reflections of the cobalt ferrite film randomly distributed around the ⟨111⟩ directions. This indicates that no preferred orientation occurs in the sample plane, i.e., no hetero-epitaxial like relationship can be satisfied between the substrate and the film.

The Orientation Distribution Function (ODF) was refined using Beartex [23] from the {311} and {333+511} experimental pole figures. The ODF minimum and maximum values are 0 and 64 m.r.d. (multiple of random distribution), respectively,

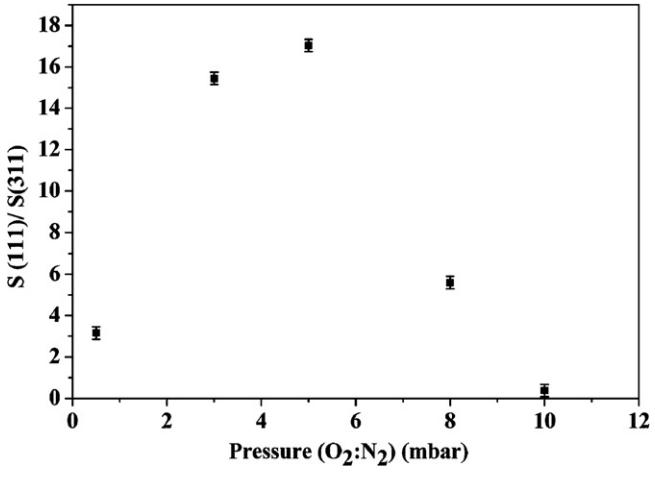

Fig. 3. Quantification of the degree of orientation given by the (111) to (311) integral peak ratio.

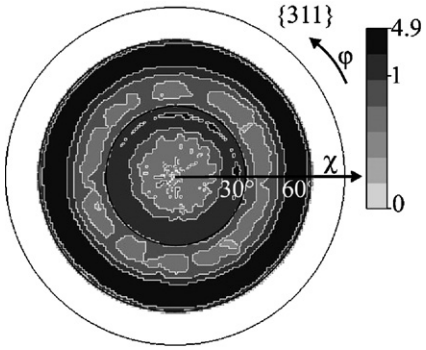

Fig. 5. Experimental {311} pole figure of $CoFe_2O_4$ for the film deposited in 5 Pa O_2/N_2. The two concentric circles represent $\chi = 30°$, 60°. Logarithmic intensity scale, equal area projection.

which underlines the fact that all the crystallites (100% of the sample volume) contribute to the texture components. The reliability of the ODF refinement is attested by the reliability factors averaged over all the experimental pole figures, RP = 26% calculated for all the density values and RP1 = 19% calculated for density values above 1 m.r.d., which for this level of texture are reasonable values [24]. From the ODF one can calculate any pole figure, and in particular those for the main crystal directions (Fig. 6). These figures show that very probably the only orientation component in the film corresponds to ⟨111⟩ directions aligned with the film' normal (⟨111⟩ fibre texture). However, not all the pole figures can be reproduced, and it may happen that a second minor texture component is present in the film, with other crystal direction aligned with the films' normal which would not be revealed in this figure.

The 001 inverse pole figure (Fig. 7) recalculated for the direction of the films' normal is a full representation of the ODF for fibre textures. It is recalculated from the orientation distribution using the WIMV (Williams–Imhof–Matthies–Vinel) algorithm [25]. It allows to reveal all the texture components along this normal. This figure clearly shows that the ⟨111⟩ component of texture is the major one and that another exists, corresponding to less than 2% of the material's volume with crystallites oriented with their ⟨123⟩ directions along the normal.

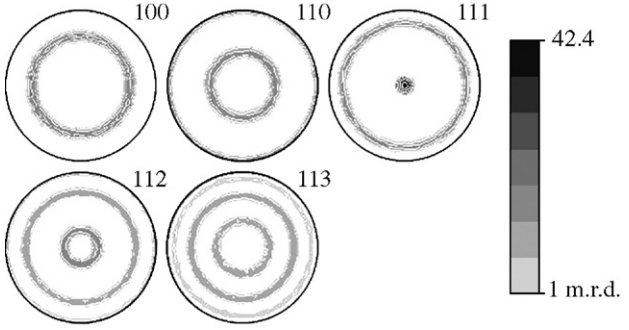

Fig. 6. Normalised pole figures recalculated from the ODF for the main crystal directions and levels above 1 m.r.d. only. Logarithmic density scale, equal area projection.

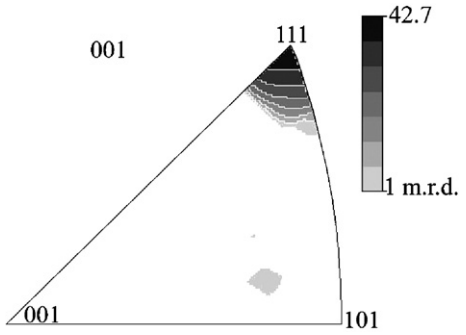

Fig. 7. 001 inverse pole figure for the direction of the sample normal. Cubic sector used, logarithmic density scale, equal area projection.

HR-TEM cross-sections images of the samples show a columnar growth of the deposited material. High-resolution images of the sample elaborated at 5 Pa O_2/N_2 (Fig. 8) strongly evidence the previously observed orientation with {111} $CoFe_2O_4$ planes ($d_{111} = 0.48$ nm) parallel to the sample surface.

The oxygen content measured by NRA for some cobalt ferrite films are shown in Table 1. The expected oxygen-to-metal ratio for stoichiometric $CoFe_2O_4$ cobalt ferrite is equal to 1.33. This value is reached for an 8 Pa O_2/N_2 pressure. Let us remark that the oxygen content of the cobalt ferrite films deposited in a 8 Pa O_2/N_2 (20:80) is higher than the one measured for films obtained in a 5 Pa pure O_2 atmosphere, although the O_2 partial pressure of the 8 Pa O_2/N_2 (20:80) is equivalent to a 1.6 Pa pure O_2 atmosphere, i.e,. lower than 5 Pa of pure oxygen. Therefore, the oxygen amount in the chamber is not the leading parameter for a good oxygen stoichiometry.

The surface of the films has been observed by AFM (Fig. 9). The root mean square roughness calculated over 5×5 μm^2 surfaces increases with the reactive gas pressures and is comparable for both O_2/N_2 and O_2 atmospheres (Fig. 10). Its maximum value is 2.0 nm and is observed for the highest pressures.

All the samples are ferromagnetic and their coercive field is of about 160 kA/m (2000 Oe) with the exception of the sample

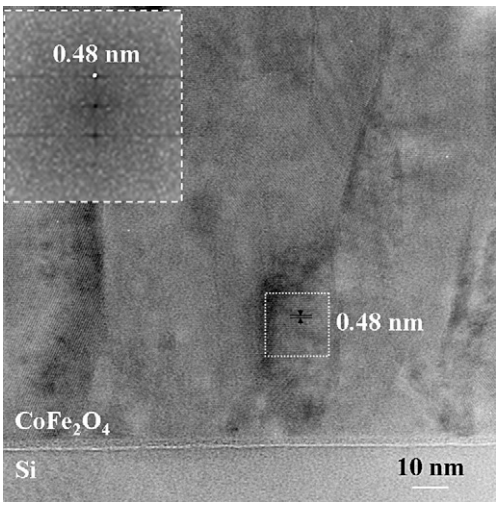

Fig. 8. HR-TEM image of $CoFe_2O_4$ cross-section for the sample elaborated in a 5 Pa O_2/N_2 pressure and its Fourier transform in insert.

Table 1
NRA results for cobalt ferrite films deposited from a $CoFe_2$ metallic target

Atmosphere	Pressure (Pa)	O/Co+Fe
O_2/N_2	0.5	1.08
O_2/N_2	8	1.33
O_2	5	1.12

elaborated in a 10 Pa O_2 pressure, which exhibits a superparamagnetic behaviour. The saturation magnetization increases with the deposition pressure and reaches, for 3 Pa O_2/N_2, an approximately constant value of 290 kA/m (290 emu/cm^3), smaller than that observed for the bulk 400 kA/m (400 emu/cm^3) [13, 14] but comparable to thin films elaborated in similar conditions [19]. The samples elaborated in O_2 follow the same trend with a slightly lower maximum magnetization value of 200 kA/m (200 emu/cm^3) reached 5 Pa.

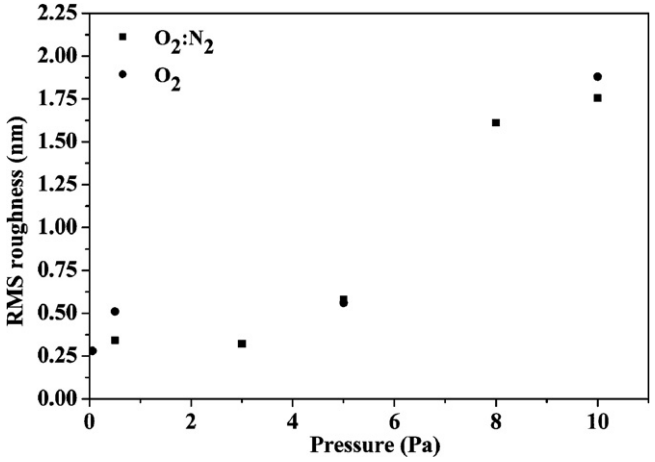

Fig. 10. Variation of the roughness with the pressures calculated for the films deposited in O_2/N_2 and O_2 atmospheres.

4. Discussion

Whatever the reactive gas used for the deposition, the same general texture trend is observed for the films. A preferred orientation with ⟨111⟩ directions of the spinel aligned with the films' normal occurs for low deposition pressures (=0.5 Pa). This preferred orientation is improved when O_2/N_2 pressure increases and disappears when the O_2/N_2 pressure reaches 10 Pa, contrary to what happens in oxygen gas for which more randomly oriented films are observed. The best ⟨111⟩ preferred orientation is observed for the O_2/N_2 mixture at a 5 Pa pressure. The quality of the films crystallisation is, on an average, better for O_2/N_2 than for O_2, as judging from the mean coherent domain sizes. This is coherent with increased oxygen content in O_2/N_2 prepared films as measured by NRA, larger crystallites being grown for higher oxygen contents in the unit cell.

Crystallisation of the $CoFe_2O_4$ phase on the substrate needs two competing fulfillments: bringing enough cationic and anionic species on the substrate, the former being decelerated in the ambient gas formed by the latter [26–28]. Too much O_2 at the level of the substrate, relative to the cation flux, may saturate crystal growth on oxygen faces, while too less would not allow the phase to form [29]. The substrate temperature is not high enough to favour ion migration throughout the layer. On another hand, the kinetic energy of the cations at the substrate may also perturb the growth, either on an orientational or crystallite size point of views. This energy is lower for high collision probabilities at higher O_2/N_2 gas contents.

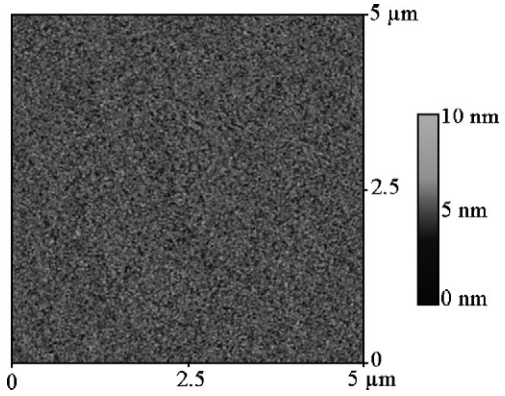

Fig. 9. AFM image of the surface of the film deposited in a 5 Pa O_2/N_2 pressure.

At low pressures, cation scattering on gas atmosphere is weak, and their kinetic energy at the substrate surface influences crystallisation and orientation by thermal transfers. In non-perturbed conditions, film crystallisation occurs as natural growing facies, provided enough O_2 is present in the atmosphere to form the $CoFe_2O_4$ phase. For such cubic crystal systems, the denser {111} planes are favoured, resulting in a regular columnar growth of the crystallites, with ⟨111⟩ directions along the films' normal. Since no heteroepitaxial relationship occurs, there is no common orientation of the crystal axes in the film plane, and a fibre texture develops. But cations' kinetic energy perturbs the growth, tends to activate other growing planes, and {311} orientation component is partially activated. In this pressure range (up to 5 Pa), increasing the O_2/N_2 pressure gives rise to a decreasing cationic kinetic energy and a more {111} natural growing and orientation.

This behaviour progressively vanishes when gas pressure increases beyond 5 Pa, a range for which the cationic species amount and speed at the substrate are limited. In such conditions, crystal growth decreases, as revealed by the mean coherent domain size reduction, and orientation schemes become more random due to insufficiently pronounced crystallisation.

In our previous experiments [19], in which the $CoFe_2O_4$ deposition was performed in O_2/N_2 at similar low temperatures with another excimer laser (XeCl, λ=308 nm with a 2 J/cm^2 fluence), the energy of the laser was not high enough to lead to any preferential orientation. The films were polycrystalline. If the substrate temperature is high enough to give sufficient mobility to the deposited species, the change of laser energy can be of much less importance on the crystallographic orientations obtained. Terzzoli et al. [18], for example, depositing at 700 °C, obtained ⟨111⟩ oriented $CoFe_2O_4$ for both a 266 and a 355 nm wavelength laser, with a very small degradation of the degree of orientation for the more energetic one.

As awaited, the oxygen contents measured by NRA increase with increasing reactive atmosphere pressures. The NRA results therefore allow to explain the variation of the lattice parameters as dependent upon the oxygen content of the lattice: the higher the oxygen content, the smaller the cell parameter. For the films deposited at low pressure ($P<8$ Pa) in one or the other reactive atmosphere, the O/(Co+Fe) value is lower than 1.33, which is the nominal value for $CoFe_2O_4$. Their lattice parameters are also larger than the bulk value. For the films deposited in 8 Pa O_2/N_2 atmosphere, the oxygen ratio O/(Co+Fe) is equal to 1.33 and the lattice parameter is around 0.840 nm, close to the bulk value (0.83919 nm). Contrary to what is observed for O_2/N_2 pressures, the oxygen content in the ferrite films deposited in pure O_2 is approximately constant whatever the used pressures. The incorporation of the oxygen in the cobalt ferrite lattice is surprisingly more efficient for an O_2/N_2 mixed gas than for pure O_2. Similar reactive gas pressures have then to be used for both gases and no gain in roughness is eventually performed with the use of O_2.

5. Conclusion

$CoFe_2O_4$ films with a strong (111) preferred orientation were elaborated on Si (100) at 300 °C by pulsed laser deposition of a $CoFe_2$ metallic target in different reactive atmospheres. The best results in terms of crystallinity and degree of orientation were obtained with the O_2/N_2 (20:80) reactive mixture at 5 Pa.

The low surface roughness and good magnetic properties of the thus elaborated films allow to consider their use in spin electronic devices.

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