

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.

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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/cm². 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/m³. The films were deposited onto 10 × 10 mm² (100) Si substrates in-situ heated at 300 °C. Before deposition, the substrates were cleaned with an HF (40%)–HNO₃ (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 ($\text{O}_2/\text{N}_2/20:80$) 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 ($\lambda_{\text{Co}}=0.1789$ 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 $\lambda_{\text{Co}}=0.15406$ 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 $^2\text{H}^+$ 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 $\langle 111 \rangle$ 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 $\langle 111 \rangle$ 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, $\lambda=308$ nm, with a fluence of 2 J/cm²) [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.

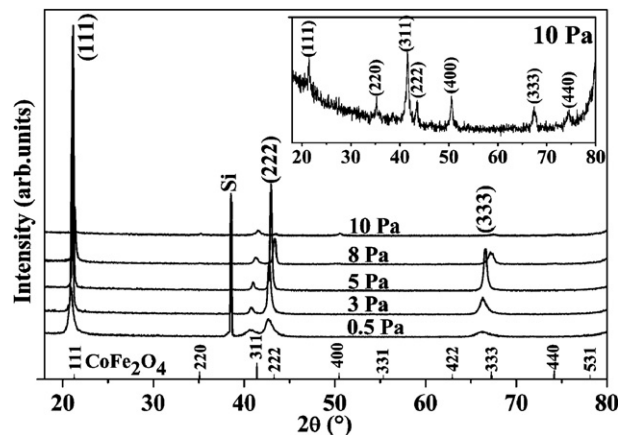


Fig. 1. X-ray θ – 2θ scans for CoFe_2O_4 films elaborated in O_2/N_2 .

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