



Co film stretching induced by lattice mismatch and annealing: The role of Graphene



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ABSTRACT

Thin Co films intercalated between a Graphene capping layer and the Ir(111) surface are of interest for spintronics applications due to their peculiar magnetic properties and to their chemical stability. The structure, and then the magnetic properties, of the Co films depend on the intercalation process which is strongly influenced by the temperature, total amount of Cobalt and quality of the capping Graphene layer. In order to identify and disentangle the effects of these contributions, we report on the structural characterisation of four Co films as a function of thickness, annealing temperature, and Graphene capping. From the structural point of view, the deposition of Co on a hot Ir substrate mimics quite well the intercalation process proving the validity of the colander model describing the Graphene role during the process.

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1. Introduction

Hcp Co is known to present a high intrinsic magnetic anisotropy [1]. In pure Co films thinner than a critical thickness $t_c \approx 14 \text{ \AA}$, the magnetic anisotropy drives the magnetization easy axis out of the film plane resulting into a perpendicular magnetic anisotropy (PMA) [2,3]. Co films showing PMA can be used to develop a new class of ultra-dense magnetic recording devices [4–8]. However, since the PMA is strongly linked to the structural and chemical properties of the film, a reliable response of these devices can be achieved only by preserving the Co film against oxidation and structural modifications.

The oxidation of the Co thin film can be avoided thanks to the chemical stability of a graphene (Gr) layer. Furthermore, Gr-covered Co films obtained through intercalation between Gr and Ir(111) [9] were also found to have higher t_c compared to uncoated films [3]. This interesting magnetic behavior may originate from chemical and structural instabilities of the intercalated layer, and motivated our previous structural characterization of Gr/Co/Ir sys-

tems [10]. For Gr-intercalated films, we observed the stretching of an in-plane lattice parameter and a broader Co-Ir interface with respect to bare Co films on Ir(111). These effects could not be reproduced on bare Co films upon annealing at the same temperature used for the intercalation ($T_{int} = 500 \text{ }^\circ\text{C}$), nevertheless, a thermally induced Co-Ir intermixing was observed at higher temperatures $T_{ann} \geq 600 \text{ }^\circ\text{C}$ [11] suggesting that the film thermal stability depends both on the temperature and on the local chemical environment of Co atoms [10,11].

A fine description of the film's structural evolution and of the parameters driving it, is still missing and requires further investigations. Here we focus on the role played by Gr, temperature, and amount of deposited Co.

Thanks to the complementary information yielded by Grazing Incidence X-ray Diffraction (GIXD) and X-Ray Reflectivity (XRR) measurements we could identify the role of each parameter in the Co-Ir interface structural evolution. Our results confirm the validity of the colander-like model proposed in [10] to describe the Gr role in the intercalation.

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2. Sample preparation and data acquisition

The sample preparation was carried out in the UHV chamber (base pressure $2 \cdot 10^{-10}$ mbar) coupled with a 6-circle diffractometer available in the second experimental hutch of the ID03 beamline (ESRF – Grenoble, France) [12]. The Ir(111) substrate was cleaned through several cycles of Ar^+ sputtering at 700 V and annealing at 850 °C. A type-K thermocouple spot welded near the sample surface was used to monitor the temperature. The surface quality was inspected by Auger Electron Spectroscopy (AES), X-Ray Reflectivity (XRR) and the peak width of the Crystal Truncation Rods (CTRs) of the substrate. The sputtering and annealing cycles were repeated until no contaminants were observed onto the surface and the width of the surface peaks indicated terraces with an average dimension exceeding 100 nm.

In the case of Gr-covered samples (Co/Gr), Gr was grown on the clean Ir(111) substrate by CVD: the Ir substrate was heated to 600 °C and exposed to ethylene at a pressure of $P_{C_2H_4} = 10^{-7}$ mbar. After 40 min the ethylene was pumped out and the sample was left to cool down. The Gr growth temperature was chosen to obtain a Gr layer with a high density of defects making the intercalation procedure possible at temperatures $T_{int} \simeq 500$ °C [9]. The two Gr-covered samples described in this study were prepared following the same procedure under the same conditions, aiming to produce two Gr layers of similar quality, i.e. with similar defects densities. Co was deposited on the Gr surface at room temperature (RT) using an electron bombardment evaporator. The evaporator calibration was performed using a quartz balance and verified with XRR by measuring the position of the Kiessig fringes [13]. All the evaporation parameters (voltage applied to the Co rod, current flowing in the filament, emission current and flux) were the same for every sample in this study. The different thickness of the samples was obtained using different the deposition times and was estimated by XRR (see below): the two films resulted to be 6 ± 1 Å (deposition time $t_d = 4$ min), and 12 ± 1 Å ($t_d = 8$ min). In the following we will refer to each sample by specifying the film deposition temperature (subscript) and thickness (in parentheses): the Gr-covered specimen will then be referred to as Co/Gr_{RT}(6) and Co/Gr_{RT}(12).

Two bare films were deposited directly on the Ir(111) while keeping the substrate at room temperature (RT) for 8 min and at $T_{dep} = 500$ °C for 6 min. The former resulted to be 14 ± 1 Å, the latter 10 ± 2 Å. In the following it will be labelled as Co_{RT}(14) and Co_{500 °C}(10), respectively.

After deposition, Co_{RT}(14) was heated to 300 ± 20 °C for 5 min. Once cooled down, a second annealing at 500 ± 20 °C for 5 min was performed to probe the thermally-induced modifications on RT-deposited films. After each experimental step (Ir cleaning, Gr growth, Co deposition, intercalation/annealing) XRR and GIXD measurements were carried out using a 2×2 MAXIPIX detector [14]. X-ray measurements were performed using an incident X-ray beam of 11 keV, i.e. just below the L-edge of Iridium, to avoid the fluorescence of the substrate and limit the background level. The beam was focussed using the toroidal mirror of the beamline to the size of 54×30 (H \times V) μm^2 . Raw data integration was performed through the BINoculars code [15,16]. XRR measurements were fitted using the GenX code [17] to evaluate the film thickness, density, and roughness, together with the dimension of the interface with the Ir substrate. GIXD data (intensity as a function of the reciprocal space coordinate parallel to the surface, H) are presented in Fig. 1. The hexagonal lattice unit cell of the Iridium surface has a lattice parameter of $a_{Ir} = 2.715$ Å at room temperature, and its diffraction peak is observed at $H = 1$ r.l.u. (reciprocal lattice units). Hcp Co, has a bulk lattice parameter of $a_{Co} = 2.507$ Å corresponding to $H = 1.08$ r.l.u. (indicated by the black line in Fig. 1) and will be used as a reference in our analysis.

3. Results

We first discuss the GIXD data obtained from the Co/Gr_{RT}(6) and Co/Gr_{RT}(12) samples (Fig. 1a and b, respectively). The diffraction from the pristine films (dashed blue lines) presents a peak at the expected position for Ir ($H = 1$ r.l.u.). In the region where we expect peaks from the Co film, a faint peak is barely visible for Co/Gr_{RT}(6) sample, while an evident but broad feature can be noticed for Co/Gr_{RT}(12). In our previous observations [10], we showed that Co deposition on Gr results in a granular film made up of small, disordered clusters. Such structures diffract in faint and barely detectable cones. This may explain why we do not observe a distinct Co diffraction peak for the pristine films.

The data collected after intercalation on both samples (red solid lines) show the Co peak appears (Co/Gr_{RT}(6)) and increases in intensity (Co/Gr_{RT}(12)) indicating an improved crystalline structure. Both peaks maxima are found at H value smaller than the reference of bulk Co $H = 1.08$ r.l.u., this is a sign of an in-plane stretched lattice. This expansion can originate from surface strains due to the lattice mismatch with the underlying Ir, and can be related to the formation of a Co-Ir alloy layer [10]. The alloy formation upon intercalation is also supported by the appearance of the feature in the Ir region of Fig. 1a: the broader Ir peak presents a small shoulder at $H = 1.01$ r.l.u. This can be associated with an Ir phase having a lattice parameter shorter than the bulk Ir. This, together with the Co peak shift, is consistent with an intermixed transition region between the substrate and the film. The increased intensity in the region between the two peaks provides a further indication of the increased disorder, compatible with a broad Co-Ir interface.

Despite the preparation of the Co/Gr_{RT}(6) and the Co/Gr_{RT}(12) samples was the same from the substrate cleaning to the Co intercalation, the thinner film exhibits wider modifications, proving the lattice stretching/alloying is thickness dependent.

The modifications found upon intercalation can be associated with high temperatures used to intercalate the Co film and/or with the presence of the Gr layer. In order to identify and isolate the thermal effects, we remove the Gr layer and study the structural evolution of films deposited directly on Ir(111) following the same preparation procedure used for Gr-capped films: RT-deposition followed by a 5 min annealing at 500 °C. The comparison of the sample Co_{RT}(14) sample to the Co/Gr_{RT}(12) highlights the Gr effects, while minimizing the thickness dependence.

The GIXD data presented in Fig. 1c refer to the Co_{RT}(14) sample. The pristine film (dashed blue line) shows a Co peak in vicinity of the black line, i.e. having a lattice parameter compatible with that of bulk Co. This suggests that Co lattice arranges differently on Ir with respect to Gr, as it was also observed by STM [10]. The Co peak is narrower and more intense than in the case of Gr/Ir (compare panel c to panels a and b), meaning this film is more ordered. After annealing at $T_d = 500$ °C (red line with circles superimposed), the Ir peak remains unchanged, while in the intermediate region a reduced intensity indicates a sharper difference between Co and Ir lattices. The Co peak position is stable while its intensity increases and its width reduces. This indicate a thermally-induced enhancement of the Co film crystalline ordering. The small shoulder observed on the low-H side of the Co peak suggests the presence of a larger crystalline phase, most likely lying at the Co/Ir interface and accommodating the strain induced by the underlying Ir.

Despite the Co deposition on the bare substrate was performed under the same conditions as for the intercalated samples, the Co structural response observed in Co/Gr_{RT} samples (lattice expansion and interface broadening) is not reproduced by annealing of the bare Co films. This implies Gr plays a fundamental role in the structural modification of the interface between intercalated film and substrate.

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