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PII: S0169-4332(17)31401-0
DOI: http://dx.doi.org/doi:10.1016/j.apsusc.2017.05.089
Reference: APSUSC 36024

To appear in: *APSUSC*

Received date: 31-1-2017
Revised date: 8-5-2017
Accepted date: 10-5-2017

Please cite this article as: Giulia Avvisati, Pierluigi Mondelli, Pierluigi Gargiani, Maria Grazia Betti, Graphene-Mediated Interaction between FePc and Intercalated Cobalt layers, *<![CDATA[Applied Surface Science]]>* (2017), http://dx.doi.org/10.1016/j.apsusc.2017.05.089

- The structural evolution for the Gr/Co system, as a function of the intercalated Co layer thickness, is herewith investigated;

- the flat-lying adsorption geometry of FePc molecules deposited on Gr/Co substrate, with different degree of corrugation, is determined;

- an interaction mechanism, between the Fe molecule centers and the intercalated Co layer(s), is proposed to be determined by a delicate balance between lateral electric dipoles (due to the rippling of the Gr sheet) and Fe/Co distance.

Graphene-Mediated Interaction between FePc and Intercalated Cobalt layers

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Abstract

The interaction strength of FePc molecules adsorbed on graphene-Co, obtained by intercalation of Co layer(s) under graphene (Gr) grown on Ir(111), has been investigated by core level photoemission and X-Ray absorption spectroscopy. Upon intercalation of a single layer of Co beneath Gr, the Co atoms are pseudomorphic with the Ir(111) lattice, inducing a rippled Gr layer with valleys and hills regions. FePc molecules, adsorbed on Gr/1 ML Co, are trapped in the valleys regions and the Fe metallic centers strongly interact with the Gr/Co system. The intercalation of further Co layers leads to a relaxation of the Co lattice up to the formation of a commensurate (1×1) Gr/Co system. The Fe centers of the molecules adsorbed on the flat commensurate Gr/Co are found to be less interacting. We have determined that, even though the organic macrocycles of the FePc molecules, flat lying on the Gr/Co system, are completely decoupled by the presence of the Gr intralayer, the Fe-Co interaction is mediated by the graphene spacer and dependent on Gr corrugation.

Keywords: Graphene, Cobalt, Intercalation, Phthalocyanine, Photoemission spectroscopy, Absorption spectroscopy

Preprint submitted to Applied Surface Science *May 8, 2017*

1. Introduction

Magnetic organic molecules are excellent candidates to design novel spintronic devices for information processing [1] and storage [2]. High magnetic anisotropy and stable magnetic state of the molecule, up to device working temperature, are mandatory requirements for these purposes. Magnetic substrates, coupled with metal porphyrines and metal phthalocynines (M-Pc), could stabilize the magnetic response up to room temperature [3, 4, 5]. However, molecule-metal interactions at the interface can strongly alter the electronic [6, 7]/magnetic [8] properties of the molecules.

A Gr sheet, interposed between the molecules and the metal surface, can electronically decouple the adsorbed M-Pcs [9, 10] while increasing their magnetic anisotropy [11]. Ferromagnetic (FM) metals, deposited at room temperature on Gr, can be easily intercalated by thermal annealing at high temperature [12, 13, 14, 15], taking advantage of pre-existing defects in the Gr sheet or via metal-induced defects and healing processes [16]. In particular, intercalation of Co (Fe) has proved to induce a magnetic activity in the Gr sheet [12] as well as tuning the height modulation of the gently-corrugated Gr/Ir moiré superstructure [13, 14, 15]. Highly corrugated moiré superstructures are ideal templates to induce molecule trapping, due to the presence of lateral dipoles/electric fields generated by the local contraction and expansion of the lattice and, therefore, of the density of charge in the Gr sheet [17]. For these systems, a definite interaction between the molecules and the intercalated metal is mediated by the rippled Gr spacer. Recently, we have investigated the FePc adsorption on Gr/Ir(111) intercalated with a single layer of Co, arranged pseudomorphic with the Ir(111) surface [18]. The Co layer, in registry with the underlying Ir(111) [13, 19], induces a pronounced moiré corrugation of the Gr layer (1.2-1.8 Å for Gr/1 ML Co [13], to be compared with 0.4 Å for Gr/Ir [20]), due to stronger C-Co(Fe) interaction than C-Ir. The FePc molecules are first trapped into the valleys of the moiré superstructure and a definite interaction of the Fe centers with the underlying Co is mediated by the Gr layer [18]. After further intercalation, the Co film structure starts to relax and recover its bulk lattice parameter, with less than 2% [21] lattice mismatch with Gr unit cell, and the Gr/Co interface releases the corrugation and becomes flat. In order to better understand the driving forces of the FePc-Gr-Co interaction, herewith we investigate the

FePc adsorption on Gr, as a function of Co intercalation and, accordingly, at decreasing rippling levels, focusing on the flat commensurate Gr/Co system. Core level photoemission and near-edge absorption spectroscopy investigation of the metallic Fe centers unveil a weakening of the interaction strength for FePc molecules adsorbed on Gr in registry with the Co multilayer, when the corrugation is attenuated.

2. Experimental Methods

Near-edge X-ray absorption fine structure (NEXAFS) measurements were performed at the BOREAS beamline of the Alba synchrotron radiation facility (Barcelona, Spain). The data were collected in the total electron yield (TEY) mode and normalized by the incident flux measured from a gold nanomesh. NEXAFS spectra were acquired in transverse magnetic (TM) and transverse electric (TE) polarizations, by changing the phase of the elliptically polarizing undulator (EPU), in an experimental geometry analogous to Ref. [18] and sketched in the top panel of Fig.4. The core level spectra were acquired at the LoTUS surface physics laboratory in Sapienza University of Rome, with Mg K-α radiation (1256.6 eV) and photoemitted electrons are collected in a normal emission geometry, with a VG Microtech Cam-2 hemispherical analyzer with an overall resolution of 1 eV. The laboratories are equipped with analogous ancillary facilities for sample preparation. The procedures adopted for Ir(111) surface cleaning and growth of the Gr layer are fully described in Ref. [18]. Metallic Co, evaporated with a conventional electron beam source, was deposited on the Gr sheet and annealed at 500-800 K for several minutes to favor intercalation of the Co atoms, following Ref. [12, 13, 15]. The Co nominal coverage, determined with a quartz crystal microbalance, was double-checked by evaluating the Ir 4f core level attenuation (at the LoTUS laboratory) or the jump-edge ratio at the Co $L_2, 3$ absorption edge, pre-calibrated with Auger Electron Spectroscopy at the BOREAS beamline. Commercially available AlphaAesar FePc molecules ($\rho = 1.52\ g/cm^3$) were evaporated with a home-made resistively heated quartz crucible, at a rate of 0.30 ± 0.05 Å/min, measured with a quartz crystal microbalance.

3. Results and Discussion

The LEED patterns of Gr on Ir(111), after the intercalation up to six Co layers, are reported in Figure 1. Graphene, grown on the Ir(111) surface,

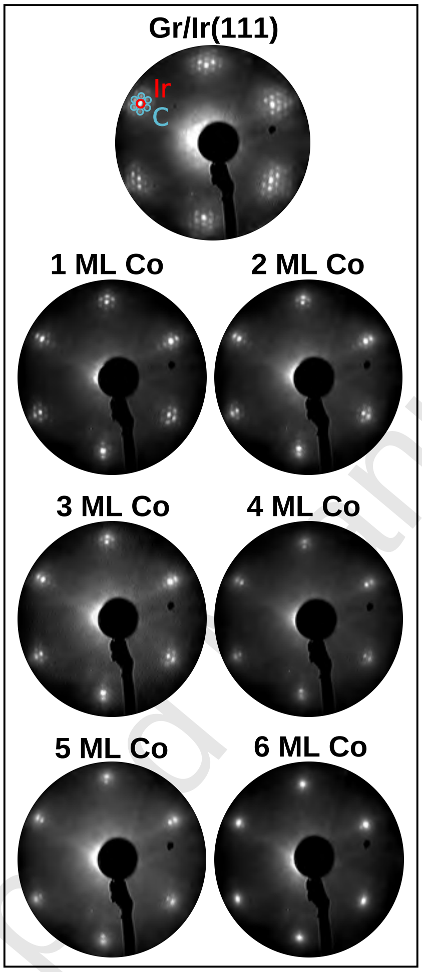

Figure 1: LEED patterns (primary beam energy=140 eV) evolution at increasing number of intercalated Co layers of Gr/Ir(111). A single Co layer is added for each step.

exhibits a moiré superstructure due to the lattice mismatch between the Gr (2.45 Å) and Ir(111) (2.72 Å) lattice parameters. Accordingly, the LEED pattern (Figure 1, top) presents the main spots of the underlying hexagonal Ir(111) surface, surrounded by the satellite spots of the C atoms arranged in a (9.3×9.3) moiré superstructure [22]. The moiré superstructure, due to the corrugated graphene, is preserved and presents the same periodicity and symmetry after the intercalation of the first Co layer. Further Co intercalation induces an attenuation of corrugation along with a progressive and gradual smearing out of the satellite spots up to 4-6 ML of Co, when the mismatch between Gr and Co starts to release and the hexagonal pattern overcomes. Finally, at the completion of the sixth layer, the LEED pattern is a well defined hexagonal (1×1) structure, i.e. the Gr layer is commensurate with the underlying Co substrate [15] and, contrary to previous measurements [23], mostly monodomain. When FePc molecules are adsorbed on Co-intercalated Gr, the symmetry and periodicity of the moiré is unperturbed [18]. The inner and outer spots, characteristic of the superstructure, maintain their distribution and relative distances, thus demonstrating that the adsorption of a single layer of FePc does not modify the Gr moiré periodicity with Co intercalated layer(s), as observed also for FePc on Gr/Ir(111) [24]. Similarly, the (1×1) LEED pattern of flat Gr/6 ML Co is preserved after deposition of a single layer FePc, as reported in the inset of Figure 2.

The adsorption geometry of FePc molecules on commensurate Gr/Co can be clarified by polarization-dependent absorption spectroscopy, performed at the N K edge (Figure 2). The spectrum can be divided into two energy regions: from 397 to 405 eV, with transitions mainly of π^* character, and above 405 eV, where the σ^*-symmetry transitions dominate the spectrum, in agreement with the existing literature [9, 18, 25]. The dichroic response of the system to linearly (TM and TE) polarized radiation reveals a planar geometry of the FePc molecular units adsorbed on the flat commensurate Gr/Co system, as observed also on both low [9] and highly corrugated [18] Gr sheets. Moreover, the absence of residual signal, due to intermixing of σ and π state in the lower energy region of the spectrum, confirms the decoupling effect of the graphene layer, avoiding any re-hybridization of the organic macrocycles. Indeed, a molecular distortion of the π^* region of the spectrum was distinctly observed for FePc molecules directly deposited on epitaxial Co(001) films [26], due to a significant mixing of the molecular orbitals located on the pyrrole macrocycles with the underlying metallic states [26]. The presence of the graphene buffer layer in FePc/Gr/Co prevents the coupling of the ligand macrocycles with the intercalated Co layer(s) at different degree of corrugation [18], as also observed for FePc/Gr/Ir(111) [9].

Although the organic macrocycle is decoupled by the Gr spacer, an interaction channel involving the central metal atom was noticed for M-Pc on Gr/Ni(111) [27, 28] and for FePc molecules adsorbed in the valley regions of the Gr/1 ML Co moiré superstructure [18]. In order to better understand the role of graphene corrugation in the interaction process between the Fe centers and the underlying Co, NEXAFS and XPS measurements have been

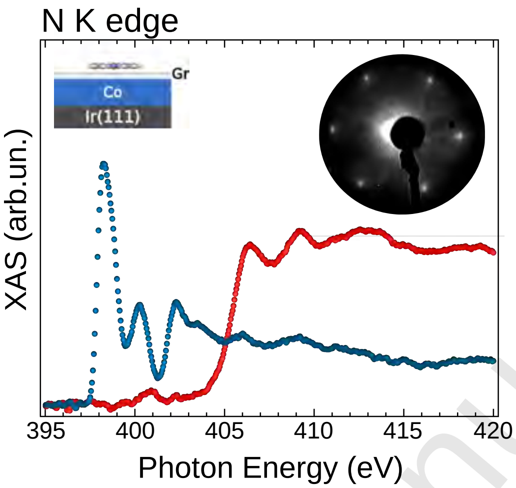

Figure 2: Polarization-dependent, TE (red) and TM (blue), absorption spectra at the N K-edge of 0.4 ML FePc/6 ML Co/Ir(111). Inset: LEED pattern for FePc adsorbed on flat Gr/Co, acquired at liquid nitrogen temperature with a primary beam energy of 140 eV.

performed at the Fe L_3 absorption edge and at $2p_{3/2}$ core level of FePc at different intercalated Co layer thickness.

The XPS spectra of the Fe $2p_{3/2}$ core level for FePc molecules adsorbed on Gr/Co/Ir(111), at increasing thickness of the intercalated Co layer, are reported in Figure 3, in comparison with molecular adsorption on the gently corrugated Gr/Ir(111). The broad and asymmetric lineshape of the Fe $2p_{3/2}$ core levels, centered at 709.5 eV, results from the multiplet structure of the Fe^{2+} central metal ion [6, 29]. In the fitting procedure, so-called partially resolved Zeeman splitting, four-fold m_J splitting $(\frac{3}{2}, \frac{1}{2}, -\frac{1}{2}, -\frac{3}{2})$ are accounted for two non-resolved Gaussian peaks located at 709.5 eV and 711.5 eV binding energy (BE), whereas a broader satellite is centered at higher BE [6]. The described fitting procedure has been applied to all the spectra reported in Figure 3. A signature of possible charge transfer and/or orbital intermixing, involving the intercalated Co layer and the central Fe atom of the molecules, can be revealed by the presence of an extra-component in the Fe $2p_{3/2}$ core level spectrum [6, 18, 30, 31]. The evolution of the Fe $2p_{3/2}$ core level lineshape indicates that a charge transfer, from the Gr/Co system to the central Fe ion, only occurs when the FePc molecules are adsorbed on the highly corrugated moiré superstructure of Gr/2 ML Co, but not when

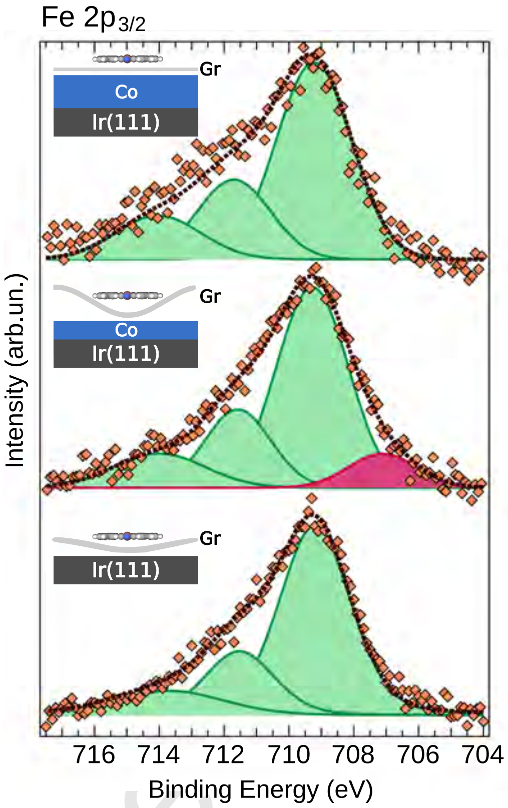

Figure 3: Fe2$p_{3/2}$ core level evolution at increasing intercalated Co layer thickness. In particular, from bottom to top: 1.3 ML FePc on Gr/Ir(111), 1.8 ML FePc on Gr/2.3 ML Co, 1.2 ML FePc on Gr/5.0 ML Co.

the molecules are deposited on a gently corrugated moiré (Gr/Ir) or on a flat commensurate (Gr/Co) system. This evidence confirms recent results on the Gr-mediated interaction between FePc molecules, adsorbed in the valley region of the highly corrugated Gr moiré superstructure on the intercalated single Co layer [18].

NEXAFS measurements at the Fe L_3 absorption edge for FePc adsorbed on Gr/Co/Ir(111) system have been performed to verify the role of Fe metallic centers in the interaction process with the underlying Co layer(s). The horizontally- (violet) and vertically-polarized (green) light absorption spectra of 0.4 ML FePc adsorbed on flat Gr/6 ML Co and corrugated Gr/1MLCo are shown in Figure 4, in comparison with a single layer FePc on Gr/Ir(111)

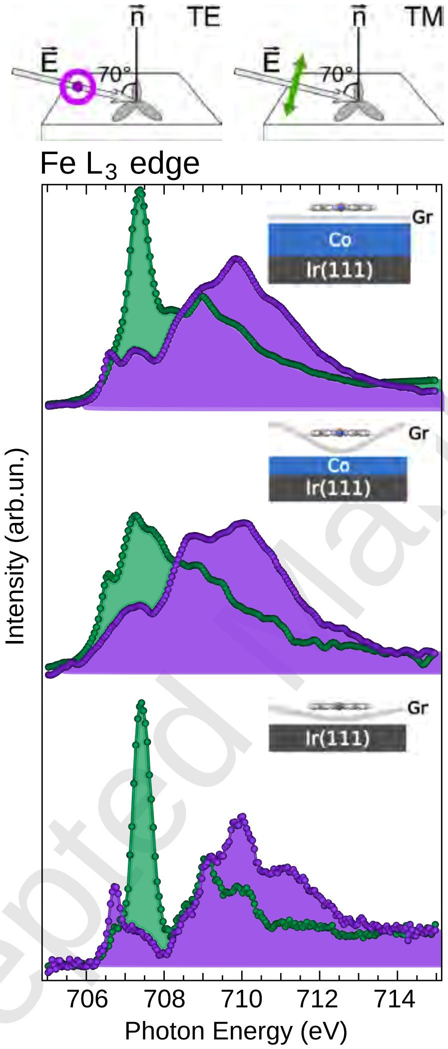

Figure 4: Absorption spectra for TE (violet curves) and TM (green curves) light polarization (see sketch in the top panel for the experimental geometry), acquired at the L_3 edge, for less than 1 ML FePc adsorbed on: Gr/Ir(111)from ref.[9], bottom spectrum; Gr/1 ML Co/Ir(111), central spectrum, and on Gr/6 ML Co/Ir(111), top spectrum.

(from Ref.[9]). Indeed, for FePc/Gr/Ir(111) the molecule-metal decoupling has been proved to be effective and the multiplet distribution of the Fe states reflects the polarization dependence of the molecular orbitals of a thin FePc film [32]. The multiplet structure in the Fe L_3 absorption edge of FePc molecules is due to the splitting of the 3d orbitals because of the ligand field interaction in the D_{4h} symmetry group. Accordingly, thanks to the planar adsorption geometry, the peaks in the TE and TM polarized absorption spectra for FePc/Gr/Ir(111) can be ascribed to transitions mainly to d states perpendicular (e_g, a_{1g}) and parallel (b_{1g}) to the molecular plane, in agreement with a $3E_{1g}$ symmetry ground-state configuration. The most intense contribution in the TM spectra, located at 707.5 eV, arises from transition to the out-of-plane a_{1g} state of d_{z^2} symmetry, while the two structures at 706.5 and 709.0 eV are assigned to the two e_g states, with $d_{xz,yz}$ symmetry. In the TE spectra the in-plane orbitals are probed, and we recognize the e_g states together with two b_{1g} states, respectively located at 710.0 and 711.3 eV, with a dominant $d_{x^2-y^2}$ character [32, 33, 8]. The intensity of the different contributions in the absorption spectrum for FePc adsorbed on both highly corrugated and flat Gr sheets, will unravel the interaction strength at different level of moiré corrugation.

A filling of the out-of-plane molecular orbitals is expected when a charge transfer from the substrate to the molecules occurs, causing a reduction [18], or even a quenching [8, 33], of the out of plane molecular orbitals. In particular, while the in-plane states seem less involved in the interaction process, the out-of-plane molecular orbitals for FePc adsorbed on the valley sites of Gr/1 ML Co are almost totally quenched, confirming a stronger interaction between the central metal ion of the molecules and the Co, when the molecules are trapped in the valley regions. On the other side, if the FePc molecules are deposited on a flat commensurate Gr/Co substrate, the out-of-plane molecular orbital only partly recover their intensity, indicating that a Fe-Co intermixing is still present after the relaxation of the moiré corrugation. Though the presence of graphene buffer layer, which completely decouples the organic macrocycles, the Fe centers are still interacting with the Co intralayer in all the adsorption configurations here investigated. The graphene-mediated FePc-Co interaction depends on the Gr rippling and it is driven by a subtle interplay between the molecular adsorption site induced by the lateral dipoles in rippled Gr substrate and the distance from the metal under the graphene spacer [20].

4. Conclusions

We have investigated the adsorption of FePc molecular units on Co layer(s), intercalated under a rippled Gr spacer at increasing Co thickness. In agreement with previous results, the Co intercalated Gr/Ir surface exhibits a moiré pattern with a decreasing rippling for increasing thickness of the intercalated Co film. The surface potential for molecular adsorption is altered for the intercalated systems compared with the bare G/Ir. In particular, when Co is commensurate with Gr (more than six layers of Co), the FePc are flat lying and the organic macrocycles are decoupled by the Gr layer from the intercalated metal, while a weak interaction of the Fe centers is revealed by the Fe absorption spectra. The strength of Fe centers interaction with the Co increases on highly corrugated moiré superstructures, when few layer of Co are intercalated (less than six Co layers). The interaction with the intercalated Co layers, driven by out-of-plane ligand splitted molcular orbitals, is mediated by the Gr spacer. The interaction mechanism is a delicate balance between the effect of lateral dipoles of the highly-corrugated moiré, inducing a trapping of the FePc molecules in the valley regions, and the distance of C atoms from the Co template, thereby allowing for locally different electronic coupling. Tuning the graphene rippling can drive the interaction between molecular units and Co magnetic layers, therefore can influence the electronic and magnetic state of the molecular network.

5. Acknowledgments

The authors thanks the BOREAS (Alba-Spain) beamline staffs for the experimental collaboration and Iolanda Di Bernardo for the experimental support for the LEED measurements. GA acknowledges funding support from University La Sapienza with grant: Avvio alla Ricerca 2016, Type 1.

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