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Oxygen-induced immediate onset of the antiferromagnetic stacking in thin Cr films on Fe(001)

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We investigated the magnetic coupling of ultra-thin Cr films grown at 600 K on a Fe(001)- $p(1 \times 1)O$ substrate by means of spin-polarized photoemission spectroscopy. Our findings show that the expected antiferromagnetic stacking of the magnetization in Cr(001) layers occurs right from the first atomic layer at the Cr/Fe interface. This is at variance with all previous observations in similar systems, prepared in oxygen-free conditions, which always reported on a delayed onset of the magnetic oscillations due to the occurrence of significant chemical alloying at the interface, which is substantially absent in our preparation. © 2015 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4918987>]

The Cr/Fe(001) interface is one of the most studied in solid state physics, mainly because of its model character as an antiferromagnetic (AF)/ferromagnetic (F) heterostructure,¹ its original importance for the discovery of the giant magneto resistance effect,^{2,3} and its leading role in applications for spintronic devices.⁴ The magnetic properties of such a system are affected by the interplay of several phenomena, such as Cr/Fe interface mixing and dimensionality. If the growth of ultra-thin layers is performed on Fe substrates kept at relatively high temperature (500–600 K), chemical mixing precludes the formation of a well defined interface.⁵ Keeping a high substrate temperature during growth is however beneficial to allow for the reorganization of atoms, leading to a smoother surface.

Many research teams studied the spin polarization of Cr layers on Fe(001) as a function of coverage, since the first observations of two different oscillation periods (2 and 10–12 Cr layers, respectively, see Ref. 6) for the relative Fe magnetization coupling (either F or AF) in Fe/Cr/Fe(001) trilayers with increasing Cr coverage. In all cases, it has been found that such oscillations start with a delay of a few monolayers (ML). This has been ascribed to chemical mixing at the Cr/Fe interface, particularly enhanced when employing high growth temperatures.⁵

Several studies have been able to demonstrate that an AF coupling occurs between the Cr overlayer and the Fe substrate, being the Cr {001} sheets antiferromagnetically coupled to each other and to the Fe substrate.^{7–9} On the other hand, on account of the aforementioned interface mixing, the magnitude of the magnetic coupling at the Cr/Fe interface has been reported to be considerably weakened, and a clear AF stacking of the Cr layers is recovered only above about 5 ML,^{10,11} interestingly at correspondence with the onset of the Fe magnetization oscillations in the trilayer system.

The presence of such a relatively extended delay of the interface magnetic coupling represents a severe drawback for applications, since nowadays the tendency in state-of-art technology is aimed towards miniaturization and the fine control of interfacial properties is a crucial issue in low-dimensional

systems. By means of spin-polarized photoemission spectroscopy (SP-PES), here we show that, by growing Cr on an oxygen passivated Fe surface, namely, Fe(001)- $p(1 \times 1)O$,^{12–14} an immediate onset of the AF stacking of the Cr layers takes place. This is at variance with the case of the oxygen-free Cr/Fe interface. This behavior is related with the abrupt character of the Cr/Fe- $p(1 \times 1)O$ interface, which shows negligible chemical mixing.^{15–17}

The samples were prepared, as described in more detail elsewhere,¹⁸ by first growing a thick Fe film onto an MgO(001) crystal through molecular beam epitaxy (MBE). The Fe(001) surface was exposed to 30 L of pure molecular oxygen (1 L = 1.33×10^{-4} Pa·s) after a 700 K annealing and then flash heated at 900 K to remove the oxygen in excess of 1 ML from the surface. This procedure leads to the formation of the well-characterized Fe(001)- $p(1 \times 1)O$ surface,^{14,19–24} in which each unit cell has one oxygen atom in the fourfold symmetrical Fe hollow site. Cr films were incrementally grown on Fe(001)- $p(1 \times 1)O$ by MBE, keeping the sample at about 600 K during the depositions. The deposition rate (~ 0.05 ML/min) was monitored by a quartz microbalance.

In such growth conditions (in particular, at such a relatively high temperature), mixing involving at least three substrate layers has been reported for the oxygen-free Cr/Fe surface.^{25,26} In our case, where, instead, the Fe(001) surface has been previously oxygen-passivated, scanning tunneling microscopy revealed that, below 1 ML coverage, two different self-assembled Cr phases [namely, a $c(4 \times 2)$ one in the 0.2–0.75 ML range, and a $(\sqrt{5} \times \sqrt{5})R27^\circ$ reconstruction over 0.8 ML of Cr] are stabilized. In such phases, no interdiffusion between the two Fe and Cr chemical species at the interface was observed.¹⁸ Moreover, after Cr deposition at 600 K, oxygen atoms are no more bound to Fe but form a monolayer-thick Cr oxide for submonolayer Cr thicknesses,¹⁸ while they float onto the topmost Cr layer for higher coverages,²⁷ leaving therefore the Cr/Fe interface in ideal conditions. Such a behavior, related to the well-known surfactant action of oxygen during growth of metallic films, is common to many other layered systems (see, e.g., Ref. 19).

The magnetic characterization of the Cr/Fe(001)- $p(1 \times 1)O$ system, with a Cr thickness up to about 6 ML, was

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accomplished by means of SP-PES. The spectra were obtained with a dedicated apparatus, equipped with a micro-Mott detector coupled to a hemispherical electron analyzer,²⁸ and with a He discharge lamp as photon source (He I photon energy $h\nu = 21.2$ eV). Samples were magnetized by a current pulse sent to a coil, producing a magnetic field of a few hundred Oe (to be compared with the rather low coercive field of our Fe films, which is below 5 Oe (Ref. 29)) parallel to one of the easy axes of the sample, which lie along one of the equivalent in-plane $\langle 100 \rangle$ directions. All spectra were acquired in magnetic remanence and at room temperature. We underline that, for oxygen-free Cr/Fe bilayers, Cr thin films were observed to retain a long-range magnetic ordering even for quite higher temperatures.¹⁰ The effect of instrumental asymmetries was averaged out by acquiring spin-polarized spectra with the sample magnetized in opposite directions. The relative intensities of the two spin channels are obtained by rescaling through the known value of the polarimeter Sherman function S (for our micro-Mott detector $S = 0.14$, see Ref. 28) following well-established methods.³⁰

In valence band (VB) photoemission spectra, Cr and Fe states overlap, and a complete separation of the two is hardly feasible.^{31,32} In order to better evidence the contribution of the Cr states, a series of spin-integrated VB spectra was acquired with increasing Cr coverage, as reported in Fig. 1. From an inspection of such results, it is possible to evidence

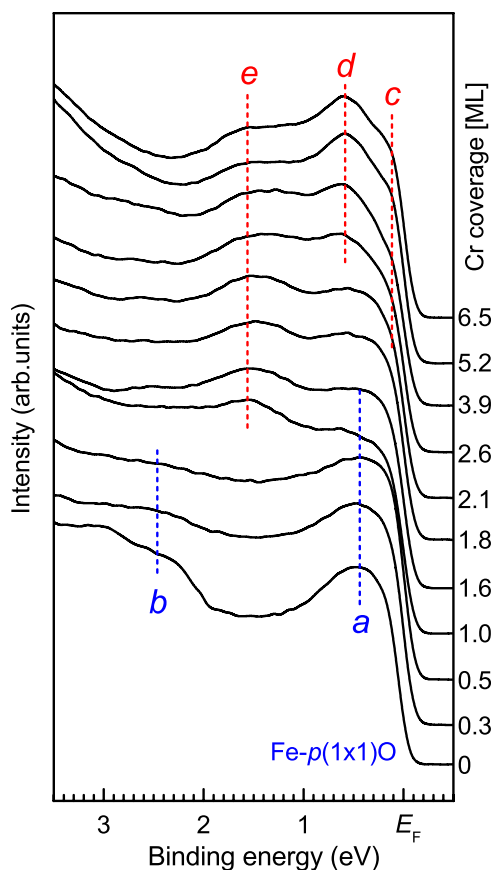


FIG. 1. Spin-integrated VB spectra taken at a pass energy of 0.5 eV on the Fe(001)- $p(1 \times 1)$ O substrate and on the Cr/Fe(001)- $p(1 \times 1)$ O system for increasing Cr coverage. He I source satellites were subtracted in all cases. Lowercase letters and dashed vertical lines mark the position of relevant features.

some relevant features, labeled with letters from *a* to *e*, attributed to either Cr or Fe VB states. Features due to states attributed to oxygen lie at a binding energy (BE) larger than 4 eV. Features *a* and *b* are related to Fe(001)- $p(1 \times 1)$ O. In particular, peak *a* can be associated to bulk Fe 3*d* states and has both majority and minority spin characters.^{13,28} Feature *b* can instead be associated with a surface resonance from the same Fe states.¹³ Features *c*, *d*, and *e* are due to states of the Cr overlayer. In particular, peak *c* is located at 0.15 eV BE and was reported as derived from Cr 3*d* states, for both oxygen-free and oxidized Cr surfaces.^{33,34} Features *d* (at about 0.6 eV BE) and *e* (at about 1.6 eV BE) are instead characteristic of oxidized Cr surfaces.^{33,35}

Both features *d* and *e* are clearly dependent on Cr thickness. On the other hand, the presence of feature *e* is evident even for the lowest Cr coverages, as visible in the 1 ML Cr spectrum of Fig. 1. The latter is therefore the best candidate to look at in order to find a Cr-derived spin-dependent state which can be exploited to characterize the magnetization of the Cr film.

This choice is validated by inspection of spin-resolved VB spectra. A selection of SP-PES spectra is reported in Fig. 2 for Fe(001)- $p(1 \times 1)$ O (Fig. 2(a)) and for Cr films of 1 ML (Fig. 2(b)) and 3.9 ML (Fig. 2(c)) thicknesses. The spin-resolved spectra of Fe(001)- $p(1 \times 1)$ O, already reported in Ref. 28, provide a reference to assign majority and minority spin character to the measured spectra, as a function of the applied magnetic field direction. When inspecting the spin-resolved spectra in Figs. 2(b) and 2(c), it appears clear that the Cr-derived feature at 1.6 eV BE (feature *e* in Fig. 1) lies in the minority electron spectrum in Fig. 2(b) and in the majority one in Fig. 2(c). This structure is thus spin-polarized, and its spin character is prevalently of minority type for 1 ML Cr thickness and of majority type for 3.9 ML Cr thickness. This indicates that the Cr film has a long range magnetic order and that its magnetization direction is thickness dependent. In particular, the measurements reported in Fig. 2(b) confirm that the first Cr layer (that is actually a monolayer Cr oxide, as remarked above) is magnetized in plane, with a magnetization oriented anti-parallel with respect to that of the Fe substrate.⁷

A typical parameter related to the average direction of the magnetic moments of the Cr layers is the polarization, which in the present case is calculated through the formula $P(1.6 \text{ eV}) = \frac{I^+ - I^-}{I^+ + I^-}$. Here, I^+ and I^- are the intensities of the spin-resolved spectra at 1.6 eV BE, for the majority and minority spin channels, respectively. The measured values of P are plotted as a function of the Cr coverage in Fig. 3.

Starting from the bare Fe(001)- $p(1 \times 1)$ O substrate, P rapidly decreases, reaching a minimum at 1 ML Cr. It then starts to increase again towards a maximum reached at 2 ML, and then an oscillation is established, with alternating maxima and minima at each ML completion. In a very simplified view based on a perfect layer-by-layer growth and AF stacking, the polarization values are given by the contribution of the attenuated substrate and the deposited Cr layers. One might expect that at 3 Cr ML the polarization value becomes negative and, after that, oscillates around zero. In our experiment, when the first Cr layer is completed, the polarization is very close to 0%, meaning

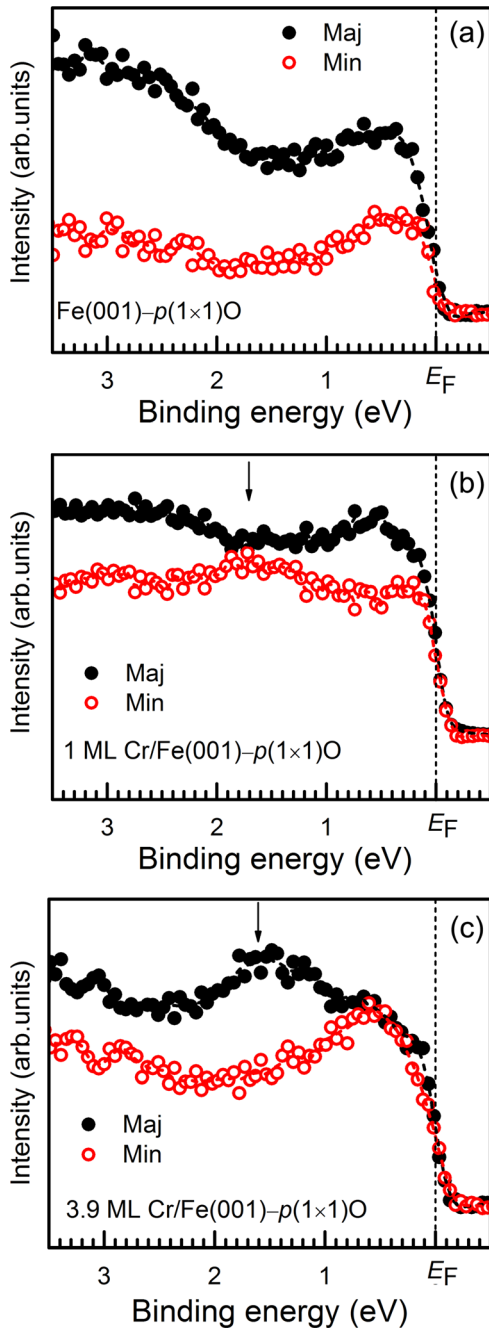


FIG. 2. Spin-resolved valence band spectra (Maj = majority; Min = minority spin channel) taken at a pass energy of 7 eV on (a) the Fe(001)- $p(1 \times 1)$ O substrate and on the Cr/Fe(001)- $p(1 \times 1)$ O system for (b) 1 ML and (c) 3.9 ML Cr thickness. He I source satellites were subtracted in all cases. The arrows mark the position of the Cr-derived feature at 1.6 eV BE (feature e in Fig. 1).

that the contribution of a single Cr layer is enough to balance the attenuated contribution from the Fe below. As soon as the second complete Cr layer is reached, the maximum is achieved at about 25%. The following values, however, do not change sign, at variance with the above expectations for the layered AF stacking of bulk Cr(001). Moreover, the oscillations following the first ones, although having the correct phase and periodicity, are much smaller and their intensity is not centered around zero.

Such discrepancies can be related, first of all, to the fact that the contribution from a spin-polarized inelastic background in the energy region of the spectrum from which

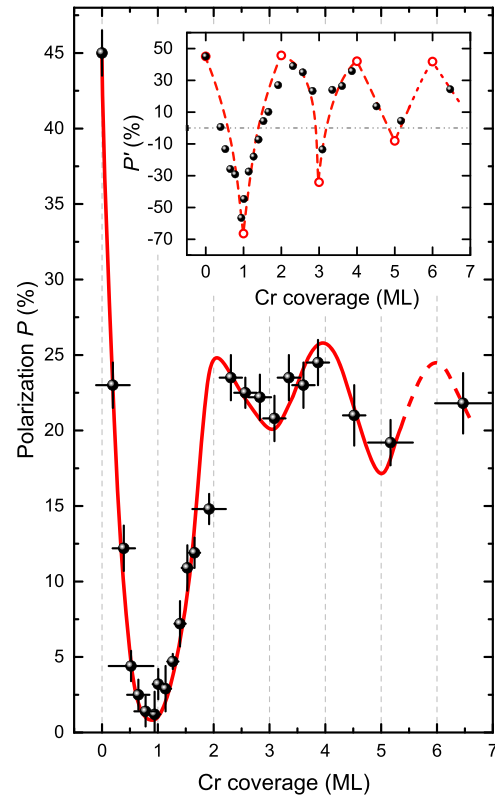


FIG. 3. Polarization P at 1.6 eV BE, as a function of the Cr coverage. The line is a guide to the eye. Inset: P' at 1.6 eV BE for difference spectra $\Delta I^\pm(i)$.

P is extrapolated may lead to a non-ideal behavior of the polarization. In order to highlight the possible presence of an AF stacking of the Cr layers, we started by using the interpolating (continuous red) line in Fig. 3, which allows to deduce the polarization values expected at each integer (say i) ML coverage. In order to decrease the large polarization bias given by the background, the contribution of each i -th Cr layer, defined as $\Delta I^\pm(i)$, has been estimated by subtracting from each SP-PES majority and minority spectrum related to the i ML coverage, namely, $I^\pm(i)$, that corresponding to the $(i-1)$ ML coverage, $I^\pm(i-1)$, attenuated by the exponential decay through the i -th layer. This gives: $\Delta I^\pm(i) = I^\pm(i) - I^\pm(i-1)e^{-d/\lambda}$, where $d = 1.44 \text{ \AA}$ is the thickness of one equivalent Cr ML and an inelastic mean free path (IMFP) $\lambda = 4.5 \text{ \AA}$ has been used, in analogy to Refs. 10 and 31. The inset of Fig. 3 shows, with open red dots, the polarization P' obtained for each $\Delta I^\pm(i)$, up to $i = 6$ ML. In analogy with P , this is defined as $P'(1.6 \text{ eV}) = \frac{\Delta I^+ - \Delta I^-}{\Delta I^+ + \Delta I^-}$. The dashed red line represents, on the basis of the above results, the expected behavior of P' for non integer Cr coverages: for a fractional $(i+f)$ coverage (where $0 < f < 1$), the retrieved difference spectrum is given by the linear combination $\Delta I^\pm(i+f)_{\text{retr}} = (1-f)\Delta I^\pm(i) + f\Delta I^\pm(i+1)$.³⁶ This can be compared with the experimental determination of P' for $\Delta I^\pm(i+f)_{\text{exp}}$, shown by black dots in the inset, obtained, for each $(i+f)$ ML data point of Fig. 3, by substituting the experimental majority and minority profiles $I^\pm(i+f)$ to $(1-f)I^\pm(i) + fI^\pm(i+1)$ in the expression of $\Delta I^\pm(i+f)_{\text{retr}}$.

According to this simplified quantitative analysis, which implies that the magnetization of each Cr layer is not

influenced when a further Cr layer is grown on top of it, the inset of Fig. 3 nicely indicates the alternating sign of P' expected for the AF stacking, although with damped oscillations especially for the negative valleys. Such a damping can be understood considering that, soon after the completion of the first Cr oxide wetting layer, pyramid-shaped islands start to grow in correspondence with screw dislocations in the substrate, becoming well evident at about 5 ML Cr coverage.²⁷ On the one hand, such three-dimensional Cr islands expose more than one Cr layer while, on the other hand, the underlying dislocations create spin frustration within the Cr layers. Both these phenomena cause an amplitude damping in the AF oscillations. Further deviations could also come through the spin dependence of the IMFP.^{37,38} Despite all these reasons that make the experimental findings deviate from the ideal behavior, Fig. 3 clearly shows that the AF stacking starts from the first deposited Cr layer on Fe(001)- $p(1 \times 1)$ O.

We can therefore conclude that the high temperature Cr growth on the Fe(001)- $p(1 \times 1)$ O surface has the advantage of avoiding chemical mixing at the interface between Cr and Fe, at variance with the oxygen-free high-temperature Cr growth on Fe(001) substrates. In the oxygen-free Cr/Fe(001) case, the chemical mixing leads to the reduction of the magnetization in the first few layers close to the interface, resulting in delayed AF stacking. In our case, instead, the presence of oxygen promotes the stabilization of a sharp interface between Cr and Fe and, as a consequence, the magnetic contribution of the first Cr layers leads to AF oscillations starting from the very interface. We finally remark that, besides the clear effect described above, a possible direct role of oxygen in enhancing the strength of the AF ordering of the Cr film cannot be excluded.

In summary, we have provided evidence of the immediate onset of an AF stacking of the magnetization of Cr {001} sheets right from the Cr/Fe(001) interface, in contrast with previous experiments, in which the stacking was observed to begin after some 5 ML, the delay being ascribed to chemical alloying at the interface. Our result was obtained when performing Cr growth on the oxygen passivated Fe(001)- $p(1 \times 1)$ O surface and offers a reliable method to obtain a well-controlled and magnetically reproducible Cr/Fe interface. This can be of significant value when embedding such an interface in low-dimensional magnetic devices.

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