Experimental evidence of the spin dependence of electron reflections in magnetic $CoFe_2O_4/Au/Fe_3O_4$ trilayers

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An original epitaxial system consisting of two ferrimagnetic insulator layers ($CoFe_2O_4$ and Fe_3O_4) separated by a nonmagnetic metallic layer (Au) has been grown. The transport properties in the current in plane geometry indicate that the conduction of the $CoFe_2O_4/Au/Fe_3O_4$ trilayer takes place within the thin metallic layer. The giant magnetoresistance (GMR) observed (2.6% at 10 K) is associated to the switching from a parallel to an antiparallel configuration of the magnetization of the two ferrite layers and corresponds to the spin dependence of electron reflection at the interfaces with a large contribution of specular reflections. The increase of the GMR (5% at 10 K) in the symmetrical interface $CoFe_2O_4/Fe_3O_4/Au/Fe_3O_4$ system and the effect of the interface roughness on the GMR confirm the presence of this spin-dependent specular reflection.

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I. INTRODUCTION

The discovery of giant magnetoresistance (GMR) by Baibich *et al.*[1] in Fe/Cr ferromagnetic (F) multilayers provoked an important research effort and created the field of spintronics. The total current flow is described as the result of two independent conduction channels with electrons of different spin.[2] In the case of parallel alignment of the magnetizations, the current is expected to be higher than in the antiparallel case, since one of the two parallel "spin" channels would be less resistive resulting in a decay of the total resistivity. Spin-dependent resistance is usually ascribed to two distinct contributions: one arising from the bulk of the F layers and the other from the diffusion of electrons at the interface between F layers and non-F spacer layers. Two approaches have been used for a theoretical description of the GMR in current in plane configuration,[3] the "semiclassical" one deduced from the pioneering work of Fuchs and Sondheimer[4–7] and the quantum-mechanical Kubo formalism.[8–10] In the semiclassical approach, spin-dependent bulk resistivities and spin-dependent interfacial resistivities are taken into account. Thus in this model the spin-dependent interfacial resistivity is treated by introducing the proportions of electrons transmitted, specularly reflected, or diffusely scattered from the interfaces.

In current in plane (CIP) magnetoresistance experiments, bulk and interface scattering contributions are usually not separable. The aim of this paper is to analyze the spin dependence of electron reflection and to experimentally examine the CIP-GMR effect which is only produced by the spin-dependent interfacial reflection by using an appropriate system. In this framework, we have grown by sputtering epitaxial trilayers consisting of the stacking of two ferrimagnetic insulating (FI) layers, i.e., $CoFe_2O_4$ and Fe_3O_4 with different coercivity H_{c1} and H_{c2} separated by a nonmagnetic metallic Au layer. In such a system, the conducting electrons are confined in the bidimensional epitaxial metallic layer and are reflected at the metallic/insulator interfaces. An antiparallel configuration of FI layer magnetization can be obtained for magnetic fields ranging between H_{c1} and H_{c2} while a parallel alignment of them is achieved for fields larger than the higher H_c. The modification of the FI/Au interface or the effect of the surface roughness on the GMR should also make it possible to highlight the presence or absence of this spin-dependent interfacial reflection effect.

Magnetite Fe_3O_4 and cobalt ferrite $CoFe_2O_4$ have an inverse spinel face-centered cubic (fcc) structure with lattice parameters of 0.8397 and 0.8392 nm, respectively, at room temperature. The measured saturation moment per formula unit is close to $3\mu_B$ for Fe_3O_4 thin films[11,12] and 1.75 μ_B for $CoFe_2O_4$ thin films.[13] Note that for thin films these magnetic properties deviate from bulk behavior and have often been ascribed to the presence of antiphase boundaries.[11,12,14] Fe_3O_4 has a low magnetic anisotropy with a coercive field of about 310 Oe at room temperature[15] and ranging between 1000 to 1200 Oe at 20 K.[16] Magnetite is a half-metallic oxide with a high resistivity of about 4×10^{-5} Ω m at 300 K and 7×10^{-4} Ω m at 130 K in the bulk sample. At the so-called Verwey temperature (T_v), the material undergoes a metal-insulator transition[17] and below T_V Fe_3O_4 behaves as an insulator: the resistivity exponentially increases and exceeds 1 Ω m below 60 K. The Verwey temperature is 120 K in bulk while it is often lower in thin films[12,18] (T_V=110 K in the single films we have grown). $CoFe_2O_4$ is a hard ferrite with a coercive field of 3000 Oe at room temperature[16,19,20] which increases progressively with decreasing temperature, up to 15 000 Oe at 150 K. The bulk $CoFe_2O_4$ ferrite presents a resistivity of 10^5 Ω m at room temperature. Gold has a fcc structure with a lattice parameter of 0.408 nm and a resistivity of 2.2×10^{-8} Ω m at room temperature.

II. EXPERIMENTAL

The trilayer $CoFe_2O_4/Au/Fe_3O_4$ fabrication had to address the delicate problem of the bidimensional epitaxial growth of a thin metallic layer onto an oxide surface. The samples used here are the result of a thorough optimization of the sputtering process which will be detailed in a forthcoming paper. The system was epitaxially grown on a α-$Al_2O_3(0001)$ substrate in a Plassys ultrahigh vacuum sputtering chamber fitted with an electron gun (20 keV) to perform reflection high energy electron diffraction experiments in order to check the surface quality of the layers after each growth step. The Fe_3O_4 and the $CoFe_2O_4$ layers were grown at 673 K with a radio frequency power under an Ar plasma pressure of 5×10^{-3} mbar. $CoFe_2O_4$ films (thicknesses between 150 and 200 nm) were grown using two cobalt ferrite targets under a 10% oxygen partial pressure. The epitaxial growth of Au on $CoFe_2O_4(111)$ has been achieved for an optimized growth temperature at 473 K and a growth rate of 0.06 nm s^{-1}. The epitaxial growth of Au on the (111) ferrite surface starts three dimensional and a minimum thickness of 6 nm has to be deposited in order to get the coalescence of the islands and a continuous bidimensional epitaxial layer with flat interfaces. Two thicknesses are studied: 10.2 and 13.4 nm. Magnetite films (thicknesses between 100 and 120 nm) were then deposited on the top of the Au layer by means of two Fe_2O_3 facing targets in a pure Ar atmosphere. Another system with symmetrical Au/Fe_3O_4 interfaces (hereafter called a "symmetrical system") was also grown. It consists in a 200 nm thick $CoFe_2O_4$ layer, on which 6 nm of Fe_3O_4 is grown, then a Au layer of 7.2 nm thickness is deposited followed by a 190 nm thick Fe_3O_4 top layer. Four similar trilayers consisting in the following stacking: α-$Al_2O_3(0001)/CoFe_2O_4$ (150 nm)/Au (6.7 nm)/Fe_3O_4 (100 nm) have also been grown and irradiated by N^+ ions at 150 keV with doses varying between 10^{12} to 5×10^{15} ions/cm^2.

The quality of the different interfaces of the trilayers and the structural properties were investigated by x-ray reflectivity, x-ray diffraction, and transmission electron microscopy (TEM) experiments on cross-sectional specimens. The TEM experiments were performed using a Tecnai FEG-Cs corrected TEM whose point resolution is 0.12 nm.

Transport measurements were performed using a standard four probe dc method with a 100 μA current in a CIP configuration with a Quantum Design physical properties measurement system. The trilayers were macroscopically patterned by using standard optical lithography and Ar plasma etching. A stripe of 1.2 mm width and 1.8 mm length was obtained by mounting a mask on top of the Fe_3O_4 layer. The magnetotransport properties were measured with the current parallel to the applied magnetic field and have been carried out between −60 and +60 kOe (and between ±16 kOe in the case of the magnetotransport measurements performed with another setup on the irradiated samples). The magnetoresistance (MR) loops were measured after field cooling through the Verwey transition. Magnetic properties of the trilayers, before patterning, were investigated using a superconducting quantum interference device.

III. RESULTS

Structural properties were checked by TEM observations on all $CoFe_2O_4/Au/Fe_3O_4$ trilayers. The TEM micrograph

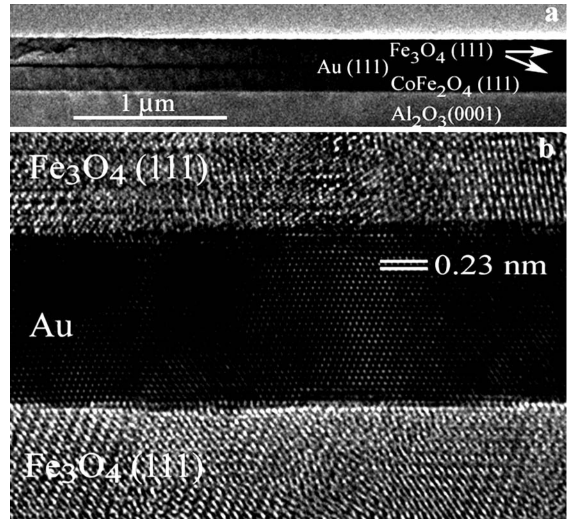

FIG. 1. Low magnification TEM micrograph (a) and HRTEM image (b) of a α-$Al_2O_3(0001)/CoFe_2O_4$ (150 nm)/Au (10.2 nm)/Fe_3O_4 (120 nm) trilayer studied along the $[\bar{1}10]$ zone axis.

in Fig. 1(a) shows the whole stacking with a continuous 10.2 nm thick Au layer. The high resolution transmission electron microscopy (HRTEM) micrograph in Fig. 1(b) confirms the high quality of the epitaxial growth of the trilayer in the [111] direction. The orientation relationships deduced from our HRTEM experiments are α-$Al_2O_3(0001)[\bar{1}100]//CoFe_2O_4(111)[\bar{1}10]//Au(111)[\bar{1}10]//Fe_3O_4(111)[\bar{1}10]$. The fully relaxed layers are separated by flat interfaces with no evidence of interface mixing. Similar structural qualities were obtained for the symmetrical system.

The resistivity versus temperature curves of $CoFe_2O_4/Au/Fe_3O_4$ patterned system with the Au layer thickness of 10.2 and 13.4 nm are reported in Fig. 2 and show a genuinely metallic behavior for all systems. Measured values at 300 K are equal to 9.5×10^{-8} Ω m for the Au-10.2 nm trilayer and $\sim6\times10^{-8}$ Ω m for the Au-13.4 nm

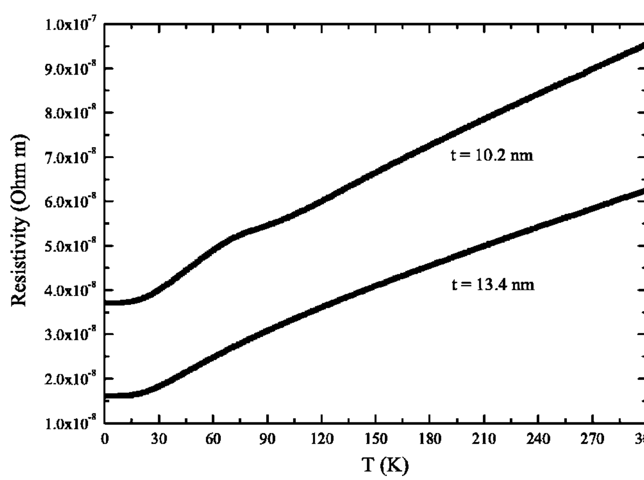

FIG. 2. Resistivity measurements as a function of temperature for the $CoFe_2O_4$ (150 nm)/Au (t)/Fe_3O_4 (120 nm) trilayers with t=10.2 and 13.4 nm.

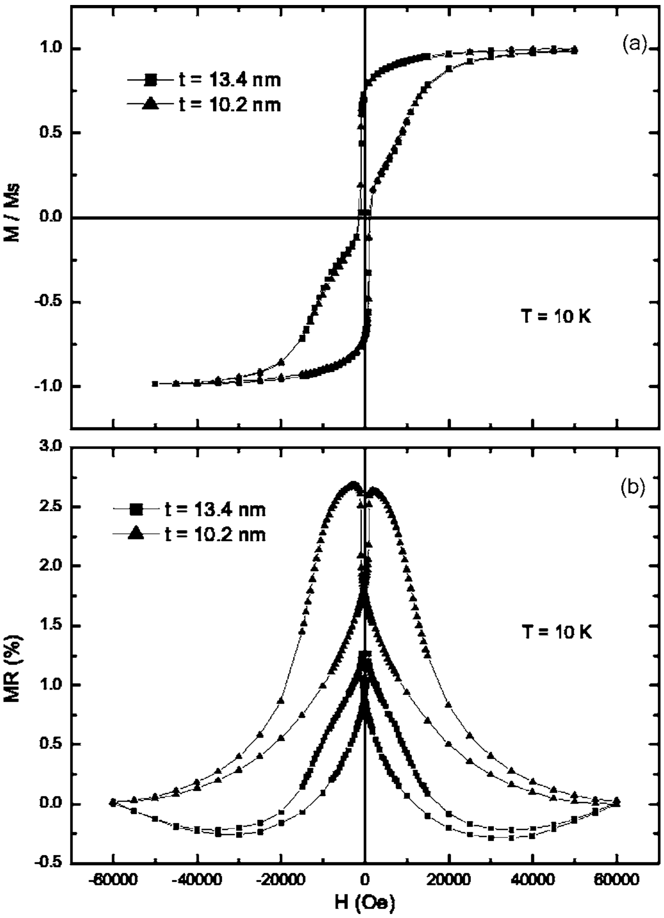

FIG. 3. Magnetic hysteresis loops (a) and magnetoresistances (b) obtained at 10 K on the $CoFe_2O_4$ (150 nm)/Au (t)/Fe_3O_4 (120 nm) trilayers with t=10.2 nm and 13.4 nm.

trilayer. These resistivities are slightly higher than that of Au pure material (2.2×10^{-8} Ω m at 300 K) but correspond nevertheless to the ones expected for a metallic thin film and indicate that the conduction takes place completely within the Au layer. The largest resistivity of the Au-10.2 nm trilayer compared to the Au-13.4 nm trilayer one is due to the FI/M interfaces whose contribution is higher as the metallic layer is thinner. This is in agreement with the Fuchs-Sondheimer theory[4] on the transport properties in thin films.

Magnetic loops of the trilayers (10.2 and 13.4 nm) obtained at 10 K are reported in Fig. 3(a). Measured values are identical for both systems indicating that the magnetic properties of the trilayers do not depend on the Au layer thickness (in the range of the Au thicknesses studied here). As already observed by many authors[11,12,14] the magnetization of ferrite thin films does not saturate because of the presence of antiphase boundaries (APBs) that create antiferromagnetic couplings. Magnetic loops exhibit the characteristic butterfly like shape corresponding to the separate switching of two magnetic layers. The first step at low field (H_c=1000 Oe) corresponds to the switching of the top magnetite layer. This field is equal to the coercive field of a single Fe_3O_4 layer below T_V. The second step at higher magnetic field is more progressive and corresponds to the switching of the cobalt ferrite. These magnetic hysteresis loops demonstrate the existence of both antiparallel and parallel magnetic configurations required for GMR (even if the fully saturated state is not reached) and the absence of any important magnetic coupling between the layers.

In Fig. 3(b) are reported the magnetoresistance plots obtained at 10 K (below T_V) on the two trilayers in a magnetic field range of −60 to +60 kOe. The magnetoresistance ratio is defined by $MR=[R(H)-R_P]/R_P$ where $R(H)$ is the resistance at a magnetic field H and R_P is the resistance in the "nearly" parallel state at high magnetic field. Similarly to the magnetization, the magnetoresistance curves do not saturate even at 60 kOe. This behavior is the result of antiferromagnetic couplings created by the APBs as explained previously. A maximum positive MR ratio of 2.6% at 10 K with a plateau of about 3000 Oe is observed for the Au-10.2 nm trilayer. Both curves present a maximum of the resistance (R_{AP}) in the range of the magnetic fields where the antiparallel configuration of the magnetization in the ferrimagnetic layers occurs. The MR abruptly increases at about 1000 Oe after the switching of the magnetite layer. Then a more slowly MR decrease happens at about 4000 Oe associated to the progressive switching of the $CoFe_2O_4$ layer. The MR curve for the Au-13.4 nm trilayer presents the same evolution with a lower maximum value (1.25% at 10 K with a ±60 kOe field).

Figure 4 shows magnetotransport measurements performed on the symmetrical system α-Al_2O_3(0001)/$CoFe_2O_4$ (200 nm)/Fe_3O_4 (6 nm)/Au (7.2 nm)/Fe_3O_4 (190 nm). The magnetic properties are identical to the ones of the previous trilayers because the thinnest Fe_3O_4 layer is ferromagnetically coupled with the $CoFe_2O_4$ layer and follows the magnetization switching of the cobalt ferrite. The GMR of this symmetrical system increases up to 5% at 10 K with a plateau of about 3000 Oe. As for the trilayers, the GMR abruptly increases at about 1000 Oe where the switching of the magnetite layer occurs.

To investigate the influence of the interface roughness on the GMR properties, we have implanted α-Al_2O_3(0001)/$CoFe_2O_4$ (150 nm)/Au (6.7 nm)/Fe_3O_4 (100 nm) trilayers with N^+ ions at 150 keV with doses varying between 10^{12} to 5×10^{15} ions/cm^2. TRIM (Transport of Ions in Matter)[21] calculations predict a nearly light ion behavior for N^+ at 150 keV, which induces very short-range displacements for target atoms and no implanted impurities in the three layers of interest. The ions pass through the trilayers and stop far below in the substrate, the main damage being located at the $CoFe_2O_4$/Au and Au/Fe_3O_4 interfaces. In Figs. 5(a) and 5(b) HRTEM micrographs obtained on the samples irradiated at doses of 10^{13} and 10^{15} ions/cm^2, respectively, are reported. No drastic changes of the interfaces show up for the 10^{13} implanted sample while a clearer roughness is visible in the trilayer implanted at the highest dose. Note that no drastic alteration of the layers is distinguishable. The magnetotransport measurements performed on the as-deposited trilayer in a magnetic field range of ±16 kOe gives a MR ratio equal to 1.4%. At such a field, the fully parallel configuration is not reached leading to a lower MR than what would be observed in ±60 kOe. In Fig. 6 the variation of the normalized MR in the ion implanted trilayers is reported. It

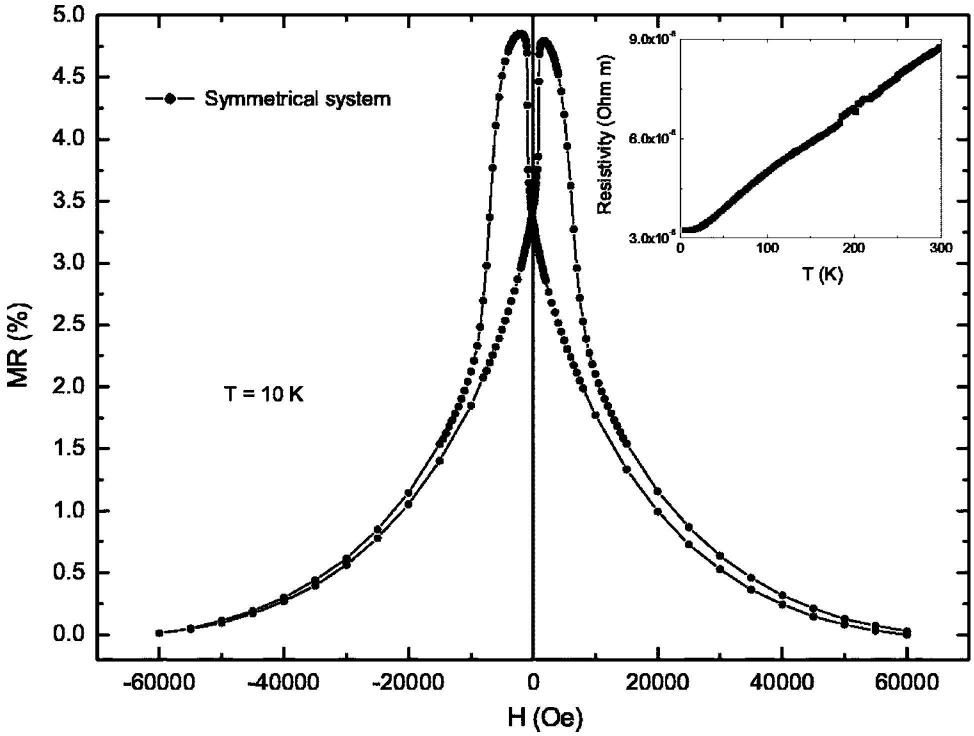

FIG. 4. Magnetoresistance obtained at 10 K on the symmetrical $CoFe_2O_4$ (200 nm)/Fe_3O_4 (6 nm)/Au (7.2 nm)/Fe_3O_4 (192 nm) and resistivity curve as a function of temperature inset.

indicates a slight increase of the MR with doses up to 10^{13} ions/cm^2 then a rapid decrease for higher doses.

IV. DISCUSSION

Spin-dependent resistance is usually described by two scattering phenomena: one arising from the bulk of the magnetic layers and the other from the interface between magnetic layers and nonmagnetic spacer layers. Bulk spin-dependent scattering into the ferrite layers on our systems seems unlikely: $CoFe_2O_4$ is a very good insulator on the whole temperature range and Fe_3O_4 also becomes a very good insulator at low temperature. At 10 K, the resistivities of the two ferrites are thus many orders of magnitude higher than that of Au. Our transport measurements indicate that the conduction is metallic for the trilayers with a resistivity very close to the one of Au pure material; therefore the electrons are perfectly confined in the metallic layer. A parasitic ferrite thin layer close to the Au/FI or FI/Au interfaces could be conducting and thus also produce the GMR effect, but the HRTEM analysis does not show evidence of such a layer. Furthermore complementary magnetoresistance measurements performed with the current perpendicular to the field rule out a contribution from possible AMR: the GMR behavior corresponds to the different switching fields of the ferrite layers. Thus, we claim that the only magnetic interactions responsible for the MR are the ones occurring when the electrons are reflected at the metal/ferrimagnetic insulator interfaces and therefore the MR is only due to the spin dependence of the electron reflections at the Au/FI interfaces.

In Fuchs-Sondheimer model,[4] two kinds of electron reflections, diffuse and specular, are taken into account. Electron reflections depend strongly of the interfacial quality: a

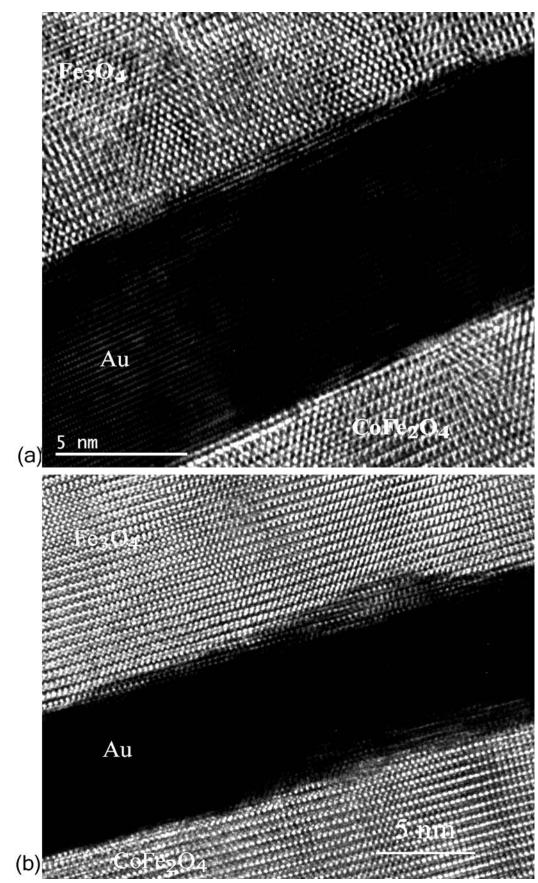

FIG. 5. HRTEM micrographs of the $CoFe_2O_4$ (150 nm)/Au (6.7 nm)/Fe_3O_4 (100 nm) trilayers implanted at a dose of (a) 10^{13} and (b) 10^{15} ions/cm^2.

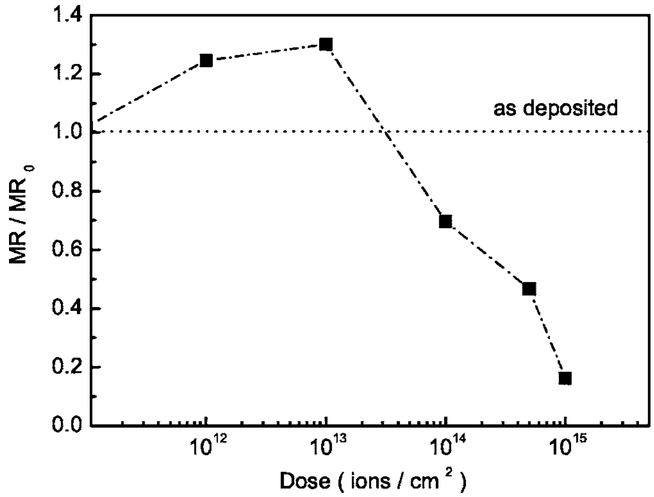

FIG. 6. Magnetoresistance variations as a function of the fluences in $CoFe_2O_4$ (150 nm)/Au (6.7 nm)/Fe_3O_4 (100 nm) trilayers irradiated with N^+ ions at 150 keV (normalized to the MR measured on the as deposited sample).

rough interface or interface with some impurities favor diffuse reflections while specular reflections are only produced in the case of a flat interface. Most papers[1,5–7] consider that diffuse reflection at the interface is the main spin-dependent contribution to the GMR. However, in the trilayers presented here, the magnetoresistance associated to this possible diffuse reflection should be extremely weak since the layers were epitaxially grown in order to obtain very flat interfaces and the electrons are not expected to be transferred far inside the ferrimagnetic insulator layers due to their extremely high resistivities. Therefore, even if the presence of defects at the interfaces on the atomic scale (which cannot be detected in HRTEM experiments) responsible for a diffuse scattering cannot be completely excluded, we assume that a part of the measured effect is described by the spin-dependent specular reflections. This assertion is supported by the MR changes observed in N^+ 150 keV ions implanted trilayers. A slight increase of the GMR with the fluence is observed for doses up to 10^{13} ions/cm^2 then a rapid decrease occurs for higher doses. Together, TEM experiments do not indicate a drastic change of the Au/FI interface roughness in the samples irradiated at doses less than 10^{15} ions/cm^2 [Fig. 5(a)]. Moreover, x-ray reflectivity performed on similar samples indicates a drastic decrease of the interface roughness with the doses for doses up to 10^{14} ions/cm^2. We claim the MR enhancement for low ion fluences is the result of the smoothing of the interfaces due to the ion implantation at such low doses while the large decrease of the MR observed for fluences higher than 10^{14} ions/cm^2 is due to an increase of the Au/FI interface roughness as observed in HRTEM [Fig. 5(b)]. These results confirm the main influence of the interface quality on the GMR we measured. We therefore assert that the GMR measured in such system is due to the spin-dependence of the electron reflections at the Au/FI interfaces with a large part of specular reflections.[5,22]

The influence of the nature of the interfaces on the GMR confirms this assumption. The GMR of the symmetrical system α-Al_2O_3(0001)/$CoFe_2O_4$ (200 nm)/Fe_3O_4 (6 nm)/Au (7.2 nm)/Fe_3O_4 (190 nm) is almost 2 times higher than the one of the α-Al_2O_3(0001)/$CoFe_2O_4$ (150 nm)/Au (10.2 nm)/Fe_3O_4 (120 nm) trilayer. A similar study has been published on the Fe_3O_4 (60 nm)/Au (5 nm)/Fe_3O_4 (20 nm) system deposited on MgO(001).[23] However, due to the absence of antiparallel magnetic configurations, no GMR effect was measured by the authors. In our system, the ferromagnetic coupling between the $CoFe_2O_4$ layer and the 6 nm Fe_3O_4 layer allows one to obtain the anti-parallel magnetic configuration essential to the appearance of the GMR. The GMR obtained here is most likely due to the increase of the number of electron reflections at the Au/FI interfaces, with decreasing Au thickness and also to the difference in the band structure of the Au/$CoFe_2O_4$ and Au/Fe_3O_4 interfaces. As Fe_3O_4 is highly spin polarized (contrary to $CoFe_2O_4$), the electron reflection is expected to be much more selective in spin at the Au/Fe_3O_4 interface than at the Au/$CoFe_2O_4$ one, resulting in a higher GMR in the symmetrical system.

V. CONCLUSION

In summary, we have realized the epitaxial growth of $CoFe_2O_4$/Au/Fe_3O_4 trilayers with flat interfaces. The interfacial flatness and the high structural quality of the multilayers have been evidenced by transmission electron microscopy. We have measured the conduction of the systems that takes place within the metallic Au layer. A CIP-GMR effect is observed at low temperature which has been ascribed only to the spin-dependent reflection at the metal/ferrimagnetic insulator interfaces. The increase of the GMR in the $CoFe_2O_4$/Fe_3O_4/Au/Fe_3O_4 symmetrical system (5% at 10 K) and the first observations of the decrease of the GMR with the Au/FI interface roughness increase highlight a CIP-GMR effect due to the spin dependence of electron reflection at the interfaces with a large contribution of specular reflections.

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