The magnetic structure of coupled Fe/FeO multilayers revealed by nuclear resonant and neutron scattering methods

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\textbf{Abstract.} We have studied the magnetic structure that forms in a Fe/native Fe oxide multilayer by nuclear resonant scattering of synchrotron radiation and polarized neutron reflectometry. Magnetic field-dependent experiments revealed a non-collinear magnetic arrangement of the adjacent metallic layers which is mediated by an antiferromagnetically ordered oxide layer. Despite its antiferromagnetic (AFM) order, the oxide exhibits a small net magnetization attributed to the presence of metallic Fe within the AFM matrix that aligns parallel to the external field. The presence of a strong uniaxial anisotropy prevents the system from forming small magnetic domains in remanence. The canting angle between the two magnetic sublattices remains close to 90° throughout the magnetization reversal on the hard axis. The results and the influence of the uniaxial anisotropy are discussed in the framework of the proximity magnetism model.

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The discovery of antiferromagnetic (AFM) coupling between ferromagnetic (FM) layers separated by metallic spacer layers has triggered a multitude of activities in fundamental and applied research. This, for example, led to the development of giant magneto-resistive read heads, which are now widely used in hard disk technology. This interlayer spin coupling is fairly well understood in terms of the RKKY model, which implies long-range exchange interaction mediated by conducting band electrons in the spacer layer [1, 2]. Transition metal monoxides (TMO) like CoO or NiO have recently attracted special interest regarding the use of them as spacer layers [3, 4] due to their unique magnetic and electric properties. They possess an AFM ground state combined with a low conductivity which prevents long-range RKKY-type exchange interactions. Instead, their magnetic coupling properties are mainly ruled by the interfacial exchange interaction between metallic FM and insulating AFM layers. This means that the interface of the TMO with the FM layers plays a determining role in the overall magnetic coupling. An FM/AFM interface is also the basic requirement for the appearance of the well-known exchange bias effect [5] where a unidirectional anisotropy is induced in the coupled FM, an effect that is characterized by an increased coercivity and a shift of the hysteresis loop. The presence of many FM/AFM interfaces in a TM/TMO layer stack could lead to a combination of long interlayer magnetic ordering and exchange bias phenomena, which has not been extensively studied so far.

In a recent study, Beach et al [6] proposed to use natively grown oxide layers (i.e. oxide layers grown by exposure of a metal to oxygen) to form metal/native oxide multilayers (MNOM) combining a large magnetic moment with a low conductivity. These two properties render these types of multilayers ideal candidates for high-frequency magnetic applications for their ability to efficiently damp eddy currents while maintaining a high net magnetization. Recently, we reported about the occurrence of non-collinear magnetic coupling between adjacent Fe layers in similar Fe/native oxide systems [7]. The magnetic field dependence of the spin arrangement pointed towards a proximity magnetism coupling model [2], which implies an AFM order of the buried native oxide. However, the microscopic spin arrangement of both the metal and the
oxide layers has not yet been resolved, thus preventing a complete understanding of the observed magnetic properties of these systems.

Element-specific x-ray scattering methods based on magnetic dichroism effects (e.g. XMCD and XMLD [8, 9]) are difficult to apply in this case to obtain layer-resolved information because the magnetic atom (Fe) is present in both the metal and the oxide layers. In principle, one can tune the photon energy to be more sensitive to the oxide or the metal signal; it is, however, difficult to completely isolate each signal. On the other hand, isotope-sensitive scattering methods like nuclear forward scattering (NFS) of synchrotron radiation and polarized neutron reflectometry (PNR) can provide a large nuclear contrast and a sub-nm depth resolution [10] if isotopic probe layers (56Fe and 57Fe) are embedded into the layer system.

In this study, we combined the two methods NFS and PNR to study the microscopic magnetic structure of isotopically enriched Fe/native oxide superlattices. The use of isotopic 57Fe probe layers placed either in the metallic or in the oxide part of the system allowed us to study independently the magnetic structure of each set of layers by NFS. PNR was applied to obtain the magnetic contrast between the metal and the oxide, ideally complementing the NFS results.

2. Sample characterization

Two different types of superlattices were prepared that are depicted in figures 1(a) and (b). The first one with a 0.6 nm 57Fe probe layer placed in the middle of the metallic 56Fe layer will be referred to as the metal-doped sample. The second one has a 57Fe-oxide probe layer embedded in the oxide part of the system and will be referred to as the oxide-doped sample. The samples were prepared by dc magnetron sputter deposition in a UHV chamber (base pressure 5 × 10−10 mbar) described in detail elsewhere [11]. The Fe/native oxide superlattices were grown on superpolished Si(100) substrates by deposition of a 2–3 nm thick Fe layer and subsequent controlled exposure to 6500 Langmuir (1 L = 10−6 mbar s) of molecular oxygen. This sequence is then repeated to produce a superlattice of 15 bilayers. A final 3 nm Al capping layer is deposited to prevent further oxidation of the samples in air. The nominal structure of the samples is Si/[Fe (1.8 nm)/FeOx (1.6 nm)]15/Al(3 nm). In [12], the chemical structure of the buried native oxide was investigated by in-situ x-ray absorption spectroscopy, which showed that the layer adopts a disordered but pure FeO-like structure upon deposition of the metal on top of the saturated oxide.

The oblique incidence of the sputtered 57Fe atoms with respect to the sample surface leads to a strong uniaxial anisotropy due to preferential alignment of polycrystalline columns during the growth process [13]. The low-field hysteresis curves along the easy and hard axes, as recorded by MOKE magnetometry, are shown in figure 1(c). The inset shows the progressive increase of the net magnetization in high fields, which saturates only above 1.2 T, as reported in [7].

3. Experimental methods

As will be shown later, Fe/FeOx multilayers exhibit a complex magnetic structure where both the metal and the oxide are magnetically ordered. The two scattering methods that we employed will yield complementary information. NFS is used as a layer-selective probe to study separately the metal or the oxide part of the system, whereas PNR is sensitive to the magnetic contrast between adjacent metal/oxide layers.
Figure 1. Schematic sample structure for (a) the metal-doped sample and (b) the oxide-doped sample. A 0.6 nm $^{57}$Fe probe layer is embedded in each metal (a) or oxide (b) layer of the superlattices. (c) Typical easy and hard axis hysteresis curves of these systems. A uniaxial anisotropy is induced by the oblique deposition of the $^{57}$Fe with respect to the sample surface. The inset shows the high-field branch of the hysteresis, which exhibits a slow increase of the net magnetization with increasing field. Saturation is reached only above 1.2 T.

NFS of synchrotron radiation is a time-resolved method, where the time-dependent de-excitation of $^{57}$Fe nuclei in the sample is recorded after pulsed excitation by synchrotron radiation. Because this de-excitation occurs on the timescale of about 100 ns, the signal from the $^{57}$Fe probe layer can be discriminated from the electronic charge scattering (which occurs on the fs timescale) using fast avalanche photodiode (APD) detectors [14]. The recorded signal is, therefore, intrinsically free of charge scattering background. The sensitivity to the hyperfine structure of the nucleus renders the techniques highly sensitive to the magnitude and orientation of the magnetic field at the position of the $^{57}$Fe nuclei [15]. The advantage of the technique here comes from the possibility to selectively probe the magnetic structure of the metal or the oxide by placing ultrathin $^{57}$Fe probe layers in these parts. The sample is typically illuminated in grazing incidence geometry and the specularly reflected signal is recorded. If this signal is recorded as a function of the momentum transfer (nuclear resonant reflectivity), one can obtain the magnetic depth profile of the $^{57}$Fe in the system. In particular, periodic magnetic structures cause a variation of the nuclear scattering amplitude that leads to superstructure peaks between the structural Bragg peaks in the nuclear reflectivity.

PNR shares some of the formalism of magnetic x-ray scattering. However, as a nuclear scattering technique, PNR can provide a large scattering contrast even for different isotopes of the same element (further details on the PNR method can be found in [16, 17]). In our case, the large difference in the nuclear scattering length density between $^{56}$Fe and $^{57}$Fe ($8.36 \times 10^{-6}$ and $1.9 \times 10^{-6}$ Å$^{-2}$, respectively) allows us to vary the nuclear contrast in the multilayer structure [18]. The magnetic scattering length, on the other hand, is due to the interaction of neutrons with the magnetic moment of the atoms (it will be the same for $^{56}$Fe and $^{57}$Fe) and is a fingerprint of the absolute magnetic moment of the atom. The full polarization analysis allows one to resolve the complete in-plane magnetization of the sample. In contrast to the NFS studies, we used PNR to determine the magnetic contrast between the metal and the oxide layers.
Figure 2. Electronic (a and c) and nuclear (b and d) x-ray reflectivity for the metal-doped (a and b) and oxide-doped (c and d) superlattices recorded with NFS. The red lines are fits. The appearance of the superstructure half-order Bragg peak accounts for a magnetic unit cell twice as large as the chemical one. The intensity of this peak is directly related to the difference in the projection of the two magnetic sublattices along the photon wavevector $\vec{k}_0$, as depicted in the inset of (b). In this case, we found a canting angle between the magnetizations of two adjacent Fe layers of 80° for the metal-doped sample and an average canting angle of 20° between the magnetizations of adjacent oxide layers in the oxide-doped sample.

The NFS experiments were carried out at the nuclear resonance beamline ID18 [19] of the European Synchrotron Radiation Facility (ESRF) in the 16 bunch mode of operation. The energy of the incoming photon was tuned to the $^{57}$Fe nuclear resonance at 14.4 keV, which corresponds to a photon wavelength of 0.862 Å. The PNR experiment was performed at the ADAM high-resolution reflectometer [20] of the Institute Laue Langevin (ILL) where the wavelength of the neutrons was set to 4.41 Å.

4. Layer-resolved magnetic structure

4.1. NFS results

The major features of the magnetic structure in the Fe/FeO superlattice were revealed using the NFS technique on the metal-doped sample. A small magnetic field of 20 mT is applied parallel to the film surface and perpendicular to the scattering plane to define the magnetization direction. As can be seen in figure 2(b), a half-order Bragg peak appears between the structural Bragg peaks in the nuclear reflectivity. Its position accounts for a magnetic period two times larger than the chemical one and is indicative of non-collinear magnetization orientation in neighboring metallic Fe layers. In this geometry, the intensity of the half-order Bragg peak is directly related to the difference in the projections of the magnetic hyperfine fields within
the two magnetic sublattices along the photon wave vector $\vec{k}_0$ \cite{21}. As depicted in the inset of figure 2(b), for a symmetric opening of the magnetization of the two magnetic sublattices relative to the external magnetic field, the projection difference increases with increasing canting angle. The intensity of the half-order peak, therefore, is determined by the relative orientation of two adjacent Fe layers, which is found to be about 80° in a 20 mT field. The nuclear reflectivity curves in figure 2 were fitted using the CONUSS program package for analysis of nuclear resonant scattering data \cite{22}. A field-dependent study \cite{7} shows that the canting angle decreases only asymptotically with increasing external field. This type of behavior is described by the proximity magnetism model developed by Slonczewski \cite{2}, which assumes an AFM order in the spacer layer.

In the present case, the coupling is mediated by direct exchange interaction at the FM/AFM interface that is then transferred via the AFM order to the top FM layer. Statistical variations of the thickness of the AFM layer result in a distribution of even or odd numbers of atomic planes in the AFM. Thus, the energetically favored configuration is a 90° coupling angle between the FM layers, a value close to what is found in our experiment. Several other studies reported similar coupling behavior for trilayer systems \cite{3,4}, where the 90° coupling is supposed to be sustained by a twist in the AFM to match the magnetization directions that are imposed by the exchange interactions at the adjacent interfaces. The occurrence of a correlated magnetic order over many bilayers implies a strong exchange coupling between the metal and the oxide.

The fact that the native oxide adopts an AFM ordering is quite surprising, because the bulk Néel temperature of FeO is 198 K. However, as was established by van der Zaag et al \cite{23}, the Néel temperature of thin AFM layers embedded between FM layers can drastically increase compared with bulk values to reach the Curie temperature of the surrounding FM in the limit of ultrathin films. In that case, the blocking temperature $T_B$ (the temperature below which exchange bias effects appear) does not match $T_N$ but is drastically reduced compared with the bulk case. Low-temperature magnetization measurements indicated a blocking temperature of 70 K for our layer system, supporting the interpretation that the oxide is antiferromagnetically ordered at room temperature.

A similar nuclear reflectivity study was performed on the oxide-doped sample, as shown in figure 2(d). Surprisingly, a half-order Bragg peak is also observed, which indicates that a long-range non-collinear magnetic order exists also within the oxide layers. A fit of this peak assuming a magnetically correlated system of oxide layers would yield an average canting angle of 20° between the magnetization direction in the oxide layers. However, the same fit could be obtained if one assumes that only a fraction (about 25%) of the moments in the oxide adopt a 90° canting angle while the rest of the layer remains in a compensated AFM state. Hence, this measurement does not provide a unique solution concerning the internal magnetic structure of the oxide. However, it still implies that the oxide layers exhibit a long-range magnetic correlation throughout the layer stack, and indicates that parts of the oxide layer are not in a purely AFM state.

4.2. Polarized neutron reflectivity

The results presented in the previous section were obtained on two different samples. So far, no direct information on the interaction between adjacent metal/oxide layers has been gained. In particular, the restriction of the information to the $^{57}$Fe probe layers does not allow us to distinguish the relative orientation of the net magnetization of the oxide compared with the

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Figure 3. Polarized neutron reflectivity curves of the metal-doped and oxide-doped Fe/native oxide superlattices. The red lines are the results of the fit. A magnetic field of 20 mT was applied in-plane of the sample and parallel to the neutron spin polarization. Top row: the appearance of a half-order Bragg peak in the spin flip channels evidences the non-collinear orientation of the magnetization in adjacent Fe layers symmetric around the magnetic field direction. Bottom row: for the oxide doped sample, the presence of a small half-order Bragg peak in the non spin flip (NSF) channels indicates that the net magnetization of the oxide layers is not collinear with the external field.

metal. The same samples were therefore studied with PNR. A small magnetic field of 20 mT was applied along the hard axis and parallel to the neutron spin polarization in both cases. The sets of reflectivity for both samples are shown in figure 3. The red lines are results of the least square routine [24] applied to simultaneously fit all four PNR curves to the theoretical model. The NSF reflectivities ($R^{++}$ and $R^{--}$) are mostly sensitive to the layer’s magnetization parallel to the neutron spin (in this case, parallel to the external field). The different positions of the critical edge for $R^{++}$ and $R^{--}$ directly relate to the net magnetization of the sample. The non-fitted high intensities at $q$ values below 0.1 nm$^{-1}$ in the spin-flip reflectivities are due to the influence of the direct beam. For the metal-doped sample, the scattering length densities of the metal and oxide layers (resulting from the weighted sum of the constituent species) are equal. This means that the first-order Bragg peak observed here is of pure magnetic origin. The half-order Bragg peak in the spin-flip channels references to a coupling angle between the metal layers of 80°, symmetric around the direction of the applied magnetic field. The fit of the reflectivity curve indicates that the metal layer possesses a magnetic moment of 2.15 $\mu_B$ per atom, a similar value compared with bulk Fe (2.2 $\mu_B$). The oxide also possesses a small net magnetic moment of 0.5 $\mu_B$ per atom pointing in the same direction as the net magnetization of the metal layers.

For the oxide-doped sample, the situation is somewhat different. The presence of the half-order Bragg peak in the spin-flip reflectivity accounts for a canting of the metal layers, which amounts here to 40°, a smaller value compared with the metal-doped sample. Additionally, it is necessary to assume a non-collinear spin arrangement in the oxide part of the layer to account for the small half-order peak present in the NSF channels. In this case, the overall net magnetization...
Figure 4. (a)–(d) Nuclear resonant reflectivity curve during magnetization reversal of the metal-doped sample. The magnetic field is applied close to the hard axis and perpendicular to the scattering plane. The decrease of the half-order Bragg peak intensity accounts for a coherent rotation of the coupled moment as one unit. In remanence, the net magnetization is aligned on the easy axis. (e) The half-order Bragg peak reappears once the sample is turned by 90°, confirming that the magnetic moments stay coupled in remanence. (f)–(i) Polarized neutron reflectivity in the remanent state. A coupling angle of 70° in remanence is derived from both the NFS and PNR measurements.

of the oxide is perpendicular to the magnetic field. The reason for this phenomenon is not clear but might be due to small structural differences of the metal-doped and oxide-doped samples, the effect of which will be discussed in a later section.

5. Magnetization reversal

The hard axis magnetization reversal was monitored by taking NFS reflectivity curves of the metal-doped sample at selected steps of the reversal. The corresponding curves are depicted in figures 4(a)–(d), where the external field was applied close to the hard axis and perpendicular to \( \vec{k}_0 \). In this geometry, the half-order Bragg peak intensity is maximum when the magnetization of the two sublattices is symmetrically tilted to the left and to the right with respect to the magnetic field. When the external field is reduced, the half-order Bragg peak intensity decreases and almost vanishes in the zero applied field. This could be due either to a reduction of the canting angle, domain formation or coherent rotation of the net magnetization of the total system (with constant canting angle) back into the easy axis direction. The first two possibilities can be ruled out by taking measurements with the sample rotated by 90° in remanence, as
The specularly reflected beam is indicated by the ridge running along the diagonal on the graph. The absence of diffuse scattering around the half-order Bragg peak indicates that the system does not decay into small magnetic domains. The lower limit for the domain size is estimated to be about 30 µm.

shown in figure 4(e). In this case, the half-order Bragg peak reappears and accounts for a 70° canting angle, indicating that the layers’ magnetization stayed canted through the magnetization reversal and that the net magnetization is now aligned along the easy axis. This is confirmed by a polarized neutron reflectivity curve recorded in remanence. As the position of the critical edge in the R++ and R−− channels is the same, we infer that there is no net magnetization along the hard axis. The half-order Bragg peak accounts for a 70° canting angle in remanence. These measurements therefore confirm the canting angle found with the NFS data. However, PNR allows us to determine the magnetic moment per atom, which is found to be 2.15 µB, the same value as was found prior to the magnetization reversal. This supports the notion of a coherent rotation of the coupled FM layers as one unit.

The formation of small magnetic domains is also ruled out by two-dimensional (2D) mapping of the off-specular neutron scattering. Magnetic domains usually lead to diffuse scattering sheets running across the magnetic Bragg peak [25]. As shown in figure 5, the absence of diffuse scattering in remanence accounts for magnetic domains that must be larger than 30 µm (the resolution limit of the instrument) and can therefore be considered as macroscopic.

6. Discussion

From the results presented above, two particular properties of the oxide can be derived. First, the oxide layer always possesses some magnetic moment, although it is supposed to be in an AFM state. Second, the uniaxial anisotropy induced by the oblique deposition of the ⁵⁷Fe probe
layer influences the system differently depending on whether the probe layer is embedded into the metal or into the oxide. In particular, it prevents the system from forming small magnetic domains in remanence.

According to the proximity magnetism model of Slonczewski [2], the 90° coupling angle between adjacent Fe layers is the result of competition between areas of differing interlayer thickness which favor either a parallel or antiparallel alignment of the FM layers. The coupling energy density $W$ can be described by the following phenomenological equation:

$$W = C_+ \theta^2 + C_- (\theta - \pi)^2,$$

where $C_+$ and $C_-$ are constants related to parallel or antiparallel alignment. The energy minimum of the system is found by setting the derivative of $W$ to 0. That is,

$$\frac{dW}{d\theta} = 2C_+ \theta + 2C_- (\theta - \pi) = 0,$$

which leads to the expression:

$$\frac{C_+}{C_-} = \frac{\pi - \theta}{\theta}.$$

This equation shows that the important quantity to determine the canting angle in remanence is the ratio $C_+/C_-$. A 90° coupling angle minimizes the system energy if both thicknesses are present in the same fraction (i.e. $C_+/C_- = 1$). Physically, the interface coupling induces a twist within the AFM which differs depending on the number of AFM layers [3], as depicted in figure 6. In this case, the resulting net magnetic moment of the AFM is close to zero. For a polycrystalline layer, the twist is supposed to be confined to one grain [26] and only a very small net moments can eventually be expected at the grain surface. However, as we have demonstrated

![Figure 6](http://www.njp.org/)
Figure 7. Schematic representation of the presence of defects in the oxide or in the metal layer. (a) Pinholes in the AFM oxide layers lead to an effective FM coupling between the two metal, reducing the overall coupling angle. (b) A defect in the metal layer would not affect the overall magnetic structure but eventually leads to a small reduction of the total moment.

recently [12], buried native oxide layers grown at room temperature incorporate a fraction of about 10% of Fe in a metallic state. In the case where the metallic regions are FM and align to the external field, they could be responsible for the measured net moment in the oxide. This picture would be consistent with the finding that the coupling angle is below 90° in low magnetic fields, which indicates a larger value of $C_+$ and therefore a predominance of FM coupling.

The influence of the oblique deposition of the $^{57}$Fe probe layers on the magnetic structure of the superlattice is somewhat more complicated to understand. It clearly provides a way to create a strong uniaxial anisotropy which allows one to have a well-defined remanent state. However, it dramatically changes the magnetic structure of the oxide spacer layer when the $^{57}$Fe probe layers are placed in its middle. According to equation (3) the reduced coupling angle of 40° between the metallic layers accounts for a ratio $C_+ / C_-$ larger than 4, which means that FM coupling is greatly favored. This large proportion of FM coupling might be a hint of deviation from a pure proximity coupling through an AFM spacer. In particular, the oblique deposition of the $^{57}$Fe might leave defects in the polycrystalline oxide layer which would allow for direct FM coupling through pinholes, as depicted in figure 7(a). This direct pinhole coupling could then be responsible for the reduced coupling angle observed. This interpretation would also be in agreement with the more pronounced coupling angle measured on the metal-doped sample. In that case, the $^{57}$Fe probe layer is in the middle of the metal. As shown in figure 7(b), defects in this layer would not influence the overall magnetic properties of the metal, and would therefore have little influence on the coupling through the AFM oxide.

7. Conclusions and outlook

Using a combination of isotope-sensitive scattering methods, we have obtained a layer-resolved magnetic depth profile in a superlattice composed of metallic Fe and its native oxide. The AFM arrangement of the oxide spacer layers leads to strong exchange coupling of adjacent metallic layers which is mediated by exchange interactions at the metal/oxide interfaces. The non-collinear arrangement is robust against an external magnetic field up to 1 T and completely reversible. We measured the magnetization reversal with the field applied along the hard axis, which takes place via coherent rotation of the coupled moments as one unit. Off-specular...
neutron scattering confirmed that the system stays single domain in remanence and the decay into magnetic domains can be excluded within the resolution limit of the instrument. The presence of metallic Fe in the natively grown oxide can account for the observed net magnetic moment in the AFM layer, which tends to reduce the coupling angles between the metallic layers. We expect that the removal of this remaining metal, for example, by mild thermal treatments should help to optimize the observed magnetic coupling properties. The ease of preparation of such MNOM systems and the strength of the 90° coupling could open interesting possibilities for spin engineering of magnetic layer systems.

The two scattering techniques used here complement themselves ideally to study complex magnetic structures. X-ray-based NFS is ideal for performing systematic studies of selected parts of the sample using isotopic probe layers, while the sensitivity of PNR to the magnetic moment of atoms is ideally suited to take snapshots of the whole system in a defined magnetic state. These considerations should trigger further joint x-ray and neutron scattering studies of magnetic multilayer systems.

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