Magneto-optical properties of Fe/Cr/Fe/MgO/Fe structures epitaxially grown on GaAs(001)

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Fe/Cr/Fe trilayers were epitaxially grown on atomically flat GaAs(001). For the thickness of Cr spacer layer corresponding to antiferromagnetic coupling, ''reversed'' minor hysteresis loops were measured with longitudinal magneto-optical Kerr effect (MOKE), i.e., a negative ''magnetization'' signal was detected when the thicker bottom Fe layer was saturated along the applied field. This behavior is interpreted by depth variations of the MOKE sensitivity. Magnetization reversal shows that both antiferromagnetic switching and spin–flop transition fields depend on the ratio of both Fe film thicknesses. The shape of the MOKE loops becomes more complex with further deposition of MgO and Fe layers on the top of the Fe/Cr/F/GaAs(001) stack. Superconducting quantum interference device measurements confirm the interpretation of the MOKE loops and demonstrate homogeneity and sharpness of the interfaces in the structures. © *2004 American Institute of Physics.* [DOI: 10.1063/1.1632016]

I. INTRODUCTION

Metal-based ferromagnetic (FM) spin aligners, which operate at weak magnetic fields and room temperature, remain a subject of great interest, in particular when grown on semiconducting substrates.[1] Recent theoretical works predict that the efficiency of the spin injection from a ferromagnet into a semiconductor can be improved for electrons created by tunneling through an insulating barrier (I) since such a process is not affected by the conductivity mismatch and results in the conservation of the spin polarization.[2]

The transport properties of FM/I/FM magneto-tunnel junctions depend on the relative orientation of magnetization in the FM layers. In order to operate the junction, the magnetization of both FM films has to be switched independently assuring antiparallel orientation of magnetization within a well defined range of the applied magnetic field. The almost perfect alignment of magnetic moments should comprise not only some small fraction of them, but has to occur homogeneously in the structure volume, at least on the scale of the junction area. In the case of symmetric Fe/MgO/Fe structures (which assure contribution of identical electronic states to the tunneling), the independent magnetization switching can be achieved: (a) by hardening of the top Fe film, e.g., by covering with Co (Ref. 3) or Ni (Ref. 4), or (b) by pinning the magnetization of one of the Fe films via its antiferromagnetic coupling (e.g., to another FM layer across a nonferromagnetic spacer[5]) or by the exchange bias effect existing in the interface between the FM layer and an antiferromagnet.[6] The MgO tunneling barrier minimizes magnetic coupling between the Fe films, however interactions between them can still be present. This magnetic interaction can be caused by any metallic ''bridges'' through the insulating barrier, magnetostatic coupling between the roughness features, stray field of the domain walls, and exchange interactions between the Fe layers.[7]

Magnetic properties of the multilayer structures are reflected in a way that depends on the experimental technique that is applied. This is important, in particular, in the case of the magneto-optical Kerr effect (MOKE) due to the depth variations of its sensitivity. Any other restriction, such as if the available magnetic field is insufficient to saturate the sample [quite common if the MOKE analysis is performed *in situ* under ultrahigh vacuum (UHV) conditions], combined with the MOKE specificity can make an interpretation difficult. The aim of this article is to show how ''exotic'' loops can be measured and how to interpret them in the frame of magnetic and magnetooptical interactions actually existing in the sample.

The Fe/Cr/Fe/MgO/Fe structures were grown epitaxially on GaAs(001). We discuss their magneto-optical properties in the case that the magnetization of the Fe electrodes is oriented antiparallel. This is achieved by pinning the magnetization of one of the Fe electrodes to another Fe layer (of

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different thickness) separated by a Cr spacer of appropriate thickness corresponding to antiferromagnetic coupling between them.

II. EXPERIMENT

The sample preparation and characterization were carried out in an UHV multichamber system equipped with molecular beam epitaxy, Auger electron spectroscopy (AES), low energy electron diffraction (LEED), scanning tunneling microscopy, and *in situ* MOKE analytical techniques. MOKE loops were collected in the longitudinal geometry by using an electromagnet (with a maximum field of 30 mT), whose axis makes a nonzero angle with respect to the specimen surface. Therefore, the perpendicular magnetization can also be detected, since it gives an ellipticity of about 1 order of magnitude higher than the in-plane magnetization.[8]

The GaAs substrates were cleaned by 500 eV Ar^+ sputtering at 590 °C. After the cleaning procedures were completed, no traces of contamination were detected in the AES spectra and sharp LEED patterns were observed. The cleaning procedure resulted in a Ga terminated (4×6)-like reconstruction, which is found to protect the Fe film against strong intermixing with As and Ga.[9,10]

Fe, Cr, MgO, and Au were deposited at a rate of 1–1.5 ML/min by electron beam evaporation from thoroughly outgassed high-purity iron, chromium, and MgO rods, as well as from a Mo crucible filled with Au. In most cases, the growth was carried out at room temperature at a pressure below 4×10^{-10} mbar (maximum pressure after a long deposition of Fe).

All MOKE data reported here are confirmed by superconducting quantum interference device (SQUID) measurements and interpreted with respect to the magnetization data obtained after the saturation field was applied. Magnetization measurements were made using a Quantum Design SQUID magnetometer. Since the magnetic field was controlled by the current, all low-field loops for $H<5$ mT were measured independently of high-field loops for $H<250$ mT to avoid the creep of the remnant field in the superconducting magnet. The remnant field which was of order of 0.5 mT was accounted for by the independent measurement of the magnetic field before and after each low-field loop.

III. RESULTS

LEED patterns and high-resolution transmission electron microscopy images confirm a good crystallographic order in the bottom Fe (or Fe/Cr) and MgO films, as well as in the Au cover layer of our GaAs(001)/Fe/Cr/Fe/MgO/Fe/Au type structures.[4] All the Fe films are magnetized in plane. The [110] direction, which is a clear easy axis of magnetization for Fe films grown on GaAs(001),[11] remains an easy axis of magnetization also for Fe/Cr/Fe multilayers grown on GaAs(001). When the thickness of the Cr spacer layer corresponds to the antiferromagnetic exchange coupling between the Fe layers, a ''layered antiferromagnet'' of uniaxial magnetic anisotropy is realized with the two Fe layers.[6] Due to the exchange coupling effect, the magnetization reversal in the bottom ''layered antiferromagnetic'' Fe films proceeds independently of that in the top Fe film of the junction, which is separated from the bottom Fe layers by the insulating MgO (Fig. 1). The top layer is thus called the ''free-Fe'' layer. In the case presented here, the magnetization of the middle Fe layer is not free but antiferromagnetically coupled to the bottom Fe layer. Due to this coupling the lower two Fe layers switch simultaneously at moderate fields below ''spin–flop'' transition. This antiferromagnetic (AFM) switching field of such a structure differs from the coercivity of the ''free-Fe'' layer and the magnetization of both electrodes switches independently.

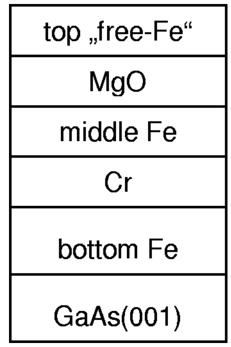

FIG. 1. Schematic view of the complete sample. The magnetization in the middle Fe layer is antiferromagnetically coupled to the bottom Fe layer. The magnetization of the top ''free-Fe'' layer switches independently.

A series of experiments were performed for GaAs(001)/Fe/Cr/Fe samples of 20 ML thickness of the bottom Fe layer and 9 ML thickness of the Cr spacer, which corresponds to AFM coupling (e.g., Ref. 12). The thickness of the top Fe layer is varied between 10 and 20 ML. Within the thickness range of the top Fe layer between 10 and ~18 ML, the minor MOKE hysteresis loops (measured at ±30 mT) are qualitatively the same (Fig. 2), showing negative ellipticity with respect to the positive ellipticity at saturation in positive fields (and, likewise, positive ellipticity in negative fields). The ellipticity in remanence varies due to the varying thickness of the top Fe layer. The net ellipticity in remanence does not correspond to the net magnetization resulting from a simple difference in the thickness of both Fe layers; however, it decreases with increasing thickness of the top Fe film.

The magnetization reversal seen within the loops (Fig. 2) corresponds to the field, at which the magnetization of both Fe layers simultaneously switches, keeping their antiparallel (AFM) orientation. The AFM switching field is dependent on the thickness of the top Fe layer with reference to the bottom one. While the thickness of the bottom Fe layer is kept constant, the AFM switching field increases with increasing thickness of the top Fe layer. This is seen in Fig. 2 plotting the MOKE signal for the samples GaAs(001)/20Fe/9Cr/xFe, $x=10$ and 15, after the external magnetic field is applied along the [110] direction.

After deposition of the MgO insulating barrier and the top Fe electrode of the Fe/MgO/Fe junction, the loops measured by MOKE for the complete structure (schematically shown in Fig. 1) become more complex. This is shown in Fig. 3 for the GaAs(001)/20Fe/9Cr/15Fe/7.5MgO/10–15Fe samples. Qualitatively, the shape of the loop can be easily

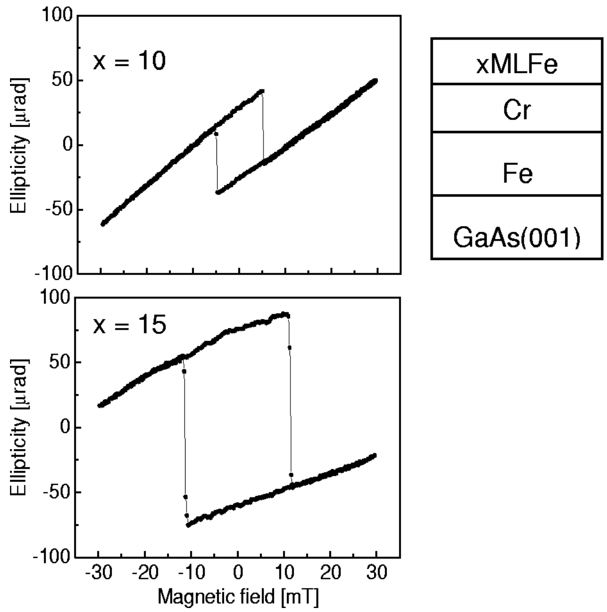

FIG. 2. MOKE loops measured at 300 K along [110] direction for GaAs(001)/20Fe/9Cr/xFe in external magnetic field up to ± 30 mT. The loops are ''reversed'' due to the magneto-optical interactions. The magnetization of both Fe layers reverses at the AFM-switching field that increases with increasing x.

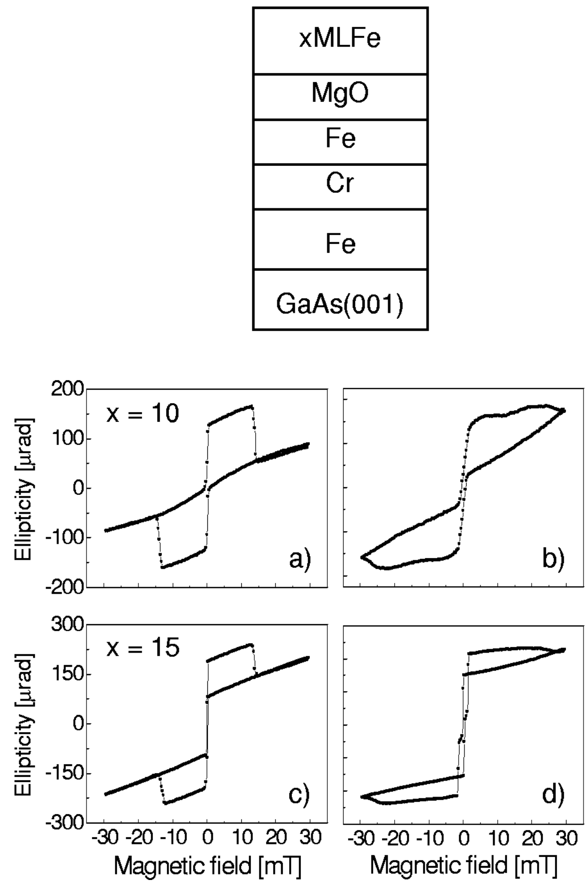

FIG. 3. MOKE loops measured at 300 K for GaAs(001)/20Fe/9Cr/15Fe/7.5MgO/xFe ($x=10$ and 15) along the [110] direction, (a) and (c), respectively, and along the [100] direction, (b) and (d), respectively. The loops result from superposition of the MOKE signal measured for GaAs(001)/20Fe/9Cr/15Fe (shown, e.g., in Fig. 2) and a low-coercivity loop of the top Fe layer. The maximum available field of 30 mT is not sufficient to reorient the magnetization of the Fe/Cr/Fe structure if applied along the [100] direction.

deduced combining the loop measured for the GaAs(001)/20Fe/9Cr/15Fe sample (shown in Fig. 2) with the loop of a single 10 ML thick Fe film characterized by a smaller coercivity. The top Fe film (10 ML) behaves like a ''free''-Fe layer because the MgO spacer magnetically separates it from the rest of the structure. The AFM switching field of the Fe/Cr/Fe structure is increased in comparison to that measured before the MgO/Fe deposition (compared to Fig. 2). This is reasonable assuming that the top ''free''-Fe layer is not perfectly separated from the Fe/Cr/Fe structure (for details see Grabowski *et al.*[13]): it is easy to imagine that it is more difficult to switch the magnetization while the ''free″-Fe layer is (weakly) ferromagnetically coupled to the rest of the structure. Any further increase of the ''free″-Fe layer thickness does not result in any qualitative changes of the loop, except of the relative contribution of both components (of the Fe/Cr/Fe structure and ''free''-Fe layer) to the total loop [Figs. 3(a) and 3(b)].

The AFM switching field is higher if the external magnetic field is applied along the [100] direction, as shown in Figs. 3(b)–3(d) in comparison to Figs. 3(a)–3(c). Even the maximum field of 30 mT that we can apply in our experimental setup seems to be insufficient to switch the magnetization along the [100] axis. This is due to the uniaxial anisotropy with the easy axis of magnetization oriented along the [110] direction, which exists in the Fe films grown on GaAs(001). This uniaxial anisotropy makes magnetization along [100] harder, although this axis is an easy axis in bulk bcc Fe(001). The relation between anisotropy along [100] and AFM coupling differs from that along the [110] direction. This can result in the increased AFM switching field along [100] or even in a coherent magnetization rotation instead of the sharp switching observed along the [110] direction.

In the case of a 2 ML MgO spacer, the minor loop measured with MOKE completely changes its character in comparison to those shown in Figs. 3(a) and 3(b). This is plotted in Fig. 4 for the GaAs(001)/20Fe/9Cr/17Fe/2MgO/10Fe sample. For a top Fe electrode of 10 ML thickness, the loop is slightly ''s-shaped'' and the AFM switching field is about 13 mT. With increasing the top layer thickness, the loop becomes more rectangular and the AFM switching field decreases (not shown in Fig. 4). The loop is no longer ''reversed.'' One can expect that the top Fe electrode is ferromagnetically coupled to the middle Fe film for this low thickness of MgO, and thus both middle and top act as one Fe layer of the GaAs(001)/Fe/Cr/Fe structure (20Fe/9Cr/(17+10)Fe). However, even then, the observed ellipticity in remanence does not correspond to that expected from the difference in thickness of the combined top and the bottom Fe layers.

IV. DISCUSSION

Let us first discuss the energetics of the ''layered antiferromagnet.'' Below the spin–flop transition the two Fe layers

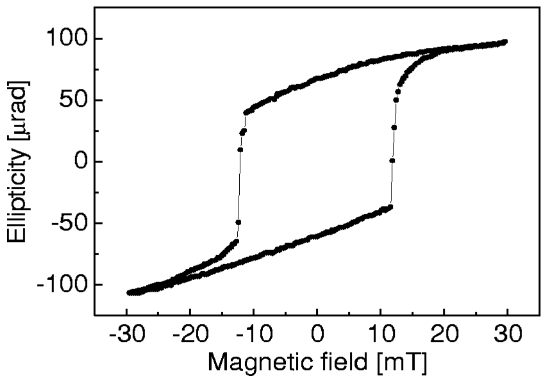

FIG. 4. MOKE loop measured at 300 K for GaAs(001)/20Fe/9Cr/17Fe/2MgO/10Fe along [110] direction. The loop is no longer reversed due to a ferromagnetic coupling between the Fe layers separated with too thin MgO layer: these two Fe layers together (17+10=27Fe) behave like a film thick enough to be magnetized along the applied field. Although the bottom Fe layer (20 ML) is magnetized in opposite direction, it contributes less to the total signal than the top Fe layers together. Finally, the net ellipticity remains positive at positive fields.

are magnetized in opposite directions. As a consequence the Zeeman energy of the ''layered antiferromagnet'' is proportional to the difference of thickness of the two layers. When a magnetic field above the coercivity is applied, the thicker layer aligns along the field. Under the assumption that both Fe layers contribute to the MOKE signal proportional to the magnetic moment, it is obvious that when the MOKE signal of the thicker Fe layer dominates over the signal of the thinner Fe layer a normal MOKE loop is measured. In the case where the contributions of both Fe layers to the total MOKE signal are exactly the same but of opposite sign, no loop is detected within the corresponding field range.[13] However, under the above assumption under no circumstances can a reversed loop be observed. The simplest explanation of the reversed loop (see Figs. 2 and 3) is a depth variation of the MOKE signal which must be taken into account above the ultrathin regime.[14] Even if the bottom Fe film is thicker and thus magnetized along the applied field, it is placed deeply inside the structure and thus contributes less to the total MOKE signal than expected from its magnetization (which is proportional to the thickness). The top Fe layer, which is thinner than the bottom one, is magnetized opposite to the applied field due to its AFM coupling to the bottom Fe layer. It can happen that this thinner top Fe layer contributes more to the total MOKE signal and thus determines the negative net ellipticity. The minor loop must change its character again when the thickness relation favors a dominant contribution to the total MOKE signal of the Fe layer that is magnetized along the applied field. Consequently, a sharp transition from reversed to normal loops has to be observed upon increasing the top Fe layer thickness. It is obvious that with increasing thickness of the top Fe layer above the thickness of the bottom one, the top Fe film becomes magnetized along the field. Being placed closer to the structure surface, the top Fe film contributes more to the net ellipticity and corresponding loops again have to be normal. This is exactly the case of the GaAs(001)/20Fe/9Cr/17Fe/2MgO/10Fe (Fig. 4). The direct magnetic coupling in this case, which is due to the very thin MgO layer, causes the structure to behave like a 20Fe/9Cr/27Fe trilayer. The top Fe layer (27 ML) is now thicker than the bottom one (20Fe) and thus the top one is magnetized along the applied external magnetic field. Due to the AFM coupling through the Cr spacer, the bottom Fe layer (20 ML) is magnetized in opposite direction. Again, due to the depth variations of the MOKE sensitivity, the contribution of the bottom Fe layer to the MOKE signal is expected to be smaller in comparison to that expected from its thickness. However, this results in an even smaller contribution of the negative component to the net ellipticity and supports a dominant positive contribution of the top Fe layer to the total MOKE signal. In this case, the top Fe layer which is thicker (27 ML) and thus magnetized along the applied field, contributes more to the total MOKE signal and thus determines the positive net ellipticity. This is why the minor loop is not reversed for this sample. The situation is equivalent to a case where the MOKE signal of the thicker top Fe layer of the Fe/Cr/Fe structure (which is magnetized along the field in this case) dominates over the signal of the thinner bottom Fe layer (magnetized opposite to the field). The net ellipticity is always determined by the Fe film most contributing to the total MOKE signal due to the combined effect of its thickness and location relative to the incoming laser beam.

The effect of the reversed minor loops originates in the same depth variation of MOKE signal which is responsible for decreasing signal from the ferromagnetic layer if it is placed deeply inside the structure in comparison to the signal measured for the individual layer of the same thickness. Such behavior is often observed (e.g., Refs. 4 and 15) and usually interpreted qualitatively by the finite optical path length through the system. A large difference in sensitivity between two different FM layers in the Kerr rotation and ellipticity responses is reported for the NiFe/FeMn/Co system and is suggested to be caused by a large Fresnel reflection at both interfaces.[16] The interpretation based on thickness variation of the MOKE signal is confirmed with individual contributions of all Fe layers to the total MOKE signal predicted from the magneto-optical calculations (published elsewhere).[17] The negative ellipticity signal occurs due to the relative phase shift between the waves reflected from the top and bottom Fe layers. The contributions from both Fe layers are close to zero and thus their magnitude is strongly influenced by the phase difference which emphasizes the signal of the thinner layer located at the top of the stack.[17]

The minor loops measured for Fe/Cr/Fe structures are characterized by the AFM switching field, i.e., the field at which both Fe layers reverse their magnetization. The AFM switching field should depend on the thickness of one of the Fe layers with respect to the thickness of the second one. This is shown in Fig. 5 where we have plotted the value of the AFM switching field versus relative thickness of the top Fe layer (d_2) against the thickness of both the Fe layers (d_1+d_2). In order to exclude any uncertainty resulting from even a small variation in thickness of the Cr spacer layer, the corresponding loops were measured step by step after each codeposition of Fe to the top Fe layer (d_2) of the particular sample of $d_1=20$ ML (the corresponding loops are shown in

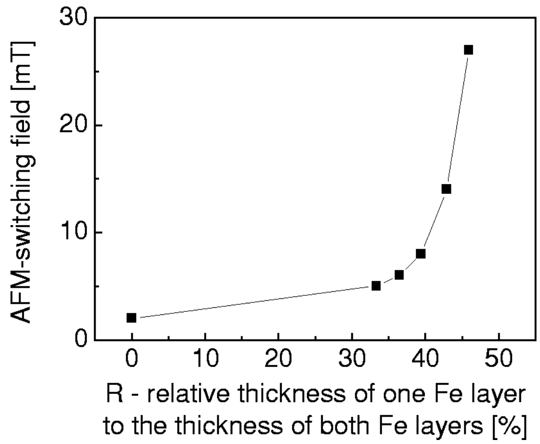

FIG. 5. AFM switching field vs relative thickness of the top Fe layer (d_2) against the thickness of both the Fe layers (d_1+d_2), i.e., $R=d_2/(d_1+d_2)$.

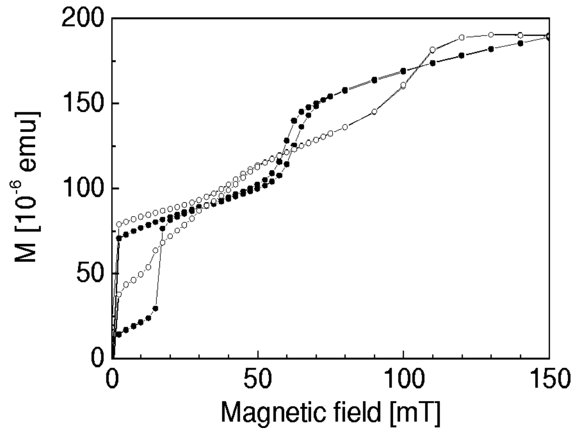

FIG. 6. SQUID magnetization curves for GaAs(001)/20Fe/9Cr/15Fe/7.5MgO/15Fe sample, measured at 300 K along [110] (full dots) and [100] (open circles) directions (only the first quadrant is shown). For the top "free" Fe layer the [100] direction appears as the easy axis of magnetization (see at very low fields), whereas the AFM switching proceeds easier if the field is applied along [110] (see at about 20–40 mT). All spins become aligned parallel just above 60 mT if the field is applied along [110]. Higher fields are required to saturate the sample along [100] direction.

Fig. 2). The experimental points follow the phenomenological expectation of increasing AFM switching field with increasing thickness d_2 of the top Fe layer. For very small d_2, i.e., for the top Fe layer very thin with respect to the bottom one, the magnetization of the Fe/Cr/Fe structure switches at the coercivity of the bottom Fe layer (which is, in this sense, equivalent to the lowest AFM switching field of the system). With increasing d_2, the AFM switching field becomes influenced by the top Fe layer, which is magnetized in opposite direction. As the AFM coupling is stronger than the Zeeman energy of the top layer, the net torque on both layers is reduced while the magnetocrystalline anisotropy does not vary much. As a consequence, the top layer compensates for part of the magnetic moment of the structure and the AFM switching field is increased. It reaches a maximum, when both layers are of the same thickness. At this point, the reversal most likely does not proceed by AFM switching anymore. A process that involves rotation processes becomes more likely, causing an upper limitation of the coercivity.

In order to verify the interpretation of the reversed MOKE minor loops (Fig. 2), SQUID measurements were performed for the GaAs(001)/20Fe/9Cr/15Fe/7.5MgO/15Fe sample which showed the reversed minor loop when measured with MOKE in magnetic fields up to ± 30 mT [see Fig. 3(b)]. The SQUID results obtained for fields (up to 200 mT) applied both along [100] and [110] direction, are shown in Fig. 6. A clear contribution of the "free"-Fe layer, characterized by a small coercivity, is visible within the loop. Along the [110] direction, the magnetization of the Fe/Cr/Fe trilayer reverses at the field of about 12 mT which agrees perfectly with the value obtained by MOKE analysis [compare to Fig. 3(c)]. After the magnetization is reversed, the magnetization of the thicker bottom Fe layer is oriented along the applied field, whereas the magnetization of the thinner top Fe layer is oriented opposite to the field. Obviously, this means a larger net magnetization of the system in comparison to the case of opposite magnetization orientation in the individual Fe layers. Thus, a sharp magnetization increase is visible within the SQUID loop in contrast to the reversed MOKE loop. As seen in Fig. 6, sharp AFM switching does not persist along the [100] direction. In this case, the antiparallel alignment with the magnetization of the thicker Fe layer oriented along the field is achieved by rotation at the field of about 50 mT and remains stable after the field is reversed. This is why the field of 30 mT, i.e., the maximum field applied *in situ* for MOKE analysis, was not sufficient to saturate this configuration [compare to Fig. 3(d)]. To reverse the magnetization of the AFM-coupled Fe/Cr/Fe trilayer along [100] is more difficult due to the uniaxial anisotropy that prefers magnetization along the [110]. The anisotropy along [100] is small and thus the AFM coupling dominates the magnetization reversal process. Consequently, a much higher magnetic field is required to reorient the magnetization of the Fe/Cr/Fe trilayer along the [100] direction. The spin–flop transition occurs only if the film is magnetized along [110], at the field of about 60 mT. This kind of behavior was predicted a long time ago from the total energy calculations of an antiferromagnetically coupled multilayer system.[18] The possible relative orientations of magnetization depend on anisotropy symmetry and direction of the applied field. In the case of uniaxial magnetic anisotropy and the field applied along the easy axis ([110] in our case), the perpendicular magnetization orientation is expected when the antiferromagnetic coupling dominates over the anisotropy energy. In the case when cubic anisotropy symmetry is considered in the total energy and the field is applied along the easy axis of magnetization, no perpendicular orientation is expected. This agrees with the result of our SQUID analysis in the case where the field was applied along the [100] direction. Nevertheless, the Fe/Cr/Fe structure is fully saturated (all spins parallel to the applied field) along [100] at lower field in comparison to that where the sample is saturated along [110]. Details concerning interlayer coupling and magnetization reversal process in the Fe/Cr/Fe trilayers grown on GaAs(001) are discussed elsewhere.[13]

V. CONCLUSIONS

We have shown complex MOKE loops measured for the combined AFM-coupled Fe/Cr/Fe trilayers and Fe/MgO/Fe magnetotunnel structures epitaxially grown on GaAs(001). While MOKE was measured *in situ*, magnetic fields up to ± 30 mT were available, allowing only measurement of minor loops which were found to be reversed. The loops were measured for samples of varying Fe thickness of one of the Fe layers with respect to the second one (i.e., varying the AFM switching field). By careful analysis and SQUID measurements we found that the minor loops are reversed due to the depth variation of the MOKE signal. We have confirmed that the net ellipticity is determined by the Fe film most contributing to the total MOKE signal due to the combined effect of its thickness and position against the incoming laser beam. The complex shape of the loops of the complete GaAs(001)/Fe/Cr/Fe/MgO/Fe structures was deduced by combining the loops measured for the GaAs(001)/Fe/Cr/Fe structures with the loop of a single Fe film characterized by a small coercivity. Finally, we have shown that the MOKE loops interpreted in this way perfectly fit the magnetization measured by SQUID.

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