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Oscillations and change of sign in indirect exchange coupling of Fe/Au/Tb trilayer structures

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Indirect exchange coupling between Fe and Tb layers through an Au layer showed oscillatory behavior with the thickness of the nonmagnetic metal. Different experimental tools such as polar magneto-optical Kerr effect, ferromagnetic resonance, and magnetotransport were used to investigate Fe/Au/Tb trilayers with Au thickness varying from 0 to 3.5 nm, prepared in an MBE system. From the experimental data we reconstruct the dynamics of the Fe and Tb magnetic moments with increasing thickness of the Au interlayer and show for the first time that there is a change of sign in the interaction between Fe and Tb, which is observed experimentally.

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Introduction

Rare earth–transition metal (RE–TM) alloys and compounds have been of fundamental and practical interest for many years [1]. The presence of various exchange interactions (RE–RE, TM–TM, RE–TM) makes understanding of physical processes complicated, though on the other hand they show the way to create new magnetic materials with novel properties. There are well known examples of high-energy permanent magnets created on the basis of $RECo_5$ and magneto-optical media for rewriteable memory storage based on RE–CoFe amorphous films. The main feature of these artificial materials is the ferrimagnetic ordering, meaning that RE and TM magnetic sublattices are oriented antiparallel with perpendicular anisotropy and have a compensation point at a definite composition. The major source of the perpendicular magnetic anisotropy is known to be single-ion anisotropy of the RE ion, which possesses an orbital angular momentum [1]. This is also valid for RE/TM multilayer films, where an interface region gives the main contribution to the perpendicular anisotropy [2,3]. The exchange interaction in this system is fairly complex: the magnetic moments of the *3d* shells of the TM atoms are thought to participate in the direct interaction exchange, whereas the orbital moments of the deep *4f* shells of the RE atoms need mediation of the conduction electrons for their indirect exchange [4]. The polarization of conduction electrons as a result of spin interaction with the TM sublattice also has a significant influence on the exchange interaction in the RE sublattice, which is manifested, for example, in a steep rise of the Curie temperature (T_C) for the Tb–Fe alloy [5] compared with the T_C for pure terbium [6]. According to the RKKY (Ruderman–Kittel–Kasuda–Yosida) model the polarization oscillates in strength and sign with increasing distance from the magnetic ion.

Oscillatory interlayer coupling between ferromagnetic metal layers separated by nonmagnetic metal spacer has been observed in many systems: Fe/Cr, Fe/Cu, Fe/Al, Fe/Ag, Fe/Au, Fe/Pd [7]. Nonmagnetic layers adjacent to the ferromagnetic layers become spin polarized and these atoms develop magnetic moments [8,9]. As the interlayer thickness is varied, the exchange coupling of the magnetic layers is found to vary in sign, oscillating between antiferromagnetic and ferromagnetic coupling. This is manifested, for example, as an oscillation in the magnitude of the GMR effect with increasing separation of the magnetic layers [10,11].

The authors of Refs. 12 and 13 investigated Co/X/Gd multilayers with X = Pt, Cu, Y, hoping to see oscillatory behavior of the coupling. However, in contrast with the results of Gd/Y/Gd [14] and Co/Cu/Co [15], where the oscillatory behavior of the coupling is well known, no oscillation of the coupling was found.

Hoffmann and Scherschlichtis reported their study of Tb and Fe multilayers separated by different nonmagnetic metal interlayers (Au, Ta, Pt) [16]. It was shown that the net magnetic moment of this system oscillates weakly with interlayer thickness, although *the indirect coupling did not show a change in sign*.

In this article we describe measurements of Fe/x Au/Tb, x = 0–35 Å, bi- and trilayers carefully grown under clean conditions in an MBE system, to investigate the effect of interlayer thickness. We show that oscillations of the net magnetic moment are seen in magneto-optical, magnetotransport, and magnetic resonance measurements. Moreover, we demonstrate for the first time experimentally that the *indirect coupling changes sign* with interlayer thickness.

Experimental details

Two sets of Fe/Au/Tb trilayers were prepared on quartz and silicon substrates by electron-beam evaporation in an MBE system having a background pressure of $1\cdot10^{-10}$ Torr and a pressure of better than $1\cdot10^{-9}$ Torr during the film growth. To minimize interdiffusion of the layers the substrate temperature during evaporation was kept near 0 °C. The rate of evaporation did not exceed 0.4 Å/s and was controlled with a calibrated quartz crystal monitor. Samples on quartz substrates were protected with 30 Å thick layer of Al_2O_3, whereas samples on Si substrate were capped with 30 Å Au layer. Polar magneto-optical Kerr effect (PMOKE), ferromagnetic resonance (FMR), and magnetotransport methods were used to characterize the films magnetically. PMOKE was measured at room temperature using a 630 nm laser in an applied field up to 1.8 T perpendicular to the film plane. The FMR was measured at room temperature by means of conventional modulation rf spectrometer at 9.41 GHz with an applied magnetic field (up to 0.7 T) in the film plane. Extraordinary Hall effect (EHE) and magnetoresistance (MR) of the trilayers were measured using standard techniques. The required five electrical contacts were made on the film samples using Ag paint, and the offset voltage in the Hall configuration was compensated in zero magnetic field. Hall voltages (in the range of millivolts for the film on the quartz substrate and a few tenths of a millivolts for those on the silicon substrate) were measured, using a 1 mA current with an applied field of 9 kOe perpendicular to the film plane, whereas the MR measurements were done with field both in plane and perpendicular to it. The thickness of the individual layers, 3 monolayers for Fe (d_{Fe} = 3 ML) and 3 monolayers for Tb (d_{Tb} = 3 ML), were chosen on the basis of the previous experiments in which the ferrimagnetic ordering of Fe/Tb multilayer has been shown [17]. Also it was shown for the control films that the 3 ML = 8 Å Fe film is ferromagnetic at room temperature, whereas the 3 ML = 12 Å Tb film is paramagnetic down to 5 K [18].

Results and discussion

Two periods of oscillation

Understanding of the magnetic coupling via different spacers between TM and RE layers (in ferromagnetic or paramagnetic state) is lacking. However, the phenomenon of magnetic coupling for two TM ferromagnetic layers separated by a nonmagnetic spacer is better understood [7,10,19,20]. Information about the properties of this latter system can be useful in understanding the nature of coupling that is being investigated here. It was shown that the amplitude of the coupling strength (exchange constant) and the period of oscillations depend on the material and thickness of interlayer, whereas the phase of the exchange coupling oscillations depended upon the properties of the ferromagnetic layers.

Theoretical works for noble-metal spacers based on the RKKY model have predicted two oscillations of the interlayer coupling with the spacer thickness, reflecting the topological properties of the Fermi surface [21]:

$$J_{\text{inter}}(d) = 1/d^2[A_1 \sin(2\pi d/\Lambda_1 + \Phi_1) + A_2 \sin(2\pi d/\Lambda_2 + \Phi_2)] \quad (1)$$

where $J_{\text{inter}}(d)$ is the interlayer exchange energy as a function of the spacer thickness d; A_1, A_2 are the amplitudes and Λ_1, Λ_2 are the periods of the energy oscillations. The phases and the amplitude ratio A_1/A_2 have been found to depend critically on sample quality and ferromagnetic layer thickness. It is only for a few trilayers that two periods of oscillations and antiferromagnetic coupling have been observed for spacers thinner than 3–4 ML, e.g., for Fe/Au/Fe [22].

In Fig. 1 the PMOKE signal measured at room temperature is plotted as a function of Au film thickness for Fe/Au/Tb trilayers (top inset), prepared on a quartz substrate. For a free Fe layer we observed an unsaturated signal (bottom inset), while the Fe/Tb bilayer showed a rectangular loop with perpendicular anisotropy [18] (right bottom quadrant). With the introduction of 1 ML of spacer layer (d_{Au} = 3 Å), the PMOKE loop showed that the magnetic moments of the Fe and Tb layers are no longer perpendicular to the film plane. For trilayers with different d_{Au} we observed very narrow loops with no remanence and with a kink, clearly showing two regions with different magnetic susceptibilities. We suppose that the low-field susceptibility is a measure of indirect exchange between Fe and Tb via Au. The difference of the loops

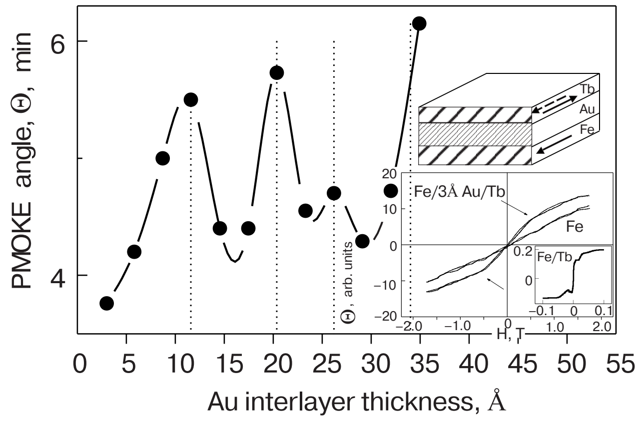

Fig. 1. Oscillation of PMOKE angle for Fe/*x* Au/Tb trilayers as a function of d_{Au} (quartz substrate). Top inset: Schematic diagram of trilayer. Bottom inset: PMOKE loop for the trilayers compared with the control 8 Å Fe film. Arrows show the kink at a critical field H_{cr} where the susceptibility changes. Bottom right quadrant: Fe/Tb bilayer shows rectangular loop with perpendicular anisotropy [18].

for the control Fe film with $d_{Fe} = 8$ Å and for trilayers shows that interaction between Fe and Tb layers still exists. Fragments of the loop at magnetic fields over the kink show that the Fe magnetization is affected by the Au. The PMOKE angle was taken at the kink point. The loop indicates the rotation of the magnetic moments of the layers from the film plane (at $H = 0$) with increasing perpendicular magnetic field: the position of the kink (values of PMOKE angle and magnetic field) oscillates as d_{Au} increased. Oscillations of the Kerr angle with Au thickness having one clear period of about 8 Å and some feature of the second period of 26 Å are observed.

Oscillation of the magnetic moment as a function of Au for thickness greater than 20 Å was seen in sputtered multilayers of $[Fe/Au/Tb]_{12}$ by Hoffman and Scherschlicht [16]. The first period (8 Å, that is 2.8 ML) observed by us on trilayers correlates well with that seen on multilayers. Interestingly, in general we observed both periods for the Fe/Au/Tb system, that is 2.8 ML and 8.9 ML. These correlate well with the oscillation periods 2.5 and 8.6 ML, which were measured for the Fe/Au/Fe system [23] and also with the values of 2.5 and 8.6 ML extracted [24] from measurements of the Au Fermi surface. The facts that the feature of the second oscillation period appears and also the high amplitude ratio, A_{1max}/A_{1min}, show that the growth conditions for the trilayers were good and the interface is sharp. The amplitude ratio of PMOKE angle, $A'_{1\,max}/A'_{1\,min}$, seen in our work, which is proportional to the net magnetic moment, are evaluated as 1.26–1.33 for trilayers on the quartz substrate and 1.64 for the silicon substrate.

The sign change of the coupling

Furthermore, we present direct evidence for the change in the sign of the coupling, alternating between ferromagnetic and antiferromagnetic interaction. From studies of RE–TM amorphous alloy films it is known that the sign of the Hall resistivity changes at the compensation composition due to the change in spin direction [25–28], meaning that either the Fe or Tb magnetic moment dominates in the perpendicular anisotropy. For Tb/Fe multilayers with different layer thickness it was also shown that the Hall voltage dependence is determined by the interface [29,30]. For Fe/Au/Tb trilayers the indirect exchange between Fe and Tb goes by means of polarized conduction electrons of Au. As the two magnetic moments, Fe and Tb, separated by a thin Au spacer with the increase of its thickness gradually come into the film plane, at a small spacer thickness they make some angle with the film plane and hence some perpendicular exchange of indirectly coupled Fe and Tb still exists. Namely polarized conduction electrons of Au become the main carriers of this exchange. On the other hand the Au layer has the lowest electrical resistivity in the trilayer studied ($2.2 \cdot 10^8\ \Omega \cdot$m at $T = 293$ K against 10 and $120 \cdot 10^8\ \Omega \cdot$m for Fe and Tb, respectively [31]). This means that magnetotransport methods, particularly the extraordinary Hall effect, can be very informative for these samples.

Magnetotransport data plotted in Fig. 2 for two sets of trilayers, prepared on quartz and silicon substrates (to increase the range of investigated spacer thickness), show oscillations of the Hall resistivity and magnetoresistance (MR) with Au thickness. The thickness of the Fe and Tb layers was kept the same at 8 Å and 12 Å, respectively, while the thickness of the Au layer was increased. We observed alternate («right» and «left») loops for the extraordinary Hall effect for different interlayer thickness, showing a sign change of the interaction (Fig. 2). The Hall resistivity in Figs. 2,*a,c*, where an Au layer is interposed between Fe and Tb films, also shows that the magnetic moments of Fe and Tb are no longer strong perpendicular to the film plane. This further shows that the Au film presents an indirect interaction between Fe and Tb with some perpendicular component of magnetization still remaining.

Also shown (Fig. 2,*c*) is the Hall resistivity for a bilayer Fe (8 Å)/Tb (12 Å) without Au in between. It is apparent that the positive («right») EHE loops correspond to the parallel coupling, while the negative («left») EHE loops are displayed for the antiparallel

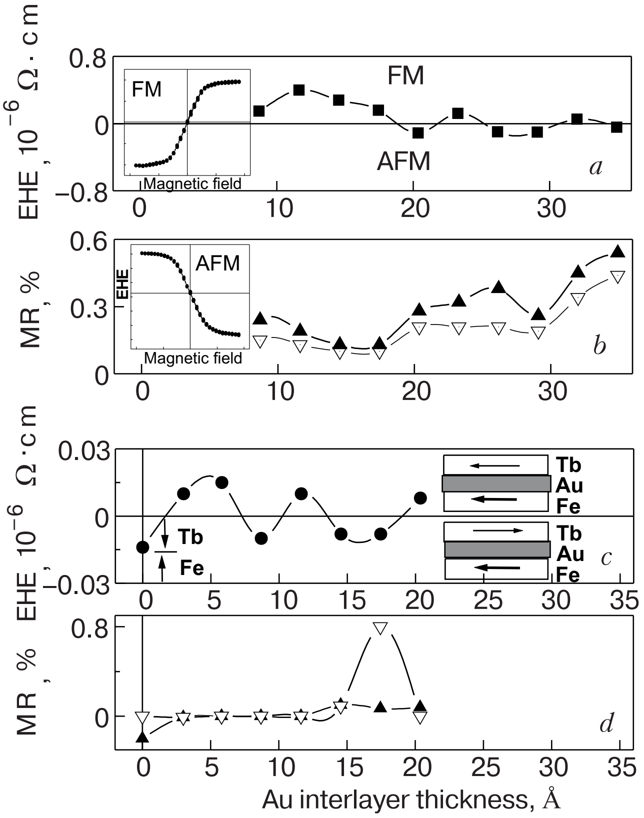

Fig. 2. (*a,c*) Extraordinary Hall effect resistivity and (*b,d*) magnetoresistance (perpendicular – white triangles and transverse – black triangles) as a function of Au interlayer thickness. Samples on (*a,b*) quartz substrate; (*c,d*) silicon substrate. The change of sign in extraordinary Hall effect loops is shown at the left of (*a* and *b*); the top «right» loop corresponds to positive values of EHE resistivity in (*a*), the lower, «left» loop corresponds to the negative values in (*a*). Inset in (*c*): Schematic of the Fe and Tb magnetization orientations as a result of coupling via the Au spacer.

arrangement (Figs. 2,*a*,*c*). The MR variation as a function of Au interlayer thickness in transverse and perpendicular geometries is plotted in Figs. 2,*b*,*d*. Increase in the MR is observed at the periods of the EHE-resistivity modulations, where the antiparallel coupling of Fe and Tb magnetic moments occurs. This is apparent for the samples with low Au thickness (the set of samples prepared on the Si substrate). As the MR signal was very low, the MR maximum could be checked only for the second AFM state ($d_{\mathrm{Au}} = 15$–20 Å). The same trend of MR increase for the antiparallel coupling is seen at higher Au thickness (the set of samples prepared on the quartz substrate).

Also the influence of substrate on the phase of oscillations can be seen in a comparison of the two sets. One observes that Hall resistivity oscillations having the same periods for the two sets of samples demonstrate some shift of phases (Figs. 2,*a*,*c*). It is quite plausible that the Fe layers (being only 3 ML thick) prepared on quartz and silicon substrates (with different surface energies) are not the same and, having some structural differences, may affect the phase of the exchange coupling oscillations.

Ferromagnetic resonance

FMR is known to be one of the most powerful experimental techniques in the study of ultrathin film magnetic properties. The main advance for our case is the high sensitivity providing detailed information about the magnetic and structural quality of thin films up to monolayer; moreover, that resonance can spread to the paramagnetic region [32].

FMR data shown in Fig. 3 display oscillations of the resonance field with Au interlayer thickness for two sets of trilayers, prepared on quartz and silicon substrates. Both kinds of them were chosen among other insulating materials to be used as substrates, with signals that do not overlap the FMR signals of the trilayers.

The H_{res} oscillations correlate well with magneto-optical and magnetotransport measurements as well. For the sets of trilayers prepared on the quartz substrate one can see the similar dependences for the

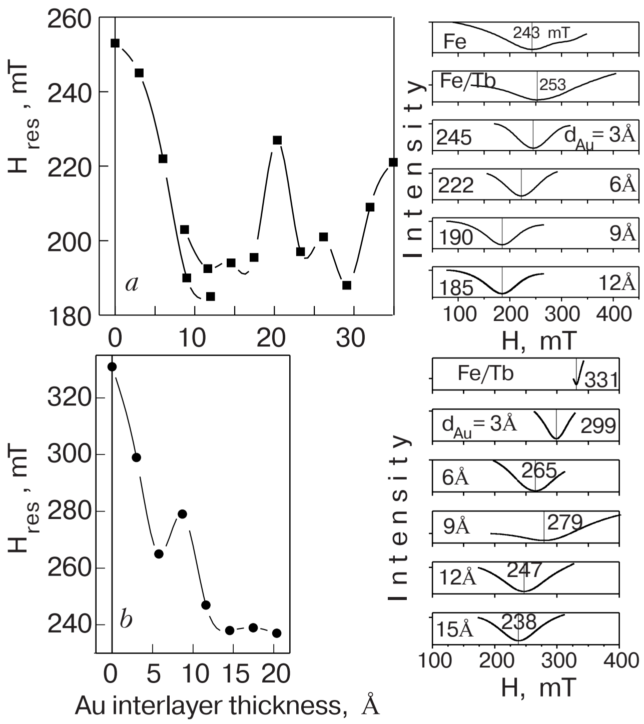

Fig. 3. Resonance fields (H_{res}), obtained (with magnetic field up to 0.7 T, applied in film plane and an rf frequency of 9.41 GHz) from FMR data, plotted as a function of Au interlayer thickness for two sets of samples on quartz (*a*) and on silicon substrate (*b*). FMR signals as a function of magnetic field are also shown at the right, where the resonant fields are also indicated in these figures.

PMOKE and FMR signals for $d_{Au} > 15$ Å, where the maxima of the Kerr angle and H_{res} are found at the same Au thickness, $d_{Au} = 20$, 26, and 35 Å. (Figs. 1 and 3,*a*). They correspond to AFM coupling for the magnetotransport data (Fig. 2,*a*). Samples prepared on the silicon substrate demonstrate AFM coupling at $d_{Au} = 9$ Å (Figs. 2,*c* and 3,*b*).

The FMR signals as a function of magnetic field present the dynamics of magnetic moments in Fe/Au/Tb trilayers. The first FMR signal shown is for the control 8 Å thick Fe film (at the right in Fig. 3,*a*). For the 8 Å Fe/12 Å Tb bilayer H_{res} is shifted to higher fields, indicating the appearance of PMA in bilayers due to the Fe–Tb interaction [18]. Introduction of 1 ML of Au at the interface causes a significant decrease of the resonance field compared to that of the 8Fe/12Tb bilayer. Now H_{res} is almost the same as for the control Fe film. With further increase of Au thickness the resonance field decays, followed by its oscillation between ferromagnetic and antiferromagnetic coupling. For the antiparallel coupling the shape of the FMR line is much wider than for the parallel ordering. The resonance fields are higher for samples prepared on the silicon substrate, again showing the influence of the substrate on the Fe layer [33].

The experimental values of H_{res} for the parallel orientation, substituted into the known Kittel equation [34]

$$\frac{\omega}{\gamma} = \sqrt{H_{res}(H_{res} + 4\pi M_{eff})} \qquad (2)$$

(where $\omega = 2\pi f$ is the microwave frequency, $f = 9.38$ GHz, $\gamma = g\mu_B/\hbar$ is the gyromagnetic ratio, g is the spectroscopic splitting factor, and $\hbar$ is Planck's constant), give the effective magnetization $4\pi M_{eff}$, which includes the input of perpendicular anisotropy due to the change of form factor and to the growth anisotropy, as well. M_{eff} and Θ measured by FMR and PMOKE for the trilayers with different Au thickness, prepared on the quartz and silicon substrates, are shown in Fig. 4. Analysis of these data shows that for both sets of samples up to the Au thickness $d_{Au} < 15$ Å the FMR and PMOKE data dependences correlate, whereas at $d_{Au} > 15$ Å the dependences are found to be in antiphase, i.e., the maximum of the one correlates with the minimum of the other. This means that $d_{Au} = 15$ Å is some critical distance for strong coupling between Fe and Tb. Earlier in a Mössbauer study of Fe/Tb multilayers with different layer thickness it was shown that the radius of coupling between Fe and Tb is about 7–15 Å [35]. Hence at interlayer thickness less than 15 Å both direct (hybridization of the Fe and Tb bands) and indirect (RKKY) interactions via the spacer exist, while at higher thickness only the indirect interaction occurs.

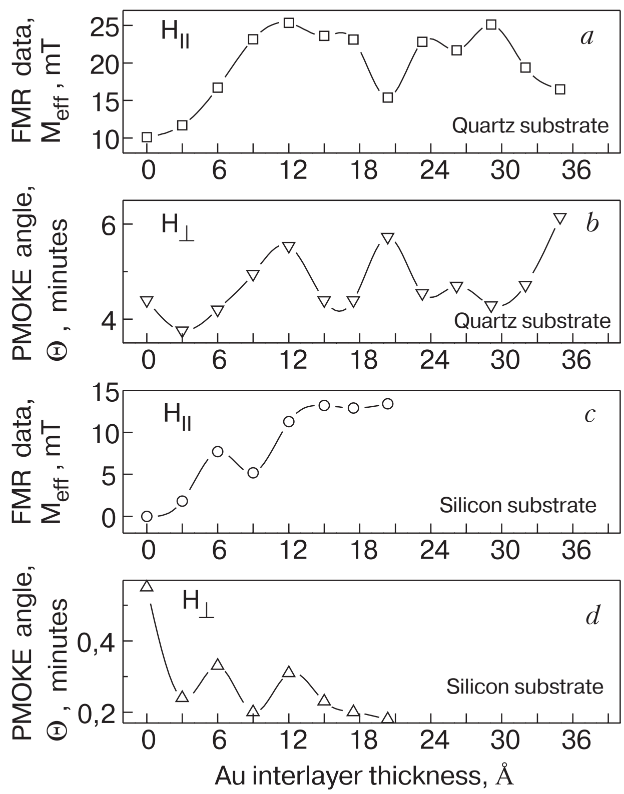

Fig. 4. M_{eff} (*a* and *c*) from FMR measurements and Θ (*b* and *d*) measured by PMOKE for trilayers with different Au thickness, prepared on quartz (*a* and *b*) and silicon (*c* and *d*) substrates.

The main feature of the direct interaction between Fe and Tb layers is the perpendicular anisotropy. When a spacer appears between them and grows in thickness the input of the direct interaction gradually decreases while the input of indirect interaction grows. Our data show that at $d_{Au} > 15$ Å the long-range indirect interaction between Fe and Tb layers has the advantage. At this condition the magnetic moment of the Fe and the induced magnetic moment of the Tb are in the film plane, and measurements by experimental methods with different geometry (FMR and PMOKE) give the opposite results.

Substrate effect

Note that in the PMOKE dependence for the samples on the Si substrate (Fig. 4,*d*), the sample with d_{Au}= 9 Å, showing AFM coupling in FMR and EHE, demonstrated a minimum in PMOKE, contrarily to samples on the quartz substrate. This can be explained by the substrate effect.

We did not use any seed layer (e.g., Ta, Ag, Cu, etc.) while preparing trilayers on Si substrates, as the latter might affect the trilayer interactions. In general using two kinds of substrates and different experimental methods we got information about the main features of interactions and also about the substrate effect. Always the signal for samples on the Si substrate was about one order of magnitude less than on the quartz. This shows that some amount of Fe did not participate in the coupling (because of the appearance of silicides at the Si/Fe interface) [33] and the interaction did not give a significant perpendicular anisotropy input. In other words, the Fe and Tb moments were in-plane (or nearly so) for the Si substrate and out-of-plane for the quartz. As a result, the PMOKE method, which is sensitive to the perpendicular input, showed a maximum for the samples on the quartz substrate and a minimum for those on Si.

Dynamics of the Fe and Tb magnetic moments

Now we can construct the dynamics of the Fe and Tb magnetic moments with an Au interlayer starting with an Fe/Tb bilayer. Without an Au layer, perpendicular magnetic anisotropy is observed in Fe/Tb interface, similar to that which is observed in amorphous Fe–Tb films close to the compensation composition: the Fe and Tb magnetic moment vectors are antiparallel to one another and perpendicular to the film plane. This is seen from the PMOKE and from the large FMR resonance field data. Only one ML of Au interposed between Fe and Tb layers is enough to decrease sharply this short-range magnetic interaction. This is supported by the change of the loop shape in PMOKE [18] as well as by the sharp decrease of the FMR resonance field. With further increase of the Au spacer thickness the magnetic moment vectors change their mutual orientations, coming closer and closer to the film plane, as seen from the EHE and FMR data. Comparison of the PMOKE, FMR, and EHE data show that when $d_{Au} > 15$ Å presumably the main character of the Fe–Tb interaction is the indirect exchange. This interaction is observed up to 12 ML of Au, with an oscillation period of 2.8 ML. At the minima of the oscillations sharp increases in the MR are seen, showing a change of sign for the long-range exchange interaction. It is known that the MR is a measure of the type and strength of antiparallel coupling [36].

The most significant feature of the oscillating mode for the indirect exchange interaction is the weak decay of the modulation amplitude [16]. The presence of oscillations up to 15–17 MLs of Au cannot be expected from RKKY theory (the r^{-3} or r^{-2} decay for a ferromagnetic layer is expected). This coupling feature is unique and needs further investigation.

Conclusions

In summary, carefully prepared Fe(3 ML)/x Au/Tb(3 ML) trilayers under clean conditions display oscillations in the exchange interaction, which can be seen with different methods of characterization. The experimental results correlate well with existing experimental and theoretical data. For the first time it is shown experimentally that:

i) Fe and Tb layers separated by a thin Au layer couple their magnetic moments parallel or antiparallel for different Au thickness, i.e., *the sign of the exchange interaction oscillates*;

ii) EHE is a powerful tool for studying indirect exchange coupling;

iii) at a spacer thickness within the radius of Fe–Tb coupling (7–15 Å) both short- and long-range exchange interactions coexist, while at higher spacer thickness the indirect interaction has the advantage;

iv) the substrate can affect the features and sign of the coupling .

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