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Antiferromagnetic coupling in amorphous Co_xSi_{1-x}/Si multilayers

C. Quirós, J. I. Martín, L. Zárate, M. Vélez, and J. M. Alameda*
Departamento de Física, Universidad de Oviedo, Avenida Calvo Sotelo, s/n, 33007 Oviedo, Spain

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Antiferromagnetic coupling has been observed in amorphous Co_xSi_{1-x}/Si multilayers prepared by co-sputtering on Si substrates. X-ray reflectivity measurements show that the multilayer structure is well defined, with cumulative roughness values around 0.8 nm. Alternating gradient magnetometry and magneto-optical transverse Kerr effect measurements show that the films have in-plane uniaxial magnetic anisotropy and that the Co_xSi_{1-x} layers are antiferromagnetically coupled for Si layer thicknesses lower than 8 nm. The magnetic field required to switch between antiparallel and parallel configurations is as low as 3 Oe. These results are in contrast with those found in reference polycrystalline Co/Si multilayers, which show no evidence of antiferromagnetic coupling.

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The discovery of antiferromagnetic (AF) exchange coupling between ferromagnetic layers separated by nonmagnetic metallic interlayers[1,2] has stimulated intensive work in the study of the magnetic and electronic properties of multilayered systems. These efforts, in combination with the improvement of techniques for fabricating ordered nanostructures,[3] have led to commercial applications in magnetic storage technology and to the development of so-called "spintronics."[4,5] However, when the nonmagnetic metallic interlayer is replaced by a semiconductor, the experimental results are more controversial and their theoretical interpretation is less clear. Particularly, in the case of the Fe/Si system, antiferromagnetic coupling has been reported several times but different oscillatory[6] or non-oscillatory[7] behaviors have been observed. Iron silicide formation at the interlayer, induced by diffusion processes, has been experimentally reported[8,9] and theoretically studied,[10] and a correlation between the composition of this layer and the oscillation of the coupling has been found.[11,12]

In contrast with Fe/Si system, Co/Si-based multilayers have been much less studied, although their technological interest may be especially high due to the suitability of Co-Si compounds as low-resistivity contacts in electronic devices,[13–15] which could save steps in the manufacturing process of devices. Most of the information available comes from the works of Fallon and co-workers.[16–20] They have found a gradual change from ferromagnetic coupling to superparamagnetic behavior when the Si layer thickness is increased. Regarding the structure, they have observed a strong mixing of the layers, especially for thicknesses smaller than 5 nm, with transition regions that, depending on the relative nominal thicknesses of the Co and Si layers, may be an amorphous alloy or a crystalline silicide. It is in good agreement with previous work of Ruterana *et al.*,[13] who have found that sputtered Co films of less than 4–5 nm react with Si atoms coming from the substrate and produce amorphous cobalt silicide. In contrast with these works, Inomata and Saito[21] have reported in a published abstract a change from ferromagnetic coupling, at Si thicknesses below 0.8 nm, to antiferromagnetic coupling, up to 1.7 nm, which disappears with no oscillation for higher thicknesses. The formation of amorphous cobalt silicide in the spacer is also mentioned in this very brief publication without figures, where the information provided is far from complete. Very recently, Luciński *et al.* have also reported results on Co/Si multilayers prepared by dc magnetron sputtering.[22] They have observed stepped hysteresis loops, which have been assigned to AF coupling, and an oscillatory behavior of the normalized remanence, which has been related to the formation of a nonmagnetic Co-Si metallic alloy. On the other hand, regarding the theoretical approach, to the best of our knowledge, only one work by Enkovaara *et al.*[23] has studied Co/Si sandwiches, proposing an oscillatory behavior of the coupling. In summary, the previous works on the system show contradictory results and no clear evidence of AF coupling while, at the same time, they suggest strong diffusion processes that are difficult to control and may be the origin of the different reported magnetic behaviors.

In this work, we have adopted a different approach in order to reduce these diffusion problems that may complicate the detection of weak antiferromagnetic couplings; our multilayers have been prepared using amorphous cobalt silicide as the magnetically active layer instead of pure polycrystalline cobalt. The amorphous material has two main potential advantages: first, due to the different structure, the interdiffusion of Si and Co could be hindered, so that the critical thickness for pinhole formation in the Si spacer layer may be reduced and the possible AF couplings could be more easily detected. Second, it is a softer magnetic material and thus can be more sensitive to any weak coupling present in the system.

Co_xSi_{1-x} films and Co_xSi_{1-x}/Si multilayers have been prepared by dc magnetron co-sputtering on oxidized Si substrates of about 8 mm × 10 mm size. Two high-purity targets of Co (normal incidence) and Si (oblique incidence) have been used at Ar working pressures of 1.0×10^{-3} mbar (base pressure $\approx10^{-9}$ mbar). This Ar pressure is one order of magnitude lower than that in previous works on Co/Si multilayers,[20] and was chosen in order to reduce the roughness that appears at high sputtering pressures[24] and that is known to degrade antiferromagnetic couplings in other multilayer systems.[25] Co_xSi_{1-x}/Si multilayers have been prepared with fixed parameters in the magnetic layer (thickness t_{CoSi} and composition x) and increasing Si layer thickness t_{Si}.

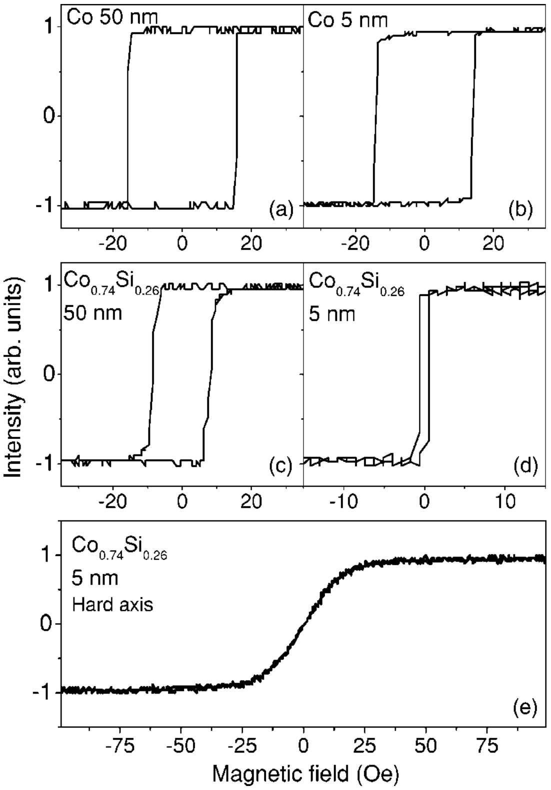

FIG. 1. MOTKE hysteresis loops of pure polycrystalline Co (*a* and *b*) and amorphous $Co_{0.74}Si_{0.26}$ films (*c*, *d*, and *e*) of 50 and 5 nm thickness [note the different field axis scale of panels (d) and (e)]. Loops in panels (a)–(d) have been measured with the field applied along the easy axis. The loop of panel (e) corresponds to the hard axis. Note that all the loops are center-symmetric, so that second order magneto-optic effects are negligible.

Then, in order to select suitable values of the parameters x and t_{CoSi} for the basic building blocks of the multilayers, the structural and magnetic properties of simple Co_xSi_{1-x} thin films must be considered.[26] For high Co concentration ($0.76<x<1$) the films are polycrystalline and the main effect of Si doping is a significant magnetic hardening, whereas for $0.7<x<0.75$, 50 nm thick Co_xSi_{1-x} films are amorphous and present low coercive fields (below 10 Oe) that are almost independent of Si concentration. Thus, alloy films with $x=0.74$ appear as a good choice for the magnetic layers in the multilayers since they have a relatively soft magnetic behavior that will not be altered by possible interdiffusion processes with the neighbor Si layers. All the films have in-plane uniaxial anisotropy, most likely induced by the oblique incidence of Si atoms during deposition, being more pronounced for the amorphous films. Actually, as the thickness of the magnetic layer is reduced the differences in the magnetic behavior of polycrystalline and amorphous Co_xSi_{1-x} films become more pronounced, as it can be seen in Fig. 1 [panels (a)–(d)]. Here, typical hysteresis loops measured by the magneto-optical transverse Kerr effect (MOTKE) applying the magnetic field along the easy axis are plotted for polycrystalline pure Co and amorphous $Co_{0.74}Si_{0.26}$ thin films with two different thicknesses, 50 and 5 nm. As already mentioned, all the films have in-plane uniaxial anisotropy, with typical anisotropy field values at room temperature around 20 Oe for the case of the amorphous alloys, as can be observed in Fig. 1(e), where the hysteresis loop taken when applying the magnetic field along the hard axis of the 5 nm thick amorphous film is shown. The most interesting feature appears when comparing the coercive fields, H_c. All the films have low H_c. For the Co samples it remains almost unchanged when reducing the thickness, from 16 Oe at 50 nm to 14 Oe at 5 nm. However, in the case of the amorphous alloy film there is a strong decrease from 8 Oe at 50 nm to only 0.6 Oe for 5 nm. Such a remarkable low value of H_c indicates that 5 nm thick layers of this amorphous alloy are actually good probes for looking for very weak AF couplings in multilayers, since the low energy needed to reverse their magnetization may be small enough not to hide AF couplings having small values of exchange coupling. Thus, a first series of $(5\ \mathrm{nm}\ Co_{0.74}Si_{0.26}/t_{Si}\ Si)_z$ multilayers has been prepared with Si spacer thicknesses $t_{Si}=2$, 4, 8, and 15 nm for each of the samples. The number of periods z has been set to 10, except for the 15 nm Si spacer multilayer, where just 6 periods have been grown in order to keep the total thickness at a value close to that of the other multilayers. All the samples start with a Si buffer layer on top of the native silicon oxide so that the first $Si/Co_{0.74}Si_{0.26}$ interface is similar to the other interfaces. The last layer is always Si in order to reduce oxidation. Deposition rates have been kept at 0.1 nm/s for Co_xSi_{1-x} and 0.05 nm/s for Si. Also, for comparison, an additional set of pure polycrystalline $(5\ \mathrm{nm}\ Co/t_{Si}\ Si)_z$ samples has been grown and studied. Structural characterization and thickness calibration have been performed by x-ray reflectivity measurements. The software package[24] SUPREX has been used to fit the reflectivity patterns. The magnetic characterization has been carried out by combining alternating gradient magnetometry (AGM) and MOTKE measurements.[27–29] The MOTKE signal has been analyzed using the method, developed in our group,[28] that allows one to obtain detailed information on the magnetization profile and reversal processes in multilayers, as will be shown later.

First, the structural quality of the multilayers has been studied by x-ray reflectivity. The corresponding patterns and fittings are shown in Fig. 2, where it can be seen that the multilayer peaks are well resolved, indicating a well-defined multilayer structure. Also, well-defined Kiessig fringes typical of smooth interfaces are present up to relatively high angles. The parameters obtained from the fitting of the scans using the SUPREX program are summarized in Table I. The experimental multilayer periods Λ^{fit} are found to be smaller than the nominal ones, 7 nm, with most of this reduction corresponding to the thickness of the Si layers. This is typical of an alloying effect at the interfaces by a solid state reaction between Co and Si so that the more dense Co_xSi_{1-x} layers grow at the expense of the Si ones, in a similar way as reported by Fallon *et al.* for Co/Si multilayers of similar modulation periods.[19] This alloying effect can result in an increase of Si concentration in the Co_xSi_{1-x} layers of the order of a few percent. It is worth noting that the final Si layer thicknesses obtained from the fit are of the order of 1 nm, which is the typical length scale where the maximum of AF couplings is observed in other magnetic/nonmagnetic multilayer systems.

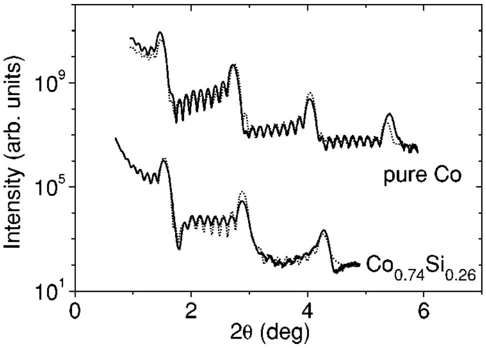

FIG. 2. (Color online). Low-angle x-ray reflectivity measurements of multilayers having a 2 nm thick Si spacer for two different nominal compositions of the 5 nm magnetic layer: $Co_{0.74}Si_{0.26}$ and pure Co. Experimental data are shown by the continuous lines and corresponding SUPREX fits by the dotted lines.

A detailed analysis of the roughness parameters obtained from the fitting reveals a clear asymmetry between the Si-on-Co and Co-on-Si interfaces for both multilayers. The first interface is much sharper than the second one and it can be described by a simple Gaussian concentration profile of width $\sigma_{\text{Si-Co}}$. In contrast, in the second interface, an intermediate $CoSi_y$ compound of about 0.5–0.7 nm thickness, is needed between the Co and Si layers in order to fit the experimental data. This asymmetry has also been found in other metal/Si multilayer systems[30] and is the result of the different diffusion rates of Co into Si from that of Si into Co. This feature is very well defined in the sample with pure Co layers, but it is much more diffused for the multilayer with amorphous $Co_{0.74}Si_{0.26}$ layers, indicating a more homogeneous density profile of the nonmagnetic layer in the latter case.

Finally, it is interesting to point out the low cumulative roughness obtained, indicative of the high quality of the layering in the multilayer that is characteristic of sputtering samples grown at very low Ar pressures. This is an important structural difference with Co/Si multilayers grown at higher sputtering pressures and lower deposition rates, which presented much rougher interfaces.[20]

The magnetic behavior of the set of $Co_{0.74}Si_{0.26}$ multilayers is shown in Figs. 3 and 4 and show the presence of an AF coupling at Si spacer distances of 2 and 4 nm. In Fig. 3, AGM measurements show the hysteresis loop of the multilayer with a 2 nm Si spacer thickness for the magnetic field (H) applied along the easy axis of the layers. The multilayers follow the same trend as the single films, showing in-plane uniaxial anisotropy. The comparison of this loop with that of the single layer used as the basic block [see Fig. 1(d)] indicates the presence of a coupling of the layers that reduces the remanence drastically. Now, MOTKE measurements (shown in Fig. 4) can provide a definitive insight in order to prove the antiferromagnetic alignment of the layers (and rule out a reduced remanence due to a transition to superparamagnetism in the magnetic layers such as that reported[20] for high-pressure-grown Co/Si multilayers). Both samples with 2 and 4 nm thick Si layers do have a remanence that is almost zero (actually it is negative), presenting a plateau around $H=0$ and oscillations after the sign of H is reversed. This nonmonotonic behavior, taking into account that even effects are not significant in our loops, which are center-symmetric, can only be the result of changes in the relative alignment of the individual layers, and is a conse-

TABLE I. Structural parameters obtained by using the SUPREX code to simulate the x-ray reflectivity patterns of multilayers having a 2 nm Si spacer thickness and different compositions of the magnetic layer: Λ^{fit} is the multilayer modulation obtained from the fitting; t_{Si}^{fit} is the Si layer thickness obtained from the fitting; $\sigma_{\text{Si-Co}}$ is the roughness of the Si-on-Co interface; t_{comp} is the thickness of the intermediate Co-Si compound at the Co-on-Si interface; σ_{cum} is the cumulative roughness.

Multilayer	Λ^{fit} (nm)	t_{Si}^{fit} (nm)	$\sigma_{\text{Si-Co}}$ (nm)	t_{comp} (nm)	σ_{cum} (nm)
$Co_{0.74}Si_{0.26}$	6.3	0.9	0.3	0.5	0.8
Co	6.6	0.9	0.0	0.7	0.5

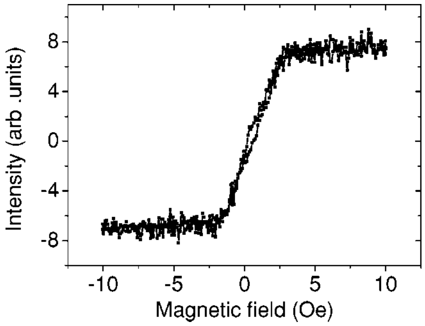

FIG. 3. AGM hysteresis loop along the easy axis of a $Co_{0.74}Si_{0.26}$-based multilayer having a 2 nm Si layer thickness. Note that the remanence is almost zero, although no plateau is observed around zero magnetic field due to the gradient field inherent to the technique, which usually affects fine measurements of soft magnetic samples.

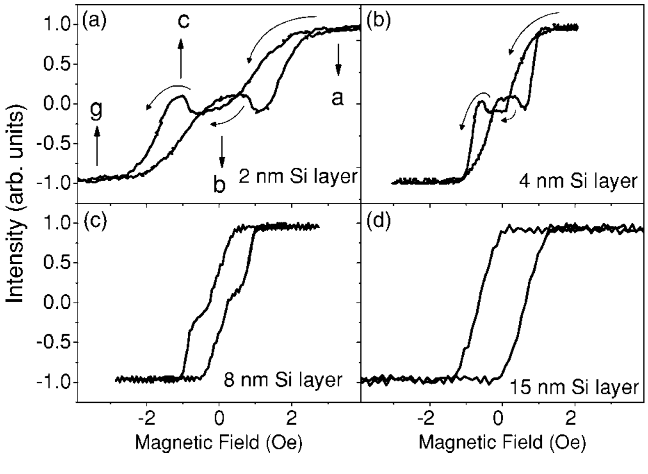

FIG. 4. MOTKE hysteresis loops along the easy axis of $Co_{0.74}Si_{0.26}$-based multilayers for different Si spacer distances: (a) 2 nm, (b) 4 nm, (c) 8 nm, and (d) 15 nm. Labels *a*, *b*, *c*, and *g* in panel (a) refer to some of the magnetic configurations discussed in Table II. Curved arrows indicate the sense of the loop.

TABLE II. MOTKE signal calculated with model of Ref. 28 for the reversal sequence, from positive (↑) to negative (↓) saturation in seven steps (from *a* to *g*), which is compatible with the experimental loop of the multilayer having a 2 nm thick Si spacer [cf. Fig. 4(a)]. *S* indicates substrate side.

Step	↑↑↑↑↑↑↑↑↑↑ S^a	↑↓↑↓↑↓↑↓↑↓ S^b	↓↓↑↓↑↓↑↓↑↓ S^c	↓↓↑↓↑↓↑↓↓↓ S^d
Kerr	1.000	(AF) −0.304	0.298	0.199
Step	↓↓↑↓↓↓↑↓↓↓ S^e	↓↓↓↓↓↓↑↓↓↓ S^f	↓↓↓↓↓↓↓↓↓↓ S^g	
Kerr	−0.269	−0.748	−1.000	

quence of the nature of the transverse Kerr effect in which the contributions of the individual layers of the multilayer to the total signal are not necessarily additive even when they are very thin.[28] This is different from bulk magnetization measurements and also from the two other Kerr effects (longitudinal and polar), which opens the possibility of probing the magnetic alignment of the layers, even in a configuration of vanishing magnetic moment, like that corresponding to the pure AF state. Thus, we have applied the model described in Ref. 28 to investigate the reversal sequences of the layers whose MOTKE signal evolution is compatible with the one observed in the loop of 2 nm Si spacer multilayer. We have obtained that all the possible reversal sequences compatible with the experimentally observed MOTKE signal [see Fig. 4(a)] always imply passing through the AF state. Then, reversing the outer layer leads to the nonmonotonic dependence of the loop. One of the sequences that agrees with the experimental loop is shown in Table II, where the calculated MOTKE signal for each spin configuration is indicated and labeled with the same notation used in Fig. 4(a).

In addition, another confirmation of the AF coupling can be obtained by looking at the magnetic field corresponding to the maximum in the loop [state *c* in Fig. 4(a)], which clearly increases when reducing the Si thickness spacer, being around 0.6 Oe for 4 nm and 1.1 Oe for 2 nm [cf. Figs. 4(a) and 4(b)]. This behavior can be explained by the increase of the AF coupling strength when reducing the semiconducting layer thickness, so that a higher magnetic field is needed in order to break the AF state, switching one of the layers. Interestingly, even the multilayer with 8 nm Si spacers still shows a narrowing of the loop around zero magnetic field and a reduced remanence that is related to this AF coupling [Fig. 4(c)]. Finally, for a Si layer thickness of 15 nm the multilayer shows a loop where the strength of the AF coupling seems to be negligible and not enough to produce the antiparallel alignment of the magnetizations.

The results obtained in the set of multilayers with nominally pure polycrystalline Co layers are completely different. Figure 5 presents the MOTKE loops of the reference series of Co/Si multilayers grown with different Si spacer thickness. In all cases, the loops are almost square, with remanence close to unity, and, therefore, without any evidence of AF coupling in contrast with the $Co_{0.74}Si_{0.26}$-based samples. Interestingly, the coercive fields in these pure Co/Si samples are significantly higher than those in the previous case, so that the usual domain wall pinning mechanisms could easily dominate over a possible AF coupling in determining the final magnetic behavior.

Thus, the magnetic measurements clearly reveal the presence of an AF coupling only in the case of the $Co_{0.74}Si_{0.26}$/Si multilayers, which can be observed due to the softness of the amorphous magnetic layers. This coupling effect has a relatively long decay distance (it is observed across at least up to 8 nm of Si), larger than the typical 2–3 nm distance found for the loss of AF coupling in Fe/Si multilayers,[8] and well above the usual decay lengths of only a few ångstroms calculated in theoretical models based on Fermi surface parameters.[23] Actually, it is interesting to point out that the observed switching fields are extremely low in comparison with reported values in other magnetic/nonmagnetic multilayer systems. For example, saturation fields between 15 Oe (Ref. 31) and 10 kOe (Ref. 8) have been found in the Fe/Si system, whereas in the present case only around 1 Oe is needed to break the AF state by reversing one layer and about 3 Oe to reach saturation, which could have important implications from the applications point of view. Taking into account this value of 3 Oe, the thickness of the films, the saturation magnetization, and the uniaxial anisotropy of the samples, the strength of the coupling can be estimated to be of the order of 10^{-6} J/m^2, which is in the lower range of the values reported for Fe/Si multilayers[31,32] (between 5×10^{-6} and 10^{-2} J/m^2). This very low coupling strength may explain why a clear signal of AF coupled multilayers has escaped detection for so long in the Co/Si system.

As a final remark, it is worth mentioning that preliminary temperature-dependent measurements show that the coercive field of single amorphous films increases continuously when the temperature decreases. As a result antiferromagnetic coupling in multilayers can be observed decreasing the temperature down to around 250 K, but not below. The results indi-

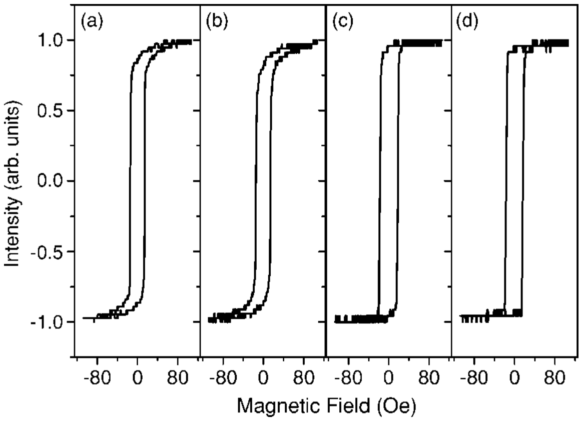

FIG. 5. MOTKE hysteresis loops along the easy axis of pure Co-based multilayers for different Si spacer distances: (a) 2 nm, (b) 4 nm, (c) 8 nm, and (d) 15 nm.

cate that further reduction of T increases the coercive field so that the multilayers start to mimic the magnetic behavior of the single layer, confirming that the coupling can only be observed if the films are magnetically soft enough.

In summary, antiferromagnetic coupling has been observed and analyzed in amorphous $Co_{0.74}Si_{0.26}/Si$ multilayers by using AGM and Kerr measurements. This AF coupling is found to be dependent on the Si layer thickness, being clearly present for 2 and 4 nm, less pronounced for 8 nm, and absent at 15 nm. The switching fields are extremely low, as only about 3 Oe are needed in order to change from ferromagnetic to antiferromagnetic alignment of the layers, what may be of great interest for technological applications.

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*Electronic address: alameda@string1.ciencias.uniovi.es

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