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Enhancement of antiferromagnetic coupling in magnetic multilayers by low energy ion beam substrate nanopatterning

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Abstract

Ion beam irradiation has been shown to be an interesting tool for tailoring the magnetic properties of thin films and multilayers. The modified properties include magnetic anisotropy, interlayer exchange coupling, exchange bias, magnetic domain structure and magnetization reversal. In this work, new results are shown concerning the enhancement, by one order of magnitude, of the antiferromagnetic coupling strength in amorphous CoSi/Si multilayers by irradiating Si(100) substrates with 1 keV Ar^+ ions. The ion beam exposure induces an increase of the substrate roughness, from 0.07 to 0.88 nm, which enhances antiferromagnetic coupling in the magnetic multilayers grown on top. One possible mechanism governing this enhancement is discussed, related to the formation of magnetic/non-magnetic regions where dipolar interactions could stabilize the antiferromagnetic alignment. The presence of non-magnetic regions is suggested by the observed trend to superparamagnetism, and is expected since the Curie temperature of the amorphous CoSi alloy used is slightly above but very close to room temperature. Accordingly, small fluctuations in the local composition, leading to an enrichment of Si, would produce non-magnetic regions enabling dipolar interactions to take place. Furthermore, the ion beam induced increase of roughness makes surface diffusion of the atoms arriving at the sample difficult, favoring the formation of local non-magnetic inhomogeneities. Finally, the role of other possible mechanisms to enhance antiferromagnetic coupling is also briefly discussed.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Low energy (∼1 keV) ion beam irradiation has been shown to be a powerful method for patterning surfaces of metals [1–3] and semiconductors [4–7]. The competition between sputtering, re-deposition, and surface diffusion phenomena induced by the ions can be adjusted by controlling the incidence angle, flux and energy of the ion beam and the substrate structure and temperature. By tuning this set of parameters, radically different morphological effects have been observed, including smoothing, roughening, and ripple or dot formation, so that surfaces structured at the nanoscale can be obtained [8, 9]. Theoretical models have also been developed which are able to explain the experimentally observed morphologies [8–11]. From the point of view of applications, each of these patterns, generated at the substrate level by an ion beam, is of paramount importance as such substrate templates will allow the properties of the thin films grown on top of them to be changed [12].

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In particular, magnetic properties are highly dependent on surface and interface morphology, and some results have been published concerning the modification by ion irradiation of magnetic anisotropy [13, 14] and interlayer coupling of multilayers [15–17]. In relation to this, two reviews have summarized results on the tailoring of magnetism by irradiation with light ions [18] and magnetic patterning by ion irradiation and implantation [19].

Interlayer exchange coupling (IEC) in magnetic multilayers has been intensively studied since it is a complex property with strong implications for the development of several technologically relevant devices, like spin-valves and magnetic tunnel junctions for read heads in magnetic hard disks, and magnetic random access memories (MRAM) [20]. Interlayer coupling can lead to ferromagnetic (F) or antiferromagnetic (AF) alignment of the layers, and the corresponding electronic transport properties of the system may be completely different for both magnetic states, as has been observed in systems having giant magneto-resistance (GMR) or in magnetic tunnel junctions [20].

In the case of IEC through metallic non-magnetic spacers, like Cu, Cr, and Ru, an oscillating behavior between F and AF alignment with the spacer thickness has been observed [20]. On the other hand, for non-conducting or semiconducting spacers like Si or Ge, the situation is less clear, although the theoretically expected exponential decays with spacer thickness [21, 22] have been experimentally observed in some systems [23]. In both cases, the thickness and quality of the interfaces strongly affect the coupling [24]. In particular, dipolar magnetostatic interactions due to correlated roughness, known as ‘orange peel’ effects [25], as well as interactions between the domain walls [26], can give additional contributions to the coupling. Also, if the lateral size of the magnetic structures is sufficiently reduced, magnetostatic stabilization of antiferromagnetic configurations through dipolar interactions from the side of the structures can be obtained [27]. In any case, it is clear that layer thickness, atomic and electronic structure at the interfaces, and roughness are critical parameters that can strongly modify the IEC.

The effect of roughness on IEC has been studied to a greater extent in the case of the Fe/Cr multilayer system. While most of the studies have focussed on the variations of GMR induced by roughness, it should be mentioned that GMR is not only dependent on IEC but also on the electronic transport properties of the multilayers. Hence, care should be taken not to attribute the modifications of GMR to the sole modifications of IEC. In some cases, roughness was varied by changing parameters like substrate temperature, deposition pressure or layer thickness [28–30]. In other cases, GMR modifications were produced by irradiation of the Fe/Cr multilayers with energetic Xe^+ ions of 500 keV [31, 32]. An increase in GMR when increasing the ion dose was observed. However, doses higher than 1×10^{13} ions cm^{-2} led to a reduction of the GMR as more regions of the samples would become ferromagnetically coupled [31]. The formation of pinholes related to the increase of roughness with increasing ion dose, and alloying at the interface due to ion beam induced mixing, have been reported as mechanisms responsible for the degradation of both the GMR and the AF IEC [32].

Concerning the effects of lower energy ions on the strength of IEC, it should be mentioned that fewer results have been reported. Irradiation with 5 keV He^+ was shown to clearly modify the AF IEC observed in Fe/Cr multilayers [15]. Depending on the Cr spacer thickness, the coupling was observed to either increase or decrease. This behavior was related to modifications of the effective interlayer thickness linked to ion induced atomic exchange processes at the interfaces. This result opened the possibility of spatially modulating the coupling of thin films by focused ion beam irradiation with 50 keV Ga^+ ions [33]. The case of synthetic antiferromagnets based on CoFeB/Ru/CoFeB trilayers deposited by ion beam assisted deposition (IBAD) has also shown a clear strengthening of the AF coupling. That effect was attributed to variations of interfacial roughness created during growth by Ar^+ ions biased at 60–100 V [17].

In this work, we report on the enhancement of AF coupling of amorphous magnetic multilayers with semiconducting spacers by using a strategy consisting of low energy ion beam pre-patterning of Si(100) substrates followed by oblique incidence growth of the non-magnetic spacer. This is different from most of the cases previously reported, in which irradiation was performed during or after deposition. The system where this AF coupling enhancement has been observed corresponds to multilayers of Co–Si amorphous films separated by semiconducting Si spacers, which have recently attracted attention because they develop AF coupling [34–36], although its origin still remains unclear. The results presented suggest new strategies for tuning interlayer coupling, and provide new indications about its possible origin for the Co–Si/Si system.

2. Experimental details

As a first step, Si(100) substrates, with their native oxide, were irradiated with ions produced by microwave plasma. Ar^+ ions of 1 keV energy and fluxes in the 10^{13} ions s^{-1} cm^{-2} range were used. The ion gun, installed in a vacuum chamber of beamline BM05 at the European Synchrotron Radiation Facility (ESRF), was set at an angle of 10° grazing incidence with respect to the substrate surface. By adjusting the exposure time, several substrates were prepared with increasing values of root mean square (rms) roughness, denoted σ.

In order to study the effect of ion beam induced nanopatterning on the AF coupling of multilayers, the same magnetic structure, which has previously been shown to be AF coupled [34, 35], was grown on top of all these substrates. This structure consisted of two periods of an amorphous CoSi alloy separated by a Si spacer, with a Si buffer and Si capping to avoid oxidation (3 nm Si/5 nm $Co_{74}Si_{26}$/3 nm Si/5 nm $Co_{74}Si_{26}$/3 nm Si/substrate). Also, samples having just one single magnetic period (3 nm Si/5 nm $Co_{74}Si_{26}$/3 nm Si/substrate) were prepared on top of some of the substrates in order to study the modifications of the magnetic anisotropy and coercive field induced by the ion pre-patterning of the substrate.

The growth of the samples was done by dc-magnetron co-sputtering in a preparation chamber different from the one

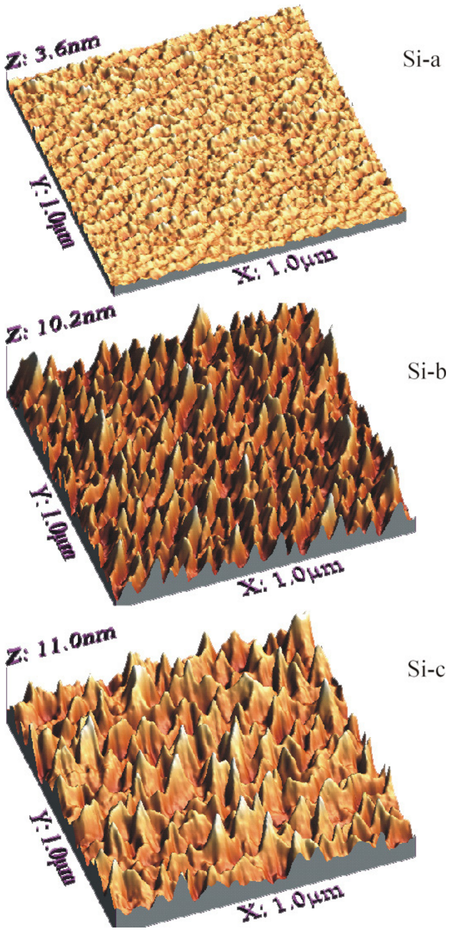

Figure 1. AFM images of Si(100) substrates for increasing times of 1 keV Ar^+ irradiation. The Z-axis scale is the same for the three images and is enlarged with respect to the X and Y axis for clarity. The corresponding rms roughnesses are 0.23 nm, 0.82 nm, and 0.88 nm, respectively.

used for Si substrate patterning. This means that the irradiated substrates were exposed to air prior to deposition. The growth was carried out with a high purity Co target, normal to the substrate, and a high purity Si target, set about 30° off the substrate normal. As a consequence of this geometry, Si atoms impinged upon the substrate at oblique incidence, and the corresponding amorphous Co–Si films and multilayers develop in-plane magnetic uniaxial anisotropy with very low coercive and switching fields [34].

The substrate morphology was studied by atomic force microscopy (AFM) [37]. Characterization of the magnetic properties was carried out using the magneto-optical transverse Kerr effect (MOTKE) [38, 39]. Magnetic hysteresis loops were obtained by applying the magnetic field parallel to either the easy or the hard axis of the samples. Grazing incidence small angle x-ray scattering (GISAXS) was used to gain insight into the mesoscopic structure of the films. The GISAXS measurements were done at the ESRF on the BM16-CRG beamline (operated by the consortium Laboratori Llum Sincrotro; LLS) using 7.64 keV photons at 0.4° incidence angle.

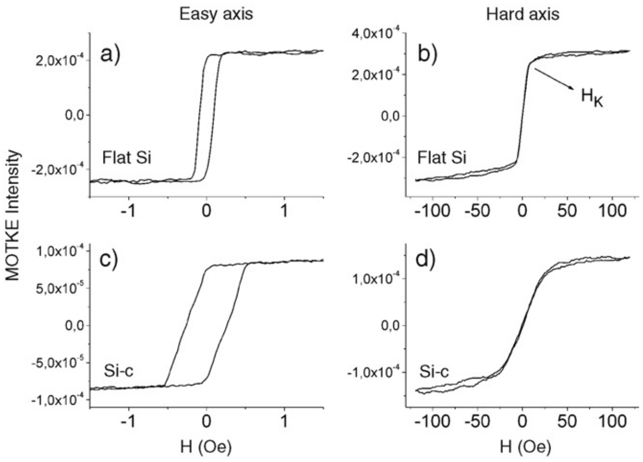

Figure 2. MOTKE hysteresis loops of one magnetic period films (3 nm Si/5 nm $Co_{74}Si_{26}$/3 nm Si/substrate) grown on top of a flat Si substrate, (a) and (b), and on top of the irradiated Si-c substrate, (c) and (d). The magnetic field H has been applied along the easy, (a) and (c), or hard, (b) and (d), axis directions.

3. Results and discussion

As already mentioned, several Si(100) substrates were patterned by exposing them to 1 keV Ar^+ ions. A series of substrates with increasing values of rms roughness were obtained by adjusting the exposure time. The irradiated substrates, denoted as Si-a, Si-b, Si-c and Si-d, had rms roughness values of 0.23 nm, 0.82 nm, 0.88 nm and 2.1 nm, respectively, while the non-bombarded flat Si reference substrate had a roughness value of 0.07 nm. The AFM images of substrates Si-a, Si-b and Si-c, obtained after air exposure, are shown in figure 1. The in-plane projection of the ion beam is approximately parallel to the X-axis. As seen in the images, the ion treatment increases the roughness and leads to the development of structures with lateral sizes of several tens of nanometers and heights of few nanometers. No ordered ripple or dot formation is observed. Interestingly, two-dimensional Fourier transform analysis of the images leads to diffuse and broad features discarding the presence of well defined in-plane correlation distances, although a slight elongation has been obtained for the Fourier signal at around 45° from the easy axis, especially for the case of the sample grown on top of Si-b substrate.

These nanopatterned substrates were used to study how these templates affect the magnetic properties of films and multilayers grown on top of them. First, two identical samples having just one single magnetic period of 5 nm of $Co_{74}Si_{26}$ (and the corresponding 3 nm Si buffer and capping) were prepared on top of the reference flat Si substrate ($\sigma = 0.07$ nm) and on substrate Si-c, which has a roughness value about ten times larger ($\sigma = 0.88$ nm). The corresponding MOTKE hysteresis loops are compared in figure 2 for a magnetic field applied along two perpendicular directions of the sample surface plane (corresponding to the easy or hard magnetic axis). The first observation is that the sample grown on the flat Si substrate is extremely soft from a magnetic point of view, with a coercive field, H_C, as low as around 0.1 Oe, as

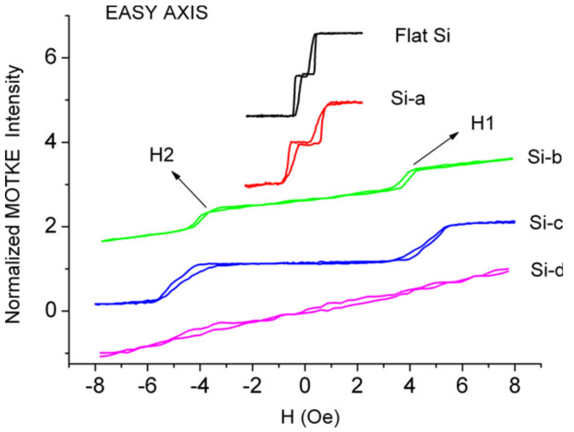

Figure 3. Normalized MOTKE hysteresis loops of a two-period multilayer (3 nm Si/5 nm $Co_{74}Si_{26}$/3 nm Si/5 nm $Co_{74}Si_{26}$/3 nm Si/substrate) grown on the substrates with increasing doses of irradiation from Si-a to Si-d (loops vertically shifted for clarity).

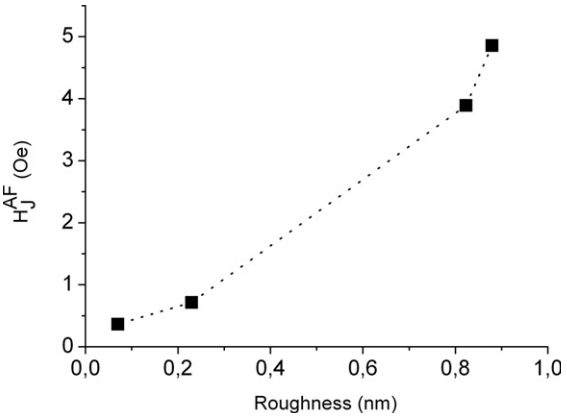

Figure 4. Dependence of the strength of the coupling, which is proportional to H_J^{AF}, with the ion beam induced substrate rms roughness.

shown in figure 2(a). Also, the loops in figures 2(a) and (b) show that it has a well-defined in-plane uniaxial magnetic anisotropy, as indicated by the closed loop around the hard axis direction, with an anisotropy field, H_K, of around 10 Oe and the square shape of the loop along the easy axis direction. This behavior is typical of amorphous magnetic Co–Si alloys and has already been observed for films in this compositional range [40]. It should be mentioned that the direction of the magnetic hard axis corresponds to the projection of the Si evaporated atoms on the surface plane. However, when the same film is grown on top of substrate Si-c, new effects are observed. The coercive field significantly increases up to 0.25 Oe, reflecting the increasing density of pinning centers for the magnetic domain walls moving in the magnetic layer grown on the rough substrate. The enhancement of the pinning leads to an increase of the magnetic field necessary for moving the domain walls and, thus, to the observed increase of coercive field. The magnetic anisotropy field increases up to 25 Oe, and the slope of the saturated part of the loops increases both along the easy and hard axis directions. This increase of slope, that can be related to superparamagnetic effects related to small magnetic inhomogeneities in the film, will be further discussed.

The changes observed in the magnetic behavior of the one-period sample grown on the ion irradiated substrate already suggest that the AF coupling, previously observed in the two-period multilayers grown on flat substrates [34, 35], could be affected by the substrate pre-patterning. In fact, this is confirmed when looking at the evolution of MOTKE hysteresis loops of the two-period multilayers grown on flat and patterned substrates, plotted in figure 3. The loops have been measured with the magnetic field applied along the easy axis direction. They have been normalized and vertically shifted for clarity. The upper loop corresponds to the reference multilayer grown on top of a non-bombarded flat Si(100) substrate ($\sigma = 0.07$ nm). This loop has a short plateau at zero field, which is the fingerprint of the antiparallel alignment at remanence of the magnetization of both CoSi layers, leading to an approximately zero net Kerr signal. The relevant fields of the reversal process are indicated in the loop corresponding to sample Si-b in the figure: $H1$, the field at which the AF state starts to be formed; and $H2$, the field at which the AF alignment starts to be broken. It should be noted that the width of the plateau, or more precisely, the difference between $H1$ and $H2$, is directly related to the strength of the AF coupling [35]. Thus, a coupling field can be defined as $H_J^{AF} = (H1 - H2)/2$ [35]. Interestingly, the sample grown on substrate Si-a, with roughness $\sigma = 0.23$ nm larger than the flat one (0.07 nm), starts forming the AF state at a magnetic field $H1$ slightly higher than the one corresponding to the flat sample, and it has also a larger plateau width, indicating a more stable AF state and a stronger AF coupling. This trend is clearly confirmed when the rougher substrates, Si-b and Si-c ($\sigma \sim 0.8$–0.9 nm), are used to grow the multilayer on top of them. Again, both the $H1$ field and the width of the plateau increase significantly, confirming the strong enhancement of AF coupling.

The changes in the coupling field as a function of the ion patterned substrate roughness are shown in figure 4. This unequivocally indicates a continuous increase of H_J^{AF} by around one order of magnitude, from 0.4 to 4.9 Oe, when σ is increased by one order of magnitude from 0.07 to 0.88 nm. This result shows that H_J^{AF} can be tuned by adjusting the substrate roughness through an adequate ion beam irradiation dose. However, there is an upper limit to this strengthening of the coupling. The sample grown on top of the roughest substrate Si-d ($\sigma = 2.1$ nm) has no plateau but a superparamagnetic like behavior, with closed loops along both the easy and hard magnetic axis. The origin of this trend to superparamagnetism is most likely related to the formation of small magnetic clusters, instead of a continuous film, the height variations of this substrate being comparable to the thickness of the subsequent magnetic layers. This means that small magnetic areas could be isolated, surrounded by Si-rich regions related to the mounds of the substrate.

In order to understand the origin of the coupling enhancement it is worth recalling that several coupling mechanisms, some ferro- and other antiferromagnetic, are simultaneously acting on this magnetic structure [35]. In particular, antiferromagnetic contributions may come from

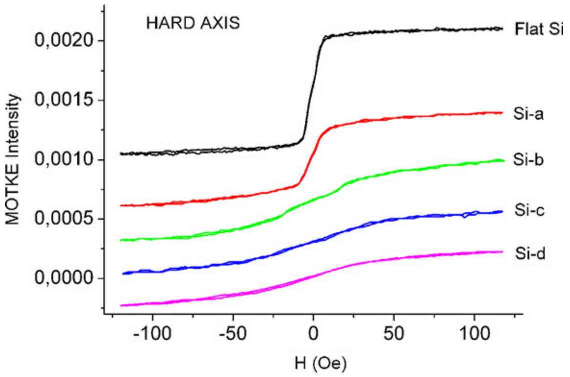

Figure 5. Hard axis MOTKE hysteresis loops of the same multilayers shown in figure 3, grown on substrates having an increasing dose of irradiation from flat Si to Si-d (loops vertically shifted for clarity).

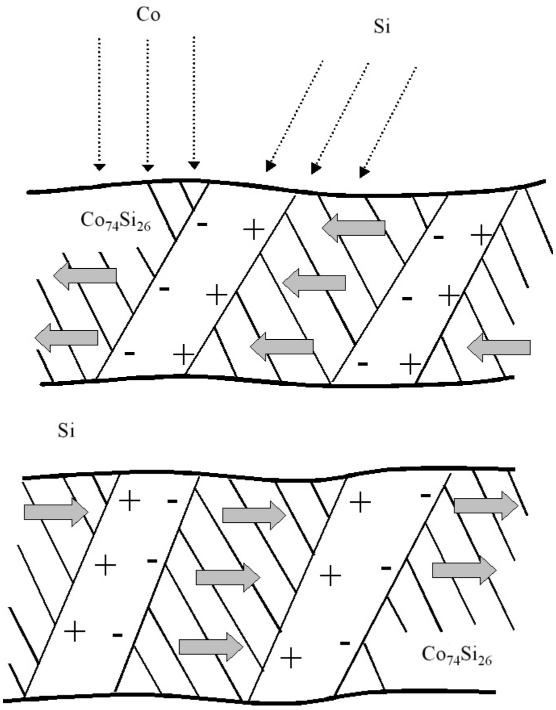

Figure 6. Sketch of the formation of magnetic areas (shaded) surrounded by Si-rich non-magnetic regions (blank) and the corresponding magnetic poles (+ and −) at the borders stabilizing AF order (thick arrows indicate magnetization).

dipolar fields arising from the finite size of the magnetic regions [27], and from electronic structure effects. On the contrary, ferromagnetic alignment is favored by orange peel coupling [25] due to the interaction between surface dipoles at fully correlated magnetic/non-magnetic interfaces. In addition, the magnetostatic interaction between domain walls from both magnetic layers can also influence the coupling [26].

One of the contributions that is expected to be affected by the combination of rough substrates and shadowing effects coming from the Si oblique incidence is the one related to the finite size of magnetic regions. As already mentioned, a trend to clustering is observed in the magnetic layers when roughness is increased, meaning that some compositional inhomogeneities are being formed. This trend is confirmed when looking at the hard axis MOTKE loops of the multilayers, shown in figure 5. In this figure it can be seen that for the multilayers grown on the rougher substrates, Si-b, Si-c and Si-d, the slope of the saturated part of the loop is significantly larger than for the samples grown on the flatter substrates, flat Si and Si-a. It should be pointed out that the Curie temperature of the $Co_{74}Si_{26}$ alloy is very close to room temperature [35]. This means that small local variations in composition could produce Si-rich non-magnetic regions where surface magnetic poles, and the corresponding AF dipolar interactions, may appear, as sketched in figure 6. These local compositional fluctuations could be enhanced by the combination of Si oblique incidence growth and roughness dependent shadowing effects. In addition, the diffusion of atoms in the surface of the growing film could be reduced for the case of rougher substrates favoring the formation of inhomogeneities. The strength of this type of AF coupling, already observed for magnetic recording media [41], can be roughly estimated using expression (9) of [27] for the sample grown on substrate Si-c, assuming grain sizes of around 100 nm (deduced from AFM data after growth) and M_s values of 30 emu cm^{-3} [35]. An AF coupling field of around 11 Oe is obtained, slightly above, but close to, the observed 4.9 Oe. In fact, larger magnetic domains, of at least around 500 nm per side, are needed in order to obtain coupling fields close to 4–5 Oe by using the model of [27], meaning that more than one surface mound is expected to participate in each domain. Actually, the lateral size of these ion beam induced structures has been determined after growth of the multilayers by AFM measurements (not shown) and does not change significantly with the ion beam dose (it is about 80 nm for the sample grown on top of substrate Si-a, 90 nm for substrate Si-b, and 100 nm for substrate Si-c). This suggests that the magnetic domains should include several of these nanostructures, and the observed enhancement in coupling field would be associated with a reduction in the lateral size of the magnetically correlated regions due to the enhanced inhomogeneities (i.e. with a reduction of the number of surface structures in each magnetic domain). In any case, it should be taken into account that in order to make a more accurate analysis with the above mentioned model, besides the lateral size of the magnetic regions, other parameters which are difficult to estimate with the available data, like the local value of magnetization or the size of the non-magnetic regions acting as gaps in between the magnetic areas, should be considered. It should also be noted that additional support for the relevance of this type of dipolar interaction comes from the temperature behavior of the coupling, which in previous work has been shown to be compatible with a mechanism mediated by compositional inhomogeneities at the spacer or at the interfaces [35].

There are other contributions that could be modified by the growth conditions. In particular, oblique growth is expected to produce oblique structures that could laterally shift the roughness between alternate layers, reducing ferromagnetic ‘orange peel’ type contributions. Interestingly, strong experimental evidence of the presence of such oblique structures in these multilayers has been obtained by acquiring GISAXS images for the two extreme cases: the sample grown on top of the flat substrate, which has the weakest coupling (GISAXS images shown in figures 7(a) and (b)), and the one grown on top of substrate Si-c, which has the highest roughness and the strongest coupling (GISAXS shown in figures 7(c) and (d)). The images represent the distribution of the x-ray scattering intensity as a function of the sample out-of-plane momentum transfer, q_z (vertical axis), and the in-plane momentum transfer, q_y (horizontal axis). Two acquisition geometries have been used: images 7(a) and (c) have been acquired with q_y probing the direction in the sample surface that is perpendicular to the projection of the Si incident atoms, whereas for images 7(b) and (d) the samples have been rotated by 90°, so that q_y is parallel to the projection of the Si incident atoms on the sample surface. As expected, the amount of diffuse scattering out of the specular plane is much smaller for the flatter sample than for the rougher one. However, the most interesting feature is observed when comparing images 7(c) and (d). Whereas the diffuse scattering on both sides of the specular plane is symmetric along the direction perpendicular to the projection of the Si incident atoms, with maxima and minima of intensity for the same q_z position, a clear asymmetry is observed when q_y probes the direction parallel to the projection of the Si incident atoms, the diffuse scattering intensity being inclined by about 25° (see dashed lines in figures 7(c) and (d)). This asymmetry, which is a typical fingerprint of the presence of oblique structures and lateral shifts of roughness [42], appears precisely when q_y is probing the direction where shadowing effects and oblique structures are expected due to the Si growth geometry. However, given the small thickness of the Si spacer layer used in these multilayers, the expected lateral shifts of roughness for about 30° oblique Si incidence are of only 1–2 nm, in comparison with the observed 80–100 nm in-plane periodicity of roughness, so that the expected reduction of 'orange peel' effects due to loss of conformity would be too small to explain the effect.

Also, the increase in roughness could mean that, locally, the effective spacer thickness would be smaller than the nominal 3 nm, leading to an increase of the AF coupling. However, x-ray diffraction measurements carried out with Cu Kα photons (not shown) indicate very small variations in the stacking periodicity, of only 0.2 nm, which cannot explain the observed enhancement. Actually, a reduction in spacer thickness from 4 to 2 nm for a multilayer grown on a flat substrate implies only a twofold enhancement in the coupling field [34], much smaller than observed here. Finally, the increase of domain wall pinning centers as roughness is increased, confirmed in the MOTKE loops shown in figures 2(a) and (c) for one-period samples, could also lead to AF states being more difficult to break, and the magnetostatic interactions between walls in subsequent layers could also be modified by the increased roughness, since the distance between them would be locally reduced. In any case, with the available data it is not easy to give a definite picture of the situation, although it is likely that the explanation for the observed coupling enhancement involves several of these mechanisms simultaneously acting on the system.

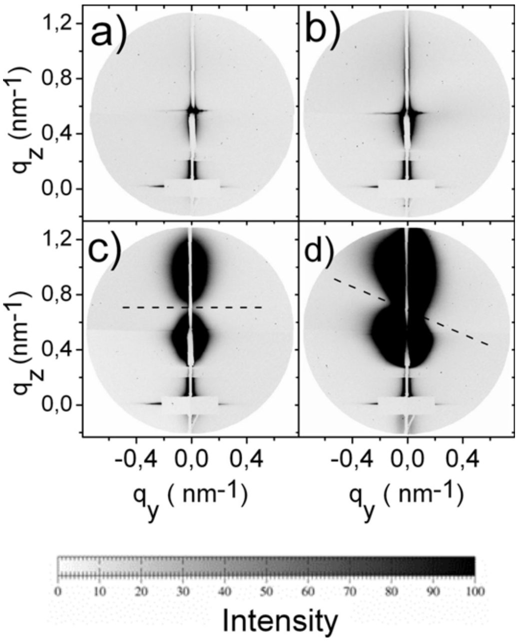

Figure 7. GISAXS images are shown on panels (a)–(d). Panels (a) and (b) correspond to the multilayer grown on top of a flat Si substrate, whereas (c) and (d) refer to the multilayer grown on top of Si-c. For (a) and (c) q_y is perpendicular to the projection of the Si evaporation atoms on the sample surface whereas for (b) and (d) q_y is parallel to that projection.

4. Conclusions

In summary, the combination of low energy ion beam induced nanopatterning of Si(100) substrates and oblique incidence growth has been shown to enhance the antiferromagnetic coupling of amorphous CoSi/Si multilayers by one order of magnitude when the roughness is increased from 0.07 to 0.88 nm. This combination of substrate ion irradiation and oblique growth opens the way for an alternative method of tuning the coupling strength of multilayered magnetic systems. An explanation for this effect, supported by the observed trend to clustering and superparamagnetism, is suggested in terms of the formation of magnetic/non-magnetic regions as a consequence of local fluctuations of the alloy composition, enhanced by the ion induced substrate roughness, although other discussed mechanisms could also play a non-negligible role.

Acknowledgments

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