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Temperature dependence of magnetically dead layers in ferromagnetic thin-films

M. Tokaç,[1,2,a] C. J. Kinane,[3] D. Atkinson,[1] and A. T. Hindmarch[1,b]
[1]*Centre for Materials Physics, Durham University, South Road, Durham DH1 3LE, United Kingdom*
[2]*Department of Fundamental Sciences, Faculty of Engineering, Alanya Alaaddin Keykubat University, 07450 Antalya, Turkey*
[3]*ISIS Neutron Facility, Rutherford Appleton Laboratory, Chilton, Oxon OX11 0QX, United Kingdom*

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Polarized neutron reflectometry has been used to study interface magnetism and magnetic dead layers in model amorphous CoFeB:Ta alloy thin-film multilayers with Curie temperatures tuned to be below room-temperature. This allows temperature dependent variations in the effective magnetic thickness of the film to be determined at temperatures that are a significant fraction of the Curie temperature, which cannot be achieved in the material systems used for spintronic devices. In addition to variation in the effective magnetic thickness due to compositional grading at the interface with the tantalum capping layer, the key finding is that at the interface between ferromagnetic film and GaAs(001) substrate local interfacial alloying creates an additional magnetic dead-layer. The thickness of this magnetic dead-layer is temperature dependent, which may have significant implications for elevated-temperature operation of hybrid ferromagnetic metal-semiconductor spintronic devices.
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Magnetic dead layers (MDLs) are non-magnetic regions, typically of a few atomic monolayer thickness, which can form at the interfaces in thin ferromagnetic (FM) films.[1–4] In most cases MDLs are suggested to form due to breaking of symmetry at the interface, or during the deposition processes due to interfacial interdiffusion. Although the details of MDL formation are unclear, the presence, and thickness, of MDLs are found to depend on, amongst other factors, the ferromagnetic material, deposition conditions, and the adjacent material across the interface.[5]

In the development of spintronic devices, FM thin films have been widely used in applications including tunneling magnetoresistance,[6] current induced magnetization switching,[7] hard disk read heads, and magnetic random access memories.[8] The functionality of these various spintronic systems depends critically on the magnetic properties of the interfaces. Therefore, MDLs have significant operational consequences is spintronic devices.[9,10] MDLs reduce the effective magnetic volume, which reduces the anisotropy and thermal activation energies, causing thermal stability issues for spintronic devices.[3,11] This problem is particularly detrimental as device miniaturization already required magnetic thin-film devices which are only a few atomic monolayers thick.

FM films grown onto III-V semiconductor (SC) substrates, such as Fe/GaAs, are a model system and have potential applications in hybrid spintronics, combining the functionalities of ferromagnetic metals and semiconductor devices.[12,13] This functionality requires efficient injection of a spin-polarized current from the FM into the SC, which necessitates that the FM material is indeed ferromagnetic right up to the interface with the SC. Unfortunately, interfaces between FM metals and SC substrates frequently feature interfacial MDLs that act to prevent effective spin

[a]mustafa.tokac@alanya.edu.tr
[b]a.t.hindmarch@durham.ac.uk

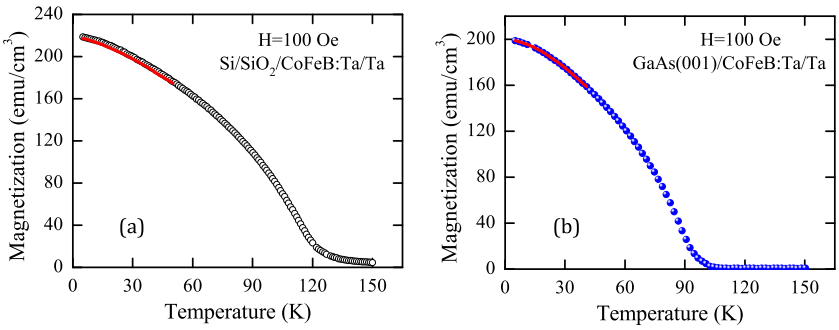

FIG. 1. Temperature dependent magnetization of 100 Å CoFeB:Ta thin-film on (a) Si/SiO_2 and (b) GaAs(001) substrates measured in a 100 Oe applied field in the temperature range from 5 K to 150 K. The red solid lines are the fit curve according to Eq. 1.

injection,[14,15] sometimes suggested to be the result of intermixing produced during the FM film deposition.

Whether the thickness of MDLs varies with temperature in a FM thin film, and the physical mechanism by which such a variation may occur, presently remains unknown. As many spintronic devices operate at elevated temperatures, understanding how temperature may influence MDLs becomes also an important technological problem. However, as the Curie temperatures of technologically relevant FM materials used in devices are typically above 700°, it is not possible to study MDLs at temperatures corresponding to an appreciable fraction of T_C without promoting significant interdiffusion in the often-delicate thin-film structures, effectively destroying the multilayer structure. In the study described here, the addition of a non-magnetic transition metal (Ta in this case) to an amorphous CoFe-based alloy was used as a model materials system in order to reduce both the magnetic moment per Co/Fe atom *and* the Curie temperature (T_C).[16] The CoFeB:Ta alloy layer used here was specifically designed to have a T_C below room-temperature in order that we may study the magnetic behaviour of the film across the magnetic phase-diagram without causing modification or damage to the structure. This enabled the MDL thickness to be easily studied in a model system without causing further interfacial diffusion to occur via thermally-driven interdiffusion at elevated temperatures.

Here, amorphous CoFeB:Ta thin-films deposited onto Si/SiO_2 and GaAs(001) substrates have been used to investigate the temperature dependence of the magnetic dead-layers. The MDL thicknesses at different temperatures are determined using magnetization depth profiles obtained from polarised neutron reflectometry (PNR). In particular, this enables us to clearly isolate the behaviour of the magnetisation at the technologically important interface with the GaAs substrate.

Amorphous Co(28%)Fe(28%)B(14%)Ta(30%) (CoFeB:Ta, nominal composition, at. %, in brackets) thin films were deposited at room temperature using dc magnetron co-sputtering from Co(40%)Fe(40%)B(20%) and Ta sputtering targets in a confocal geometry under ultra-high vacuum (UHV). Films with thickness of 100 Å were deposited onto Si/SiO_2 and GaAs(001) wafer substrates. The GaAs substrate was chemically etched in dilute HCl, then annealed at 600° for 1 hour and allowed to cool to ambient temperature under UHV prior to film deposition. To prevent oxidation the CoFeB:Ta layers were capped with 30 Å Ta. The deposition process had been previously optimised to produce high quality thin-film samples with low surface roughness and sharp interfaces.[17,18] Structural characterisation by x-ray reflectivity (XRR) was obtained using a Rigaku Smartlab laboratory reflectometer at room temperature.

Temperature dependent magnetic characterization was performed using SQUID magnetometry in the temperature range from 5 K to 150 K in order to extract T_C for both structures, as shown in Fig. 1. The temperature dependent magnetization $M(T)$ of a FM material can be fitted at low-temperatures using the spin-wave model and follows the Bloch $T^{3/2}$ power law,

$$M(T) = M(0)\left[1 - \left(\frac{T}{T_C}\right)^{3/2}\right], \tag{1}$$

where the solid line through the data is a fit to this equation, and $M(0)$ is the spontaneous magnetization at zero temperature. T_C values are estimated from these fits to the experimental data as $T_C \sim 110$ K, and $T_C \sim 90$ K for CoFeB:Ta films on Si/SiO_2 and GaAs(001) substrates, respectively. The difference in T_C for both thin-film structures is attributed here to a slight sample-to-sample compositional variation in the co-deposition process between those films.

PNR was used to obtain structural and magnetic scattering length density (SLD) depth profiles. Measurements were performed at the ISIS pulsed neutron source, using the CRISP time-of-flight reflectometer.[19] Data is presented as normalised reflected neutron intensities as a function of the scattering vector, $Q_Z = 4\pi \sin\theta/\lambda$, with resolution $\Delta Q_Z/Q_Z = 4$ %. θ is the angle of incidence, and λ is the neutron de-Broglie wavelength. Neutrons were polarized parallel and anti-parallel to a magnetic field applied in the plane of the sample and perpendicular to the scattering plane, resulting in spin-up (R^+) and -down (R^-) reflectivity profiles from which spin-asymmetry, $SA = (R^+ - R^-)/(R^+ + R^-)$, was derived. The sample temperature was controlled using a helium gas-flow cryostat.

PNR measurements at room temperature were performed under a 'large' magnetic field $\mu_0 H = 0.3$ T to enhance the sensitivity to possible residual magnetization at this temperature. As expected, no magnetic signal was observed at room temperature (i.e. above T_C). Low temperature PNR measurements were made for both films at temperatures of ~0.1 T_C (10 K) and ~0.6 T_C (55 K for the structure on GaAs(001) and 70 K for the structure on Si/SiO_2 substrate.) These measurements were made in a magnetic field of $\mu_0 H = 0.02$ T, which had previously been determined to be sufficient to saturate the magnetization. Over the temperature range studied, the variation of film thickness due to thermal expansion/contraction is anticipated to be around 0.03 %,[20] which is far below the resolution of PNR measurements.

XRR and PNR data were both fitted using the GenX code.[21] The parameters describing the structure of the two CoFeB:Ta structures on Si/SiO_2 and GaAs(001) were obtained from simultaneously fitting room temperature PNR and XRR data for each sample, and are presented here as neutron structural scattering length density (SLD) profiles. The magnetization depth profiles for each combination of sample and temperature were then obtained by fitting the low-temperature reflectivities, consistent with these previously determined structures; only the magnetic parameters were allowed to vary as a function of temperature. The magnetization depth profiles for the CoFeB:Ta layers are presented here as depth-resolved neutron magnetic SLD profiles. Note that only the *reflectivity* data is fitted, and the simulated spin-asymmetry calculated from these fits: The solid lines showing simulated spin-asymmetry are *not* direct fits to the spin-asymmetry data.

Figure 2 shows x-ray reflectivity for the two CoFeB:Ta/Ta films on Si/SiO_2 and GaAs(001) substrates. The fits to the XRR data for both films assume the CoFeB:Ta layer is compositionally uniform. The feature around $Q_Z = 0.1$ Å^{-1} for both samples cannot be reproduced under this assumption. We note that the intensity reduces more quickly with increasing Q for the sample with GaAs(001) substrate due to the anticipated long wavelength topological roughness of the GaAs(001) substrate after etching and annealing.[22]

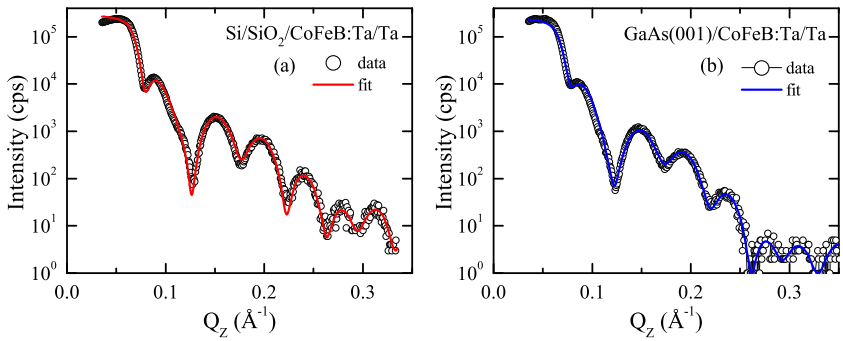

FIG. 2. Specular x-ray reflectivitiy of CoFeB:Ta thin-films on (a) Si/SiO_2 and (b) GaAs(001) substrates. The solid lines in reflectivity plots are the best fit calculations, using a model which assume that the CoFeB:Ta layer is compositionally uniform.

PNR measurements and spin-asymmetries, derived from the corresponding spin-up and spin-down reflectivities for CoFeB:Ta films on Si/SiO_2 and GaAs(001) substrates, measured at T ~ 0.1 T_C and ~0.6 T_C are shown in Fig. 3. The spin-asymmetry simulations show good correspondence with the data, despite being calculated from the fits to the reflectivity rather than direct fits to the spin-asymmetry. This correspondence demonstrates that we have accurately determined both the physical and magnetic structure of the two films. For both structures, the spin-asymmetries show several prominent 'dip' features indicated by dashed vertical lines. The first dip, at around Q_Z = 0.04 $Å^{-1}$ for the structures on both substrates show no significant shifts with temperature within Q_Z resolution. However, the second dip shifts to higher Q_Z as the temperature increases, indicating a reduction of the *effective total magnetic thickness* for both samples. The shift is larger for the FM film on GaAs(001) than for the film on Si/SiO_2, immediately indicating that the change in total MDL thickness with increasing temperature is greater for the film on GaAs(001).

The PNR data for both samples was not well described using a simple 3 layer model consisting of substrate/CoFeB:Ta/Ta; i.e. a description of the sample structure assuming the CoFeB:Ta to be compositionally uniform. A slightly more complex model was required: including a surface layer corresponding to oxidized Ta,[22] denoted by Ta_2O_5 and subdivision of the CoFeB:Ta layer into 4 'slabs', 2 fully magnetized slabs and two interfacial slabs where the magnetization was allowed to vary from the bulk value. Note that these 'slabs' represent only the approach to parameterise the SLD profile; from which the reflectivity is then calculated.

Such multiple-slab models describe CoFeB:Ta layers which are not compositionally uniform, as is suggested by the x-ray reflectivity data and fits shown in figure 2, and provide realistic descriptions of the magnetic structure of the samples. These models provide fits which describe both the x-ray and neutron reflectivity data very well *and* shows very good correspondence between the derived

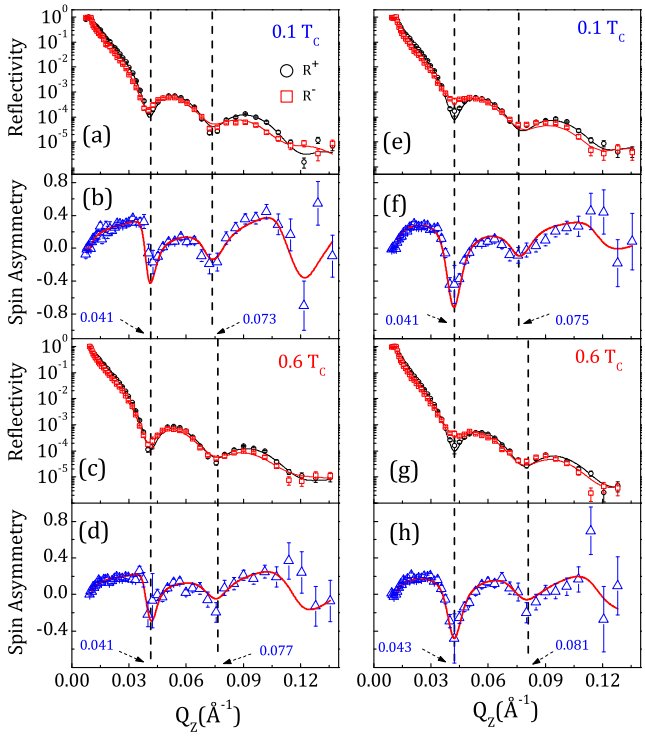

FIG. 3. Measured polarised neutron reflectivity (circles and squares) and derived spin-asymmetry (triangles) of CoFeB:Ta films on Si/SiO_2 (a-d) and GaAs(001) (e-h) substrates measured at T ~ 0.1 T_C and ~ 0.6 T_C. The solid lines in reflectivity plots are the best fit calculations based on the previously determined structure, and from which the corresponding solid lines shown in spin-asymmetry plots are derived. Note that the solid lines through spin-asymmetry are *not* direct fits to the spin-asymmetry data.

neutron spin-asymmetries. We reiterate that the spin-asymmetry data is *not* fitted directly, but the model spin-asymmetry derived from the reflectivity fits agrees very well with the data. Adding further subdivisions ('slabs') to the models resulted in no further improvement of the fit quality,[23] evaluated based on the reduced χ^2 statistic for each fit.

The real part of the SLD profiles extracted from the fits shown in figure 3 are shown in figure 4 for structures on Si/SiO_2(a) and GaAs(001)(c), with corresponding schematics of the sample structures. In order to more clearly illustrate the positions of the structural and magnetic interfaces in these structures, the derivative of the structural and magnetic SLD profiles are plotted in figure 5. Note that this representation of the data is purely for illustrative purposes. In these derivatives, layer interfaces in the model structures are highlighted as peaks and dips.

If figure 4 the 'bulk' CoFeB:Ta structural SLD[24] profile for the film deposited on Si/SiO_2 is slightly larger than that for the film on GaAs(001), indicating that the film on Si/SiO_2 has a slightly lower Ta concentration due to sample-to-sample variation in the co-deposition process. This interpretation is consistent with the slightly higher T_C measured for the film on Si/SiO_2. The structural SLD profiles for both structures varied gradually from 'bulk' values in the CoFeB:Ta layers close to the capping layer interfaces. This suggests that compositionally-graded layers are formed in the region of these interfaces, between bulk CoFeB:Ta layers and the Ta capping layers, with spatially varying Ta doping concentration. Such a compositional grading is likely to have formed due to Ta diffusion during film deposition. The imaginary part of the structural SLD profile, as shown in Fig. 4(b) and (d) is sensitive only to boron, and shows the expected uniform CoFe-to-B composition through the CoFeB:Ta layers; confirming that these changes in structural SLD profile are due to Ta migration.

For the FM film on Si/SiO_2, the film/substrate interface width appears relatively broad, attributed to the diffuse interface between the Si substrate and the thin native SiO_2 layer which has a thickness ~1–2 nm. For the structure on GaAs(001) the structural SLD profile shows a clear 'layer' with intermediate SLD over a thickness of ~ 15 Å at the film/substrate interface. This may be a result of interfacial interdiffusion; such interdiffusion produces, for example, various inter-metallic alloys of Fe, Ga and As at GaAs/Fe interfaces,[12,25,26] Co_2GaAs at the GaAs/Co interfaces,[2] and $(CoFe)_3GaAs$ at GaAs/CoFe interfaces.[27] Such interfacial alloying has been found to appear irrespective of growth temperature, substrate orientation, and substrate reconstruction — and remains poorly understood.

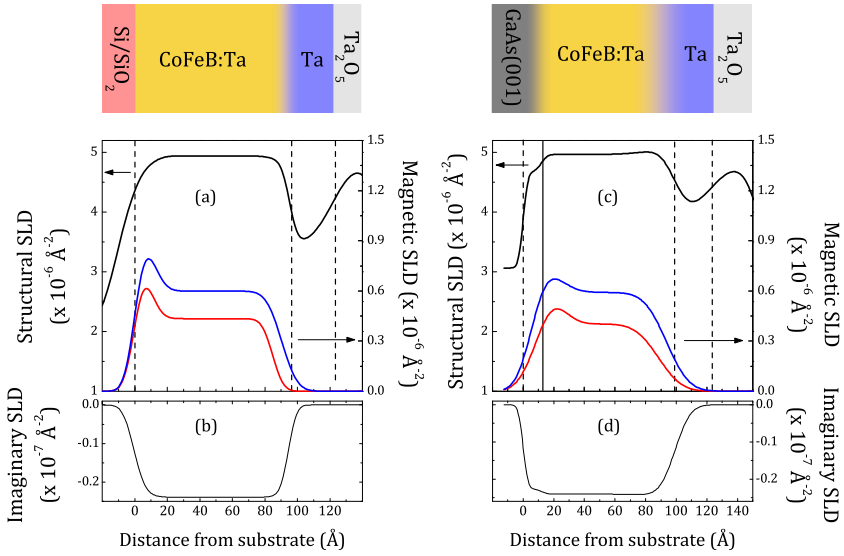

FIG. 4. Structural SLD profiles (left axes) for CoFeB:Ta films deposited onto Si/SiO_2 (a) and GaAs(001) (c) substrates. The corresponding imaginary parts of the structural SLDs are shown in (b) and (d). The vertical dashed lines correspond to the positions of the layer interfaces in the model structures; the layer structures are shown schematically at the top of the figure. Magnetic SLD profiles at temperatures corresponding to $T \sim 0.1\ T_C$ and $\sim 0.6\ T_C$ are shown on the right axes.

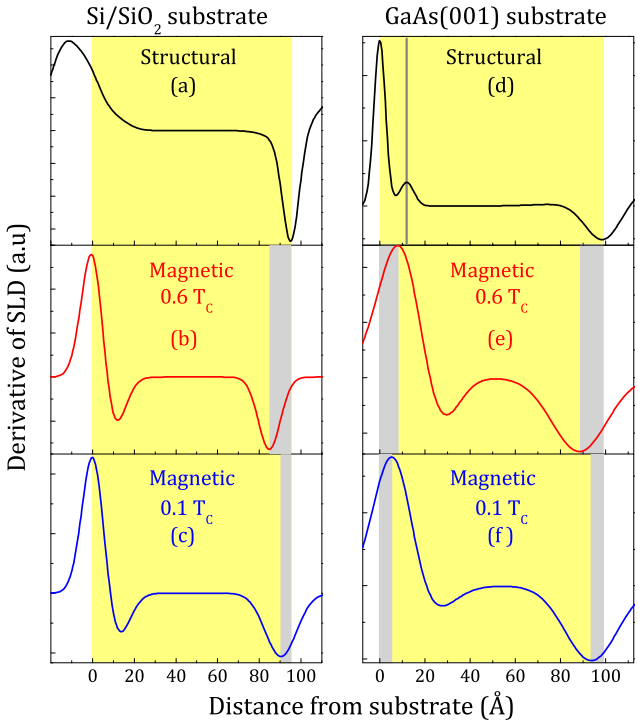

FIG. 5. The derivative of room temperature structural and magnetic SLD profiles for 0.6 T_C and 0.1 T_C to extract layer interfaces are shown for CoFeB:Ta films deposited onto Si/SiO_2 (a-c) and GaAs(001) (d-f) substrates. The vertical dashed lines correspond to the positions of the layer interfaces in the model structures. The shaded regions show magnetic dead layer formations.

Much of the two sets of magnetic SLD profiles – for films deposited on Si/SiO_2 and GaAs(001) substrates – are qualitatively similar. For each structure, increasing temperature causes the magnetic SLD (which is proportional to the depth-resolved magnetic flux density) to decrease across the entire FM film thickness, as expected. Ta doping of CoFeB dilutes the local magnetization and reduces the local Curie temperature in proportion to the local Ta concentration.[16] The magnetic SLDs show that magnetizations are roughly constant within the volume of the CoFeB:Ta layers, and reduce toward the interfaces with the Ta capping layers due to the compositional grading observed in the structural SLDs.

In both FM films, a small region close to the interface with the substrate shows enhancement of the magnetic SLD; this enhancement is greater for the film on Si/SiO_2 than for the film on GaAs(001). The structural SLDs in these regions do not deviate significantly from those in the volume of the films, indicating that this enhanced magnetization is not due to local reduction in Ta concentration. This locally enhanced magnetization may be a result of charge transfer due to interface bonding,[22,28] which may differ for bonding at the sharp oxide-metal SiO_2/CoFeB:Ta interface compared to that at the slightly intermixed semiconductor-metal GaAs/CoFeB:Ta interface.

Starting with the derivative of the structural model derived for the film on Si/SiO_2, Fig. 5(a), we see that in addition to a broad peak indicating a diffuse substrate/oxide interface and a sharper dip indicating the film/capping-layer interface, there is also a slight inflection corresponding to the interface between the SiO_2 native-oxide layer and magnetic CoFeB:Ta film. From previous measurements using conventional magnetometry it has been suggested that a 0.5 nm-thick MDL is found at the SiO_2/CoFeB interface, attributed to interdiffusion.[4] In contrast to this, here peaks in the derivatives of the magnetic SLDs for the Si/SiO_2/CoFeB:Ta structure, Fig. 5(b) and (c), illustrate that the 'magnetic' film/substrate interface is co-located with the structural film/substrate interface for *both* measurement temperatures in the ferromagnetic phase. The key point here is that *no* MDL arises at the SiO_2/CoFeB:Ta interface.

In contrast, Fig. 5(b) and (c) also illustrate that the upper, CoFeB:Ta/Ta, 'magnetic' interface is shifted to lower thickness, away from the nominal structural interface, with increasing temperature in both films. There is a MDL at the CoFeB:Ta/Ta interface and the thickness increases with increasing temperature; reducing the effective ferromagnetic thickness of the film. This is the reason for the previously described shift of features in the PNR spin-asymmetries shown in Fig. 3(b) and (d). Ta diffusion into the CoFeB:Ta layer from the Ta capping layer is the reason for the broad film/cap structural interface in Fig. 5(a). It is also the reason why the interfacial MDL thickness increases with increasing temperature; the local Curie temperature of this compositionally-graded region determines the position of the effective magnetic interface.

This is consistent with studies of room-temperature MDL formation in nominally un-doped, CoFeB/Ta interfaces, which have shown that deposition of a Ta capping layer onto CoFeB creates a 6 Å thick MDL at the CoFeB/Ta interface at room temperature[29] (corresponding to $T \sim 0.4\ T_C$), due to the diffusion of Ta into CoFeB.[4,29,30] In previous PNR investigations of CoFeB thin-films sandwiched by MgO and Ta layers, a formation of 20 Å thick MDL is observed at CoFeB/Ta interfaces due to diffusion of boron atoms.[31] The MDL thicknesses at CoFeB/Ta interfaces are comparable to those due to further interfacial Ta diffusion into CoFeB:Ta in Fig. 5(b) and (c); (5.6 ± 0.5) Å MDL thickness at $T \sim 0.1\ T_C$, increasing to (9.2 ± 0.9) Å at $0.6\ T_C$.

Turning now to the derivatives of the SLDs for the film deposited onto GaAs(001) substrate, Fig. 5(d)–(f), compositional grading and a corresponding temperature-dependent MDL is also seen at the CoFeB:Ta/Ta interface, although the interface is slightly broader in this structure.

There is also an additional peak in the derivative of the structural SLD near the nominal film/substrate interface, which highlights the interface between the intermixed CoFeB:Ta-GaAs interfacial region and CoFeB:Ta film. Fig. 5(e) and (f), clearly illustrates a MDL at the lower magnetic interface of the CoFeB:Ta film on GaAs(001). This MDL is indicated in Fig. 5(e) and (f) by the shaded regions at the film/substrate interface.

MDLs at FM/SC interfaces are severely detrimental for spin-injection applications as the 'FM' material directly adjacent to the SC is no longer actually ferromagnetic. As the MDL appears to be confined to the interfacial region it is likely that the MDL forms as a result of interfacial intermixing at the film/substrate interface. Magnetic depth profiles of CoFe/GaAs(001) using PNR have previously been reported;[27] measurements have only been performed at room temperature, which corresponds to $T \sim 0.4\ T_C$ for CoFe. In reference 27 the magnetic thickness of the CoFe layer was found to be reduced from the structural thickness by around 6 Å. Reducing the growth temperature below room temperature can result in an interface free of MDL, and annealing of epitaxial CoFe/GaAs(001) films at 250 $^\circ$ C has been found to result in an 11 Å interfacial MDL for films grown at any temperature up to 95 $^\circ$C[32] demonstrating the interfacial degradation which occurs in conventional materials upon heating to an appreciable fraction of their T_C.

Fig. 5(e) and (f) illustrate that, in our CoFeB:Ta/GaAs(001) structure, the lower magnetic interface is shifted (5.3 ± 0.4) Å into the volume of the CoFeB:Ta film at $0.1\ T_C$, and (8.5 ± 1) Å into the film at $0.6\ T_C$; a result that is compatible with what was found in Refs. 27 and 32 at a similar reduced temperature. Note that the values which we extract are taken directly from the modeling, rather than from any examination of the illustrative differentiated SLDs shown Fig. 5.

The critical point here is that the thickness of the MDL at the FM/GaAS(001) interface *varies with temperature*; a result which has not previously been observed, and is distinct from the enhanced MDL thickness which occurs in conventional materials due to interfacial degradation at elevated temperatures. The demonstration that the thickness of the MDL at a III-V/FM interface varies with temperature, while the structure is unmodified, is the key novel result reported in this work.

In summary, the depth-dependence of the magnetic structure in CoFeB:Ta/Ta thin films on Si/SiO_2 and GaAs(001) substrates was studied as a function of measurement temperature using polarized neutron reflectometry. The amorphous CoFeB:Ta alloy, having a reduced Curie temperature due to addition of Ta, allows investigation into interface magnetism and magnetic dead-layers at temperatures close to the Curie temperature. In these films, the magnetic interface position corresponding to the film/capping-layer interface changes with temperature due to a compositional grading which causes the local Curie temperature in the vicinity of the interface to vary; the magnetic thickness varies with temperature. With this knowledge, we are then able to robustly determine

also the presence of an additional magnetic dead-layer at the III-V semiconductor/ferromagnet interface; the thickness of this magnetically dead-layer also varies with temperature, but is confined within a localised interfacial alloyed region. Such a result, decoupling magnetic changes from thermally induced modification to the interface structure, can only be achieved using low-temperature measurements on a designed model system such as that used here. This work shows the potentially significant influence of subtle structural intermixing on magnetic thin-films: understanding interface magnetism and magnetic dead-layers is of critical importance for thin-film magnetic devices.

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