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On the magnetic ordering in interleaved Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML) superlattices

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Abstract

The magnetic properties of superlattice films with alternating Fe thicknesses, Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML) with $4 \le y \le 15$, prepared by dc magnetron sputtering on MgO(001) substrates are studied by superconducting quantum interference device measurements. By investigating the basic magnetic observables of the films: the transition temperature T_c , the magnetic remanence M_r , and the saturation field H_s , which change in a correlated manner as a function of the vanadium spacer thickness, y, we find antiferromagnetic (AFM) coupling in Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML) for 9 < y < 11. The derived values of the spin wave parameter B and the ground state magnetic moment m_{s0} also change synchronously with T_c , M_r , and H_s . The peak of the AFM coupling energy per unit area, I, is estimated to be ≈ 0.06 mJ m⁻² using the H_s versus y dependence.

1. Introduction

Ever since the first investigations on the interlayer exchange coupling (IEC) in layered magnetic structures [1], and the oscillatory ferromagnetic/antiferromagnetic (FM/AFM) character of the IEC [2], there has been a tremendous amount of research within this field. (For a review article on transition metal multilayers, see e.g. [3].) There are not so many works, however, on the effect of the IEC on the basic magnetic observables of superlattice (SL) films, such as their transition temperatures T_c , the remanent magnetization M_r , and the saturation field H_s . Some recent experimental and theoretical studies investigate the influence of the interlayer coupling on the intrinsic [4, 5] and extrinsic [6] properties of the SLs.

Generally, the basic elements in the heterostructure determine its fundamental properties. Additionally, the strength of the interlayer coupling can be tuned by the thickness of the

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spacer y, while T_c , M_r , and H_s are influenced by the thickness of the magnetic layer, as well as the thickness of the spacer.

Microscopic characteristics, such as the thermal spin wave excitations (SWE) in a material, are closely related to the exchange coupling between spins. The spin wave stiffness constant, D, is proportional to the average exchange integral and equals $B^{-2/3}$, where B is the spin wave parameter of the system under study. The SWE approach is used mainly for bulk materials, although satisfactory results have been reported [4] in the case of three-dimensional thin films also. D and B are modified by the presence of the IEC, and oscillate synchronously with T_c , M_r , and H_s for the Ni(7.3 Å)/Au(4–80 Å) heterostructural system studied in [4].

Fe(3 ML)/V(y ML) SLs were investigated [7]; it was found that the IEC is AFM for a V spacer thickness in the range of 12–14 monolayers (ML).

The basic characteristics of the Fe(2 ML)/V(y ML) system were studied [8]; it was established that the Fe(2 ML) layers are AFM coupled in the same spacer range as the Fe(3 ML) layers. The strength of the AFM coupling and the magnetoresistivity ratio were estimated to be 0.0075 mJ m⁻² and 2%, respectively [8].

It is a common assumption that the strength of the coupling between two neighbouring FM layers separated by a non-magnetic spacer will change when the thickness of the magnetic ML is altered. The change of the IEC will affect all the macroscopic and microscopic properties discussed herein.

In this work we investigate a modified Fe(x ML)/V(y ML) system with alternating Fe thicknesses: Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML) with $4 \le y \le 15$. The two bilayer prototype systems, Fe(3 ML)/V(y ML) and Fe(2 ML)/V(y ML), are interleaved and the IEC between the Fe(3 ML) and Fe(2 ML) ML is changed. We study the development of T_c , M_r , H_s throughout the system and estimate the AFM coupling in Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML)/Fe(2 ML)/V(y ML).

2. Experimental details

Ten repetitions of Fe(3 ML)/V(*y* ML)/Fe(2 ML)/V(*y* ML) with vanadium thickness *y* between 4 and 15 ML were grown epitaxially at 330 °C on MgO(001) substrates of dimensions $10 \times 10 \times 0.5 \text{ mm}^3$, and capped with ~60 Å of Pd to protect against oxidation in an ultrahigh-vacuum dc sputtering system [9]. Each sample was characterized by conventional low- and high-angle XRD analysis to investigate the structural quality and obtain the total repeat thickness, Λ . The SL fringes with minor increase of their widths from sample to sample, and therefore clearly defined repeat distances for Fe(3 ML)/V(*y* ML)/Fe(2 ML)/V(*y* ML), $4 \leq y \leq 15$, were seen in the range of 0° to 12°. The high-angle XRD data showed the Bragg peak of the film and, in general, two SL satellites on each side. The full widths at half-maximum (FWHM) of the (002) Bragg peaks for the films in the radial and transverse directions are typically 0.6° and 0.8°, respectively. Figures 1(a) and (b) show the results from the low-and high-angle XRD experiments for Fe(3 ML)/V(9 ML)/Fe(2 ML)/V(9 ML) in particular.

To estimate the average interface roughness, the reflectivity data were simulated using the program GIXA [10]. The calculated interface roughness is $\leq \sim 1.4$ Å, which corresponds approximately to <1 atomic layer. Line *n* in figure 1(a) shows the result of the simulation for Fe(3 ML)/V(9 ML)/Fe(2 ML)/V(9 ML).

The XRD characterization was followed by Rutherford back-scattering (RBS) measurements [11] carried out to identify the ML thicknesses of the Fe and V layers. The RBS spectrum was simulated using existing programs (see e.g. www.genplot.com), to estimate the Fe and V thicknesses of the total repeat distance Λ to a precision of 0.6 Å.



Figure 1. Low-angle (a) and high-angle (b) XRD data for Fe(3 ML)/V(9 ML)/Fe(2 ML)/V(9 ML). Curve n in figure 1(a) (translated vertically) shows the simulation of the data using GIXA.

Physical properties such as the temperature and field dependences of the magnetization, magnetic hysteresis, and remanence at relevant temperatures were measured using superconducting quantum interference device magnetometry. The magnetic field during the measurements was applied in the plane, in the [110] direction of the film, in view of the fact that the Fe(3 ML)/V(y ML) SLs are isotropic over a wide temperature range beginning from the lowest utilized temperatures [12], which was confirmed to be true for the Fe(3 ML)/V(y ML) system also.

The temperature dependence of the magnetization was measured using an applied field of 1200 Am^{-1} . The *M* versus field *H* dependence was measured at 20 K to obtain the saturation field H_s and the corresponding saturation magnetization M_s . We chose 20 K as the temperature for the *M* versus *H* measurements, because the magnetic contribution of the substrate at this temperature is two orders of magnitude smaller than that of the magnetic films, for fields up to 240 kA m⁻¹. Nonetheless, the magnetic moment of the substrate is taken into account in the analysis of the *M* versus *H* results. The remanent magnetization M_r at a temperature *T* is the magnetization measured in zero field after removing a field of 800 kA m⁻¹.

The magnetization per Fe atom, m, in Bohr magnetons (μ_B) was calculated using the total volume of the Fe layers in each film as the magnetic volume. The contribution of the induced moments in the V spacer was not taken into account directly⁴.

⁴ Our measurements show that the average Fe moment is considerably reduced ($\sim 0.65 \mu_B$), compared to the average moment of bulk Fe, $-2.2 \mu_B$. Similar results were reported in [13], and explained on the basis of the specific responses of these SL when the number of the Fe ML is reduced to 3 ML, or less than 3 ML. Then the contribution of the V spacer to the total measured magnetic moment is much more crucial. Effectively we measure the difference between Fe and V interface moments, which are coupled antiferromagnetically [13].



Figure 2. The dependence of the transition temperature, T_c , on the V spacer thickness, y (ML) for Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML), $4 \le y \le 15$. T_c is presented as estimated from m^2 versus T (solid circles) and from dm/dT versus T (open circles), with 5% error bars. The solid curve connecting the experimental data points and the dotted vertical lines in the figure serve as guides for the eye. The inset shows the determination of T_c for Fe(3 ML)/V(8 ML)/Fe(2 ML)/V(8 ML) from (a) m^2 versus T, and (b) from the minimum of dm/dT (m is presented in Bohr magnetons (μ_B)).

3. Results and discussion

Generally, Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML) SLs are magnetically soft materials with coercive fields of $H_c < 4.8$ kA m⁻¹ at T = 5 K. The temperature dependence of the magnetization of the different SLs reveals the interplay between the interlayer and intralayer magnetic coupling of the Fe layers as well as the destruction of the magnetic ordering upon heating.

3.1. Transition temperature

The transition temperature T_c was determined from the measured M(T) dependence, either by linear extrapolation of $M^2(T)$ to M = 0 according to molecular field theory, or from the minimum of dM(T)/dT versus T [4, 8].

The dependence of the transition temperature on the V layer thickness is illustrated in figure 2. The insets of the figure show the determination of T_c by both methods for Fe(3 ML)/V(8 ML)/Fe(2 ML)/V(8 ML) in particular.

A strong decrease of T_c from ~400 to ~140 K is observed with V increasing from 6 to 9.5 ML. Minima of T_c are seen for y = 9.5 and 11 ML, and a local maximum of T_c (~210 K) is indicated at y = 10 ML. Upon further increase of the V thickness from 13 to 15 ML, the ordering temperature slowly increases. Similar dependences of the ordering temperature with the spacer thickness have been observed for Ni(7.3 Å)/Au(4–80 Å) [4] and Fe(2 ML)/V(y ML) [8] multilayers.

Within mean-field theory, the transition temperature T_c is proportional to the interlayer exchange energy per magnetic atom, ε . A change in sign of the interlayer coupling corresponds to reversal of the relative alignment of the sublayer magnetizations, so ε is always negative, depends only on the absolute value of the coupling coefficient, and has a period of oscillation

half that of the exchange coupling [4]. Therefore, a maximum in the thickness dependence of $|\varepsilon|$ will correspond either to FM or AFM coupling. Correspondingly, the $T_c(y)$ function will have a local maximum at the same thickness of the spacer layer. The local minima in $|\varepsilon(y)|$ will correspond to uncoupled or weakly coupled layers. Consequently the function T_c should exhibit local minima at the same y as $|\varepsilon(y)|$.

In terms of the coupling of the Fe(3 ML) and Fe(2 ML) layers, figure 2 shows minima in the ordering temperature (coupling energy) for V thicknesses of 9.5 and 11 ML. Furthermore, a local maximum of the coupling energy, indicative of FM or AFM ordering, is seen at y = 10 ML. The first inflection point in the $T_c(y)$ curve, at $y \approx 7$ ML of V in figure 2, corresponds roughly to the onset of the weak coupling of magnetic layers in the Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML) system.

Complementary magnetic hysteresis measurements at 10 K showed a progressive decrease of the coercive field H_c from ~3.2 kA m⁻¹ at y = 6 ML to 1.2 kA m⁻¹ for y = 9 ML of V. H_c retains its value in the range of 9–11 ML of V and increases thereafter to 3.2 kA m⁻¹ as the V thickness increases from 12 to 15 ML.

We have calculated the interlayer coupling energy per unit area, I, using the function $T_c(y)$ and assuming that the IEC affects all Fe atoms equally. Using $\Delta T_c = T_{max} - T_{min} \approx 55^{\circ}$ results in coupling energy per unit area $I \approx 31 \text{ mJ m}^{-2}$ (31 erg cm⁻²), which is larger than theoretical values cited in the literature—0.1–0.4 erg cm⁻² [14], but close to some reported experimental values [4] obtained using the T_c versus y dependence. One reason for the quantitative discrepancy between experimental and theoretical estimates could be the fact that T_c is affected by the maximum value of the coupling energy, not by its average value [4]. Experimentally, however, it is not possible to extract how the coupling strength varies inside a magnetic layer.

Qualitatively the periodic change of T_c observed for Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML) within the range of $4 \le y \le 15$ gives unique information on the gradual change of the interlayer coupling upon increasing the spacer thickness. By measuring only T_c , however, it is not possible to distinguish between FM and AFM coupling in a system.

To learn more about the nature of the magnetic transition, T_c was estimated independently as a free parameter along with the critical exponent β using the scaling law for the magnetization as a function of temperature (Barber [15]):

$$M \propto (1 - T/T_c)^{\beta} \qquad \text{for } T < T_c. \tag{1}$$

The best fit to equation (1) was obtained by varying T_c ; the values of T_c so obtained are generally close (~ -3 K) to the values obtained from dM/dT versus T. The exponent β decreases from 0.40 to 0.37 (± 0.01) for the FM samples investigated in the range of $5 \le y \le 8$, correspondingly. These values of β are at the lower end of a 3D Heisenberg spin–lattice system ($\beta = 0.37$) (Barber [15]) and higher than the values of β for finite-size 2D XY magnets (0.23) (Bramwell [15]). One reason for the low-end values of β may be the fact that we are not dealing with a standard 3D bulk material. Nevertheless, the values of β for all FM samples of the Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML) series investigated show that they undergo a standard second-order FM–paramagnetic transition.

3.2. Exchange coupling in Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML)

Figures 3(a) and (b) depict the variation of the saturation field and the magnetic remanence of Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML), both measured at 20 K, as a function of the V layer thickness. As seen in figure 3(a), the saturation field increases by one order of magnitude as the thickness of the V layers increases from ~9 to 10 ML. However, the remanent magnetization



Figure 3. (a) The saturation field H_s versus y (ML). The base dashed line marks the behaviour expected without interlayer coupling. (b) The remanence M_r/M_s versus y (ML). The dotted horizontal line is the remanence for uncoupled layers.

 M_r/M_s , displayed in figure 3(b), drops sharply in the same thickness range, and has its minimum value of ~0.5 for y = 10 ML. This is the same V thickness for which the functions $T_c(y)$ in figure 2 and $H_s(y)$ in figure 3(a) have a local maximum.

While from figure 2 it was not possible to reach a conclusion as regards the sign of the interlayer coupling in Fe(3 ML)/V(10 ML)/Fe(2 ML)/V(10 ML), figure 3 adds to our understanding on this. A minimum in M_r/M_s , accompanied with a simultaneous sharp increase and local maximum in H_s , is indicative of antiferromagnetically coupled Fe ML. Hence from the results presented in figures 2 and 3 it is possible to conclude that the Fe(3 ML) and Fe(2 ML) layers are aligned oppositely in a ferrimagnetic configuration in the Fe(3 ML)/V(10 ML)/Fe(2 ML)/V(10 ML) film⁵.

The ferrimagnetic structure in Fe(3 ML)/V(10 ML)/Fe(2 ML)/V(10 ML) is one plausible reason for the value of its magnetic remanence ~0.5 (see figure 3(b)). Another factor contributing to the remanence of Fe(3 ML)/V(10 ML)/Fe(2 ML)/V(10 ML) is the presence of uncorrelated interface roughness (~0.3 ML) and local thickness fluctuations, which result in interface regions with FM coupling, coexisting with the general AFM ordering for the y = 10 ML film. Close values for M_r/M_s (0.6) [4], and not-so-close values (0.2) [8] and (-0.1) [16] are reported and interpreted similarly.

 $^{^5}$ The magnetic structure of the film is ferrimagnetic, because the magnetic moment of Fe(3 ML) is larger than that of Fe(2 ML).

Comparing the different results displayed in figures 2 and 3, it is easy to see that the local maximum in $T_c(y)$ in figure 2 coincides with the maximum in the $H_s(y)$ function in figure 3(a) and the minimum in $M_r(y)/M_s(y)$ in figure 3(b). The gradual increase of $T_c(y)$ beginning at y = 13 ML marks the beginning of the next-order periodic change of T_c . This higher-order coupling initiates the general change of the physical quantities observed in figure 3. However, it is not an objective of this work to investigate the higher-order couplings in the present system.

The H_s -values for all ferromagnetically coupled and uncoupled samples were interpolated to estimate the strength of the AFM interlayer coupling per unit area I. The result of this interpolation is presented as the base dotted line in figure 3(a). We used the assumption that the interlayer exchange energy is balanced by the magnetostatic energy of the Fe layers in an applied field, H_{eff} . The effective field H_{eff} is defined as $H_{eff} = H_s^{max} - H_{av}$, where H_s^{max} is the maximum field needed to align the antiferromagnetically coupled layers ferromagnetically, and H_{av} is the estimated field for the same V thickness if the Fe layers were uncoupled. As seen from the base line in figure 3(a), H_{av} is considerably lower than H_s^{max} .

The estimated value for the peak in the AFM coupling energy per unit area is $I = 0.06 \text{ mJ m}^{-2}$, for Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML) in the range investigated, $4 \le y \le 15$. This value of *I* is smaller than the theoretically anticipated values ~0.1–0.4 erg cm⁻² [14]. A possible reason for the divergence of our result could be that in [14] the calculations are for smaller spacer thickness ~4.5 ML and the first AFM coupling peak, while in this work, and in the Fe/V systems in general, the first AFM coupling peak, expected to occur at y = 12 Å, is suppressed due to the transient FM moments induced in the V spacer [16]. We observed experimentally and estimated the second AFM coupling peak in Fe/V.

3.3. Spin waves in Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML)

The temperature-dependent magnetization curves of the FM coupled and uncoupled samples from the Fe(3 ML)/V(y ML)/Fe(2 ML)/V(y ML) series were measured using an applied field larger than their low-temperature saturation fields H_s . The *M* versus *T* curves of the ferrimagnetic samples were measured, after we had first demagnetized the samples at room temperature, and then applied a field of 400 A m⁻¹ in their [110] directions. This field value is less than 1% of the saturation fields for these samples. In the following, the measured magnetization will be given as the magnetic moment per Fe atom, and denoted by $m_s(T)$. The data for $m_s(T)$ were fitted to the expression [4, 17]

$$m_s(T) = m_{s0}(1 - BT^x)$$
(2)

where x may vary from 1 to 2 depending on the type of the interlayer coupling [17]. The exponent x is ≈ 1 for uncoupled layers, $\approx 3/2$ for ferromagnetically coupled layers, and ≈ 2 for antiferromagnetically coupled layers [8, 17]. *B* and m_{s0} in equation (2) are the spin wave parameter and the average ground state magnetic moment, respectively. Equation (2) is valid for temperatures $T < T_c/2$ according to the spin wave theory. As pointed out in [4] and [18], equation (2) may hold for ultrathin films also, with changes expected in the parameter values due to the reduced coordination at the interfaces. Our results on the critical exponent β showed that we could still regard these materials as three-dimensional systems.

Reasonable fits (standard deviation: $\leq 3\%$) of the $m_s(T)$ data to equation (2) were obtained using $x \approx 3/2$. The parameter values extracted from this procedure, B(y) and $m_{s0}(y)$, are shown in figures 4(a) and (b). A minimum of *B* (maximum of *D*) occurs at y = 10 ML as seen from figure 4(a), while *B* exhibits maxima (minima of *D*) at $y \approx 9$ and 11 ML. We established that for y = 10 ML the coupling is AFM and for y = 9 and 11 ML the Fe layers are uncoupled. The results for *B* (and *D*) may indicate that the AFM interlayer coupling



Figure 4. (a) Spin wave parameter *B* versus *y* (ML). (b) Average ground state magnetic moment per Fe atom at $T \rightarrow 0$, m_{s0} (μ_B), versus *y*. The inset in (b) shows a representative m (μ_B) versus *T* plot for Fe(3 ML)/V(8 ML)/Fe(2 ML)/V(8 ML) and the result of the data simulation (the line) according to equation (2). The solid curves connecting the experimental data points in (a) and (b), and the dotted vertical lines, serve as guides for the eye.

stabilizes the magnetic order against thermal fluctuations, since a local maximum in D implies that there is a local maximum in the average coupling energy per atom [4]. For y = 9 and 11, the D versus y curve exhibits minima, and therefore minima in the coupling energy, which is consistent with the results from sections 1 and 2.

As introduced, m_{s0} is the amplitude of the spin density wave at T = 0 K. The parameter m_{s0} is expected to vary synchronously with the average coupling energy per magnetic atom [19]. Thus, m_{s0} should decrease from the ferromagnetically coupled samples to the uncoupled samples and increase back for a stable AFM coupling and structure. In figure 4(b) a 15–20% increase of m_{s0} is observed as y reaches 10 ML, which may be explained as indicated above—a general stabilization of the magnetic order in the AFM state of this heterostructure compared to the uncoupled samples with $y \approx 9$ and 11 ML of V.

To summarize, the basic magnetic characteristics of the SL system with alternating thicknesses of Fe, Fe(3 ML)/V(*y* ML)/Fe(2 ML)/V(*y* ML), have been investigated by a number of independent techniques; we find that the Fe layers in the system are antiferromagnetically coupled for $9.5 \leq y \leq 11$ ML. The AFM coupling is found at the same V thickness in measurements of T_c , M_r/M_s , H_s , D, and m_{s0} . The peak of the coupling energy per unit area in the ferrimagnetic Fe(3 ML)/V(10 ML)/Fe(2 ML)/V(10 ML) SL, I is estimated to be ≈ 0.06 mJ m⁻² using the H_s versus y dependence. This method of estimation can be utilized with satisfactory precision, in our opinion, to compare the coupling strengths in modified systems such as Fe(3 ML)/V(y ML) and Fe(2 ML)/V(y ML) [8].

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