

Magnetization of uncovered and V-covered ultrathin Fe(100) films on V(100)

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We used polarized neutron reflectometry (PNR) to determine the absolute magnetic moment of uncovered and V-covered Fe films in the thickness range from 0.3 to 5.5 nm. The films were prepared by molecular beam epitaxy on a V(100) buffer layer grown on a MgO(100) crystal. The magnetic moment shows a linear dependence on the Fe film thickness with a reduction (compared to the Fe bulk value) of the magnetic moment equivalent to 0.1 nm bulk Fe for the V-covered films and a reduction equivalent to 0.03 nm bulk Fe for the uncovered Fe films. For the case of the V/Fe/V samples we observe a much smaller reduction of the magnetic moment than reported for experiments on Fe/V multilayers. As theoretical calculations show a strong decrease of the magnetic moment for an interface alloy we conclude that the larger reduction of the magnetization in Fe/V multilayers is due to an increase in interface roughness with increasing film thickness. For the uncovered Fe(100) films we find a much smaller reduction of the magnetic moment than in earlier *in situ* PNR experiments on V(110)/Fe(110) where we observed a reduction equivalent to 0.4 nm bulk Fe.

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I. INTRODUCTION

The investigation of the magnetic moment of thin ferromagnetic films in contact with non-ferromagnetic materials has attracted the interest of many researchers over the last two decades. It is a challenging task from an experimental as well as theoretical point of view because in many cases the deviations from the bulk values are very small. Therefore, a lot of measurements were not performed on single films but rather on multilayers to increase the magnetic signal. There are only a few methods available which are capable of determining the absolute magnetic moment with an accuracy in the range of a few per cent of a Fe monolayer. Besides the magnetometry techniques like torsion oscillation magnetometry¹⁻³ and alternating gradient magnetometry⁴ also polarized neutron reflectometry^{5,6} was used to determine the absolute magnetic moment of thin films. The widely used superconducting quantum interference device (SQUID) magnetometry suffers from the huge diamagnetic background signal of typically used substrates. X-ray magnetic circular dichroism (XMCD) has become a very powerful tool because of its element sensitivity. On the other hand, there remains always the uncertainty of the applicability of the sum rules.

The vanadium atom has a magnetic moment of $3\mu_B$ in its ground state whereas the V bulk is nonmagnetic. The prediction of a nonmagnetic-magnetic transition by increasing the lattice parameter⁷ shows that vanadium is not that far away from magnetism. This leads to the interesting question whether a ferromagnetic film in contact with a V film could trigger a magnetic ordering as well. The choice of Fe as the 3d-ferromagnet is logical because it also crystallizes in the bcc structure which is a good precondition for epitaxial growth.

All theoretical calculations⁸⁻¹² on the Fe/V system predict an induced magnetization for the V interface antiferromagnetically coupled to the Fe magnetization which itself is

reduced at the interface. The antiferromagnetic coupling of the V polarization was verified experimentally by spin-polarized electron energy loss spectroscopy,¹³ by spin-polarized Auger electron spectroscopy,¹⁴ and x-ray magnetic circular dichroism.^{9,15-17} Some authors even claim an oscillatory behavior of the V polarization¹⁴ but that has never been observed by other groups.

The goal of the present work was to determine in a straightforward way the absolute magnetic moment of uncovered and V-covered single Fe(100) films in order to investigate whether results from multilayers by averaging over many nonidentical interfaces should be taken to infer properties of a single Fe/V interface. Therefore, we studied the epitaxy of V and Fe in great detail in order to get films with smooth interfaces and high crystalline quality.

II. SAMPLE PREPARATION

The most important issue in thin film magnetism is the preparation of a chemically clean sample of homogeneous thickness with a high crystalline quality. In the case of vanadium it is well-known that it is very difficult to clean V single crystals.¹⁸⁻²⁰ Therefore, instead of using a V single crystal substrate we prepared a V buffer layer on top of a MgO(100) crystal by molecular beam epitaxy (MBE) using an electron beam evaporator. The MgO crystal was cleaned in alcohol before it was introduced into the ultrahigh vacuum (UHV) chamber which was operated at a base pressure below 10^{-10} mbar. The substrate was annealed for 20 minutes at 900 K to remove contaminations from the surface and then checked by low energy electron diffraction (LEED) and Auger electron spectroscopy (AES).

The V buffer layer was evaporated at a rate of 0.01 nm/s at room temperature and had a thickness of 85 nm. To improve the crystalline quality the film was annealed at 700 K. As can be seen in Fig. 1 the LEED picture shows an oxygen

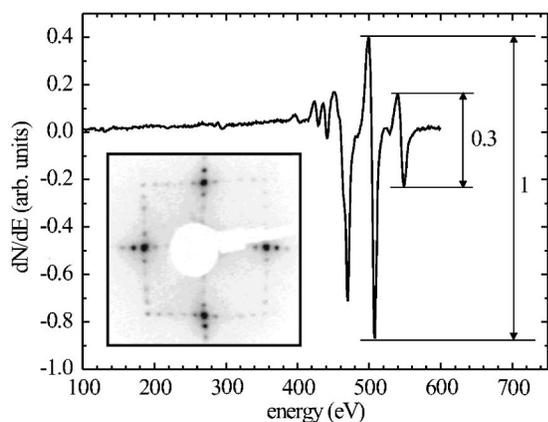


FIG. 1. AES data and LEED pattern (as inset) of a 85 nm thick V buffer layer prepared on MgO(100) after annealing at 700 K for 20 minutes. Please note the shift of the whole spectrum by 33 eV due to electrical charging of the sample.

induced 5×5 superstructure. Turban *et al.*²¹ revealed that this LEED pattern is due to a superposition of 1×5 and 5×1 domains. After deposition of additional 3 nm V at room temperature we got an oxygen-free surface of high crystalline quality as can be seen from Fig. 2. The LEED pattern is more sensitive to oxygen contamination than the AES measurement because the V and O peaks overlap and cannot be resolved as two peaks. A quantitative measure of an oxygen-free surface is the ratio of the peak at 547 eV to the one at 506 eV. The oxygen-free surface results in a ratio of 0.25, whereas for an oxygen contamination the ratio is larger than 0.25, as can be seen in Fig. 1, where the ratio is 0.3. This additional intensity due to oxygen is also reported in Refs. 18 and 21. In our case the whole spectrum is shifted to higher energies by 33 eV due to electrical charging.

The Fe and V films were evaporated at a rate of 0.007 nm/s at room temperature. For the *ex situ* measurements all films were covered by a 10 nm thick V layer. For the *ex situ* experiments we always used a new substrate for each sample, whereas for the *in situ* experiments we used the same substrate crystal. In the latter case the Fe film was

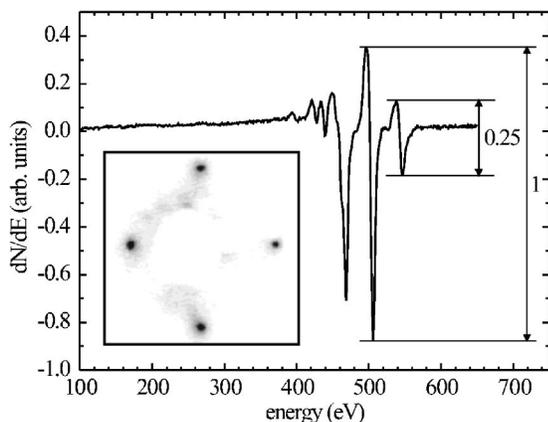


FIG. 2. AES data and LEED pattern (as inset) of a 85 nm thick V buffer layer after annealing at 700 K for 20 minutes and evaporation of additional 3 nm V. Please note the shift of the whole spectrum by 33 eV due to electrical charging of the sample.

removed by sputtering before the preparation of a new sample was started.

III. EXPERIMENTAL SETUP AND METHOD

All PNR experiments were performed on the neutron reflectometer V6 of the Hahn-Meitner-Institut in Berlin.²² The graphite monochromator was set to deliver neutrons with a wavelength of $\lambda=0.466$ nm. The higher order neutrons with a wavelength of $\lambda/2$, $\lambda/3$, etc., were removed by a Be filter. The neutrons were polarized by using an Fe-Co/Si supermirror in transmission.²³ The instrument has a vertical scattering geometry (the sample is mounted horizontally) and the magnetic field is applied in the plane of the sample. For the *ex situ* measurements we used ³He pencil detectors mounted on the standard detector arm which moves by an angle 2θ if the sample is moved by an angle θ . For the *in situ* measurements we had to remove the standard detector arm along with the electromagnet in order to clear the space for the MBE chamber. Therefore, we used a Helmholtz coil arrangement for the magnetic field and a stationary position sensitive detector to record the reflected intensity. More details on the *in situ* setup can be found in Nawrath *et al.*⁶

For the case of neutron reflectometry the interaction of neutrons with a film can be described by the Fermi pseudo-potential V^\pm

$$V^\pm = \frac{2\pi\hbar^2}{m} \rho(b_n \pm b_m), \quad (1)$$

where m denotes the neutron mass, ρ the atomic density, b_n the nuclear scattering length, and b_m the magnetic scattering length. The superscripts $+$ and $-$ indicate that the scattering potential is different for neutrons with their spins aligned parallel ($+$, up-neutrons) or antiparallel ($-$, down-neutrons) to the external field. The magnetic scattering length is directly proportional to the magnetization of the sample with $b_m = c\mu$, where $c=2.695$ fm/Bohr magneton is a conversion constant and μ is the magnetic moment per atom. In the specular reflection geometry the scattering vector $\mathbf{q}=\mathbf{k}_f-\mathbf{k}_i$ is perpendicular to the sample's surface with k_f and k_i being the wave vector of the reflected and incoming beam. Therefore, perpendicular magnetization components do not contribute to the potential V .

The calculation of the neutron reflectivity is analogous to the calculation of the Fresnel reflectivity in light optics. The index of refraction for neutrons n_N is given by

$$n_N = \sqrt{1 - \frac{2mV}{\hbar^2 k^2}}, \quad (2)$$

with $k=2\pi/\lambda$ being the neutron wave vector in the medium where the reflection process takes place. Details on calculations of reflectivity curves can be found elsewhere.^{24,25} We used the software Parratt32²⁶ which is based on the recursion formula developed by Parratt.²⁷

Contrary to x rays, the nuclear scattering length b_n of neutrons is not proportional to the atomic number. For some elements small or even negative scattering length density values can occur like for V for which $\rho b_n = -27.6 \mu\text{m}^{-2}$,

whereas for other elements a large scattering length density is observed as for Fe with $\rho b_n = +802 \mu\text{m}^{-2}$. For comparison, the magnetic scattering length density of bulk Fe at room temperature is $\rho b_m = 498 \mu\text{m}^{-2}$.

It is important to note that we cannot determine where the reduction of the magnetic moment is exactly located, i.e., in the V interface layer or the Fe interface layer or in both. The reason for that is that the deviation from bulk behavior is confined to about two layers. The corresponding q vector of about 20 nm^{-1} is way beyond the accessible q range of the used reflectometer because of lack of intensity. Hence, with PNR we measure the averaged magnetic moment of the whole film as in classical magnetometry.

IV. MAGNETIC MOMENTS OF THIN FILMS

The spontaneous magnetization is the basic property of a ferromagnet which distinguishes it from other materials. In ultrathin films the absolute value as well as the temperature dependence deviates from the bulk behavior.^{1,28} Therefore, it is important to obtain experimental data in order to compare to theoretical calculations.

Bloch's law describes the temperature dependence of the magnetization $M(T)$ of a bulk ferromagnet far below its Curie temperature,

$$M(T) = M(0) \cdot (1 - bT^{3/2}). \quad (3)$$

The spin wave parameter b describes the deviation of the magnetization from the ground state $M(0)$.

Surprisingly, Bloch's law describes the temperature dependence of thin Fe(110) films²⁹⁻³¹ very well in contrast to the linear temperature dependence predicted by the spin wave theory.^{32,33}

The magnetization of thin films with a thickness t can generally be written as

$$\mu \cdot t = \mu_{\text{bulk}} \cdot t + \hat{\mu}_{\text{size}} + \hat{\mu}_{\text{int}}, \quad (4)$$

where the magnetization μ is given in units μ_B per atom which makes it easy to compare to theoretical papers. The relation to the magnetization M is simply

$$M = \mu \frac{\rho N_A}{u}, \quad (5)$$

where ρ denotes the density, u the mass number, and $N_A = 6.022 \times 10^{23}$. The size effect $\hat{\mu}_{\text{size}}$ accounts for the reduced magnetic moment due to the reduced Curie temperature of thin films and shows a strong temperature dependence. The interface effect $\hat{\mu}_{\text{int}}$ accounts for the change of the electronic structure at the two interfaces³⁴ and is virtually independent on temperature. So, in order to distinguish between these two effects, it is inevitable to measure the magnetic moment as a function of temperature.

We use Eq. (4) in the following sections to fit our results. By plotting $\mu \cdot t$ versus t the slope of the linear fit represents the Fe bulk magnetization and the intersection with the ordinate represents the size and interface effect. The intersection with the abscissa yields the reduction of the total magnetic moment corresponding to exactly that thickness of bulk Fe.

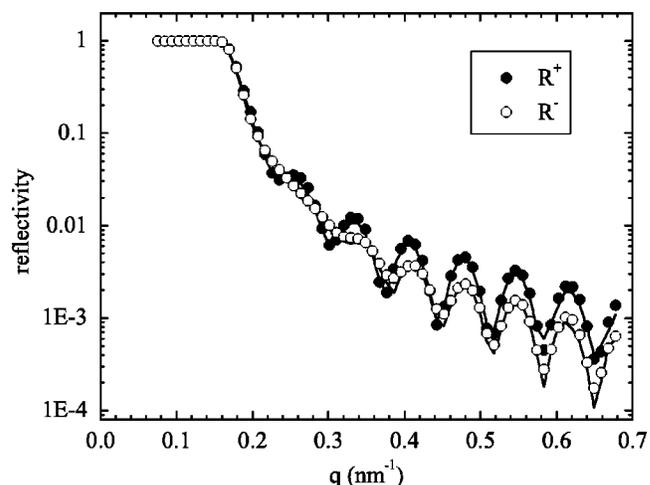


FIG. 3. PNR data of a 0.97 nm thick V-covered Fe layer on a 88 nm thick V buffer. The Kiessig fringes for up-neutrons (solid circles) and down-neutrons (open circles) are clearly visible. The solid lines represent the fits.

By assuming that the reduction is confined to the interface or surface atoms only we calculate in the following sections a reduction per Fe interface atom. This can be easily compared to theoretical calculations.

V. RESULTS

A. V-covered Fe films

As the scattering contrast between V and Fe is very large the reflected neutron intensity is very sensitive to a small amount of Fe on V. This has been shown already in earlier PNR experiments on the V/Fe system^{6,35,36} and can be seen in Fig. 3, where the reflected intensity of an Fe film with a thickness of $t_{\text{Fe}} = 0.97 \text{ nm}$ on a 88.3 nm thick V buffer is shown. The reflected intensity of the up-neutrons is given by the solid circles, whereas the reflectivity of the down-neutrons is given by the open circles. The solid lines are the fits. From the PNR fits we conclude a magnetic moment per Fe atom of $\mu = 1.94 \mu_B$ which is considerably reduced from the bulk value at room temperature³⁷ of $\mu = 2.18 \mu_B$ per atom. As the V cap layer was exposed to air we must take into account that it was partially oxidized consisting of a pure V layer with a thin vanadium oxide layer. This vanadium oxide layer has a large scattering length density compared to V because oxygen has a scattering length of 5.803 fm compared to V of -0.38 fm . Therefore, the separation Δq of two maxima of the Kiessig fringes³⁸ is proportional to $1/(t_{\text{V,sub}} + t_{\text{Fe}} + t_{\text{V,cap}} + t_{\text{V}_2\text{O}_3})$ with $t_{\text{V,sub}}$ denoting the thickness of the V substrate layer and $t_{\text{V,cap}}$ the thickness of the V cap layer. For the case of a pure V cap layer Δq would be proportional to $1/(t_{\text{V,sub}} + t_{\text{Fe}})$ because the scattering length density contrast between V and air is too small to show up in the Kiessig fringes. We also performed x-ray reflectometry (XRR) experiments in order to confirm the fitted oxide layer thickness. Within the error limits the XRR and PNR data gave the same value for the thickness of the vanadium oxide layer.

The thickness dependence of the magnetic moment, plotted as the magnetic moment per atom times t_{Fe} , is shown in

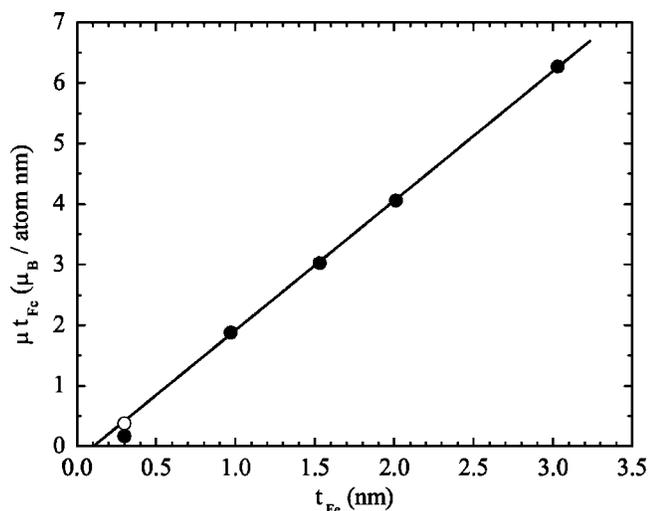


FIG. 4. Magnetization values as determined from PNR are plotted as the product of the magnetic moment per atom times the Fe film thickness t_{Fe} . The solid circles are from experiments at 295 K, the open circle is from measurements at 10 K, and the solid line is a linear fit to the data for $t_{\text{Fe}} > 0.4$ nm.

Fig. 4. The data for $t_{\text{Fe}} > 0.3$ nm show a perfect linear behavior with a slope of $(2.14 \pm 0.02) \mu_B/\text{atom}$ and an intersection with the abscissa at $t_{\text{Fe}} = (0.1 \pm 0.01)$ nm. The slope represents the magnetization of bulk Fe and the intersection is proportional to the surface and size effect. From the intersection it is easily calculated that the magnetic moment of the samples is reduced by an amount of magnetic moment equivalent to 0.1 nm bulk Fe. Assuming that both interfaces are equal this corresponds to a reduction of $(0.75 \pm 0.05) \mu_B/\text{Fe}$ interface atom.

The reduction of the magnetization increases with decreasing t_{Fe} and for the smallest Fe thickness $t_{\text{Fe}} = 0.3$ nm we determined a magnetization of $\mu = 0.57 \mu_B/\text{Fe}$ atom. This data point (solid circle in Fig. 4) is off the fit line inferred from the data points for $t_{\text{Fe}} > 0.3$ nm. However, at a temperature of 10 K it is back on the fit line (open circle in Fig. 4) with $\mu = 1.28 \mu_B/\text{Fe}$ atom. The temperature dependent measurements for the sample with $t_{\text{Fe}} = 0.3$ nm are shown in Fig. 5. The dashed line is a phenomenological power law fit

$$\mu = \mu_0 \cdot \left(1 - \frac{T}{T_C}\right)^\beta \quad (6)$$

with $\mu_0 = 1.28 \mu_B/\text{atom}$, $\beta = 0.57 \pm 0.12$, and $T_C = (398 \pm 45)$ K but we want to take it only as a guide for the eye to get a rough estimate for the Curie temperature.

B. Uncovered Fe films

All *in situ* PNR experiments were performed at 295 K. A typical reflectivity curve obtained from *in situ* PNR on a film with $t_{\text{Fe}} = 2.36$ nm is shown in Fig. 6. The reflectivity curves of the *in situ* experiments look very similar to the *ex situ* ones. The only difference is that the *ex situ* samples have an additional vanadium and vanadium oxide layer. Therefore, as discussed above, the separation Δq of two maxima of the reflectivity curve is larger for the *in situ* than the *ex situ*

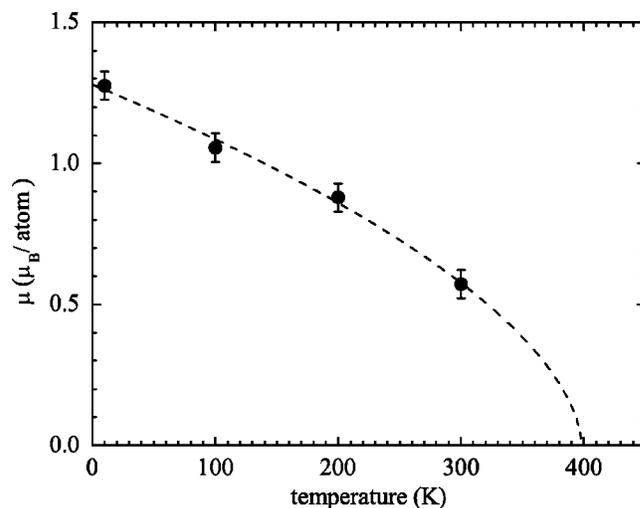


FIG. 5. Magnetic moment of a 0.3 nm thick Fe film as a function of temperature. The dashed line is a fit according to Eq. (6) with $T_C = (398 \pm 45)$ K.

experiments. For the *ex situ* experiments Δq is proportional to $1/(t_{\text{V,sub}} + t_{\text{Fe}} + t_{\text{V,cap}} + t_{\text{V2O3}})$, whereas for the *in situ* experiments Δq is proportional to $1/(t_{\text{V,sub}} + t_{\text{Fe}})$.

The magnetic moment of the films also shows a linear dependence on t_{Fe} as can be seen in Fig. 7. The value for the slope is $(2.14 \pm 0.04) \mu_B/\text{atom}$ and the intersection with the abscissa is at $t_{\text{Fe}} = (0.03 \pm 0.06)$ nm. The slope representing the magnetic moment of bulk Fe is the same as for the V-covered Fe films, whereas the reduction equivalent to 0.03 nm bulk Fe is much smaller than for the V-covered films. Assuming that both interfaces were equal this would correspond to a reduction of $(0.23 \pm 0.45) \mu_B/\text{Fe}$ interface atom.

VI. DISCUSSION

From Fig. 4 we can infer that the V-covered Fe films show a total reduction of 0.1 nm bulk Fe magnetic moment or

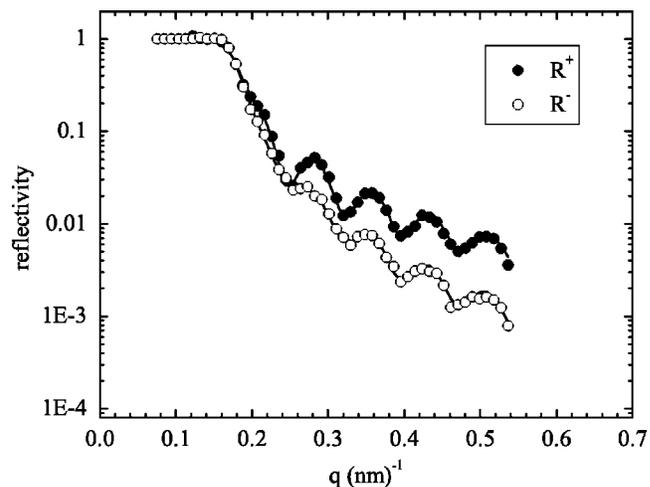


FIG. 6. PNR data of a 2.36 nm thick uncovered Fe layer measured *in situ*. The solid circles represent the reflectivity of up-neutrons, whereas the open circles represent the reflectivity of down-neutrons. The solid lines represent the fits.

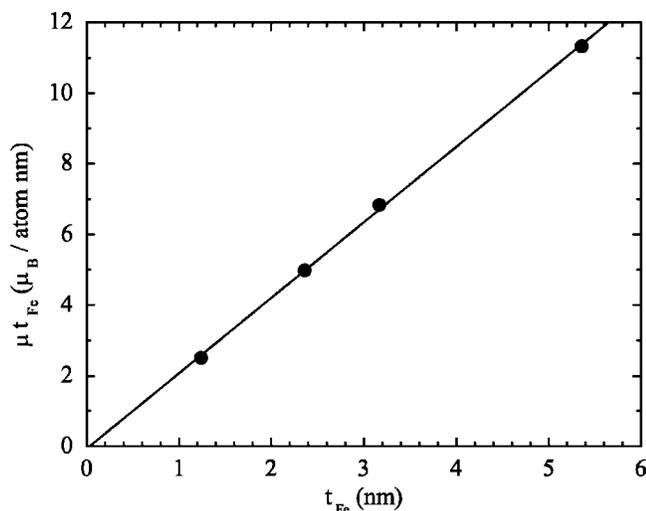


FIG. 7. Magnetization values as determined from *in situ* PNR are plotted as the product of the magnetic moment per atom times the Fe film thickness t_{Fe} . All experiments were performed at 295 K. The solid line is a linear fit to the data.

$0.75\mu_B$ per Fe interface atom at both Fe/V interfaces. This is only right under the assumption that both interfaces are identical. However, they are not identical from a structural point of view because the Fe starts with pseudomorphic growth on the V substrate layer and relaxes with increasing film thickness towards the Fe bulk lattice constant²⁰ which is about 5% smaller than the one of V. On the other hand, the vanadium on top of the Fe layer must adapt to the Fe lattice constant. Hence, both Fe/V interfaces are structurally different because calculations done by Niklasson *et al.*¹¹ show indeed that both the reduction of the Fe magnetic moment and the polarization of the V layer depend on the lattice parameter. However, these effects cancel each other and the sum of these two effects for a V/Fe interface with a specific lattice constant is virtually independent on the lattice parameter what finally justifies our assumption that the total reduction is equally distributed among the two V/Fe interfaces with a reduction of $0.75\mu_B$ per Fe interface atom at each interface.

Our experimentally derived value of $0.75\mu_B$ reduction per interface atom is in perfect agreement with the XMCD experiments of Tomaz *et al.*¹⁵ on V/Fe multilayers with a 11 monolayer thick V-layer revealing a total reduction of $0.7\mu_B$. However, these experiments show the reduction only in the V layer and claim bulk behavior in the Fe film. Our thickness dependent magnetization data differ substantially from those deduced from SQUID or VSM data^{39,40} on V/Fe multilayers. For the case of $t_{\text{Fe}}=0.3$ nm the multilayer magnetization is only $0.66\mu_B$ per Fe atom in contrast to our finding of $1.28\mu_B$ per Fe atom. The measured Curie temperature of 200 K also strongly deviates from our data which imply a Curie temperature of about 400 K.

This substantial difference between Fe/V multilayers and a single Fe film sandwiched between V layers is most probably due to interface roughness in the multilayers because it is a general rule of epitaxy that the interfaces will never get smoother with increasing film thickness, even for the case of homoepitaxy for, e.g., vanadium²⁰ or iron.⁴¹ So, the

multilayer experiments just average over Fe/V interfaces with increasing roughness leading to a reduction of the magnetic moment in agreement with theoretical calculations^{9,42} showing that alloying reduces the magnetic moment drastically. Therefore, also the temperature dependence of the magnetic moment³⁹ is qualitatively different from the behavior shown in Fig. 5 for a single Fe layer.

Our results of a total reduction of $0.75\mu_B$ per Fe interface atom is in perfect agreement with theoretical calculations of Izquierdo *et al.*¹⁰ and Schwickert *et al.*,⁹ both predicting a reduction of $0.9\mu_B$. Niklasson *et al.*¹¹ find a slightly higher reduction of $1.1\mu_B$. All theoretical results show a reduced magnetic moment for the Fe interface layer and a negative polarization of the V interface layer.

The *in situ* PNR data on uncovered Fe films show a total reduction of 0.03 nm bulk Fe magnetic moment or $0.23\mu_B$ per Fe interface atom, assuming that the Fe/V interface and Fe surface contribute equally to the reduction. However, taking the deduced $0.75\mu_B$ reduction of the Fe/V interface from the *ex situ* data into account we can deduce an enhancement of $(0.3\pm 0.9)\mu_B$ for the free Fe surface. Unfortunately the errors are quite large because of the lack of more data points on the one hand and the problem of subtracting two quantities of similar magnitude on the other hand. This leads to the problem that the error gets larger than the calculated value. But despite this problem that the error bars are quite large we definitely can say that the samples with a free Fe surface have a larger magnetic moment than the V-covered ones. Furthermore, the inferred value of $0.3\mu_B$ per Fe surface atom is in perfect agreement with calculations⁴³ finding a total increase of $0.44\mu_B$.

The reduction of the magnetic moment in the Fe(100) films is much smaller than in Fe(110) films for which we found a reduction according to 0.4 nm bulk Fe.⁶ As already pointed out by Izquierdo *et al.*⁴² the most probable reason for that huge reduction is a surface alloy at the interface between the V(110) surface and the Fe layer. For these earlier experiments we used a V(110) single crystal which must be prepared by many sputtering and annealing cycles. So, it is conceivable that the surface of the single crystal is rougher than the surface of an epitaxial V layer on top of a smooth MgO crystal.

VII. CONCLUSION

We determined the magnetic moment of uncovered and V-covered Fe(100) films with PNR. The measurements on the V-covered Fe films reveal a reduction of $0.75\mu_B$ per Fe interface atom which is in good agreement with theoretical papers.^{9,10} We find a smaller reduction of the magnetic moment than was found in V/Fe(100) multilayers earlier^{39,40} which is probably due to a higher interface roughness present in multilayers compared to single films. The additional *in situ* PNR experiments show a much smaller total reduction. By taking the value of $0.75\mu_B$ reduction per Fe interface atom we can deduce an enhancement compared to the Fe bulk value of the magnetic moment at the free Fe surface by $(0.3\pm 0.9)\mu_B$ per Fe surface atom.

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