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Direct probe of interdiffusion effects on the induced V spin polarization at Fe/V interfaces

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X-ray magnetic circular dichroic measurements have been performed at room temperature at the V $L_{2,3}$ edges on Fe/V/Fe(110) trilayers to determine the induced V spin polarization. Contrary to similar studies on magnetic multilayers, the sample preparation and characterization were performed thoroughly *in situ* under ultrahigh vacuum conditions. This allowed us to modify the interfaces by tuning the deposition temperature and to probe directly the influence of interface quality on the induced V moments. The magnitude of the induced V moment increased by about a factor of 2 upon changing the deposition temperature from 300 K to 600 K. Comparison with *ab initio* calculations and with spectra of FeV alloys suggests that alloying at the interface results in the formation of large induced V moments.

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Induced magnetism in bulk materials has received some interest in 1960s and 1970s due to progress in magnetic-moment determination techniques based on nuclear physics, see, for example Refs. 1–3. Recently, the subject has been revitalized and transferred to the field of magnetic interfaces owing to developments in thin-film deposition techniques under ultrahigh vacuum (UHV) conditions and experimental methods with element specificity and monolayer (ML) sensitivity such as the x-ray magnetic circular dichroism (XMCD) and x-ray magnetic scattering. Indeed, element-specific studies during the past ten years have probed considerable induced magnetic moments at the interfaces,[4,5] have allowed for constructing magnetic-moment profiles,[6–9] and have revealed extraordinary effects such as the apparent breakdown of the third Hund's rule.[10] A fundamental question that still remains open is the effect of interdiffusion on the interface magnetic moments. This is, however, of great importance if one compares experimental with theoretical results. Frequently, quite different values of the interface magnetic moments have been measured with respect to *ab initio* theoretical predictions. Two characteristic examples are (i) the Ni moments at the Ni/Pt(111) interface, where experiment revealed by a factor of 2 reduced Ni moments as compared to theory,[9] and (ii) the V moments at the Fe/V(100) and the Fe/V(110) interface, where the experimentally deduced V moments are at least a factor of 2 larger than the ones theory predicts.[7,11] The observed discrepancies were considered to occur due to intermixing at the first 1-2 interfacial atomic layers.[12–16] However, to the best of our knowledge, no direct probe of the effects of atomic intermixing on the interface magnetic moments exists and the theoretical considerations remain still hypothetical.

In this work we demonstrate directly the influence of interdiffusion on the induced V spin polarization at Fe/V interfaces in terms of polarization range and magnitude of magnetic moments. For the determination of the V magnetic moments, we have analyzed the relation between the V XMCD and the magnetic ground-state properties in a previous work.[17] To probe directly the effects of interdiffusion, we have circumvented the conventional process for the determination of the induced magnetic moments in layered structures. Usually, one prepares magnetic multilayers or superlattices under UHV and caps them with a protective layer. Then, these samples are removed from the UHV chamber and measured at a synchrotron radiation or neutron source facility. The optimum deposition rate and/or temperature for the highest quality samples is selected by comparing measured with calculated x-ray-diffraction spectra. However, this method cannot accurately discriminate between interdiffusion and roughness, see, e.g., Ref. 18. Moreover, for practical reasons, one cannot vary the deposition parameters in order to modify *in statu nascendi* the interfaces and inspect the outcome of this variation on the magnetic moments. On the contrary, the great advantage of working thoroughly *in situ* is that one can control the growth and interface quality step by step and monitor the resultant magnetic moments. We succeed in a direct observation of the influence of interdiffusion on the magnitude of the induced V magnetic moments in the prototype system Fe/V/Fe(110).

Fe/V/Fe(110) trilayers were grown on a Cu(100) single crystal via evaporation from high-purity rods *in situ* under UHV conditions ($p_{base} \sim 2 \times 10^{-10}$ mbars). The Cu surface was prepared by several cycles of Ar^+ bombardment and annealing to 900 K. First, a thick Fe-buffer layer (thickness $d \sim 50$ ML) was deposited and then annealed softly to 450 K.[19] Low-energy electron-diffraction spots showed a bcc(110) structure. Scanning tunneling microscopy (STM) images for the (a) as deposited and (b) annealed Fe-buffer layer in Fig. 1 confirm that by soft annealing at 450 K the surface roughness reduces dramatically, see the corresponding line scans. On the annealed surface one may see atomically flat terraces with a broadness of about 15 nm. This recipe of first evaporating a layer, then annealing to decrease surface roughness, and then reevaporating the next layer has been introduced to produce the highest interface quality with minute amount of interface atomic exchange.[20] However, this type of growth is practically impossible to be employed in conventional multilayers, mainly due to time limitations in the fabrication process.

In order to study the influence of interface quality on the induced V moments we prepared two series of trilayers with variable thickness of the V spacer layers between 1 and 8

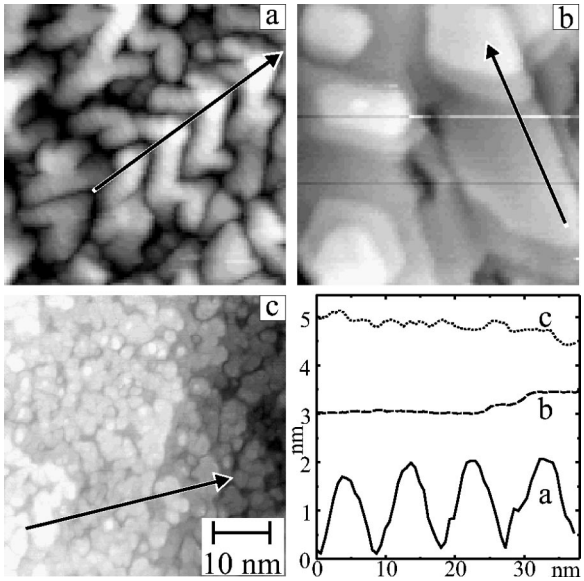

FIG. 1. STM images illustrate the step-by-step preparation of the trilayers grown at 300 K (a) 50 ML Fe(110) as grown on Cu(100) and (b) after annealing softly to 450 K. (c) Deposition of 3.0 ML V on the Fe(110) buffer. (d) Topographic view for (a), (b), and (c) via representative line scans. In (c) the steps are 1 ML high, as expected for layer-by-layer growth.

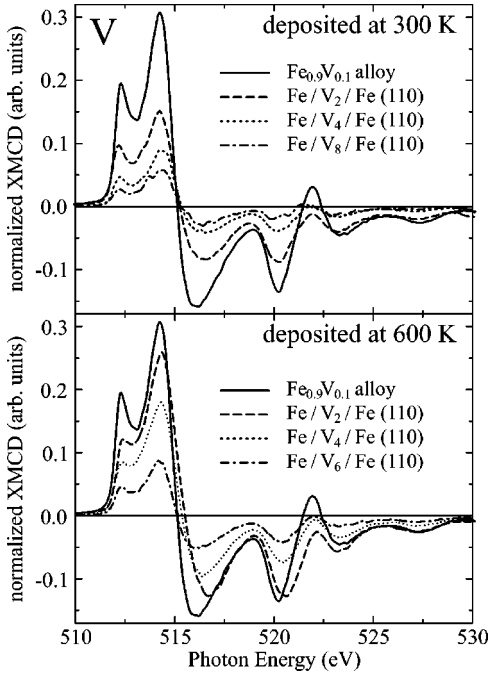

FIG. 2. Vanadium XMCD spectra in Fe/V/Fe(110) trilayers grown at (a) 300 K and (b) 600 K. The thickness of the V spacer layers is indicated by an index. A spectrum for V in a $Fe_{0.9}V_{0.1}$ alloy with an induced V moment $\sim 1\mu_B$/atom is included as a reference in both datasets.

ML. The deposition rates for V and Fe were 1 Å per minute. For the first series the V spacer and the top Fe ($d_{Fe}=5$ ML) films were successively deposited at room temperature (300 K) without further annealing. This reduces atomic exchange processes across the Fe/V interface. The STM image (c) indicates that the growth of V on top of the annealed Fe buffer enhances slightly the surface roughness. However, the arithmetic mean roughness for 3 ML V on Fe(110) is 0.09 nm and, thus, comparable to the one of 5 ML Ni/Cu(100) for which a layer-by-layer growth up to 5 ML is well established.[21] Since interfaces in our trilayers are atomically flat, any effect of roughness on the interface moments may be neglected. By eliminating the roughness, we stay with one free parameter concerning interface quality, i.e., interdiffusion. In our experiments we were able to *switch on* interdiffusion effects by raising the deposition temperature from 300 K to 600 K for the second series of trilayers. The thickness calibration was done via Auger electron spectroscopy and the Fe/V $L_{2,3}$ edge jumps of the x-ray absorption spectra (XAS) and cross-checked with the STM study.

All samples were investigated *in situ,* i.e., no capping layers were needed and, consequently, no oxygen contamination hindered the measurement of the V XAS in the full required energy range. The data were taken at the third generation synchrotron radiation facility of Berlin (BESSY II). The XAS spectra were measured at the $L_{2,3}$ edges of Fe and V using the total electron yield detection mode. The degree of circular polarization was 90(1)% and 85(1)% for the V and Fe $L_{2,3}$ energy range, respectively. The XMCD spectra were carried out by reversing the magnetization of the samples with an electric pulse driven coil and alternatively by inverting the helicity of the x rays. Hence, this cross-check can rule out artificial magnetic backgrounds in the magnetic dichroism. The easy axis of magnetization of the Fe/V trilayers is in plane along the [001] Cu direction. Therefore, the samples were measured under 20° grazing incidence of the light and, consequently, the Fe and V XAS spectra were corrected for self-absorption effects. The samples were probed at magnetic remanence. The Fe layers exhibited a magnetization equal to the bulk value[17] ensuring that the remanent magnetization can be safely translated into magnetic moment per atom units. Note that the spectra presented here (normalization of the edge jump to unity) correspond to parallel alignment of the photon wave vector and the magnetization, and 100% degree of circular polarization.

In Fig. 2(a) we plot the vanadium XMCD spectra at the $L_{2,3}$ edges for the series of samples grown at 300 K. The overall line shape of the XMCD spectrum and its relation to the V ground-state spin and orbital moments have been discussed in detail with the help of theory elsewhere.[17] To determine the V magnetic moments we have evaporated a $Fe_{0.9}V_{0.1}$ alloy for which the V moment is equal to $0.99\mu_B$/atom according to Ref. 17 and, independently, to measurements based on polarized neutrons.[22] Clearly, the XMCD signal, which is proportional to the total V magnetic moment, decreases with the V spacer thickness. Comparison of the V spectra for the alloy and the Fe/V_2/Fe trilayer reveals that the interface V moment should be of about $0.5\mu_B$/atom, see Fig. 2(a). In Fig. 2(b) we plot the corresponding V spectra for the series of samples grown at 600 K. One may notice the large increase of the V moment for the Fe/V_2/Fe trilayer. The magnitude of the induced V moment approaches the value of V in the alloy. Interestingly, *ab initio* calculations for the Fe/V(110) interfaces predict such a large

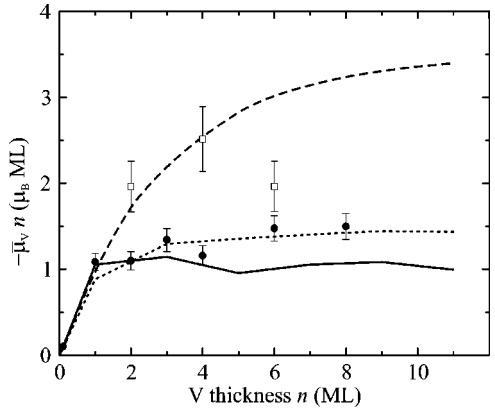

FIG. 3. Thickness dependence of the integrated V magnetic moments in Fe/V(110) trilayers grown at 300 K (closed circles) and deposited at 600 K (open squares). Contrary to theoretical calculation that expect a short-range spin polarization of the integrated V moments for perfect (solid line) as well as for diffused interfaces (dotted line), a long-range spin polarization (dashed line) of V in Fe/V(100) superlattices was experimentally deduced, see Refs. 11 and 23.

increase of the interfacial V moment as we move from the ideal (nonmixed) to the diffused Fe/V interface.[15,22]

In Fig. 3 thickness-dependent integrated V magnetic moments in Fe/V(110) trilayers deposited at 300 K (closed circles) and at 600 K (open squares) are plotted. This type of plot has the advantage of revealing whether the internal atomic layers of V are magnetized or not: if the integrated-moment curve does not saturate then also the internal layers are magnetized. The large difference in the magnitude of the V induced moment which is exhibited in Figs. 2(a) and 2(b) between the trilayers deposited at 300 K and 600 K is also reflected on the datasets of Fig. 3. The curve for the trilayers deposited at 300 K approaches very fast the saturation ($\sim$3 ML). This suggests that the inner layers contribute much less to the total V moment than the interface V layers, and the induced V moment is mainly located at the interface. This observation of a ''short-range'' V polarization is expected from *ab initio* calculations.[15,23] Moreover, the slight deviation of the monotonous increase of the integral might be due to small variation of the V moments of the interface and the inner layers with the total V thickness. Indeed, theoretical calculations found such variations in the layer resolved moment profiles of V with the thickness.[15,11] However, these variations are small with respect to the magnitude of the induced V moments. Hence, the theoretical curve for the ideally sharp interfaces (solid line) is practically constant with small oscillations as a function of the V thickness. The main behavior of the integral curve, i.e., the short-range polarization of V, does not change upon interdiffusion at the Fe/V interface in the theory. The dotted curve corresponds to calculation where the Fe and V interface layers are modeled as a single intermixed interface layer and termed as ''diffused'' interfaces.[23] Direct comparison with our datasets manifests that the current deposition on atomically flat Fe(110) terraces at 300 K leads to integrated V magnetic moment curves similar to the ones expected for almost ideal interfaces. However, the integrated-moment curve for trilayers grown at 600 K (open squares) shows a considerable increase. This result is consistent with an experimentally deduced dashed-line curve,[11] which does not saturate, and it corresponds to the so-called ''long-range'' order polarization of V in Fe/V superlattices on MgO(001). This term was used to characterize the polarization of the internal V for at least 3–4 atomic layers beyond the Fe/V interface. Here, we demonstrate on the basis of our experimental datasets that deposition at 600 K results in the formation of more than one interdiffused layers at the Fe/V interface. Furthermore, the deviation of the integrated moment for $n=6$ ML of the 600 K deposited trilayers with regard to the Fe/V superlattices reveal a reduction of the V spin polarization. The magnetic moments of Fe in the intermixed layers reduce strongly with increasing number of V neighbors[15,16,23] and, therefore, the surrounding V atoms acquire smaller induced moments. The considerable decrease of the polarization for $n=6$ ML suggests that the V thickness is sufficient enough to separate the Fe-buffer layer from the top Fe layers.

TABLE I. Averaged and layer-resolved V magnetic moments with regard to the interface I in μ_B/atom for the Fe/V(110) trilayers grown at 300 K and 600 K. The negative V moments indicate antiparallel alignment between Fe and V. The errors of the absolute values $\bar{\mu}_V$ are 10% and 15% for the 300 K and 600 K series, respectively.

n	$\bar{\mu}_V$	$\mu_V(I)=\bar{\mu}_V(2\ \mathrm{ML})$	$\mu_V(I-1)$
4 ML (300 K)	-0.29	-0.54	-0.04
4 ML (600 K)	-0.63	-0.98	-0.28

The V magnetic moments at the interface I and at the second inner V layer $(I-1)$ given in Table I are deduced under two approximations similar to the ones described in Ref. 9 for Ni/Pt(111) multilayers: both Fe/V interfaces in the trilayers exhibit the same structural and magnetic properties and the induced V moments depend only on the distance of the layer to the Fe/V interface. Both approximations are justified for perfect Fe/V interfaces with regard to theoretical calculations.[15,23] The values of the V moments for the series deposited at 300 K are similar to the theoretical ones of Refs. 15 and 23 deduced for the short-range V polarization. Since the approximations for the layer-resolved moments became invalid for the interdiffused samples, the long-range polarization of V is an apparent one. The large V moments for the interdiffused trilayers are close to the experimental ones shown for Fe/V superlattices grown on MgO(001).[7,11,16] Interestingly, the enhanced interface V moments and the long-range polarization of V were probed in both series of superlattices grown at lower temperatures of 370 K (Ref. 7) and higher temperatures of 570 K.[11] Moreover, the induced V magnetic moments as a function of the V-layer thickness for the (001) and the (110) growth orientations were found to be indistinguishable.[7] At this point, one has to recall that Fe/V superlattices deposited at temperatures slightly above room temperature suffer from roughness.[24] Therefore, we conclude that roughness also results in the formation of large interface V moments and long-range polarization profiles. In our

trilayers, though deposited at room temperature, we could avoid roughness as we have demonstrated in Fig. 1. This may be understood by the fact that the trilayers are ultrathin (2 nm or thinner) while superlattices are typically more than one order of magnitude thicker. Roughness is known to increase with the total film thickness mainly due to kinetics. So it is easier to have an ultrathin trilayer with flat interfaces than a superlattice grown at the same temperature. Deposition of superlattices at higher temperatures, on the other hand, leads to atomically flat layers and improves considerably x-ray-diffraction patterns.[24] However, it has been considered that the high-temperature deposition results in some intermixing and, subsequently, to the formation of large V moments at the Fe/V interfaces.[11,16] This has been directly probed for the first time, to the best of our knowledge, via the present results; the *in situ* control of interfaces by tuning the deposition temperature helped us in bridging experiment and theory. This demonstrates the effects of interdiffusion on the induced V spin polarization at the Fe/V interfaces.

In summary, we have performed both structural and magnetic investigations on Fe/V/Fe(110) trilayers by means of STM and XMCD. The deposition temperature was found to play a dramatic role in the modification of the induced V spin polarization. Deposition on atomically flat Fe(110) layers at lower temperatures (300 K) resulted in smaller interface V moments in agreement with theoretical calculations for ideally sharp Fe/V interfaces. However, upon depositing at 600 K, the induced V moment at the interface almost doubles as theory predicts for a mixed Fe/V interface. The formation of more than one interdiffused layer at the Fe/V interface results in an apparent long-range V polarization. Our results serve as a direct demonstration of the effects of interdiffusion on the induced magnetic moments at the interfaces of easily polarizable elements.

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[1] R.M. Bozorth, P.A. Wolff, D.D. Davis, V.B. Compton, and J.H. Wernick, Phys. Rev. **122**, 1157 (1961).

[2] G.A. Westenbarger and D.A. Shirley, Phys. Rev. **138**, A161 (1965).

[3] R.E. Parra and J.W. Cable, J. Appl. Phys. **50**, 7522 (1979).

[4] G. Schütz, S. Stähler, M. Knülle, P. Fischer, S. Parkin, and H. Ebert, J. Appl. Phys. **73**, 6430 (1993).

[5] W. Grange, M. Maret, J.-P. Kappler, J. Vogel, A. Fontaine, F. Petroff, G. Krill, A. Rogalev, J. Goulon, M. Finazzi, and N.B. Brookes, Phys. Rev. B **58**, 6298 (1998).

[6] J. Vogel, A. Fontaine, V. Cros, F. Petroff, J.-P. Kappler, G. Krill, A. Rogalev, and J. Goulon, Phys. Rev. B **55**, 3663 (1997).

[7] M.A. Tomaz, W.J. Antel, Jr., W.L. O' Brien, and G.R. Harp, J. Phys.: Condens. Matter **9**, L179 (1997).

[8] L. Séve, N. Jaouen, J.M. Tonnerre, D. Raoux, F. Bartolomé, M. Arend, W. Felsch, A. Rogalev, J. Goulon, C. Gautier, and J.F. Bérar, Phys. Rev. B **60**, 9662 (1999).

[9] F. Wilhelm, P. Poulopoulos, G. Ceballos, H. Wende, K. Baberschke, P. Srivastava, D. Benea, H. Ebert, M. Angelakeris, N.K. Flevaris, D. Niarchos, A. Rogalev, and N.B. Brookes, Phys. Rev. Lett. **85**, 413 (2000).

[10] F. Wilhelm, P. Poulopoulos, H. Wende, A. Scherz, K. Baberschke, M. Angelakeris, N.K. Flevaris, and A. Rogalev, Phys. Rev. Lett. **87**, 207202 (2001).

[11] M.M. Schwickert, R. Coehoorn, M.A. Tomaz, E. Mayo, D. Lederman, W.L. O' Brien, T. Lin, and G.R. Harp, Phys. Rev. B **57**, 13 681 (1998).

[12] P. Poulopoulos, F. Wilhelm, H. Wende, K. Baberschke, D. Benea, H. Ebert, M. Angelakeris, N.K. Flevaris, A. Rogalev, and N.B. Brookes, J. Appl. Phys. **89**, 3874 (2001).

[13] S. Lounis, M. Benakki, S. Bouarab, and C. Demangeat, Surf. Sci. **518**, 57 (2002).

[14] S. Frota-Pessôa, A.B. Klautau, and S.B. Legoas, Phys. Rev. B **66**, 132416 (2002).

[15] J. Izquierdo, A. Vega, O. Elmouhssine, H. Dreyssé, and C. Demangeat, Phys. Rev. B **59**, 14 510 (1999).

[16] A. Scherz, H. Wende, P. Poulopoulos, J. Lindner, K. Baberschke, P. Blomquist, R. Wäppling, F. Wilhelm, and N.B. Brookes, Phys. Rev. B **64**, 180407 (2001).

[17] A. Scherz, H. Wende, K. Baberschke, J. Minár, D. Benea, and H. Ebert, Phys. Rev. B **66**, 184401 (2002).

[18] Eric E. Fullerton, Ivan K. Schuller, H. Vanderstraeten, and Y. Bruynseraede, Phys. Rev. B **45**, 9292 (1992).

[19] A. Scherz, P. Poulopoulos, H. Wende, G. Ceballos, K. Baberschke, and F. Wilhelm, J. Appl. Phys. **91**, 8760 (2002).

[20] X.W. Jhou and H.N.G. Wadley, J. Appl. Phys. **84**, 2301 (1998).

[21] J. Shen, J. Giergiel, and J. Kirschner, Phys. Rev. B **52**, 8454 (1995).

[22] I. Mirebeau, G. Parette, and J.W. Cable, J. Phys. F: Met. Phys. **17**, 191 (1987).

[23] R. Coehoorn, J. Magn. Magn. Mater. **151**, 341 (1995).

[24] P. Isberg, B. Hjörvarsson, R. Wäppling, E.B. Svedberg, and L. Hultman, Vacuum **48**, 483 (1997).