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1998 J. Phys.: Condens. Matter 10 5791

(http://iopscience.iop.org/0953-8984/10/26/007)

J. Phys.: Condens. Matter **10** (1998) 5791–5797. Printed in the UK PII: S0953-8984(98)91374-1

Magnetic and Mössbauer studies of Fe/V multilayers

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Received 5 February 1998, in final form 8 May 1998

Abstract. The structural and magnetic properties of rf-sputtered Fe/V multilayers with the elemental Fe and V layer thickness $t_{Fe} = t_V$ and with the structural modulation period Λ ranging from 2 nm to 24 nm have been studied by high-angle x-ray diffraction, vibrating-sample magnetometry, and conversion-electron Mössbauer spectrometry methods at room temperature. The results show that the Fe/V interfaces are paramagnetic. The magnetic behaviour of the multilayers, hence, originates from the α-Fe at the centres of the individual subsystems and the iron-rich crystalline Fe(V) alloy lying near the interface. The spin orientation in the Fe layers is strongly aligned in the film plane. However, evidence for a weak perpendicular spin orientation associated with the magnetic topmost Fe layer is found.

1. Introduction

Artificially layered ferromagnetic/non-magnetic materials exhibit several unusual phenomena, such as oscillating interlayer coupling, giant magnetoresistance, and perpendicular magnetic anisotropy [1]. These multilayers have, as a result, been receiving considerable attention. An important key to understanding such effects is the knowledge of the magnetic behaviour at the interfaces, on the basis of which questions concerning the magnetic moments, the type of the magnetic exchange coupling, and the spin orientation may be answered. Of particular interest for applications, as well as from the physical point of view, is the modification of the magnetic moment which takes place when a magnetic T (=Fe, Co, Ni) surface (or, more generally, interface) is covered by a few layers of a non-magnetic transition element X. It has been realized that the magnetic moment of these added layers is induced, and is antiparallel to that of the T subsystem for the light transition-metal elements (LT = Ti, V, Y, Zr, Lu, ...) and is parallel to it for the heavy elements (HT = Cu, Ag, Pd, Pt, ...). This effect is a consequence of a systematic change in hybridization of the d states on going from the light to the heavy transition elements [2, 3]. The spin orientation in the interfaces has been described in the same manner [4]. It is well known, for the T/HT multilayers, that the magnetic anisotropy of the interface between individual magnetic and non-magnetic layers is perpendicular. On the other hand, the interface of the T/LT multilayers is predicted to show a parallel anisotropy. Experimentally, however, a random spin

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0953-8984/98/265791+07$19.50

orientation was observed at room temperature in Fe/LT multilayers (with e.g., LT = Ti [5, 6], Zr [7]).

Fe/V multilayers are good systems on which to base a study of the non-magnetic-layer-induced magnetization. Oscillatory interlayer exchange coupling as a function of V layer thickness was reported for sputtered Fe/V multilayers [8]. Antiparallel alignment of the Fe and V magnetic moments has been indicated by a theoretical study [9]. This was confirmed by x-ray magnetic circular dichroism experiments [10].

The aim of this paper is to study the structural and magnetic properties of sputtered Fe/V multilayers with the elemental Fe and V layer thickness $t_{Fe} = t_V$ and with the structural modulation period ranging from 2 nm to 24 nm by means of x-ray diffraction, conversion-electron Mössbauer spectrometry (CEMS), and magnetization measurements. The attention is focused on the magnetism and magnetic anisotropy of the interfaces.

2. Experiments

Fe/V multilayers, with the Fe and V thicknesses $t_{Fe} = t_V = 1$ nm, 2 nm, 3 nm, 5 nm, 7 nm, 9 nm, 12 nm and with the structural modulation period Λ ranging from 2 nm to 24 nm, were deposited on a glass substrate at 300 K by using a triode rf-sputtering system. The samples were deposited with the number of periods n varied from 6 to 120. The thicknesses of the elemental layers were controlled during deposition by monitoring the reflectivity of the low-angle x-rays. To avoid corrosion and oxidation, the Fe/V stack was covered with a V top layer 10 nm thick.

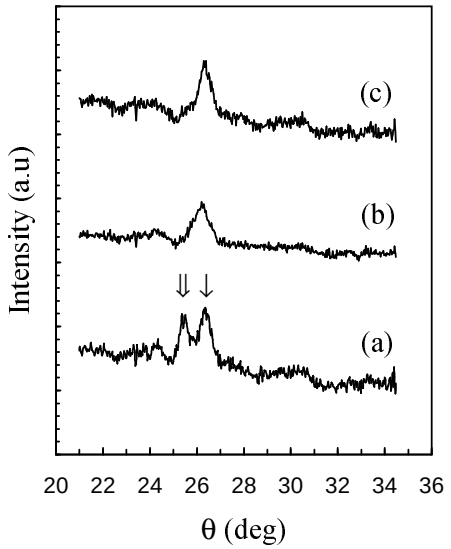

Figure 1. High-angle x-ray diffraction patterns for the Fe/V multilayers. (a) $\Lambda = 2$ nm, (b) $\Lambda = 10$ nm, (c) $\Lambda = 24$ nm; ⇓: vanadium-rich disordered V(Fe) alloy; ↓: iron-rich disordered Fe(V) alloy.

The global structure of the samples was investigated by high-angle x-ray diffraction using a cobalt anticathode ($\lambda_{Co\,K\alpha} = 0.1790$ nm). Examples of the measurements are displayed in the figure 1. The results show that for the film with the smallest Fe thickness ($t_{Fe} = 1$ nm), the sample consists of a disordered structure of iron-rich Fe(V) layers and a

disordered or amorphous vanadium-rich V(Fe) phase. As the Fe layer thickness increases, pure crystalline α-Fe layers start to appear at the centres of the individual subsystems. Within the experimental errors, no characteristic peak of V is evident for the samples under consideration.

Table 1. Magnetic and Mössbauer data for the Fe/V multilayers: the structural modulation period (Λ), the saturation magnetization referred to the thickness t_{Fe} ($\mu_0 M_S$), the saturation magnetization referred to the thickness t_m ($\mu_0 M_{\mathrm{Fe}}$), the mean hyperfine field $\langle B_{hf}\rangle$ and the mean Mössbauer angle $\langle\beta\rangle$ (see the text), and the relative fraction of Fe atoms in the α-phase compared to all of the Fe atoms ($A_{\alpha-\mathrm{Fe}}$).

Λ (nm)	$\mu_0 M_S$ (T)	$\mu_0 M_{\mathrm{Fe}}$ (T)	$\langle B_{hf}\rangle$ (T)	$\langle\beta\rangle$ (deg)	$A_{\alpha-\mathrm{Fe}}$ (%)
2	1.38	1.97	25.1 ± 0.3	32 ± 5	47
4	1.63	1.92	25.6 ± 0.3	70 ± 5	55
6	1.89	2.1	26.4 ± 0.3	73 ± 5	59
10	1.93	2.05	28.9 ± 0.3	70 ± 5	75
14	1.96	2.05	27.7 ± 0.3	73 ± 5	73
18	1.96	2.03	29.7 ± 0.3	69 ± 5	80
24	1.96	2.01	29.0 ± 0.3	69 ± 5	80

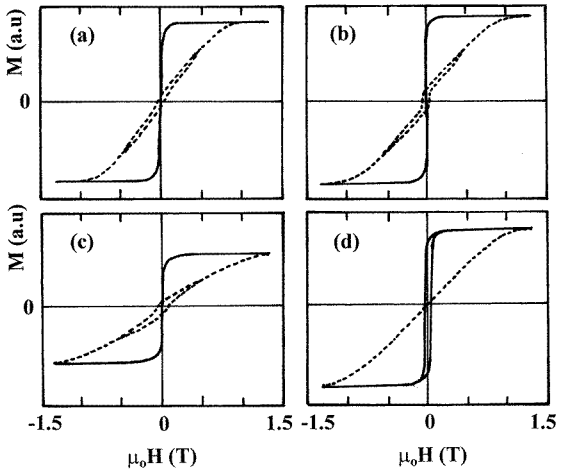

Figure 2. Magnetic hysteresis loops obtained with the applied magnetic field in the film-plane (solid curve) and in the film-normal (dashed curve) directions for the Fe/V multilayers. (a) $\Lambda = 2$ nm, (b) $\Lambda = 4$ nm, (c) $\Lambda = 6$ nm, and (d) $\Lambda = 10$ nm. For the values of the saturation magnetization, see table 1.

The magnetization was measured with a vibrating-sample magnetometer (VSM) in magnetic fields up to 1.4 T applied in the film-plane and film-normal directions.

The CEM spectra at room temperature were recorded using a conventional spectrometer equipped with a home-made helium–methane proportional counter. The source was a ^{57}Co in rhodium matrix. The films were set perpendicular to the incident γ-beam. The spectra were fitted with a least-squares technique using a histogram method relative to discrete distributions, with the linewidths of each of the elementary spectra constrained to be the same. The isomer shifts are given relative to that of α-Fe at 300 K. The average 'cone angle' $\langle\beta\rangle$ between the incident γ-ray direction (which is the film-normal direction) and

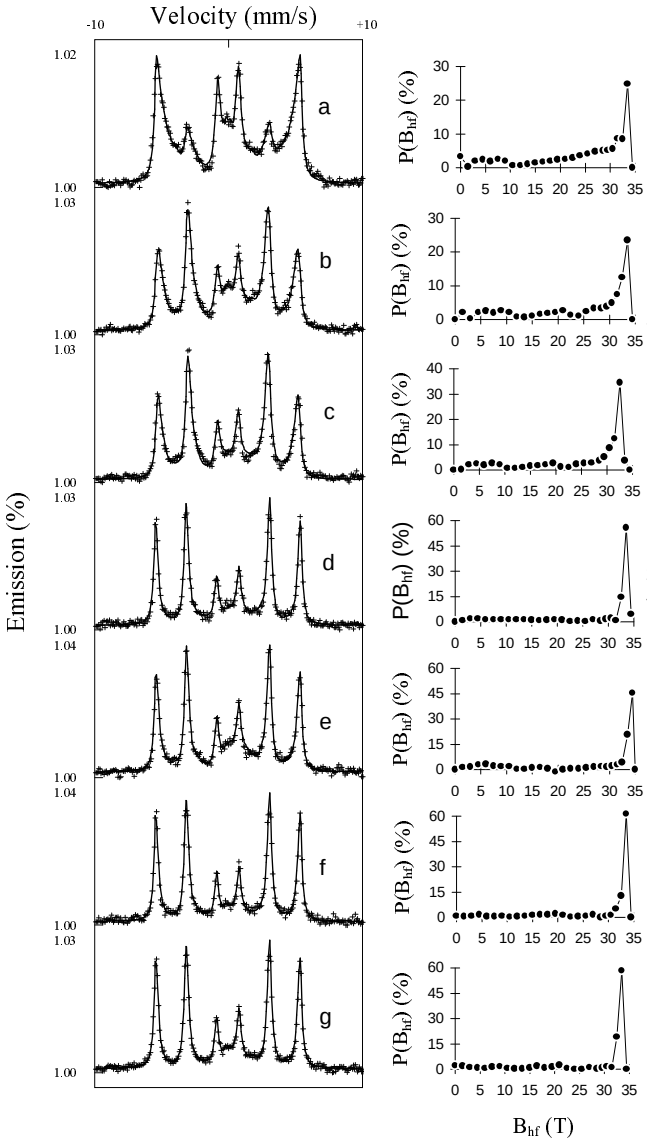

Figure 3. CEM spectra and their corresponding hyperfine-field distributions obtained at 300 K for the Fe/V multilayers. (a) $\Lambda = 2$ nm, (b) $\Lambda = 4$ nm, (c) $\Lambda = 6$ nm, (d) $\Lambda = 10$ nm, (e) $\Lambda = 14$ nm, (f) $\Lambda = 18$ nm, and (g) $\Lambda = 24$ nm.

that of the hyperfine field B_{hf} (or the Fe magnetic moment direction) is estimated from the line-intensity ratios 3:x:1:1:x:3 of the six Mössbauer lines, where x is related to $\langle\beta\rangle$ by $\sin^2\langle\beta\rangle = 2x/(4+x)$.

3. Experimental results and discussion

Figure 2 presents the magnetic hysteresis loops obtained with the applied magnetic field in the film-plane and film-normal directions for several Fe/V multilayers at 300 K. For all

of the samples, the in-plane direction is always easy to magnetize. The anisotropy field $\mu_0 H_A$ slightly increases with increasing t_{Fe}. In addition, one observes also the existence of a weak-magnetization component, which is also easy to magnetize in the perpendicular direction (see, e.g., figures 2(b) and 2(c)). This finding may reflect a weak random spin orientation at the interfaces. The saturation magnetization $\mu_0 M_S$ referred to the Fe thickness (t_{Fe}) increases with increasing Fe thickness and tends to almost saturate for the value of 1.96 T (see table 1). These data will be analysed later.

In figure 3, the CEM spectra and their corresponding hyperfine-field distributions $P(B_{hf})$ are shown for the Fe/V multilayers. Each spectrum is fitted with three components, i.e. a major ferromagnetic, a minor ferromagnetic, and an additional non-magnetic component. The major component is a sharp sextet corresponding to α-Fe, which is localized at the centre of the Fe layers. In the distributions of the hyperfine fields $P(B_{hf})$ shown in figure 3, this is manifested by the presence of a peak located at 33.4 T. The relative fraction of Fe atoms in the α-Fe phase compared to all of the Fe atoms was obtained by adjusting the area of this sharp sextet to the total spectrum area. The values are listed in table 1. The minor ferromagnetic component with smaller hyperfine fields is due to the Fe-rich alloy perturbed near the interfaces. The contributions of $P(B_{hf})$ just below the value of $B_{hf}(\alpha\text{-Fe})$ can be related to this contribution. A low-field component attributed to an interface disordered phase is observed in addition to the interface ferromagnetic component. The mean hyperfine field $\langle B_{hf}\rangle$, which is the average of the values of the $B_{hf}(\alpha\text{-Fe})$ and lower-field contributions, is reported in table 1. Coherence of the magnetization and mean-hyperfine-field data can be observed in figure 4, where the normalized values $\langle B_{hf}\rangle / B_{hf}(\alpha\text{-Fe})$ and $M_S/M_S(\alpha\text{-Fe})$ for the Fe/V multilayer are plotted as functions of the Fe thickness.

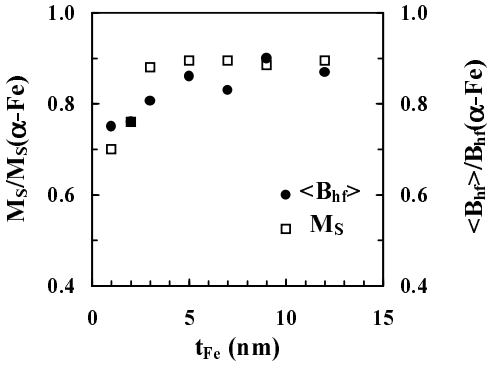

Figure 4. The normalized $\langle B_{hf}\rangle / B_{hf}(\alpha\text{-Fe})$ and $M_S/M_S(\alpha\text{-Fe})$ values as functions of the Fe thickness for the Fe/V multilayer.

The data presented in figure 5 are average values, determined over the whole Fe thickness. In agreement with the results of the structural and Mössbauer studies, one can describe the multilayers by considering that (i) the inner V subsystem is non-magnetic and (ii) each individual Fe subsystem of thickness t_{Fe} is divided into a non-magnetic layer of thickness t_{nm} at each V interface and a magnetic layer of thickness t_m (in more detail, one can even separate t_m into two parts corresponding to an α-Fe core and an Fe top layer). In this context, the magnetization of the samples is related the Fe magnetization $\mu_0 M_{\mathrm{Fe}}$ as follows:

$$\mu_0 M_S \Lambda = \mu_0 M_{\mathrm{Fe}} t_m = \mu_0 M_{\mathrm{Fe}} (t_{\mathrm{Fe}} - 2t_{nm}). \tag{1}$$

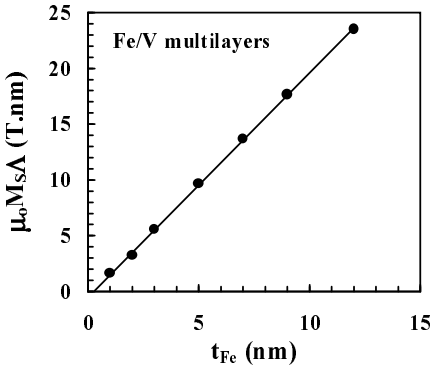

Figure 5. The variation of M_S as a function of t_{Fe} for the Fe/V multilayers.

A plot of $\mu_0 M_S$ versus t_{Fe} is presented in figure 5, from which we obtain $t_{nm} = 0.15$ nm. The magnetization referred to the true magnetic Fe thickness (t_m) is now given as $\mu_0 M_{\mathrm{Fe}} = \mu_0 M_S(t_{\mathrm{Fe}}/(t_m))$. Its values are listed in table 1. For all of the samples, $\mu_0 M_{\mathrm{Fe}}$ is almost constant (2 T) and is below the value for bulk iron by about 7%. A V magnetic moment, which is induced by the hybridization between the 3d(Fe) and 3d(V) states near the interfaces, is usually expected to be detected in the samples with small thickness. At present, however, it is hardly in evidence.

The direction of the Fe magnetic moment in the Fe/V multilayers can be deduced from the fitted value of the average 'cone angle' $\langle\beta\rangle$. At room temperature, a value of 32° was found for the Fe(1 nm)/V(1 nm) sample, which strongly supports the results from the magnetization investigations of the perpendicular magnetic anisotropy. For the other multilayers, $\langle\beta\rangle$ is about 70° (table 1). This is governed by the iron moments, which strongly align in the film plane.

The effective anisotropy constant K_{eff} was evaluated from $K_{eff} = K_A - \mu_0(M_S^2)(t_{\mathrm{Fe}})/2$, where the anisotropy energy $K_A = (\mu_0)^2 H_A M_S/2$. The phenomenological relationship between the interface (K_S), the volume (K_V), and the effective (K_{eff}) anisotropies is usually given as

$$t_{\mathrm{Fe}} K_{eff} = 2K_S + t_{\mathrm{Fe}} K_V. \tag{2}$$

From the plot of $t_{\mathrm{Fe}} K_{eff}$ versus t_{Fe} (figure 6), it is apparent that the interfacial perpendicular magnetic anisotropy constant $K_S = 0.05$ mJ m^{-2} and the volume contribution $K_V = -0.16$ MJ m^{-3}. The results obtained are smaller than those deduced for the Fe/Ti multilayers [6]. However, they can explain the existence of the weak-magnetization component, which is oriented in the film-normal direction as observed in several hysteresis loops.

The weak perpendicular magnetic anisotropy mentioned above is usually connected to the interface. In the present case, the interface is paramagnetic, so the anisotropy may relate to the topmost Fe layer. As already discussed above, the magnetic moment of these Fe atoms is not modified by the hybridization. The only change is that the reduced symmetry at the topmost layer results in anisotropies differing greatly from those of the bulk. As proposed by Bruno [11] and Wang *et al* [12], the anisotropy can be understood by considering the modification of the band structure of the out-of-plane states (OPS) that occurs because they cannot form bonds directed towards the surface. The positive sign of the anisotropy, then, was derived for the Fe surface because in the vicinity of the Fermi

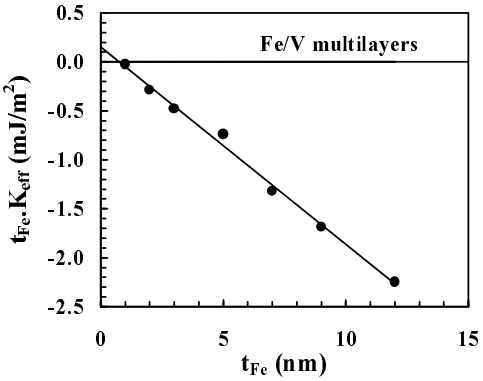

Figure 6. A plot of $t_{\mathrm{Fe}}K_{eff}$ versus t_{Fe} for the Fe/V multilayers.

level the OPS symmetry states are dominant (in comparison to those of the in-plane states (IPS)). These arguments were able to provide a basis for interpreting the perpendicular anisotropy observed in the Fe/Ti multilayers [6]. This simple model leads us to suggest that the observed weak perpendicular anisotropy component in the Fe/V multilayers is not related to the Fe–V alloy in the interface, but is created essentially from the topmost Fe layers.

Acknowledgment

The stay of NHD at the University of Rouen was supported by the Ministére Français de l'Education Nationale, de la Recherche et la Technologie.

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