

UAs/Co multilayers studied by x-ray magnetic circular dichroism at the U $M_{4,5}$ edgesN. Kernavanois,^{1,2,*} D. Mannix,^{3,4} P. Dalmas de Réotier,² J.-P. Sanchez,² A. Yaouanc,² A. Rogalev,³ G. H. Lander,⁵ and W. G. Stirling³¹*Institut Laue-Langevin, BP 156, 38042 Grenoble Cedex 9, France*²*CEA/Grenoble, Département de Recherche Fondamentale sur la Matière Condensée, SPSMS, 17 rue des Martyrs, 38054 Grenoble Cedex 9, France*³*European Synchrotron Radiation Facility, BP 220, 38043 Grenoble, France*⁴*Department of Physics, The University of Liverpool, Liverpool L69 3BX, United Kingdom*⁵*European Commission, Joint Research Centre, Institute for Transuranium Elements, Postfach 2340, D-76125 Karlsruhe, Germany*
(Received 25 August 2003; published 11 February 2004)

X-ray magnetic circular dichroism (XMCD) measurements have been performed on UAs/Co multilayers at the $M_{4,5}$ edges of uranium. The linear relation between the polar Kerr rotation previously measured and the $5f$ magnetic moment deduced from XMCD has been demonstrated. Temperature and field dependences have been studied. The ferromagnetic character of the UAs layers is clearly shown and room-temperature measurements indicate the presence of an induced uranium moment through exchange coupling with the Co magnetic moments.

DOI: 10.1103/PhysRevB.69.054405

PACS number(s): 75.70.-i, 87.64.Ni, 31.70.-f

I. INTRODUCTION

Much of the recent scientific interest in magnetic multilayers has been driven by their potential use in practical device applications such as magneto-optical storage media. Following the discovery of large magneto-optical Kerr effects (MOKE) in U compounds by Reim and Schoenes,¹ researchers at IBM attempted to make a room temperature ferromagnet film using uranium.^{2,3} They started to work on amorphous USb and UAs which they found to be ferromagnetic with a Curie temperature $T_C \leq 140$ K.⁴ When other elements, such as Fe, Mn, Cu were mixed into the film, T_C did not increase.^{5,6} They then turned to the production of sputtered multilayer films of Co and UAs layers with the aim of inducing polarization in the U through exchange coupling to the Co layers.^{2,3} Among the limited initial studies, magneto-optical experiments showed that there was a moment on the uranium site at low temperature in all samples and even at room temperature in the $12 \times [\text{UAs}(80 \text{ \AA})/\text{Co}(20 \text{ \AA})]$ multilayer.³ Subsequent efforts by some of us using polarized neutron reflectivity (PNR) have not confirmed these results unambiguously.⁷ Although analysis of the PNR data indicates that there is indeed magnetism in the amorphous UAs layers, the main part of the magnetic PNR signal arises from the Co moment, both within the multilayer and in the buffer material. This has a value close to that of elemental Co and considerably larger than any moment at the U site.

This paper deals with x-ray magnetic circular dichroism (XMCD) measurements performed at the M_4 (3726 eV) and M_5 (3551 eV) edges of uranium. The XMCD technique allows one to probe the $5f$ magnetism separately thus overcoming the difficulty encountered in the PNR measurements. XMCD measurements were made on three of the UAs/Co samples from IBM, namely, $12 \times [\text{UAs}(80 \text{ \AA})/\text{Co}(20 \text{ \AA})]$, $15 \times [\text{UAs}(60 \text{ \AA})/\text{Co}(20 \text{ \AA})]$, and $20 \times [\text{UAs}(40 \text{ \AA})/\text{Co}(20 \text{ \AA})]$.

These bilayered films were all grown on glass substrates

and on a 200 Å buffer layer of cobalt. They are amorphous with a total thicknesses of ~ 1400 Å and the UAs layers were sputtered from their elements to give a [U]/[As] atomic ratio of 1.5.^{2,3} The structures were deposited under computer control. The Co layers were prepared by holding the glass substrates over the source and opening the shutter for a precalibrated time; the U-As alloy layers were prepared by sequentially depositing subatomic layers of 1 and 1.5 Å of U and As, respectively. To avoid possible degradation due to moisture, the samples were covered by a top protective cobalt layer and stored in a dry atmosphere in between the different measurements. Our experience with new multilayers, with Fe as the top layer, has shown that the change is slow (over many years) even when the multilayers are stored in air (and Fe is much reactive than Co).

In Sec. II of this paper, investigations by x-ray reflectivity are presented to check the quality and periodicity of the samples. We introduce the XMCD technique and the experimental procedure in Sec. III. The low-temperature XMCD results for the three multilayers will be discussed in Sec. IV; here, we compare the $5f$ moment obtained by XMCD with the previous polar Kerr rotation measurements. Section V is dedicated to the study of the temperature and field dependences of the $5f$ magnetization in the $12 \times [\text{UAs}(80 \text{ \AA})/\text{Co}(20 \text{ \AA})]$ sample. A small induced $5f$ moment at room temperature in this multilayer is found to exist. Finally, conclusions are drawn in Sec. VI.

II. SAMPLE CHARACTERIZATION

The structures of the multilayers were investigated by x-ray reflectivity using the rotating anode x-ray diffractometer at Keele University (U.K.). The x-ray reflectivity spectra of the $12 \times [\text{UAs}(80 \text{ \AA})/\text{Co}(20 \text{ \AA})]$ sample together with the best fit using a simple bilayer model is presented in Fig. 1(a). This model is clearly inadequate to account for the experimental scattering profile; a more sophisticated simula-

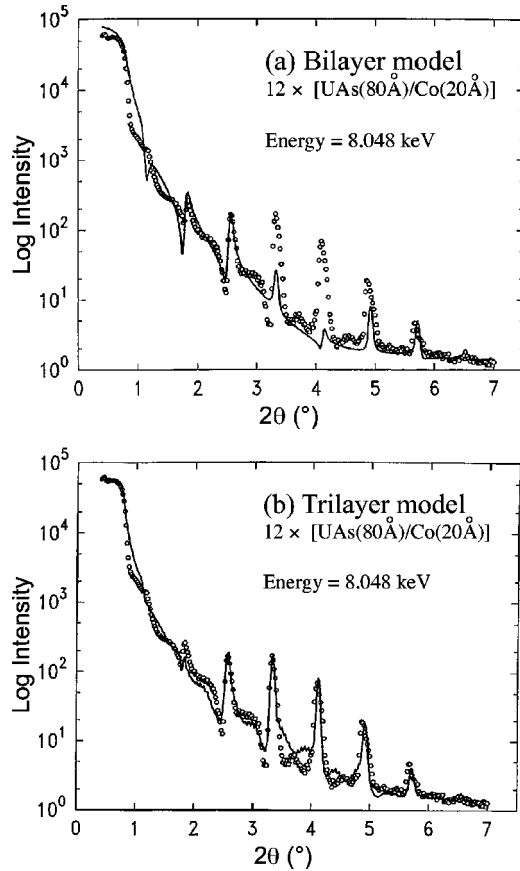


FIG. 1. Fits to x-ray reflectivity data for the $12 \times [\text{UAs}(80 \text{ \AA})/\text{Co}(20 \text{ \AA})]$ film using a bilayer model (top panel) and a trilayer model (bottom panel).

tion is required due to the relatively poor quality of the sample. Indeed, further evidence that the sample is not perfect can be found in the fact that the Kiessig fringes are not resolved in the x-ray scattering. Such effects are often associated with layers that are not flat. A trilayer model for the multilayer structure was then considered, introducing an additional “alloy” region of UAs-Co with 50% mixing of the electron densities. This model gives a much better description of the scattering and the fit is presented in Fig. 1(b) for the 12 bilayer sample. The thickness of the alloy region was determined to be around 10 \AA in agreement with the dead-layer thickness suggested by the previous bulk magnetization measurements.³

The x-ray reflectivity for the $15 \times [\text{UAs}(60 \text{ \AA})/\text{Co}(20 \text{ \AA})]$ and the $20 \times [\text{UAs}(40 \text{ \AA})/\text{Co}(20 \text{ \AA})]$ samples was also measured, resulting in a similar thickness of the UAs-Co alloy region. In all cases, the x-ray reflectivity data indicate that these samples present relatively large interdiffusion and possibly topologically rough interfaces that prevent the Kiessig fringes being resolved. For this reason a detailed reflectivity analysis is not worthwhile. Nevertheless, the presence of (relatively) sharp peaks in the reflectivity spectra of all three samples is evidence of the periodicity of the multilayers, despite the existence of interdiffusion regions.

III. XMCD TECHNIQUE AND EXPERIMENTAL PROCEDURE

A. Background

X-ray magnetic circular dichroism is an element-sensitive technique which allows the evaluation of the expectation values of the z projections of both the orbital moment $\langle L_z \rangle$ and the spin angular momentum $\langle S_z \rangle$ with the help of magneto-optical sum rules.^{8,9} This magnetometry tool uses the x-ray absorption orbital selectivity of a given element and the sensitivity provided by a circularly polarized x-ray beam to the magnetic state of matter. The experiment consists of measuring the absorption of a magnetic sample submitted successively to a right and a left polarized x-ray beam at the threshold excitation energy of a chosen core hole in the unfilled part of the valence band. The difference between the two absorption spectra is the dichroism signal. In our case of interest, the $5f$ shell of uranium can be investigated by performing XMCD measurements at the $M_{4,5}$ edges corresponding to $3d_{3/2,5/2} \rightarrow 5f$ transitions. Sum rules allow the quantities $\langle L_z \rangle/n_h$ and $\langle L_z \rangle/\langle S_z^e \rangle$ with $\langle S_z^e \rangle \equiv \langle S_z \rangle + 3\langle T_z \rangle$ to be extracted. $\langle T_z \rangle$ is the expectation value of the z projection of the magnetic dipole operator of the $5f$ shell and n_h is the number of holes in this shell. Further details about XMCD at the U- $M_{4,5}$ edges technique can be found in Refs. 10–12.

B. Experiment

The XMCD measurements were performed at the ID12A beamline of the European Synchrotron Radiation Facility (ESRF, Grenoble, France), which is dedicated to polarization dependent x-ray absorption studies.^{13,14} The x-ray source is a helical undulator (Helios-II) which provides a beam characterized by high flux, high circular polarization (about 97%) and tunable helicity. A double Si(111) crystal monochromator was used. At the energy of the M_4 and M_5 edges of uranium, the monochromator Bragg angle is large and the degree of circular polarization of the monochromatic beam is then reduced to 35% (M_5) and 45% (M_4).¹⁵

In order to polarize the sample, a 4 T magnetic field produced by a superconducting cryomagnet was applied. The XMCD signal was obtained by flipping the helicity of the incident photon beam; this is equivalent to reversing the magnetic field direction (parallel or antiparallel to the incoming photon beam). The normal to the multilayer surface made a 30° angle with the field direction. Due to the high absorption of uranium the different spectra were measured in the fluorescence yield mode. This mode is the easiest method to employ in the presence of a magnetic field; nevertheless self-absorption corrections to the measured fluorescence spectrum are required to obtain the absorption coefficient.

The relation between the absorption coefficient and the normalized fluorescence intensity $I^{X,0}(E) = I^X(E)/I_0(E)$ where $I^X(E)$ is the measured fluorescence intensity and $I_0(E)$ is proportional to the incident flux, can be written as^{16,17}

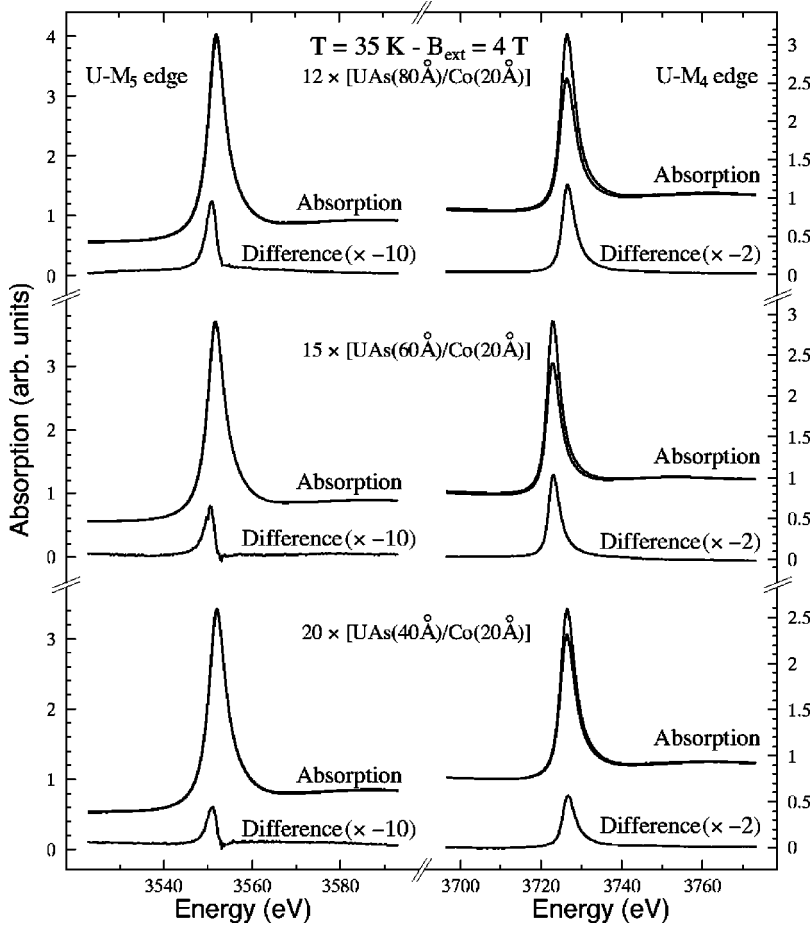


FIG. 2. Absorption and dichroism spectra at the $M_{4,5}$ edges of uranium for the 12, 15, and 20 bilayered films at 35 K and 4 T. These spectra have been deduced from the fluorescence spectra which have been corrected for self-absorption and for the energy dependence of the circular polarization rate of the monochromatic beam.

$$I^{X,0}(E) = B \frac{\mu_X(E)}{\mu_{tot}(E) + A} f(E) \quad (1)$$

with

$$f(E) = 1 - \exp\left[-\left(\frac{\mu_{tot}(E)}{\sin \alpha} + \frac{\mu_{tot}(E_f)}{\sin \beta}\right)d\right], \quad (2)$$

where d is the thickness of the sample under study and $\mu_X(E)$ is the absorption coefficient associated to the production of a core hole in the level X of interest. $\mu_{tot}(E) = \mu_X(E) + \mu_{other}(E)$ is the total absorption coefficient with $\mu_{other}(E)$ ascribed to absorption due to levels other than X and to the other atomic species. We have defined $A = (\sin \alpha / \sin \beta) \mu_{tot}(E_f)$. α is the angle between the sample surface and the incident photon beam of energy E . β is the angle between the outgoing fluorescence photon and the sample surface, and E_f is the energy of the photon resulting from the hole decay. B depends only on the experimental setup and can be determined in order to obtain typical characteristics of the “jumps” at the actinide absorption edges.¹⁸

Two simple assumptions can generally be made, from Eqs. (1) and (2), (i) For thin samples (low absorption), $(\mu_{tot}(E)/\sin \alpha + \mu_{tot}(E_f)/\sin \beta) d \ll 1$ leading to $\mu_X(E) \propto I^{X,0}(E)$. (ii) For thick samples (high absorption), $(\mu_{tot}(E)/\sin \alpha + \mu_{tot}(E_f)/\sin \beta) d \gg 1$ and then $\mu_X(E) = I^{X,0}(E)[\mu_{other}(E) + A]/[B - I^{X,0}(E)]$.

In the case of interest here, the multilayers containing uranium are characterized by $\{[\mu_{tot}(E)/\sin \alpha] + [\mu_{tot}(E_f)/\sin \beta]\} d \geq 0.3$ so neither of the two assumptions above is valid. Therefore, Eq. (1) has been solved numerically by iterative methods starting from the absorption coefficient calculated for a thick sample. This correction does not lead to significant variations of the $\langle L_z \rangle$ and $\langle S_z \rangle$ values derived from the data. Nevertheless, the presence of uranium in the multilayers under study legitimates its use. All the following results have been corrected for self-absorption effects in this way.

IV. LOW-TEMPERATURE XMCD RESULTS FOR DIFFERENT UAS THICKNESSES

This section is dedicated to the low-temperature XMCD results obtained for the 12, 15, and 20 bilayered films. Absorption and dichroism spectra corrected for self-absorption effects are presented in Fig. 2.

As for several previously measured uranium compounds^{10,11,19,20} the M_4 edge XMCD signal reproduces the white line shape while at the M_5 edge the signal vanishes for energies larger than the absorption maximum. Furthermore, the dichroism spectra at the M_4 and the M_5 edges do not decrease with UAs layer thicknesses in the same way. The decrease of the M_5 edge XMCD amplitude seems to be proportional to the UAs thickness while the M_4

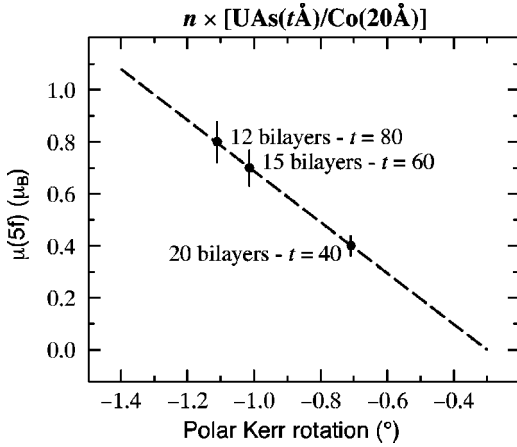


FIG. 3. The $5f$ magnetic moment deduced from our XMCD measurements (35 K, 4 T) is plotted vs the maximum polar Kerr rotation (MOKE;³ 10 K, 3 T) for the three multilayers.

edge amplitude is roughly the same for the 12 and 15 bilayered films and drops sharply for the 20 layer sample. This feature does not arise from a badly corrected saturation effect since the variation of the $5f$ magnetic moment exactly reproduces the magneto-optical Kerr effect³ as shown in Fig. 3.

The orbital and spin magnetic contributions deduced from the sum rules^{8,9} are summarized in Table I, taking the n_h and $\langle T_z \rangle$ values corresponding to each of the two uranium valence states, $5f^2$ (U^{4+}) and $5f^3$ (U^{3+}).²¹ We are not able to determine the uranium configuration since both $5f^3$ and $5f^2$ assumptions give reasonable orbital to spin ratio values when compared to the free-ion ones. Nevertheless, we note the good agreement between the $5f^3$ magnetic moment deduced from the XMCD measurements, and the magnetization value for UAs amorphous films, reported by Fumagalli *et al.*, of $\approx 0.7 \mu_B$.^{3,5} Furthermore, previous studies performed on uranium monpnictides^{22,23} strongly suggest a U^{3+} ($5f^3$) configuration, confirming the agreement between our results and the MOKE interpretations. For simplicity, we will thus assume the $5f^3$ valence state only in the following parts of this paper.

We also note that the uranium moment for the 20 bilayered film is much smaller than that of the 12 and 15 bilay-

ered samples, confirming the trend of the $U-M_4$ edge XMCD signal. This feature could be ascribed to the interdiffusion of Co in the UAs layers associated with the formation of a “dead” layer at each interface (see Sec. II and Ref. 3). Indeed, if such a dead-layer exists, one would measure an average $5f$ contribution with a mixing of the magnetic (within the layer) and nonmagnetic (dead-layer) parts of the UAs layers. Assuming the dead-layer thickness Δ to be constant from one multilayer to another and taking the magnetic moment carried by the magnetic part of UAs layers to be $\mu^{mag}(5f) = 0.7 \mu_B$,^{3,5} one can estimate a value of $\Delta \sim 13 \text{ \AA}$ from our XMCD results, in agreement with Ref. 3. Thus, even if this result is considered as an indication of tendency (the XMCD technique is not optimized for such a determination), it again favors the interdiffusion scenario.

We stress the direct relationship between the maximum polar Kerr rotation measured by Fumagalli *et al.*³ at a temperature of 10 K and under a 3 T applied magnetic field and the $5f$ magnetic moment deduced from our XMCD experiment, which were performed at 35 K and 4 T (cf. Fig. 3). We note the linear relation between the two sets of data, confirming that the MOKE results are directly related to the magnetic contribution of the uranium.

Note that for a zero $\mu(5f)$ we have a nonzero Kerr rotation angle, the so-called optical constant effect, which is always present in Kerr rotation measurements.³ The aim of our study is then to see whether the uranium moment contributes to the room-temperature Kerr effect.

V. XMCD RESULTS AS A FUNCTION OF TEMPERATURE AND FIELD FOR THE $12 \times [\text{UAs}(80 \text{ \AA})/\text{Co}(20 \text{ \AA})]$ MULTILAYER—EVIDENCE OF THE U POLARIZATION

In this section we concentrate on the 12 bilayered sample since it is the one which exhibits the largest uranium magnetic moment and as a consequence the largest polar Kerr rotation. The $U-M_4$ edge XMCD signal area has been measured as a function of temperature and field. The associated $5f$ magnetic contribution was then deduced using the scaling factor which connects the $U-M_4$ edge XMCD integrated signal measured at 10 K and the corresponding $5f$ magnetic

TABLE I. Magnetic contributions deduced from XMCD measurements performed at 35 K and 4 T for the 12, 15, and 20 bilayered films (corresponding to UAs layer thicknesses t of 80, 60, and 40 \AA , respectively). Orbital, spin and total $5f$ moments $\mu_L(5f)$, $\mu_S(5f)$, and $\mu(5f)$, respectively, are given in μ_B . Experimental orbital to spin moments ratios, $-\mu_L(5f)/\mu_S(5f)$, should be compared to the theoretical values (2.54 and 3.36 for the $5f^3$ and $5f^2$ configurations, respectively²¹).

	Bilayers	$t(\text{UAs}, \text{\AA})$	$\mu_L(5f)$	$\mu_S(5f)$	$\mu(5f)$	$-\frac{\mu_L(5f)}{\mu_S(5f)}$
$5f^3$	12	80	1.4(1)	-0.59(6)	0.8(2)	2.37(41)
	15	60	1.3(1)	-0.59(6)	0.7(2)	2.20(39)
	20	40	0.74(7)	-0.34(3)	0.4(1)	2.18(40)
$5f^2$	12	80	1.5(1)	-0.41(4)	1.1(2)	3.66(60)
	15	60	1.4(1)	-0.41(4)	1.0(2)	3.41(58)
	20	40	0.81(8)	-0.23(2)	0.6(1)	3.52(65)

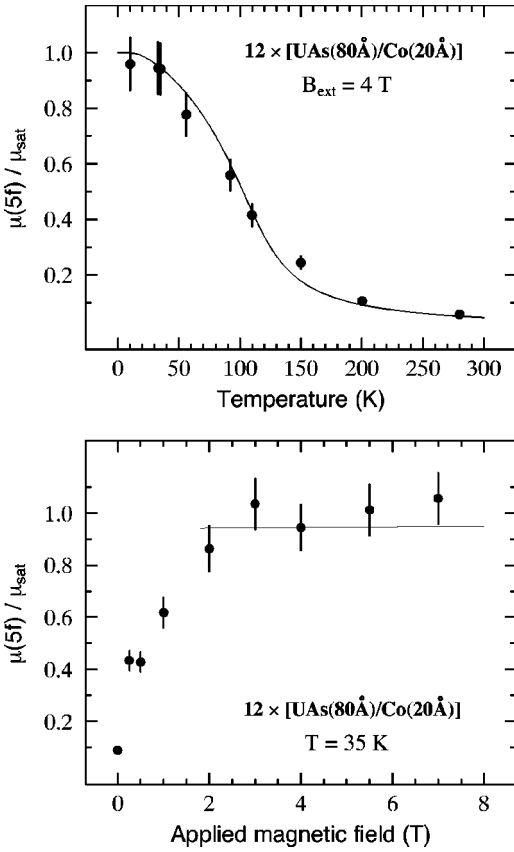


FIG. 4. Temperature dependence for a 4 T applied field (top panel) and field dependence at 35 K (bottom panel) of the $5f$ magnetic contribution assuming a U^{3+} valence state. The $5f$ moment calculated in a mean-field scheme is shown as the solid lines (both panels).

moment resulting from the application of the sum rules. The results are shown in Fig. 4.

Assuming the exchange field due to cobalt to be constant in the region of interest here, we also present in Fig. 4 the variation of the $5f$ moment calculated within the mean-field approximation. In this scheme, we define

$$\frac{\mu(5f)}{\mu_{sat}} = B_J \left(\frac{g \mu_B J [\lambda \mu(5f) + H_{eff}]}{k_B T} \right) \quad (3)$$

with,

$$\lambda = \frac{3k_B T_C}{\mu_{sat}(g \mu_B)(J+1)}. \quad (4)$$

$B_J(x)$ is the Brillouin function. $\mu(5f)$ is the $5f$ magnetic moment and $\mu_{sat} = 0.85 \mu_B$ its saturation value. $H_{eff} = H_{app} + H_{exch}^{Co}$ represents the effective field as the sum of the applied field and the exchange field due to the cobalt ions. Considering a U^{3+} configuration, we have $J = 9/2$ and the Landé factor calculated in the intermediate coupling scheme, $g = 0.759$. A rather good agreement between experimental and calculated $5f$ magnetic contributions is obtained using the UAs Curie temperature $T_C = 100$ K and $H_{exch}^{Co} = 6$ T. This model oversimplifies the physics of these multilayers,

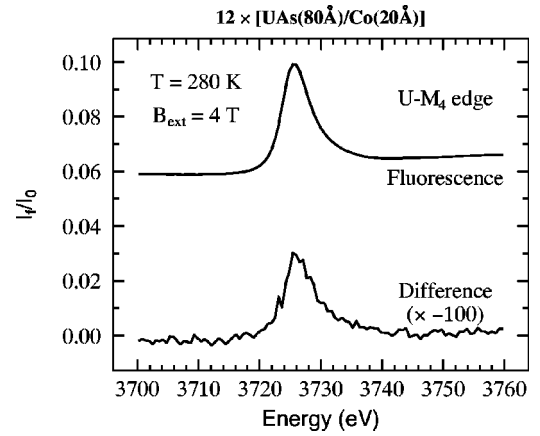


FIG. 5. Raw fluorescence and dichroism spectra at the M_4 edge of uranium for the 12 bilayered film at 280 K and 4 T.

but it shows that the applied field alone is not sufficient for the polarization of the uranium; the role of the cobalt is clearly demonstrated.

Saturation is achieved above ≈ 2 T as shown by the field dependent measurement; *a fortiori*, 4 T applied along the direction used is enough to saturate the sample. The saturated uranium moment reaches $\approx 0.85(15) \mu_B$. Finally, Fig. 5 also shows that at room temperature there is a small XMCD signal at the $U-M_4$ edge. This corresponds to an induced moment on the uranium of $\approx 0.05 \mu_B$ (assuming the $5f^3$ configuration) which is considerably lower than the $0.14 \mu_B$ predicted by the IBM group.³ This result shows that, due to the strong spin-orbit coupling, even a small uranium contribution can lead to a sizeable magneto-optical effect. A magnetic moment can be induced between two layers of a multilayer via exchange coupling.

VI. CONCLUSIONS

A series of uranium arsenide/cobalt multilayers was studied using x-ray reflectivity and XMCD. The three samples consisted of 12, 15, and 20 bilayers of UAs/Co with differing UAs layer thicknesses. The x-ray reflectivity results indicate that, while the films are of relatively poor quality, the reflectivity profile can be modeled by a trilayer model in which there is a 10 \AA interdiffused layer of UAs-Co alloy in agreement with previous published results.^{3,5}

XMCD measurements have been performed on these multilayers at 35 K with a 4 T applied field. For the $12 \times [\text{UAs}(80 \text{ \AA})/\text{Co}(20 \text{ \AA})]$ multilayer, both temperature and field dependent measurements, have been performed. A linear relation between the polar Kerr rotation and the $5f$ magnetic moment has been demonstrated, confirming previous interpretations of the magneto-optical effect. For the $12 \times [\text{UAs}(80 \text{ \AA})/\text{Co}(20 \text{ \AA})]$ and $15 \times [\text{UAs}(60 \text{ \AA})/\text{Co}(20 \text{ \AA})]$ multilayers, the uranium moment is in good agreement with magnetization measurements. The moment decreases considerably for the 20 bilayered film corresponding to the thinnest UAs layers (40 \AA). This feature seems to confirm the presence of a dead UAs-Co layer at each interface. The temperature and field dependences

of the 12 bilayered film probe the ferromagnetic behavior of the UAs layers. T_C is not enhanced by the presence of cobalt and saturation is achieved above ≈ 2 T. Finally, an important observation is the small uranium moment at room temperature of $0.05 \mu_B$ induced by the cobalt magnetization through exchange coupling. Nevertheless, this very small uranium moment is responsible for important magneto-optical effects.

ACKNOWLEDGMENTS

The authors want to warmly thank the Keele University(U.K.) for its support and assistance during the x-ray reflectivity measurements and the IBM researchers (T.S. Plaskett *et al.*) who provided us the samples. We are particularly grateful to Dr. S. Brown for his assistance and advice with the x-ray reflectivity measurements.

*Email address: kerna@ill.fr

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