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Investigation of (Fe/Dy) multilayers by ^{57}Fe Mössbauer spectrometry

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Abstract

The structural and magnetic properties of (Fe t_{Fe} nm/Dy t_{Dy} nm) multilayers deposited at 320 K by thermal evaporation in ultra-high vacuum (UHV) have been investigated by conversion electron ^{57}Fe Mössbauer spectrometry (CEMS) at 300 K. These measurements enabled to determine in which conditions the perpendicular magnetic anisotropy (PMA) is optimised as a function of the layers thickness. The Fe thickness, t_{Fe} , is varied between 1 and 4 nm, and the Dy thickness, t_{Dy} , equals 1 or 2 nm. The Mössbauer spectrometry measurements reveal that the PMA is the largest when t_{Fe} is the smallest, i.e., when the multilayers are structurally close to an Fe–Dy amorphous alloy with modulation of composition.

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1. Introduction

Transition Metal (TM)/Rare Earth (RE) systems have already been widely investigated in the past because of their potential applications in magneto-optical recording [1–7]. From a fundamental point of view, numerous models have been proposed to explain the physical origin of the perpendicular magnetic anisotropy (PMA) in the TM–RE amorphous alloys, based on dipolar interactions [8], anisotropic distribution of atomic pairs [9], microstructural anisotropy [10], or local structural anisotropy [11]. The key-element is the strong spin–orbit coupling acting on the RE ions, combined with the asphericity of its 4f orbital, leading to the so-called single-ion anisotropy. Since TM/RE multilayers have been found to enhance the PMA in certain conditions [12], a large number of studies have been devoted to examine the source of the PMA in TM/RE multilayers. Among them, (Fe/Dy) multilayers are one of the most interesting in terms of PMA. If (Fe/Tb) multilayers have already been extensively investigated in the past [12–22], much less studies have been carried out on (Fe/Dy) multilayers [23–29]. In such multilayers, the structural and

magnetic properties strongly depend on the thickness of the individual layers. For small layer thicknesses, typically less than 1 nm, the samples can be properly described as compositionally modulated amorphous films with strong perpendicular magnetic anisotropy [23,24,27]. For intermediate values of individual layer thicknesses (2–3 nm), crystallisation occurs and a reversible reorientation of the magnetisation from in-plane orientation at 300 K to out-of-plane orientation at low temperature has been evidenced [28]. For thicker layers (more than 4 nm), the PMA disappears and the magnetisation lies in the sample plane [26]. The temperature dependence of the spin textures of Fe and Dy has been studied by ^{161}Dy and ^{57}Fe Mössbauer spectrometry [29]. The average orientations of moments at 4.2 K were found random for Dy but out-of-plane for Fe. However, there has been no systematic study of the influence of the layer thickness on the Fe spin texture in (Fe/Dy) multilayers. The present study is part of a wider work consisting of probing whether the physical origin of the PMA is rather due to the symmetry breaking at the interfaces [30] or to the crystal field irregularities induced by chemical mixing between the elements [25]. ^{57}Fe Mössbauer spectrometry technique was used to study the influence of the individual Fe and Dy layer thicknesses on both the structure and the average Fe-spin orientation at room temperature.

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2. Experimental

The samples were prepared in ultra-high vacuum (UHV) by thermal evaporation of high-purity elements onto (1 0 0) Si substrates at 320 K. The number of bilayers was chosen in order to enhance the total amount of iron atoms for improving the counting rate during the Mössbauer analysis (Table 1). The background pressure was close to 5×10^{-8} Pa, and the pressure during deposition was lower than 5×10^{-6} Pa. The deposition rate was approximately 0.01 nm s^{-1} and the thicknesses were controlled by a quartz monitor close to the substrate holder. All the samples were characterised by conversion electron Mössbauer spectrometry (CEMS) at room temperature under normal incidence with a home-made helium–methane gas proportional counter, with ^{57}Co in a Rhodium matrix as the source. Isomer shift is given with respect to α -iron at 300 K. The experimental spectra were fitted with a least-squares technique [31] using the histogram method relative to discrete distributions constraining the linewidths of each elementary spectrum to be the same.

3. Results and discussion

The CEMS spectra of the multilayers with a Dy layer thickness (t_{Dy}) of 1 nm and various Fe layer thicknesses (t_{Fe}) are shown in Fig. 1. The spectra have been fitted with a wide distribution of magnetically splitted sextets with different hyperfine field and isomer shift values to take into account all the structural and chemical environments experienced by the Fe atoms. From this fitting procedure, average values of the isomer shift (δ) and hyperfine field (B_{hf}) can be obtained. These mean parameters are the weighted sum of the values of each distributed sextet. For the thinnest Fe layers ($t_{\text{Fe}} = 1 \text{ nm}$), the broad lines on the Mössbauer spectrum and the shape of the distribution of hyperfine field $P(B_{\text{hf}})$ (Fig. 1(a)) are characteristic of a

magnetic Fe–RE amorphous alloy [32,33]. This can be understood by a strong mixing of the Fe and Dy atoms at the interfaces during deposition [34]. For $t_{\text{Fe}} = 1.5 \text{ nm}$ (Fig. 1(b)), the spectrum is narrower and the low field contributions on $P(B_{\text{hf}})$ are enhanced, indicating a less magnetic sample. Increasing t_{Fe} to 2 nm leads to a spectrum with only a sharp line characteristic of a paramagnetic phase, with an isomer shift of -1.17 mm s^{-1} (Fig. 1(c)). This paramagnetic phase is revealed on the hyperfine field distribution by a peak at very low magnetic fields and is attributed to an Fe-rich amorphous alloy or to pure amorphous Fe in the centre of Fe layers [35–37]. It should be emphasized that the low field contributions cannot originate from a compensation state in a ferrimagnetic structure, since the Mössbauer technique can only probe the Fe magnetic subnetwork. For $t_{\text{Fe}} = 2.5 \text{ nm}$ (Fig. 1(d)), the Mössbauer spectrum exhibits, in addition to the non-magnetic central line, a small sextet with hyperfine parameters ($\delta = 0 \text{ mm s}^{-1}$ and $B_{\text{hf}} = 33 \text{ T}$) close to those of crystalline BCC-Fe phase. This clearly evidences the crystallisation of a fraction of Fe atoms (15% of all Fe atoms) inside the Fe layers. When t_{Fe} is further increased from 3 to 4 nm (Figs. 1(e)–(g)), the BCC-Fe contribution on the spectrum and on the $P(B_{\text{hf}})$ increases in intensity at the expense of low-field contributions, indicating the thickening of the crystallised iron layers and a reduction of the amorphous fraction located at the interfaces. Such a structural transition from amorphous to crystalline Fe layers has already been observed in many studies concerning (Fe/Tb) multilayers [19,20,22].

Fig. 2 shows the evolution as a function of t_{Fe} of the Mössbauer spectra and $P(B_{\text{hf}})$ for the second set of (Fe/Dy) multilayers with $t_{\text{Dy}} = 2 \text{ nm}$. For an iron layer thickness of 1 nm, the shape of the spectrum (Fig. 2(a)) exhibits a central line with broadening at the base. The hyperfine field distribution is wide due to a large variety of Fe atom environments, in agreement with an amorphous layer structure. In comparison with the (Fe 1 nm/Dy 1 nm) multilayer (Fig. 1(a)), the spectrum is typical of a less magnetic amorphous alloy and the $P(B_{\text{hf}})$ distribution has a maximum that is shifted towards lower fields, with a value of the mean hyperfine field (B_{hf}) of 9.4 T, instead of 16.1 T for $t_{\text{Dy}} = 1 \text{ nm}$. These small values can be explained by a modulated composition due to the layered structure of the sample. For an increasing Fe layer thickness, the non-magnetic central peak increases in intensity on the spectra, as previously observed for $t_{\text{Dy}} = 1 \text{ nm}$, until the ferromagnetic contribution totally disappears for $t_{\text{Fe}} = 2.5 \text{ nm}$ (Fig. 2(d)). This behaviour is explained by the increasing Fe concentration in the Fe–Dy amorphous alloy, since pure amorphous Fe is paramagnetic at room temperature [35–37]. The appearance of the α -Fe sextet in addition to the non-magnetic component on the spectra occurs for $t_{\text{Fe}} = 3 \text{ nm}$ (Fig. 2(e)), clearly showing the beginning of the iron layers crystallisation. For larger t_{Fe} values, the BCC-Fe sextet grows in intensity and is superimposed to a broad component attributed to the interfaces, where

Table 1
Number of bilayers (n) and Fe atomic fraction (X_{Fe}) in the multilayers as a function of Dy (t_{Dy}) and Fe (t_{Fe}) individual layer thicknesses

t_{Dy} (nm)	t_{Fe} (nm)	X_{Fe}	n
1	1.0	0.73	80
	1.5	0.80	70
	2.0	0.84	45
	2.5	0.87	40
	3.0	0.89	35
	3.5	0.90	30
	4.0	0.91	30
2	1.0	0.57	80
	1.5	0.67	60
	2.0	0.73	45
	2.5	0.77	40
	3.0	0.80	35
	3.5	0.82	30
	4.0	0.84	30

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