



# Magnetic properties of Fe–Co ferromagnetic layers and Fe–Mn/Fe–Co bilayers obtained by thermo-ionic vacuum arc

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## ABSTRACT

Simple Fe–Co ferromagnetic films and Fe–Mn antiferromagnetic films of different compositions were grown on epitaxial Si substrates with Ta buffer layers via thermo-ionic vacuum arc method. Fe–Co/Fe–Mn bilayer structures were obtained by following similar growing conditions as for the simple films. The magnetic behavior concerning easy axis distribution, anisotropy energy and coercive field of the Fe–Co films exchange coupled to the antiferromagnetic layers were discussed with respect to the magnetic behavior of the simple, uncoupled, ferromagnetic films. The composition of the antiferromagnetic films has a sensible influence on the magnetic parameters of the exchange coupled ferromagnetic films. The largest coupling and the narrowest easy axis distribution are induced in the bilayer system with equatomic composition of the Fe–Mn antiferromagnetic layer.

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## 1. Introduction

Since the discovery of the exchange bias effect more than 50 years ago, by Meiklejohn and Bean [1], a new and exciting branch of modern magnetism has been rapidly grown up. The origin of the exchange bias effect is related to the interfacial exchange coupling of a thin ferromagnetic (F) layer sharing a common interface with an appropriate antiferromagnetic (AF) layer. The exchange coupling leads either to a shift of the hysteresis loop of the F layer with respect to the direction of the applied field, when an unidirectional anisotropy energy is involved at the interface (the shift is called exchange bias field) or to an increased coercivity, when only a common uniaxial anisotropy energy is involved. Clearly, both features lead to a different magnetization reversal of the F layer interfacially pinned to the AF layer, as compared with the case of a free F layer. Therefore, new spin valve devices were developed on basis of giant magneto-resistance (GMR)/tunneling magneto-resistance (TMR) effects generated in conductive/insulator thin films sandwiched between free and pinned F layers [2–4]. The equatomic composition of Fe–Mn was used for long time as a convenient AF layer and Fe<sub>20</sub>Ni<sub>80</sub> (permalloy) as a suitable low coercive F layer in exchange biased spin valves [3,5]. In the state of art spin valve devices, Ir–Mn has replaced Fe–Mn due to enhanced intrinsic magnetic properties (e.g. higher Néel temperature as well as

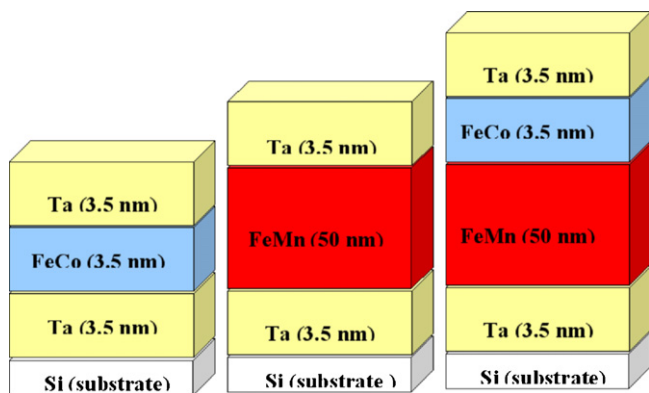
exchange anisotropy energy) and Co–Fe has replaced Ni–Fe due to a decreased atomic inter-diffusion at the F/Cu (conductive layer) interface. However, to optimize Fe–Mn as a valuable AF layer in AF/F bilayers for particular applications could become more convenient with respect to lower fabrication costs. This paper promotes a detailed study of the interfacial coupling in Fe–Mn/Fe–Co bilayer systems of different Fe concentrations in the Fe–Mn coupling layer, taking the advantage of the combinatorial processing procedure of the thermo-ionic vacuum arc method [6,7]. The orientation dependence of the anisotropy energy and coercive fields in the bilayer systems were compared and discussed with respect to the equivalent parameters of the free Fe–Co thin films.

## 2. Experimental

Simple ferromagnetic Fe–Co and antiferromagnetic Fe–Mn thin films as well as Fe–Mn/Fe–Co bilayers were deposited by thermo-ionic vacuum arc method on Ta buffer layers grown on (100) Si plackets (Ta thin films were also top deposited as protective cap layers). The currently used experimental arrangement of the thermo-ionic vacuum arc method, involves two electron beams generated by heated cathodes and accelerated by high anodic voltages. The materials, placed in special crucibles at the anodes, are evaporated via electron bombardment. The evaporating species, metals and alloys in our case, can be deposited on a series of substrates, located at different distances from the anode positions. The experimental arrangement using four different crucibles at each of the two anodes (the crucibles are rotating in the same anodic position via a special device) allows either the simultaneous deposition from two different crucibles or the simple deposition from only one crucible. However, using this design, each different substrate can be indexed by only one (in case of one source deposition) or two (in case of two sources deposition) distances from the anodic positions. Hence, a two sources deposition leads to a combinatorial preparation of thin film alloys of different concentrations, depending on the position of the substrate with respect to the two anodic positions where

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**Fig. 1.** The geometrical structure of the analysed samples, as follows: (a) set  $F.n$ , (b) set  $AF.n$  and (c) set  $AF/F.n$ .

the evaporating materials are placed. Let us note the distance from anode 1 by  $d_{A1}$  and the distance from anode 2 by  $d_{A2}$ . Four substrate positions (1, 2, 3, and 4) have been considered for each preparation, involving the following couple of distances  $d_{A1}/d_{A2}$  (in mm): 160/200, 185/175, 200/165 and 215/155, for the mentioned positions, respectively. The three prepared sets of samples are presented in Fig. 1. A Fe–Co alloy closed to the equatomic composition was previously prepared by arc-melting and introduced in one of the four crucibles at anode 1. Similarly, Ta foils were introduced in a crucible at anode 2. In conditions of one source evaporation, almost similar Fe–Co relative contents are expected for the thin films grown on all the four substrates placed at positions 1–4, but also a diminished thickness of the Fe–Co thin films when moving from position 1 to 4 (and similarly a higher thickness of the Ta films, when moving in the same sense). It is worth mentioning that the generic thicknesses given in Fig. 1, correspond to average thicknesses per preparation, measured by only one quartz balance (Kressington) placed between positions 2 and 3. For preparing the Fe–Mn thin films, a two sources preparation procedure was used, by inserting Fe in a crucible at anode 1 and Mn in a crucible at anode 2 and initiating a simultaneous deposition. Due to the experimental design, Fe–Mn thin films with increasing Mn content when moving from position 1 to 4 of the substrates are expected. The relative variation of the thickness of the Fe–Mn films has to be this time almost insignificant, due to the much higher thickness of the film (more than one order of magnitude thicker than in the case of the Fe–Co film) on one hand and, on the other hand, to the simultaneous evaporation with almost similar rates from both sources at anode 1 and 2. Finally, in case of the bilayer system, the preparation was done with materials inserted in crucible exactly as above mentioned for the simple films and following the same evaporation procedure. Therefore, the thicknesses and composition of each of the two films in the bilayer structure belonging to one position of the substrate, have to be identical with the ones in single films corresponding to the same substrate position. However, a different magnetic texture would be expected for the Fe–Co thin films grown on Fe–Mn layers of different Fe relative content, as compared with the films grown on the Ta buffer. The interfacial coupling can also modify the coercive force and the shift of the hysteresis loop in the exchange coupled Fe–Co films. Therefore, the magnetic behavior of the Fe–Co layers exchange coupled to Fe–Mn layers of different relative concentrations has been analysed essentially with respect to the behavior of the corresponding free (uncoupled), Fe–Co layers. The prepared samples were labeled as follows (see also Fig. 1): (a)  $F.n$  with  $n = 1, 2, 3, 4$  indexing the substrate position, for simple ferromagnetic Fe–Co films with Ta both as buffer and cap layer, (b)  $AF.n$  for simple antiferromagnetic Fe–Mn films with Ta deposited as buffer and cap layer and (c)  $AF/F.n$ , for Fe–Mn/Fe–Co bilayer systems with Ta again as buffer and cap layer. The relative content of Fe in the  $AF.n$  films as well as in the  $F.n$  films was estimated by Energy Dispersive X-ray (EDX) technique and the film surface quality was checked by Scanning Electron Microscopy. The room temperature magnetic reversal processes in the free as well as in the coupled Fe–Co films were characterized by longitudinal magneto-optic Kerr effect (MOKE). A MOKE device (type AMACC) working in longitudinal geometry and using a magnet with laminated sheets with essentially zero remanence and negligible hysteresis was utilized. The device is provided with a step motor for rotating the sample in its own plane, with a precision better than  $0.5^\circ$ . The wavelength of the incident light, provided by a laser diode, is about 640 nm. The angle of incidence is  $45^\circ$  and the incident light is linearly polarized along the sample plane. A brief low temperature characterization of the magnetization reversal at 5 K was performed via VSM magnetometry, in a close cycle Cryogenics cryomagnet. All the magnetic measurements were performed on square-shaped samples, with an area of about  $81 \text{ mm}^2$  for MOKE measurements and of about  $10 \text{ mm}^2$  for VSM measurements and with the magnetic field applied along the sample plane. The cutting edge of the sample with respect to the crystallographic axis of the Si substrate was validated by X-ray diffraction.

### 3. Results and discussions

The relative content of Fe and Co in the 2 extremities samples  $F.1$  and  $F.4$ , as determined by EDX, was 50–50 at.% in sample  $F.1$  and 52–48 at.% in sample  $F.4$ , respectively. Within the error limits of 1–2 at.%, we may consider that all the  $F.n$  films present the same composition, close to the equatomic ratio. This consideration is also in agreement with the preparation procedure based on the evaporation of an Fe–Co alloy from only one evaporation source. However, the ratio between the relative wt.% of Fe–Co and of the Ta decreases from 1.5 in sample  $F.1$  down to 0.7 in sample  $F.2$ , proving clearly a decreased thickness of the Fe–Co film and an increased thickness of the Ta buffer, when moving from position 1 (closer to anode 1, where the Fe–Co crucible was placed) to position 4 (closer to anode 2, where the Ta crucible was placed). Roughly, from the wt.% EDX delivered data, the thickness of the Fe–Co film in sample  $F.4$  was estimated at about 0.7 from the thickness of the ferromagnetic film in sample  $F.1$ . Starting from the indication of the quartz balance placed between positions 2 and 3, it can be inferred that the thickness of the Fe–Co films decreases from about 4 nm for  $F.1$  down to 3 nm for  $F.4$ . The magnetization reversal in samples  $F.1$  and  $F.4$  was analysed by longitudinal MOKE, at different orientations of the Si substrate edge, with respect to the direction of the applied magnetic field. X-ray diffraction scans were performed on Ta grown on the Si substrate, with the radiation incident on the sample plane at a fix small angle and moving only the detector in the incidence plane from  $15^\circ$  to  $60^\circ$ . With the edge of the sample along the radiation beam, only the 3 1 1 diffraction line of Si with face center cubic structure (fcc), space group  $Fd\bar{3}m$ , as well as 1 1 1 and 2 0 0 diffraction lines of Ta with fcc structure, space group  $Fm\bar{3}m$ , were observed. Rotating the sample by  $45^\circ$  in its own plane, the Si line disappeared completely whereas the two lines of Ta remained both present, just with a slightly diminished intensity ratio  $I_{111}/I_{200}$ . That proves clearly the edge of the mono-crystalline Si is cut along the [1 1 0] crystallographic direction and a poly-crystalline layer of Ta with a slight texture also along [1 1 0] is formed. The hysteresis loops of the  $F$  films with extreme thickness,  $F.1$  and  $F.4$ , collected at three different orientations,  $\theta = 0^\circ$ ,  $45^\circ$  and  $90^\circ$  (where  $\theta = 0^\circ$  means that the sample edge, which is along the [1 1 0] axis of Si, is parallel to the applied field, whereas  $\theta = 90^\circ$  means that it is perpendicular to the applied field) are shown in Fig. 2(a) and (b) respectively. At a very first glance two important aspects can be mentioned from these results: (i) the hysteresis loops evolve with  $\theta$  angle, from a more rectangular shape and higher coercivity at  $\theta = 0^\circ$  to an almost closed shape of much lower coercivity at  $\theta = 90^\circ$  and (ii) at  $\theta = 0^\circ$ , the hysteresis loop of sample  $F.4$  is more rectangular than the hysteresis loop of sample  $F.1$  whereas at  $\theta = 90^\circ$ , sample  $F.4$  show no coercivity as compared with the finite coercivity of sample  $F.1$ . These results can be qualitatively explained via the simple Stoner–Wolfarth model [8,9] of a ferromagnetic film (when a single domain state is considered) with in-plane uniaxial anisotropy. It is worth mentioning that Conversion Electron Mossbauer spectra of the Fe–Co films enriched in the  $^{57}\text{Fe}$  isotope have proven the in-plane spin orientation of the analysed films [10]. Defining  $K_F$  as the anisotropy constant,  $\theta$  as the orientation of the applied field with respect to the anisotropy direction (easy axis of magnetization),  $M_F$  the saturation magnetization and  $\beta$  the orientation of the magnetization with respect to the anisotropy direction, the magnetic energy per unit volume of the ferromagnetic film is:

$$E = -\mu_0 H M_F \cos(\theta - \beta) + K_F \sin^2 \beta \quad (1)$$

where the first term represents the magnetic Zeeman contribution and the second one is the contribution of the magnetic anisotropy. Angle  $\beta$  is deduced from the condition of the minimization of the magnetic energy with respect to this angle, for each value and

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