INTERFACIAL EFFECT IN Fe/ InGaAs MAGNETISM

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ABSTRAT

Fe layers 4, 8, 20 and 60nm-thick, were epitaxially deposited by Molecular-Beam-Epitaxy on InGaAs/InP(100) wafers. We found that significant changes in cristallinity and microstructure occur with increasing the Fe thickness from the AFM measurement. Also, we have used both complementary techniques: VSM and FMR measurements to display how change the magnetic anisotropies as a function of thickness Fe layer. A strong in-plan uniaxial magnetic anisotropy with magnetic axes along the both major crystallographic directions is present at Fe/InGaAs/InP(100) interface for the 4-nm-thick Fe layer. The 8-nm-thick Fe film have a cubic anisotropy that is mixed with an interface induced uniaxial anisotropy. The magnetic anisotropy of the bulk Fe dominates for 20-nm-thick Fe film. On the other hand, in our FMR experiments, we observed unconventional two-picks FMR spectra for the thinner sample as compared to the bulk. This anomalous peaks, allowing one to discriminate between various in plane magnetic anisotropies. Moreover, after the thermal treatment a drastic change of the hysteresis loop has generated and the disappearance of the peak associated to the Fe (110) plane. In addition to this, we have performed a depth profile of thermally diffused Fe/InGaAs/InP using phoemission spectroscopy combined with Ar+ ion sputtering. We found that Fe ion was thermally diffused into the deep region of the wafer and the strong segregation of metallic indium at the surface under the UHV annealing treatment.

Keywords: Fe/InGaAs/InP; AFM, VSM and FMR; uniaxial and cubic magnetic anisotropy, DRX and XPS, annealing treatment

1. INTROCUCTION

Over the last decades, the characterization of Ferromagnet / Semiconductor interfaces has been a topic of great interest because of the possible future development of spintronics, e.g., spin injection through the ferromagnetic thin film to the semiconductor [1]. Particularly, the Fe/GaAs system has been extensively studied [2] due to its ferromagnetism at room temperature and the small lattice mismatch (1.3 %) between the two materials. The numerous investigations performed on such a system have revealed the crucial role played by the interface, particularly on the magnetic properties of iron. Indeed, elaboration of ultrathin films of iron has evidenced a reduction of its magnetization with respect to the bulk, and thus suggested the existence of a magnetically-deficient region at the interface [3]. Moreover, uniaxial magnetic anisotropy has been reported for iron ultrathin films. Its origin has been explained, at first, by Krebs *et al.* [4] by the dangling bond at GaAs surface. However, more recently, such an anisotropy has been observed for different GaAs surface reconstructions and consequently interpreted by magnetoelastic effects due to the lattice mismatch at the interface [5].

Another promising candidate for spintronic is the Fe/$In_{0.5}Ga_{0.5}As$ system. However, contrarily to Fe/GaAs, only few studies have been reported [6,7]. Magnetization reversal investigations [7] have demonstrated the presence of a uniaxial anisotropy for an iron thickness of less than 6 nm as well as a reduction of the magnetization at low iron coverage. Investigations of the interface after 1-hour annealing at temperatures in the range 350 - 450°C were performed by X-Ray Diffraction and Mosbauer techniques [6] and led to the identification of metallic indium, Fe-As compounds and $Fe_3Ga_{2-x}A_x$ (x = 0.2 – 0.3). The presence of indium at the interface is of high interest since it is expected to modify its magnetic properties with respect to GaAs. This is of first relevance since the indium atoms are known to strongly diffuse, even at moderate temperatures, and to not make alloy with iron. In the present paper, we focus on the characterization of Fe / $In_{0.5}Ga_{0.5}As$ / InP (001) heterostructure. Iron layers with different thicknesses were epitaxially deposited. The magnetic properties of the as-deposited samples will be studied and explained by a competition between interfacial and bulk iron magnetism. To point out the main moving species at the interface, thermal treatment of the heterostructures will then structurally and magnetically be investigated.

2. EXPERIMENTAL DETAILS

Iron films with thicknesses from 4 to 60 nm were grown by Molecular Beam Epitaxy (MBE) deposition onto oriented semiconductor $In_{0.5}Ga_{0.5}As$ / InP(100) substrates. $In_{0.5}Ga_{0.5}As$ was grown by metal-organic chemical vapor deposition (MOCVD) onto an InP (100) substrate within the *Laboratoire des Multimatériaux et Interfaces*. Because of the mismatch agreement (a = 0.5868 nm) between InGaAs layer and InP(001) substrate, there is a limited amount of constraints. Iron growth was achieved in a UHV chamber at a base pressure of 5 x 10^{-10} Torr. InGaAs substrates were preliminary degreased with acetone, and the native oxide was removed by Ar^+ spttering (800 eV). The Fe deposition (4, 8, 20 and 60 nm) was performed with a Knudsen cell, and the substrate temperature was 100 ± 10 °C. The deposition rate was calibrated with a quartz microbalance and was fixed at 2.0 Å / min

The deposition of 4, 8, 20 and 60 nm of iron was magnetically characterized by Ferromagnetic Resonance (FMR) and Vibrating Sample Magnetometer (VSM) experiments. FMR measurements were made with a standard spectrometer ELEXYS 500-BRUKER operating in X band at 9.7 GHz under application of the field in the sample plane. The morphology and the surface roughness of the samples were controlled through Atomic Force microscopy (AFM) experiments. The eventual diffusion of species was studied by both X-Ray Diffraction (XRD) and X-ray photoelectron spectroscopy (XPS) depth profile; the latter permits one to investigate the different layers after controlled Ar^+ etching [8].

3. RESULTS AND DISCUSSION

Figures 1(a) and 1(b) show the morphologies of 4- and 20-nm-thick iron layers. They clearly highlight differences in roughness and morphology. The 4-nm-thick Fe film looks flat, whereas granular structures features are observed for the higher thickness. The root mean square is 0.4nm for the first sample and 1.4 nm for the other. These differences in roughness and morphology are induced by the difficulty to growth epitaxial 3D films; they likely affect the magnetic properties, i.e. magnetic moments, magnetic anisotropy or pinning of domain wall.

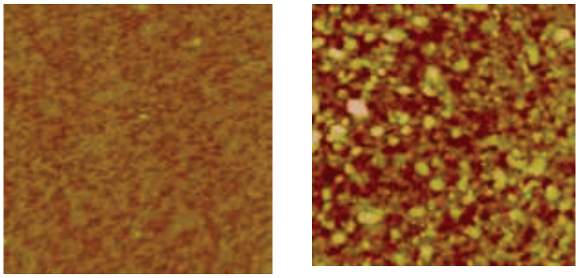

Fig 1. Atomic force microscope (AFM) images of 4-nm-thick (a) and 20-nm-thick (b) Fe layer on InGaAs (001) substrate. Scan area was 1 µm x 1 µm.

Figure 2 shows the magnetization reversal for 4 different iron thicknesses at room temperature. It displays clearly an increase of the coercivity field, H_C, with iron thickness. This behavior seems thus be correlated to the roughness increase observed by AFM. However, since the magnetization reversal is a rather complex mechanism, the relationship between coercivity and roughness for a rather thick film is not a simple monotonic relationship [9]. A similar effect was recently been reported on Fe/GaAs (001) [10]. The comparison of the different pictures highlights a second magnetic effect evidenced through the occurrence of a uniaxial anisotropy when the thickness is 4 nm: it is clearly evidenced in Fig. 2(a) by the existence of magnetic easy and hard axis. Increase in thickness causes a reduction of the uniaxial anisotropy (Fig. 2(b)) prior to its complete disappearance (Figs. 2c and 2d). Iron layer being essentially single domain by nature, this observation confirms the competition between the uniaxial anisotropy related to the first layers and the anisotropy associated to the Fe bulk as described by Richomme et al. [7]. The shape of the hysteresis loops is unchanged, and the squareness is almost constant from 8 nm to higher coverage. For the 4 nm sample, the coercive field, H_C, in the easy axis was measured at 15 ± 2 Oe. For comparison, the H_C value for a similar iron layer deposited on Si (100) wafer under the same conditions was measured and found equal to 6 ± 1 Oe. This low value highlights the strong influence exerted by the interface on H_C for the lowest thickness sample. Its observed enhancement in Fe/InGaAs might be due to species diffusion and / or alloy formation as already reported by Haque et al. [11] for Nickel thin films deposited on Si and GaAs substrates.

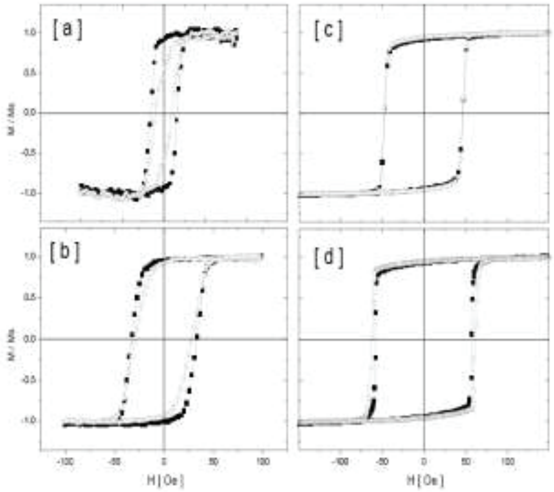

Fig. 2. Hysteresis loops for Fe films of different thicknesses (a: 4-, b: 8-, c: 20- and d: 60-nm) deposited on InGaAs (001). Both cycles (hard o, easy ■ axis) were perpendicularly acquired. Magnetization was normalized to M_S at saturation.

To elucidate this point, the samples were characterized by FMR, a technique highly sensitive to interfaces. Figure 3 illustrates the results of measurements and shows that, at high iron coverage (8 and 20 nm), only one peak is observed. It corresponds to the uniform mode due to the precession of layer magnetization around the applied field. The observed increase of the FMR peak width at higher iron thicknesses is correlated with the number of inhomogeneities in the layer due, for example, to the increase of the roughness. Decreasing the iron thickness makes appear several modes: the 4-nm spectrum shows a double peak structure. These structures are explained by a uniform mode in relation with the iron layer at low field and to an interface mode, respectively. They constitute evidence of the presence of two magnetic iron species at the interfaces. Furthermore, the shift towards high fields for the two peaks observed at 4 nm is explained by the reduction of the magnetization at low coverage.

As shown by the interfacial FMR mode and by the enhancement of H_C for the 4 nm sample, the interaction between the substrate and the firsts iron layers is a key parameter in the magnetic properties of the bilayer. To investigate the mobility of ion during the deposition and to thermal annealing at 250°C for 90 minutes in an Ultra high Vacuum chamber. Figure 4 presents the magnetization reversal of a 60-nm-thick Fe layer without and with thermal annealing. It shows a drastic change of the hysteresis loop generated by the thermal treatment; the coercivity value being about 650 Oe. Moreover, the cycle shape is no longer a square, and discontinuities appear. The observed decrease of the saturation magnetization by a factor of 3 suggests the creation of non magnetic alloys. Similarly, no ferromagnetic signature was observed after the thermal treatment of the 20 nm layer. These results prove the strong interdiffusion process in the innerlayers in contrast to the indication of only a weak interdiffusion below 350°C reported by Monterverde and al. [6].

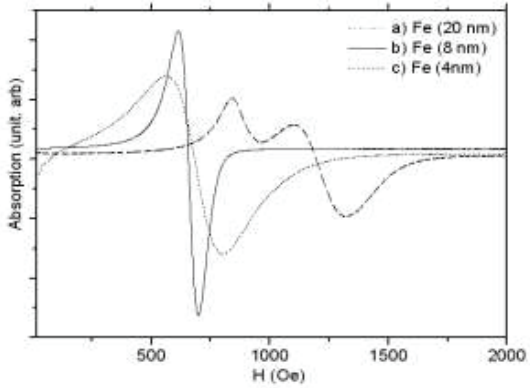

Fig. 3. FMR Spectra of Fe (t) / InGaAs (001): t = 20 nm (a); t = 8 nm (b) and t = 4 nm (c)

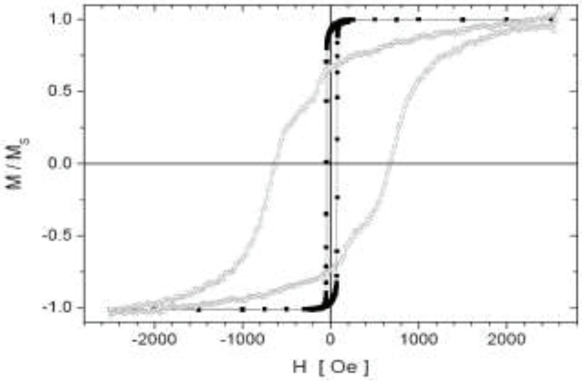

Fig. 4. Hysteresis loops for 60-nm-thick Fe layer deposited on InGaAs (001). The black and open dots correspond to data before and after annealing, respectively.

The 60-nm-thick iron sample was studied by XRD before and after annealing. The θ-2θ XRD pattern is dominated by the (002) and (004) reflections from the substrate InGaAs / InP (001). Figure 5 presents a restricted (40° to 48°) XRD pattern located around the Fe (110) peak located at 44.68°. One should note, after the annealing treatment, the disappearance of the peak associated to the Fe (110) plane. This is consistent with the study by Montverde *et al.* [6] who also reported on the disappearance of the Fe(110) at 400°C. However, in contrast to this report, one should note, here, after the annealing at low temperature, the lack of the additional peaks attributed to the presence of Indium and Fe_2As. This suggests a subsequent process consisting in a strong migration of atoms followed with crystallization at higher temperature. To gain more insight into the interdiffusion process, XPS measurements were made after different ion bombardment times, *i.e.* at different depths. To extract the depth profile composition, the Ga(3p), As(3p), P(2p), In ($3p_{3/2}$) and Fe(3p) spectra were fitted and subsequently corrected by their atomic

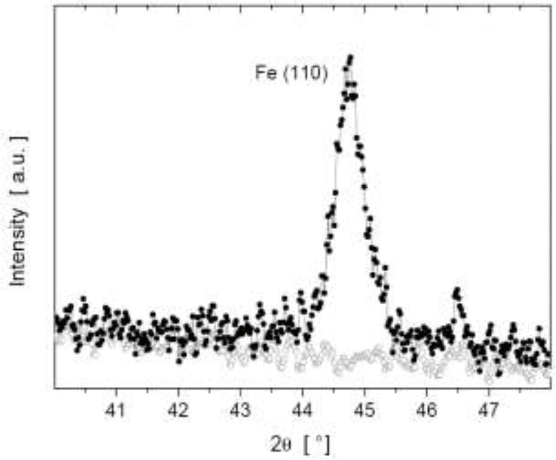

Fig. 5. θ/2θ diffraction pattern obtained for a 60-nm-thick Fe film deposited on InGaAs (001). The black and open dots correspond to data before and after annealing, respectively.

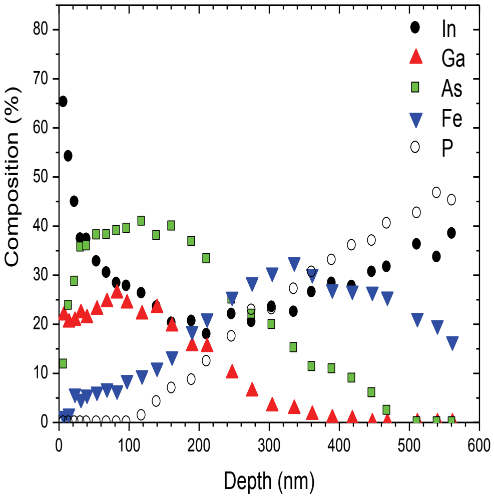

Fig. 6. XPS depth profile of Fe (60 nm) / $In_{0.5}Ga_{0.5}As$ (190 nm) / InP after *thermal annealing*

atomic sensitivity factors and sputtering yields, which were determined from SRIMS simulations. On Fig. 6, it is worth noting the strong interdiffusion of Iron within the whole system with a shallow maximum at about the 300-observation, at a depth of about 5 nm, of an outerlayer nm depth. This explains the reduction of the magnetization as well as the increase of the coercitive field. Besides, the region mainly composed of indium is indicative of the strong segregation of metallic indium at the surface under surface.under the UHV annealing treatment. Between 5 and 200 nm, Fig. 6 also shows an increase of the arsenic content and a decrease of the indium one, which lead to nearly the original InGaAs composition. The observation of P atoms released from the InP substrate from 120 nm indicates a migration towards the

4. CONCLUSION

The epitaxial growth of Fe films of different thicknesses (up to 60 nm) was achieved on InGaAs/InP(100) substrates by Molecular beam Epitaxy. FMR and VSM techniques provided evidence of the strong influence of the interface on the magnetic properties. Among them, a uniaxial magnetic anisotropy as well as an FMR interface mode were both revealed for the 4-nm Fe/InGaAs/InP(100) film. The thermal annealing of iron deposited at moderate temperature (250°C) in an Ultra High Vacuum chamber highlighted the strong mobility of atoms, which is a directly correlated dramatic change in magnetic properties. It results in both a diffusion of iron atoms within the multilayers and a strong segregation of indium atoms towards the surface. Consequently, even low temperature annealing has to be taken into account for technical applications.

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