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Structural and magnetic properties of Fe films grown on GaAs

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Abstract. — A study is presented of magnetic and structural properties of thin Fe films deposited on (111) GaAs substrates for thicknesses ranging from 65 to 120 nm. The films grown by ion-beam-sputtering exhibit both bcc phase and ferromagnetic structure. Transmission electron microscopy has allowed to determine the morphological growth stages and the grain size of the films. Both vibrating sample magnetometry and Mössbauer effect measurements show that the magnetic moments are lying in the film plane for every film. The average magnetization and $^{57}$Fe hyperfine field decrease with decreasing thickness which indicates the existence of two iron sites. An in-plane uniaxial anisotropy is found for the 65 nm thick film. Finally at film thickness approaching 120 nm, the film assumes the character of bulk $\alpha$-Fe.

1. Introduction.

The interest in Fe/GaAs interfaces arises for fundamental and technological reasons:

i) the interface strongly affects the magnetic properties of thin metallic films, possibly because of band hybridization and spin-orbit interaction [1]. The interfacial effects may be due to a perturbation of the crystal field caused by hybridization with the substrate electronic structure;

ii) iron thin films provide an opportunity for integrating magnetic devices with semiconductor circuits located on the same GaAs chip [2].

The properties of single-crystal films of iron grown by molecular-beam-epitaxy (MBE) or ion-beam-sputtering (IBS) on GaAs substrates have been already studied [3-6]. The focus, in these studies, was on the experimental conditions (substrate orientation and temperature, growth mode...) required to yield good quality magnetic single-crystal films of bcc Fe on fcc GaAs substrates. It was shown that the properties strongly depended on the growth conditions. Let us note that these first studies will be used for comparison.

In this paper, we report on a study of the influence of the interface on the magnetic and structural properties of the Fe/GaAs system. The deposition conditions were different from
2. Experimental procedure.

Iron films with thicknesses ranging from 65 to 120 nm were deposited using a sputtering system equipped with an ion-beam source operating in a vacuum chamber [7]. The (111)-oriented GaAs substrates were polished by mechanical abrasion and diamond paste; substrate orientation was confirmed by Laue patterns. The substrates were kept at room temperature during deposition by using a water cooling system. The starting pressure was $5 \times 10^{-5}$ Pa and then the pressure was maintained at $5 \times 10^{-3}$ Pa during the growth. The evaporation rate was monitored by a calibrated quartz oscillator and fixed at the value of 0.04 nm/s. The film thickness was measured using a DEKTAK 3030 profilometer.

Films deposited on GaAs substrates were used to prepare samples for VSM, CEMS, SIMS and TEM experiments. Concerning the cross-sectional TEM, the layers were glued together face-to-face in a sandwich structure and then cut in vertical sections which were first thinned by mechanical polishing to a thickness of about 80 µm. Final thinning to electron transparency was achieved by Ar$^+$ ion-beam milling. The observations were performed in a Jeol 200 CX electron microscope operating at 200 kV. As far as the CEMS technique is concerned, the 7.3 keV conversion and 5.6 keV Auger electron spectra were taken at 293 K using a source of 50 mCi $^{57}$Co in rhodium, a proportional counter with a He-5% CH$_4$ gas flow and a conventional Mössbauer spectrometer. The counter is earlier described in reference [8]. Magnetization, coercive force and hysteresis loops were obtained using a VSM as function of field strength. The VSM measurements were performed at the Laboratoire de Magnétisme et Matériaux Magnétiques, Bellevue (France). For studying concentration profiles, SIMS in the dynamic mode is a suitable technique, combining high detection sensitivity with good depth resolution. The SIMS analyses were carried out with a MIQ 156 system consisting of a duaplasmatron gun coupled to a quadrupole mass spectrometer and using 5 keV Xe$^+$ primary ions.

3. Results and discussion.

3.1 STRUCTURAL STATE. — The films were structurally analyzed using a combination of cross-sectional TEM and SIMS analyses. Figure 1 shows both a dark field image (a) and diffraction rings (b) observed for a 65 nm thick Fe film. In figure 1a, we can see from bottom to top four morphological parts. The first one corresponds to the GaAs substrate. The next part is a narrow band which stands between the substrate and the film; we suggest it is the interfacial zone, the thickness of which is close to 15 nm. The third part, 50 nm in thickness, exhibits a randomly oriented distribution of bcc Fe grains ranging in diameter between 4 and 13 nm. Finally, in the upper part, we can observe the remaining glue. The diffraction patterns shown in the figure 1b clearly indicates that the film exhibits the bcc structure. In the dark field image of the 120 nm Fe/GaAs sample (Fig. 2), we observe larger grains which have both a columnar structure and a specific orientation. The grain diameters are between 18 and 50 nm. The columnar grains grow normal to the GaAs surface. We can also notice that the interfacial zone appears to be more abrupt than in figure 1a. This is probably because it becomes too small a fraction to see.
Fig. 1. — Transmission electron microscopy dark field image (a) and diffraction pattern (b) of a 65 nm thick Fe film deposited on (111) GaAs substrate. A, B, C, D stand for substrate, interface, Fe-film and glue respectively.

Fig. 2. — Transmission electron microscopy dark field image of a 120 nm thick Fe film on (111) GaAs. The arrow indicates a $10 \times 30 \text{nm}^2$ grain.
The SIMS study was performed on a 80 nm thick Fe film deposited onto a (111) GaAs substrate. From preliminary bar graph spectra, the main sputtered species were identified as Fe, As and Ga. Only C and O atoms are significant surface contaminants. Their amounts were determined by complementary XPS and found to be less than 8 at% and 2.8 at%, respectively. The Ga and As concentration profiles (Fig. 3) show the diffusion of these species into Fe during the deposition. In fact, the Fe profile shows that Fe diffuses into the substrate as well but at a slower rate. The result is that As, Ga and Fe species coexist through a depth of about 10 nm. This interface width is defined as the average interval where the intensities drop from 84 % to 16 % of maximum signal [9].

![Diagram](image)

**Fig. 3.** — Gallium (a), arsenic (b) and iron (c) SIMS profiles *versus* depth for a 80 nm thick Fe film on (111) GaAs. Note the As and Ga out-diffusion into Fe.

### 3.2 Magnetic properties.

#### 3.2.1 Global properties. — Magnetic hysteresis curves of 1.2 × 1.0 cm² rectangular samples were measured by the VSM at RT. Curves for 65 and 120 nm thick Fe films were taken with the magnetic field applied along the [11\(\bar{2}\)], [\(\bar{1}\)0] and [111] substrate axes, respectively (Fig. 4a). Figure 4b shows the hysteresis loop taken from a 120 nm thick film with the field applied parallel to the [1\(\bar{1}\)0] substrate axis. The loop shows a rectangular shape with a small coercive field of 8.2 Oe. No significant difference was detected between the two in-plane [11\(\bar{2}\)] and [\(\bar{1}\)0] directions. The VSM results are summarized in table I.

It is quite obvious that the magnetization easy axis lies in the film plane. This spin orientation was observed for the two Fe films and is also confirmed by CEMS measurements. The magnetization of the thicker Fe film is equal to that for bulk Fe. Thus the magnetic properties of this film appear not to be significantly influenced by the interface (see Tab. I).

For the 65 nm thick Fe film, however, in addition to in-plane anisotropy, we also find that the magnetization exhibits a uniaxial anisotropy in the film plane, which is a good indicator for the film-substrate interaction [1]. The value of this anisotropy was not measured here, but an order of magnitude can be found in reference [5]. The saturation magnetization is less than that of bulk iron, the average magnetization being only 80 % of the bulk so that 20 % of the Fe
PROPERTIES OF Fe FILMS GROWN ON GaAs

(111) GaAs Plane

(a)

[111] [110]

(b)

Fig. 4. — a) Crystallographic directions for magnetic field application. b) Magnetic hysteresis loop taken at 293 K from a 120 nm Fe film with magnetic field applied parallel to the [110] direction.

Table I. — Magnetic properties at room temperature for both 120 and 65 nm thick Fe films with field applied parallel to (a) [110] and (b) [112] directions. \( H_c \), \( M_s \) and \( t \) stand for coercive force, saturation magnetization and film thickness respectively.

<table>
<thead>
<tr>
<th></th>
<th>120</th>
<th>65</th>
</tr>
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<tbody>
<tr>
<td>( H_c ) (Oe)</td>
<td>8.2(a) - 7.6(b)</td>
<td>10.3(a) - 5.9(b)</td>
</tr>
<tr>
<td>( M_s ) (emu/g)</td>
<td>217.6(a) - 217.9(b)</td>
<td>173.6(a) - 173.2(b)</td>
</tr>
<tr>
<td>( M_s / \text{bulk } M_s )</td>
<td>1.00</td>
<td>0.80</td>
</tr>
</tbody>
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atoms appear to be magnetically inactive. It is reasonable to attribute this to Fe atoms at the interface.

In the case of Fe grown by MBE on (001) GaAs, an in-plane uniaxial anisotropy has been observed and was suggested to be induced by the tetrahedral Ga(As)-Fe bonds at the (100)
interface. The decreasing magnetization was attributed to the formation of antiferromagnetic Fe₄As microcrystals in the α-Fe film resulting from As out-diffusion [5].

3.2.2 Local properties. — The CEM spectrum of the 120 nm thick Fe film (Fig. 5a) exhibits six lines, indicating a clear ferromagnetic state of bcc Fe as expected. The full width at half height is however slightly more (= 10%) than the peak width of bulk iron. This difference can be attributed to the fluctuations of the interatomic Fe distances in the layers. The Lorentzian fitting procedure led to the following parameters: the average hyperfine field is 330 kOe, as in bulk iron, the mean angle between the field and the film normal is 90° which corresponds to magnetic moments lying in the film plane. This latter result is in good agreement with the VSM measurements. The spectrum collected from the 65 nm Fe/GaAs sample (Fig. 5b) also exhibits a six line pattern, indicating that the magnetic moments are also lying in the film plane.

Fig. 5. — a) CEM spectrum taken at 293 K from a 120 nm Fe film. b) CEM spectrum taken at 293 K from a 65 nm Fe film. Note the asymmetry in outer lines due to the interfacial contribution.
However, the spectrum cannot be fitted by a single sextet. A residual sextet representing about 22% of the area has to be introduced to explain the asymmetry of the peaks. This is attributed to the coexistence of two different Fe species: Fe atoms located at the interface and usual bcc Fe in the layer. The hyperfine field of the residual sextet is close to 310 kOe. Since As and Ga atoms are magnetically inactive, this value can be attributed, as in Fe-Si alloys [10], to Fe atoms involved in a bcc phase and surrounded by 7 Fe atoms and 1 As or Ga nearest neighbour.

4. Conclusion.

Structural and magnetic properties of thin α-Fe films ion-beam sputtered onto (111) GaAs have been studied by a combination of TEM, SIMS, VSM and CEMS. We have found the following:

i) the thinner film, 65 nm in thickness, exhibits two Fe atom species and a uniaxial inplane anisotropy, clearly indicating an interaction between the film and the substrate. An interfacial layer, resulting essentially from the As(Ga) out-diffusion, is formed;

ii) in contrast the properties of the thicker films, 120 nm in thickness, appear not to be significantly influenced by the interfacial zone, this may be a consequence of the relatively large film thickness. The films assume nearly the character of bulk α-Fe. The evolution of Fe/GaAs interfaces as a function of annealing temperature is at present under investigation.

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References