Out of plane component of the magnetization of sputtered $Fe_{72}Ga_{28}$ layers

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Abstract

In this paper we present an investigation about the out of plane component of the magnetization of Ga-rich sputtered FeGa thin films. To study this magnetic component, we have used magnetometric measurements and magnetic force microscopy combined with a structural characterization by means of x-ray diffractometry. For a more profound analysis, we have examined samples in both, as-grown and annealed state. The out of plane component of the magnetization promotes a magnetic ripple observed by magnetic force microscopy in all the studied samples. To quantitatively monitor the out of plane component of the magnetization, we have used the ratio between the magnetic remanence and the maximum magnetization (M_r/M_{max}), i.e. the squareness, measured in the perpendicular hysteresis loops. The experimental results indicate that the out of plane component of the magnetization is reduced upon annealing at a moderate temperature of 400 °C. The experimental results can be understood considering that phase coexistence is the most likely origin for the observed magnetic ripple.

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1. Introduction

In the last years, there is a huge interest in the metallic FeGa system due to the discovery in 2000 of a large magnetostriction in these free rare-earth alloys [1-2]. Many of the published works have been devoted to the understanding of the origin of this enhanced magnetostriction in contrast with pure Fe [3-12]. It is remarkable that the largest deformation in Fe can be reached in the [100] direction with a (3/2)λ_{100} value of 22 ppm, whereas in FeGa alloys it can be obtained 430 ppm in that same direction [5]. In addition, there is a number of investigations that shown the strong impact that structural characteristics have on the magnetic properties of these alloys [4, 12-18]. Very important are the studies about the origin of the magnetic anisotropy in either out of or in the sample plane of FeGa thin films [3, 9, 16-17, 19-22]. It seems that Ga-pairs together with a tetragonal distortion are related to the in-plane magnetic anisotropy [12, 16-18].

Works about the out of plane (OOP) component of the magnetization have reported both, stripes and magnetic ripple [19-24]. Magnetic ripple has been observed in electrodeposited FeGa films with a Ga content around the first peak of magnetostriction [22], and FeGa layers deposited by molecular beam epitaxy (MBE) on MgO with Ga above 20 at. % [21], and on sputtered $Fe_{73}Ga_{27}$ layers deposited on Si<100> [24]. Therefore, regardless of the growth method, substrate, and composition there seems to be an OOP component of the magnetization in FeGa films. In fact, MBE FeGa layers on GaAs substrates with Ga ranging between 14 and 29 at. % show an enhanced perpendicular magnetic anisotropy presenting stripes above a critical thickness [19]. Therefore, the magnitude of this OOP component is different from work to work being difficult to correlate it with the structural properties, the Ga content, or the deposition technique since, for example, MBE FeGa films with Ga > 20 at. % can exhibit stripe domains [19-20] and magnetic ripple [21]. Largest values of magnetostriction are obtained in the so-called second peak of magnetostriction (Ga ca. 28 at. %) [5], and therefore, it is important to systematically explore that Ga-rich range. We have already performed a detailed investigation about the influence of the sputtering conditions and thickness on the structural properties of Ga-rich FeGa thin films [12, 16-18]. We have observed that Ga-pairs and tetragonal distortion are related to the in-plane magnetic anisotropy. However, magnetostriction seems to be related to medium range order [12].

The aim of this work is to look for a possible explanation for the OOP component of the magnetization in Ga-rich FeGa thin films. We have investigated layers deposited by sputtering that exhibit magnetic ripple, in the as-grown and annealed case. We have explored which of the hypothesis proposed so far can better explain our experimental results [21, 23-24]. Among them, phase coexistence seems to be the most probable origin for magnetic ripple. This can explain why it has been observed in FeGa obtained by different growth methods being difficult to correlate with the structural properties.

2. Experimental techniques

All the growth processes were carried out by DC magnetron sputtering in oblique incidence with an angle between the vapor beam and the perpendicular to the sample plane of about 25° and a distance of 9 cm between targets and substrate [16-18, 25]. Oblique deposition can induce columnar structure, but for technical limitations we can only grow FeGa in this configuration. However, since the sputtering process is always performed in the same geometry, the effect of the oblique deposition is the same in all the samples, and it is possible to extract conclusions from their comparison. $Fe_{72}Ga_{28}$ layers with a thickness ranging from 25 to 150 nm were grown in the ballistic regime at room temperature on glass substrates capped with 20-nm thick Mo layers. It was used a $Fe_{72}Ga_{28}$ target with a diameter of 5 cm and a thickness of 2 mm, and the sputtering deposition was performed using an Ar pressure of 3×10^{-3} mbar and a growth power of 90

W, in all cases. In order to avoid effects related to the target ageing, the samples were consecutively deposited. FeGa layers were capped with a 20-nm thick Mo to avoid oxidation. Mo buffers and cappings were grown with a power of 90 W at an Ar pressure of 3×10^{-3} mbar. Samples were post-growth annealed in Ar atmosphere during 1 hour at 400 °C. From here on, samples will be denoted with a number that specify their thickness in nanometers, followed by -AG or -Ann to indicate their as-grown or annealed state. For example: 150-AG indicate a 150 nm thick FeGa layer in its as-grown state.

X-ray diffractometry (XRD) in the Bragg-Brentano configuration was performed in a Philips X'Pert MPD using the Cu K_{α} wavelength (1.54056 Å). At room temperature, in-plane and OOP hysteresis loops were carried out in a vibrating sample magnetometer (VSM). The data were normalized to the highest magnetization value of the corresponding loop obtained for the maximum applied magnetic field of 12.5 kOe. Magnetic Force Microscopy (MFM) images were recorded by a Digital Instruments Nanoscope IIIa, using the phase detection mode, i.e., monitoring the cantilever's phase of oscillation while the magnetic tip was scanning the sample surface at a distance of 40 nm on average (lift mode). The MFM measurements were performed at remanence after an in-plane magnetic field of 15 kOe was applied. With the MFM technique, we can only obtain qualitative information about the out of plane component of the magnetization, because the colour contrast was not calibrated. However, MFM images provide an insight into the magnetic properties of the studied layers, as it will be shown in the next section.

3. Results and discussion

A sample will show perpendicular magnetic anisotropy if two conditions are fulfilled: i) it is magnetically isotropic in the sample plane, and ii) the OOP direction is an easy axis in comparison to any direction in the sample plane. Therefore, either in their as-grown or annealed state, none of the studied samples present perpendicular magnetic anisotropy (Figure 1). Nevertheless, the VSM hysteresis loops show a perpendicular component of the magnetization that is also observed in the MFM images (Figure 2), as it is explained below.

In all cases, the roughness of the samples is rather low. The height variation observed in the topography is around 6 nm, that is much lower than the height of 40 nm used during MFM imaging. Therefore, we do not expect much influence of the topology on the magnetic contrast measured with the MFM. Apart from that, there is a Mo layer of 20 nm between the FeGa and the tip of the MFM. Stripe domains are formed in systems with low perpendicular magnetic anisotropy above a critical thickness due to alternating out of plane orientation of the magnetization [26-27]. We have not observed stripes in either the as-grown or the annealed samples (Figure 2), and therefore, the perpendicular magnetic anisotropy is rather small as indicated by the VSM hysteresis loops (Figure 1). The MFM contrast present in our samples is a corrugation known as magnetic ripple that has been previously observed in other polycrystalline magnetic systems, including FeGa thin films [21-22]. This corrugation reflects the competition between different contributions, and its origin is an inhomogeneous magnetization due to fluctuations of the magnetic anisotropy [21, 28-29]. Therefore, a random magnetic anisotropy contribution is able to break the uniform orientation of the magnetization giving raise to the irregular magnetic contrast as that presented in figure 2.

In FeGa films, the origin of random anisotropy can be due to several factors [21]: coexistence of crystal phases, inhomogeneous distribution of both internal strain and Ga–Ga next nearest neighbor pairs, and interface magnetic anisotropy due to Fe–O bonds. The latter can be ruled out in this work since FeGa is sandwiched between Mo buffer and capping layers to avoid oxidation either from air exposure or oxygen coming from the glass substrate. In fact, x-ray absorption near edge structure (XANES) has ruled out oxidation in our samples [12, 16-18]. Thanks to XANES, we have also found that as-grown sputtered Ga-rich FeGa thin films are comprised of three different structural phases: A2, B2, and $D0_3$ [16-18]. Thus, it is very likely that phase coexistence can be the reason to explain the magnetic ripple in our samples.

From the OOP hysteresis loops it can be inferred the M_r/M_{max} ratio, also denoted as squareness, between the magnetic remanence (M_r) and the magnetization at the maximum applied magnetic field (M_{max}). The squareness can be used to quantitatively monitored the evolution of the OOP component of the magnetization since the higher the M_r/M_{max} ratio, the larger the OOP component of the magnetization. In Table I it is summarized the dependence of the squareness with the thickness. Although the dependence is quite negligible, it seems there is a small increase of the squareness with the thickness. In permalloy it was found that the columnar growth promoted the formation of stripes above a thickness of 250 nm [30]. The almost negligible dependence between thickness and squareness makes improbable that columnar growth is the origin for the magnetic ripple observed in the studied FeGa films with a thickness between 25 and 150 nm. However, in sputtered FeGa alloys we have observed that the phase ordering can vary as the thickness is increased [17]. Then, the increase of the squareness with the thickness can be an indication of the dependence between the magnetic ripple and the phase mixture.

In agreement with previous reports [16-17], XRD patterns of these samples show two main diffraction peaks related to the (110) reflections of Mo and FeGa (Fig. 3). It is obtained a <110> texture in both, Mo and FeGa, because sputtering promotes the stacking of the most dense planes that in the bcc structure correspond to (110) planes [31]. To analyze the evolution of the crystalline texture we have used a parameter that considers the ratio between the intensity of the FeGa(110) ($I_{FeGa(110)}$) and Mo(110) ($I_{Mo(110)}$) diffraction peaks defined as $I_{FeGa(110)}/I_{Mo(110)}$. This parameter increases with the FeGa thickness indicating the enhancement of the <110> texture as the layers become thicker (Table I). $I_{FeGa(110)}/I_{Mo(110)}$ is not modified by the thermal treatment pointing out that the crystalline texture cannot be affected by post-growth thermal treatments at 400 °C (Table I). For each thickness, $I_{FeGa(110)}/I_{Mo(110)}$ is the same for as-grown and annealed samples, whereas the M_r/M_{max} ratio is higher in the as-grown. Thus, it seems that the OOP component of the magnetization is not related to the crystalline texture. This is also in agreement with the fact that we observe a similar magnetic ripple in our <110> texture FeGa samples that in <100> MBE FeGa films by Begué *et al.* [21].

The correlation between the squareness and the thermal treatment is clear (table I). For each thickness, M_r/M_{max} is always higher in the as-grown in comparison to the annealed samples. Typically, a thermal treatment promotes an enhancement of the crystalline quality. We can use the Scherrer's formula to have a first approximation about the crystallite size (D), and to check the influence of the thermal treatment on the structural disorder:

$$D = \frac{Q\lambda}{\beta cos\theta} \qquad (1)$$

where Q is a shape factor taken as 0.9, λ is the radiation wavelength (1.5406 Å), β is the full width at half maximum of the diffraction peak -FeGa(110) in this case-, and θ is the Bragg angle for that peak. As it is shown in table I, for each thickness D increases with the thermal treatment revealing a reduction of the crystal disorder with the annealing. Therefore, this indicates that the OOP magnetization increases with the structural disorder, because for each thickness the squareness is higher in the as-grown layers.

We have also calculated the lattice parameter (*a*) from the FeGa(110) diffraction peak considering a cubic structure (Table 1). As-grown films have a larger lattice parameter than annealed probably because of a more disorder state.

We can clearly observe two magnetization reversals in the perpendicular hysteresis loops of the as-grown samples that reflect two magnetic phases (figure 1). This double contribution is subtle for the annealed layers, indicating that although not completely reduced, the presence of at least two magnetic phases is reduced upon annealing. The absence of this double contribution in the in-plane hysteresis loops can be explained by the fact that in our samples, the OOP component of the magnetization is rather small. The magnetization is almost in the sample plane being impossible to observe any effect related to the OOP component of the magnetization in the in-plane hysteresis loops. There seems to be a correlation between the OOP component of the magnetization and the existence of at least two magnetic phases, since the two magnetization reversals are only clearly present in the samples with the highest squareness, i.e. the as-grown layers.

In sputtered FeGa thin films, the in-plane magnetic anisotropy has been related to Ga-pairs and tetragonal distortion [12, 16-18], and therefore, a different mechanism is needed for the development of the OOP component of the magnetization. As commented above, we have not found indications of oxidation in none of the studied samples, and considering all the previous discussion about our experimental results, from the possible origins proposed by Begué et al. for random magnetic anisotropy to eventually promote magnetic ripple in FeGa thin films [21], the coexistence of different crystal phases is the most likely origin in this study for the OOP component of the magnetization.

The main conclusions obtained from our experimental results are: i) the decrease of the squareness of the perpendicular hysteresis loops with the thermal treatment, ii) the existence of at least two magnetic phases in the as-grown layers that are partially reduced upon annealing, iii) the presence of magnetic ripple instead of stripes that indicate a random magnetic anisotropy contribution, and iv) decrease of structural disorder upon the annealing. Therefore, from the possible origins for the magnetic ripple to exist discussed by Begué et al. [21], our experimental results indicate that phase mixture is the most probable.

As we have already commented, the A2, B2 and $D0_3$ phase mixture have been observed by other groups and also by us in FeGa [5, 12, 16-18, 21, 24, 32-34], and this can explain the existence of an OOP component of the magnetization in the as-grown samples. Theoretical calculations based on atomic density field theory have shown that, rather from direct nucleation, the formation of $D0_3$ occurs through a cascade of congruent orderings from A2, following the sequence A2 → B2 → $D0_3$ [33]. This is in accordance with the experimental evidence that the ordered phase B2 segregate from an A2 matrix [6]. Thus, when the samples are annealed, the formation of ordered phases (B2 and $D0_3$) is promoted [33, 17-18], the structural disorder is reduced as indicated by the increase of the crystallite size (Table I), and the squareness of the perpendicular hysteresis loops is decreased in the annealed state (Table I). The behavior of the hysteresis loops points in this same direction. The phase coexistence promotes different magnetic phases clearly observed in the as-grown samples (Figure 1). Upon annealing, this phase coexistence is decreased, although not completely reduced, and the two magnetizations reversals are subtle but still present in the annealed samples (Figure 1). In fact, this can explain why magnetic ripple is observed not only in as-grown, but also in annealed layers (Figure 2), because phase mixture is still present in the annealed case. Our results are in agreement with the work of Begué et al. based on layers deposited by MBE [21], and in a recent paper by Chelvane and coworkers it has also been reported that phase coexistence is behind the magnetic ripple in Ga-rich FeGa thin films [24]. Actually, phase mixture can be one of the reasons because of the lack of a clear explanation for the OOP in FeGa alloys since the proportion of crystal phases is extremely dependent on the growth process.

4. Conclusions

We have studied the evolution of the OOP component of the magnetization in sputtered Ga-rich FeGa thin films. It has been crucial the investigation of both, as-grown and annealed samples, to achieve a better correlation between magnetic and structural properties. The squareness between the magnetic remanence and the maximum magnetization measured in the perpendicular hysteresis loops has been used to quantitatively monitored the OOP component of the magnetization. Its reduction upon annealing indicates that a certain degree of structural disorder is necessary for the magnetic ripple to appear. The experimental results can be understood considering that phase coexistence is the most likely origin for the OOP component of the magnetization that produces magnetic ripple in Ga-rich FeGa thin films. This can explain why it has been observed in FeGa layers obtained by different growth methods being difficult to correlate with the structural properties.

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Table I. Summary of the OOP M_r/M_{max} ratio extracted from the perpendicular magnetic hysteresis loops, the $I_{FeGa(110)}/I_{Mo(110)}$ intensity ratio between the (110) diffraction peaks for FeGa ($I_{FeGa(110)}$) and Mo ($I_{Mo(110)}$), and the lattice parameter (a).

Sample	OOP M_r/M_{max} (arb. units)	$I_{FeGa(110)}/I_{Mo(110)}$ (arb. units)	a (nm)	D (nm)
25-AG	0.4	0.13	0.292	5
50-AG	0.3	0.22	0.291	6
100-AG	0.3	0.42	0.290	9
150-AG	0.6	0.80	0.290	10
25-Ann	0.1	0.14	0.289	5
50-Ann	0.1	0.21	0.289	9
100-Ann	0.1	0.42	0.288	11
150-Ann	0.2	0.80	0.288	12

Figure captions

Figure 1. In plane (■) and OOP (○) hysteresis loops for FeGa layers with different thickness as-grown: (a) 25 nm, (b) 150 nm, and annealed: (c) 25 nm, and (d) 150 nm.

Figure 2. MFM images taken as remanence for as-grown FeGa layers with different thickness: (a) 25 nm, (b) 150 nm, and annealed (c) 25 nm, and (d) 150 nm.

Figure 3. X-ray diffraction patterns of the FeGa thin films studied in this work. Curves are vertically shifted for clarity. Black curves are used for the as-grown, and red curves for the annealed. The thickness of each pair of samples (as-grown and annealed) is indicated in the graph.

Figure 1.

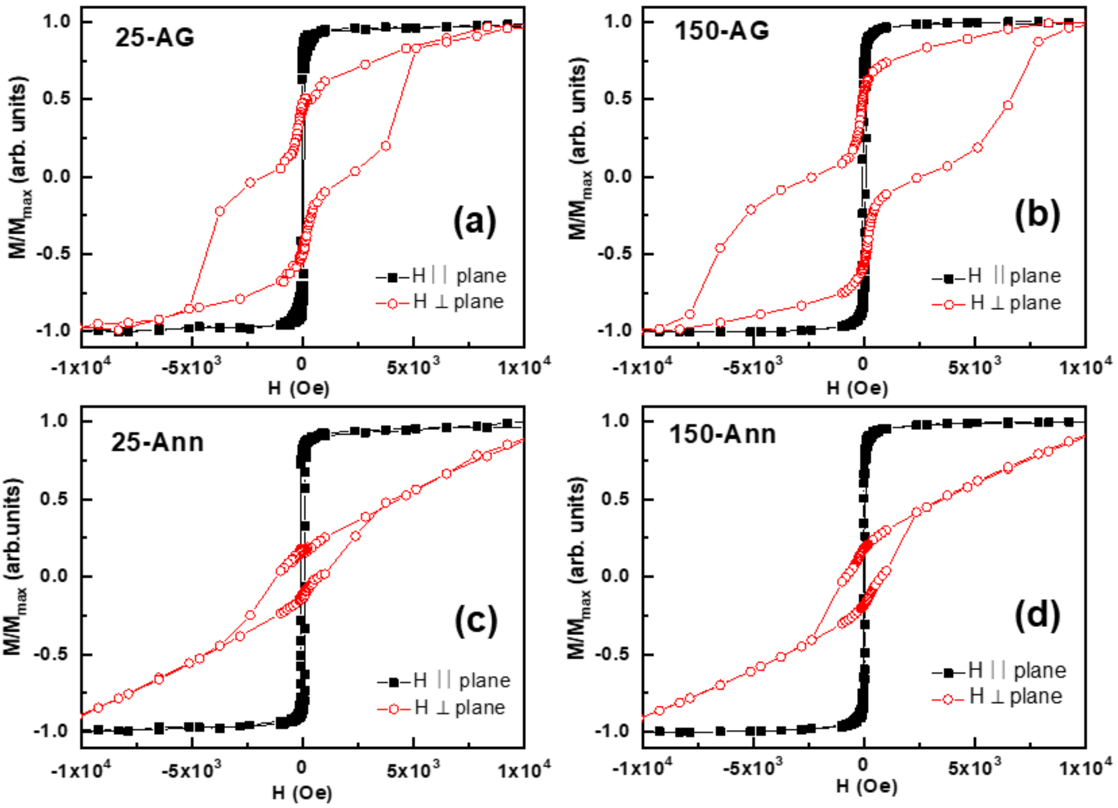

25-AG
(a)
150-AG
(b)
25-Ann
(c)
150-Ann
(d)
H || plane
H ⊥ plane
M/M$_{max}$ (arb. units)
H (Oe)
1.0
0.5
0.0
-0.5
-1.0
-1x10^4
-5x10^3
0
5x10^3
1x10^4

Figure 2

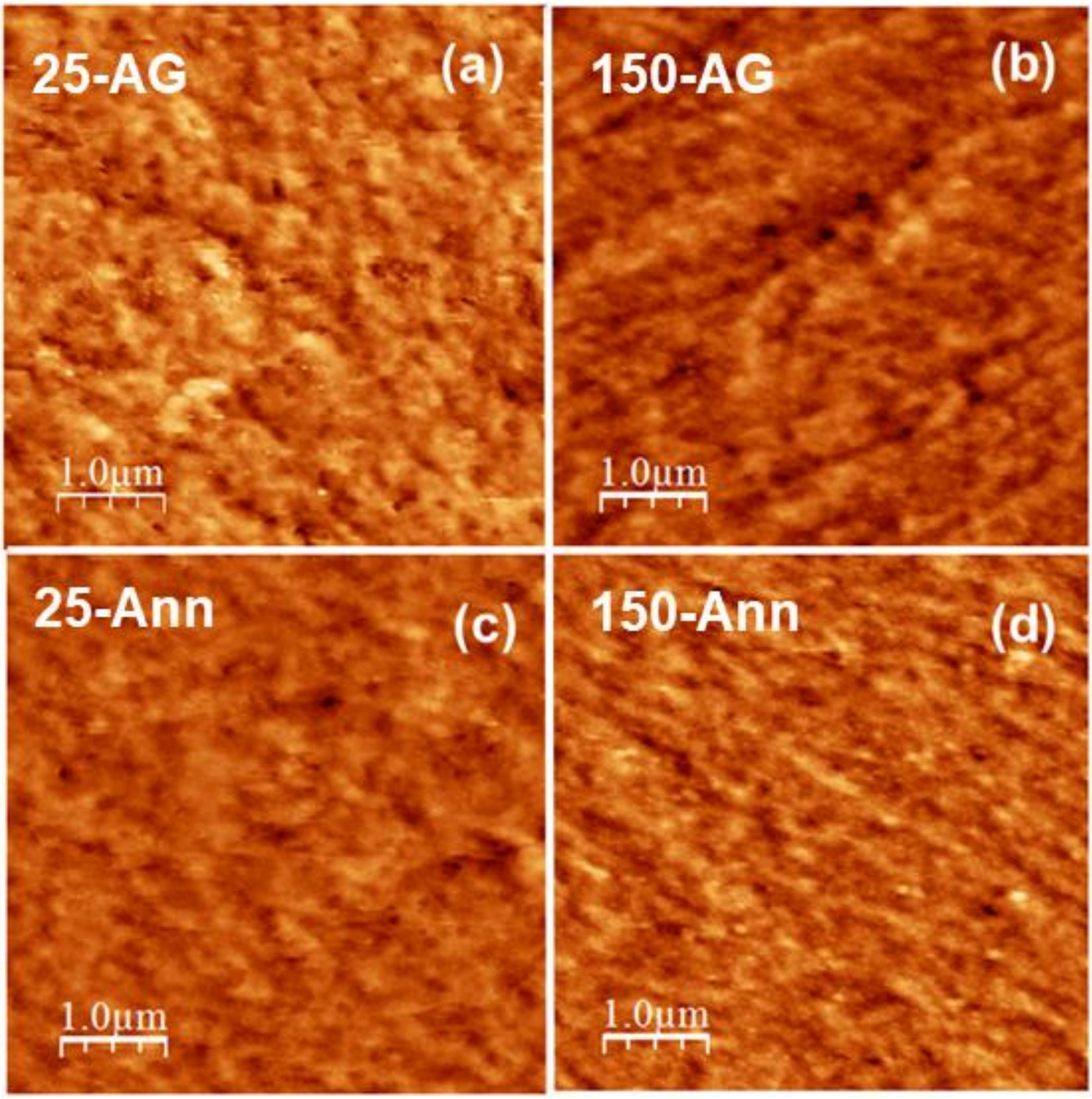
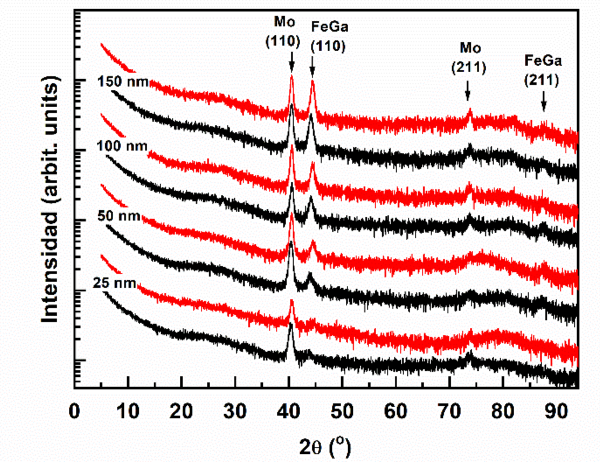


Figure 3


Mo
(110)
FeGa
(110)
Mo
(211)
FeGa
(211)
150 nm
100 nm
50 nm
25 nm
Intensidad (arbit. units)
0
10
20
30
40
50
60
70
80
90
2θ (°)