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Citation: [Journal of Applied Physics](#) **119**, 245304 (2016); doi: 10.1063/1.4954872

View online: <http://dx.doi.org/10.1063/1.4954872>

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Effect of low-temperature post-growth annealing on anisotropic strain in epitaxial Fe layers deposited on GaAs(001)

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(Received 13 April 2016; accepted 14 June 2016; published online 27 June 2016)

We study the effect of low-temperature post growth annealing on the Fe layer in an epitaxial Fe/GaAs(001) heterojunction. High resolution X-ray diffraction and X-ray reflectivity were used to probe the Fe layer before and after annealing. No change in morphological features like annealing induced intermixing and thickness variation of the Fe layer are observed. However, annealing leads to increase in the compressive strain and improves isotropy of the ferromagnetic layer as revealed by measuring both lateral and out-of-plane lattice components. *Published by AIP Publishing.*
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I. INTRODUCTION

A thin film Fe on bulk GaAs is widely used as a ferromagnet (FM)/Semiconductor (SC) model system for spin injection studies using electrical and optical techniques.^{1–3} One advantage of this system is the ease with which Fe layers can be epitaxially deposited on GaAs⁴ due to a relatively small lattice mismatch of 1.4% between substrate and twice the bulk Fe lattice constants. Another advantage is the high Curie temperature exhibited by the Fe films⁵ which allows the study of spin injection even at room temperature.¹

The Fe/GaAs heterojunction is very sensitive to post-growth annealing. Fleet *et al.* observed a reduction of intermixing at the interface as a result of low-temperature post-growth annealing.⁶ Other reports also indicate a strong influence of post-growth annealing on spin injection efficiency.^{7–9} We also observed a similar effect,¹⁰ where the non-local spin signal was observed only after a soft post-annealing of the structures to 200 °C.

To date, several studies were attempted to correlate post growth annealing and spin injection by analyzing the atomic structure of the FM/SC interface. Zega *et al.* proposed a model of ordered and coherently intermixed interface of Fe and As atoms.¹¹ Fleet *et al.* reported coexistence of intermixed and abrupt interfaces.⁷ On the other hand, Lebeau *et al.* reported an interface consisting of a partially occupied Fe layer.¹² The nature of interface as discussed in these studies will indeed influence the strain state of the epitaxial layers. Therefore, by studying structural and strain properties of the Fe layer before and after annealing, it might be possible to reveal mechanisms controlling spin injection efficiency.

The existing reports on annealing studies in Fe/GaAs heterojunctions using diffraction techniques are conducted either on very thick or ultrathin Fe films.^{13–16} Some of the studies were performed to investigate effect of annealing on atomic ordering at the interface.^{7,11} Since accumulated stress present in a Fe layer depends on the thickness of the

film,^{17,18} we focused on film thicknesses which are typically used for spin injection experiments in a non-local geometry. In this study, X-ray reflectivity (XRR) is combined with High Resolution X ray Diffraction (HRXRD) to observe the strain evolution in Fe layers. We also discuss the observed anisotropic compressive strain and its variation with low-temperature post-growth annealing.

II. EXPERIMENTAL CONDITIONS

The Fe/GaAs heterojunctions were prepared in a multi-chamber MBE system consisting of a Riber C21 III/V semiconductor chamber and a metal deposition chamber connected by an ultra-high vacuum channel. All the heterojunction layers were deposited on undoped GaAs (001) wafers. Before deposition, the wafers were degassed at 400 °C for 1 h in a degas station. Afterwards, the wafers were transported to the semiconductor growth chamber where the native oxide was removed at 540 °C under background As flux. An undoped GaAs buffer layer was deposited followed by a 300 nm of n-doped GaAs channel with the carrier density of $5 \times 10^{16} \text{ cm}^{-3}$ (n^+). Later, a 30 nm transition layer ($n^+ \rightarrow n^{++}$) was grown and terminated with 15 nm of highly doped GaAs layer (n^{++} , $3 \times 10^{18} \text{ cm}^{-3}$). An As terminated (2×4) reconstructed surface was observed by Reflection High Energy Electron Diffraction (RHEED) before transferring the sample into the metal MBE chamber. In the metal chamber, a 5 nm Fe layer was deposited at the growth rate of 0.4 nm/min. The structure was finalized by deposition of 15 nm Au layer as a protective cover with a growth rate of 0.2 nm/min. Both the metal layers were deposited at room temperature. The sample layout designed for spin transport experiments¹⁰ with nominal thicknesses is shown in Fig. 1.

The as-deposited sample was studied using Atomic Force Microscopy (AFM), XRR, and HRXRD. The laboratory X-ray measurements were performed at the energy of characteristic Cu K α radiation ($E = 8048 \text{ eV}$) using an X'Pert-PRO four-circle diffractometer equipped with a Ge (220) 3-bounce analyzer and a homemade incident

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FIG. 1. Layer sequence of the MBE-grown Ferromagnet/Semiconductor spin injection device.

collimator. Afterwards, the same sample was soft annealed to 200 °C for 10 min under nitrogen atmosphere. The sample before annealing will be referred in further text as “as-deposited” and the sample after annealing as “annealed.” We applied the same techniques as mentioned before to study the annealed sample. An additional HRXRD measurement was performed on the annealed sample at the P08 beamline of Petra III at DESY using a photon energy of 8319 eV.

III. RESULTS AND DISCUSSION

A. Morphology of Fe/GaAs heterointerface

Uniformity of the top Au protective layer is critical for preventing the Fe layer from oxidation. The layer uniformity will be reflected in the surface morphology which we accessed using AFM in tapping mode. The AFM image of the as-deposited sample in Fig. 2(a) reveals the presence of small island like features on the surface. The total volume of

FIG. 2. $2 \times 2 \mu\text{m}^2$ AFM images of (a) as-deposited and (b) annealed samples. (c) Comparison of height distribution functions of the samples.

these islands corresponds to an equivalent Au layer thickness of 2.2 nm. This equivalent thickness is significantly smaller than the nominally deposited layer of 15 nm. This implies that the Au layer is a combination of small islands on a thick continuous layer.

An AFM image of annealed sample as shown in Fig. 2(b) reveals that in annealing process the islands transform into smaller features with sharper edges. The equivalent thickness of these islands decreases to 0.7 nm which indicates that these islands partially merge into the continuous Au layer during annealing.

Height distribution functions (HDF) of both samples are shown in Fig. 2(c). A logarithmic scale is used to enhance details of the distribution. The peak at 0 nm is assigned to the surface of the continuous Au Layer. The shape of the HDFs also supports the suggestion that the surface is a combination of islands and a continuous Au layer. Sharp decrease in the counts below -1 nm indicates the absence of pinholes which would expose the ferromagnetic material. The broad tail on the right is attributed to the islands with a statistical height distribution on the surface of the Au layer. Comparison of the HDF plots before and after annealing reveals a decrease in the height and volume of the islands after annealing. The observed changes in Au layer topography are later used to explain changes in the reflectivity curves.

Information about the interfaces is obtained from X-ray reflectivity measurements. The difference in electron density of different heterojunction layers leads to reflection of X-rays at the interfaces of the layer stack. Interference of the reflected X-rays when measured as a function of incident angle results in an oscillatory pattern of reflected intensity (as seen in Fig. 3) called Kiessig fringes. The scans are performed in θ - 2θ (incident-reflected angle) geometry, and the incident angle is transformed into the out of plane reciprocal scattering vector q_z , with $q_z = 2k \sin \theta$, where k is the X-ray wave vector. The intensity and period of the oscillations are then used to model thickness (t) and interface roughness (r) of individual layers in the heterojunction. The fitting was

FIG. 3. X-ray reflectivity profiles of as-deposited (red line) and annealed sample (blue line). Upper inset: comparison between experimental and simulated profile of as-deposited sample. Lower inset: Layout used for simulation of reflectivity.

FIG. 4. Reciprocal space maps measured on the as-deposited sample at the (a) 004 Bragg reflex of GaAs and (b) 224 Bragg reflex of GaAs. q_z and q_y are along the $[110]$ and $[-110]$ crystal directions, respectively. The colour scale represents diffraction intensity in arbitrary units. The regions in black rectangles highlight Fe reflexes. The white rectangle in (b) represents the analyzer streak.

performed using a recursive Parratt algorithm¹⁹ for converging the experimental and simulated profiles.

The X-ray reflectivity profiles of both as-deposited and annealed samples are shown in Fig. 3. The bottom inset shows the sample layout used for the fitting. The upper inset shows the comparison between experimental and simulated reflectivity patterns of the as-deposited sample indicating good agreement. The best fit for as-deposited sample yields following thicknesses: $t_{Fe} = 4.7 \pm 0.06$ nm, $t_{Au} = 14.3 \pm 0.04$ nm for the Fe and Au layers, respectively. The roughness of Fe layer and substrate are: $r_{Fe} = 0.32 \pm 0.04$ nm and $r_{substrate} = 0.12 \pm 0.02$ nm, respectively.

Surprisingly, the best fit for the annealed sample does not indicate a change in either thickness or roughness of the Fe layer. The layer seems to remain unaltered after annealing. Clearly, there is only little contrast between the reflectivity profiles of both samples (as shown in Fig. 3), and the difference is mainly due to the change in the surface roughness of the Au layer (as-deposited: $r_{Au} = 0.13 \pm 0.01$ nm, annealed: $r_{Au} = 0.10 \pm 0.01$ nm). This difference can be qualitatively correlated to the change in root mean square roughness (rms) of the continuous Au layer ($rms_{as-deposited}$: 0.56 nm, $rms_{annealed}$: 0.33 nm) measured with AFM. We note that one may not expect the roughness values derived from XRR and AFM to coincide. The difference arises from many factors like different ranges of lateral spatial frequencies covered by the two techniques²⁰ and also, due to the fact that, in the XRR simulation algorithm diffuse scattering from rough surface is not considered.

Previous studies observed the presence of few monolayers of binary and ternary Fe-Ga-As compounds at the interface either immediately after deposition^{21,22} or their formation after annealing.^{9,13,14} Although we cannot completely exclude the existence of these compounds in our samples, we can conclude from XRR data that no continuous layer of these compounds is present in the studied heterojunctions. Incorporation of even few monolayers of such compounds into our reflectivity model led to significant disagreement between experimental and simulated profiles. Within the resolution of X-ray reflectivity measurements, we observe no formation of new compounds at the Fe/GaAs

interface. Yet isolated islands of new compounds may exist at the interface.

B. Crystallinity of Fe layer

HRXRD was used to access the strain state and crystallinity of the Fe layers. We collected reciprocal space maps (RSM) around different reflexes of the Fe layer and GaAs substrate which revealed information about the crystalline nature, orientation, and strain of the epitaxial layers. RSM's were collected in two different geometries called symmetric and asymmetric scans. In the former geometry, the diffraction vector is normal to the surface allowing measurement of the out-of plane lattice parameter a and layer inclination in $[001]$ crystal direction. In the latter geometry, the diffraction vector of the measured reflex is tilted by an angle with respect to the surface normal allowing evaluation of lateral distortions in $[110]$ and $[-110]$ crystal directions by measuring the inter-planar spacing d .

A bulk cubic system like Fe should have the same lattice parameter in all three crystal directions. But in a thin film, due to epitaxial relation with the substrate, the lattice parameters slightly differ in normal and lateral direction. To observe these changes, we have extensively studied reciprocal space maps around (004) (shown in Fig. 4(a)), (224) (shown in Fig. 4(b)), and (-224) reflexes of the GaAs substrate. The scans were large enough so that the corresponding (002), (112), and (-112) reflexes of Fe are also observed. By analyzing both symmetric (004) and asymmetric scans ((224), (-224)), we obtain the deformation of elementary cells in the crystalline layers. This approach complements previous studies^{18,23} which use only the out-of-plane lattice constant to evaluate strain in thicker Fe films.

The RSM's are shown as colour maps of diffraction intensity plotted as a function of reciprocal space coordinates (q_z , q_y). In both maps, the most intense and sharp peak belongs to GaAs, while the elongated streak highlighted by a black rectangle arises from the Fe layer. The reflexes of Fe are elongated because of the small layer thickness. The intensity tails highlighted by a white rectangle around the GaAs peak in the asymmetrical scan are a typical analyzer streak.

FIG. 5. Comparison between diffraction profiles of annealed sample obtained using lab source and synchrotron source.

Symmetric maps are projected to q_z axis to obtain line profiles along q_z . (224) and (-224) maps are projected to q_y axis to obtain line profiles along [110] and $[-110]$ crystal directions of GaAs, respectively. Fig. 5 shows the comparison between line profiles of annealed sample extracted from a symmetric map and line scan obtained using synchrotron radiation. Peaks from the line profiles were fitted with pseudo-Voigt functions to obtain the exact q_z and q_y values which are later used to calculate out-of-plane lattice parameters and inter-planar spacing, respectively. The results of fitting are summarized in Tables I and II.

From Table I, it can be seen that the inter-planar spacing d for as-deposited sample is less than the expected bulk value of Fe (2.026 Å). Such an in-plane contraction is expected for the epitaxial structure²⁴ and has been observed before using polarization-dependent X-ray absorption fine-structure spectroscopy.²⁵ As a result of annealing, it shrinks further towards the bulk value of GaAs (1.998 Å). To allow this compression, the out-of-plane lattice should expand, and that is exactly what we observe in Table II where the out-of-plane lattice constant is found to be greater than the bulk Fe value (2.866 Å) and increases further after annealing. In other words, the Fe film is inherently strained in the as-deposited sample and this compressive strain increases after annealing. The increase might be caused by improved atomic level ordering at the interface after post-growth annealing.^{7,11} Compressive strain resulting from the matching between in-plane lattice spacing of substrate and Fe layer also supports our inference that there is no continuous intermediate layer of Fe-Ga-As compounds at the interface because their existence would result in tensile strain of Fe layer as observed by Filipe *et al.*²¹

TABLE I. Inter-planar spacing of as-deposited and annealed sample.

Sample	q_y (Å ⁻¹)	d (Å)
As-deposited 110	3.1276	2.008 ± 0.001
As-deposited -110	3.1398	2.001 ± 0.001
Annealed 110	3.1488	1.995 ± 0.002
Annealed -110	3.1468	1.996 ± 0.002

TABLE II. Out-of-plane lattice parameters of as-deposited and annealed sample.

Sample	q_z (Å ⁻¹)	a (Å)
As-deposited 004	4.333	2.900 ± 0.001
Annealed 004	4.319	2.909 ± 0.001
Annealed (beamline) 004	4.318	2.910 ± 0.003

It can be further noted that the inter-planar spacing for the as-deposited sample differs in both [110] and $[-110]$ directions which indicates anisotropy in the strain distribution which is also expected from first principle calculations.²⁴ Similar behavior was already observed in the size of Fe coherent domains on GaAs¹⁶ and also in strain relaxation of the Fe film on InAs²⁶ and was attributed to preferential bonding of Fe to As dimers present on the reconstructed surface.²⁷ We believe the anisotropy observed in our sample also stems from the same effect. Structural anisotropy of the interface is expected to influence the magnetic anisotropy of the Fe film.¹⁶ Recently, Tivakornsasithorn *et al.* proposed that the structural anisotropy is also responsible for widely reported uniaxial anisotropy in thin Fe/GaAs films.²⁸

The lateral strain anisotropy seems to disappear after annealing. The in-plane components in both crystal directions have the same inter-planar spacing with compressive strain still being preserved. So we speculate that the observed reduction in the structural anisotropy will also cause a reduction of the uniaxial magnetic anisotropy of Fe film. Similar behavior was reported in previous studies where the interplay between uniaxial and cubic magnetic anisotropy of Fe layer is found to be affected by structural anisotropy resulting from Ge²⁹ and ZnSe²⁸ buffer layers deposited between the GaAs substrate and the Fe layer.

As evident from Figure 5 and Table II, we got very consistent data from the lab diffractometer and synchrotron measurements, which validate the resolution and accuracy of our measurements. In an ideal Fe/GaAs heterojunction, four Fe unit cells are epitaxially connected to one GaAs unit cell. A fully relaxed iron layer would show lattice mismatch of 1.4%. In our as-deposited sample, difference between the lateral components of the Fe and GaAs unit cells calculated from the inter-planar spacing d was found to be 0.5% and becomes more isotropic after annealing decreasing to the value of 0.12%.

IV. CONCLUSIONS

In summary, we studied the effect of post growth annealing on epitaxial Fe layers deposited on GaAs (001). We did not observe any significant change in morphology of the Fe layer. From our XRR results, we also can exclude the formation of a continuous interface layer. On the other hand, post growth annealing is found to result in a significant variation in the strain of the Fe layer. The as-deposited sample was found to be compressively strained. This strain was observed to increase further after annealing and to become isotropic. The observed strain variation makes us believe that interplanar spacing of Fe unit cell shrinks to match that

of the GaAs unit cell. Perfectly coordinated interfaces assumed in some simulations^{24,30–32} may be extended to include the annealing induced change in the inter-planar spacing of Fe unit cell. We speculate that increase in the matching of inter-planar spacing of Fe and GaAs at the interface after annealing might contribute to enhanced spin injection efficiency. This might be due to a change in the Fe-As local hybridization configuration resulting from the strain variation.^{7,31}

ACKNOWLEDGMENTS

We are grateful to Professor Viktor Asadchikov and Professor Vitalii Dugaev for their advice and valuable discussions. The authors also like to acknowledge the funding from Joachim Hertz Stiftung foundation and from Bundesministerium für Bildung und Forschung via the European Project Era.Net.Rus “Nanostructured MgO tunnel barrier for metal-semiconductor spin injection (SpinBar).”

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