Smoothening of ultrathin Fe films grown on GaAs(100) observed by scanning tunneling microscopy and Brillouin light scattering

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We report on an *in situ* scanning tunneling microscopy study of the growth of Fe on the GaAs(100)-pseudo- 4×6 reconstructed surface in the thickness range of 1–4 ML (monolayers). We observe that at 1 ML, the Fe islands have nucleated on the As dimer rows, and that subsequent growth is primarily directed along the $[0\bar{1}1]$ direction. Initially, the islands are well separated, but as more Fe is deposited, the islands start to coalesce in both the [011] and $[0\bar{1}1]$ directions. Percolation probably occurs between 2 and 3 ML. Scanning tunneling microscopy studies of a nominally 2 ML thick film over a period of 30 h reveal that the islands relax primarily growing in the $[0\bar{1}1]$ direction, reducing the roughness of the film. The smoothening of the film is also corroborated by Brillouin light scattering measurements on slightly thicker films of 4.25 and 6.2 ML as evidenced by an increase in the spin-wave frequency and a decrease of the linewidth over a period of 11 h.

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I. INTRODUCTION

Fe on GaAs(100) is a widely studied model system for the growth of magnetic films and spin transport across an interface between a ferromagnetic metal and a semiconductor (SC)-see Ref. 1 and references contained therein. This heterostructure is currently of interest for spintronics due to the Schottky barrier that forms at the interface, enabling spin transport through the structure via electron tunneling.²⁻⁵ Initially, however, the system was studied because Fe grows epitaxially on various GaAs surfaces with a small lattice mismatch ($\sim 1.3\%$). It was quickly realized that for thin films, there was a uniaxial magnetic anisotropy (UMA) present with the easy axis aligned along the [011] direction, rather than along the cubic easy axes corresponding to the $\langle 001 \rangle$ directions. Since this behavior of the UMA is the same for Fe grown on different GaAs reconstructions, it suggests that the interface bonding plays a considerable role in determining the direction of the UMA.¹ Another property of the system is the late onset of ferromagnetism at 3.8 ML (monolayers) at room temperature (RT). High temperature growth (above 175 °C) or growth on As rich surfaces can lead to $Fe_rGa_{1-r}As_v$ compounds being formed;⁶ these may lead to reduced or even zero magnetic moments of the Fe creating so-called magnetically "dead" layers. At RT, FerAs, are present at the interface due to intermixing; from the work we reported in Ref. 7, we estimate that this is limited to 1.5 ML. It has been suggested that Fe also grows via a threedimensional (3D) growth mode, which prevents long range magnetic order.^{1,8} However, the role these mechanisms play in the evolution of magnetism in Fe/GaAs is beyond the scope of the current study.

We have previously reported on the growth of Fe on the GaAs(100)-2 \times 6 surface for a nominal thickness of less than 1 ML.⁷ In this paper, we demonstrated that Fe islands nucleate on the As dimer rows, probably as Fe₂As clusters. These clusters, which are estimated to be 1.5 ML thick, act as seed crystals for the subsequent growth of bcc Fe. These islands largely grow by diffusion along the As dimer rows, and in the submonolayer regime, they rarely bridge across the Ga

dimer rows. Hence, a nominally 1 ML thick film is not a continuous layer but is made of Fe islands, and structural percolation must occur at a greater thickness, probably in the thickness range of 1–4 ML, where superparamagnetism⁹ and ferromagnetism (at both room¹⁰ and low¹¹ temperatures) are observed.

Here, we present the results of scanning tunneling microscopy (STM) studies of the time and thickness dependent structure of Fe films in the thickness range of 1–4 ML. In the time dependent study, we look for structural evidence that the as-deposited surface is metastable and hence the film may relax by changing the surface morphology. If this occurs, the surface may either become rougher or smoother which may change the magnetic properties. Thus, in addition to the STM measurements, we have also performed the Brillouin light scattering (BLS) experiments to probe the spinwave modes of ferromagnetic films.

II. EXPERIMENTAL DETAILS

The measurements were carried out *in situ* in an ultrahigh vacuum (UHV) system with a base pressure of 2×10^{-10} mbar. Thin films were grown by molecular beam epitaxy (MBE) using an e-beam evaporator. The films were structurally characterized using a VG rear view low energy electron diffraction (LEED) system and a Burleigh UHV STM (in a separate chamber). Magnetic characterization was done using *in situ* magneto-optical Kerr effect (MOKE) and BLS setups. The sample was transferred between the growth and STM chambers without breaking the UHV.

A commercial Si *n*-doped GaAs(100) wafer $(N_D=10^{18} \text{ cm}^{-3})$ used during these experiments had been prepared in another SC UHV MBE chamber beforehand. A buffer layer with thickness $t \approx 0.5 \ \mu\text{m}$ of homoepitaxial GaAs was grown on the wafer to provide an as smooth as possible surface and subsequently As capped. After transferring the sample to the MBE chamber, the As cap was removed by annealing at $T \approx 400$ °C for 15 min. Afterwards, the temperature was increased to $T \approx 550$ °C and maintained



FIG. 1. (Color online) $50 \times 30 \text{ nm}^2$ filled state STM images of films with nominal Fe thicknesses of (a) 1 ML, (b) 2 ML, (c) 3 ML, and (d) 4 ML. These were taken within 5 h of deposition, with the exception of the 2 ML sample (b) which was taken 30 h after deposition, but the inset of (b) shows a $20 \times 13 \text{ nm}^2$ image of the asdeposited surface. (e) and (f) show line profiles from the lines along the (e) [011] and [011] directions indicated in (d). Several of the images show more than one GaAs terrace, which are separated by the expected 0.15 nm.

there for another 30 min to obtain a clean and ordered surface before the Fe growth. This treatment leads to the pseudo- 4×6 reconstructed GaAs surface,^{7,12–14} which is made up of two phases the 4×2 and 2×6 .

The Fe was deposited at RT at a constant rate of 1 ML/min. The deposition rate was monitored with the help of a quartz microbalance. The surface reconstruction of the substrate was examined by LEED. The STM was run in constant current mode with a dc-etched tungsten tip. The STM images were acquired with currents in the range of 0.4-1.2 nA and sample biases in the range of -0.4 to -0.1 V, hence tunneling from the filled states of the sample occurred, which are dominated by the contribution from Fe states. The average Fe island sizes and areas were determined for all coverages by using the two-dimensional autocorrelation function for a certain amount of images (not all shown here). In situ BLS measurements were performed in the Voigt geometry with the magnetic field applied perpendicular to the light beam, and the optical setup was located in a separate room, isolated from the mechanical vibrations of the chamber. Further details of the BLS setup can be found in Refs 9 and 15.

III. RESULTS

For our GaAs(100) substrates (prepared as described above), it was found by STM and LEED that we had the pseudo-4×6 reconstruction and the 2×6 phase was the dominant phase, although small patches of the 4×2 phase were observed. The 2×6 reconstruction is Ga rich but As terminated, with As dimer rows aligned parallel to the $[0\overline{1}1]$ direction and separated by 2.4 nm in the [011] direction. This appears to play an important part in determining the growth of the Fe on this surface. For more details of the substrate and Fe nucleation, see our previous work in Ref. 7.

The STM data in this study were gathered primarily from one substrate and the values of the thickness refer to the nominal thickness assuming a continuous film. The 1–4 ML films were grown in 1 ML steps within a few hours of each other except between 2 and 3 ML when the film was allowed to relax for 40 h before the 3 ML film was grown. STM measurements on the 2 ML film were made within 5 h and after 30 h. Thus, there are three regimes that we need to consider for the increase in thicknesses: 1–2 ML, 2–4 ML, and the relaxation at 2 ML. At all points, the sample was kept under UHV conditions and at RT; we will address the possibility of oxidation later in the paper.

In the following sections, we repeatedly talk about Fe islands, although it is probable that the islands are not made of pure Fe. It is known that the GaAs segregates and that Ga and As diffuse to the surface of Fe films¹⁶ for coverages exceeding 2–3 ML, so any islands will probably be made up of Fe and As. Using STM, we cannot tell the difference between Fe, Ga, and As atoms within an island, but we believe that this terminology is appropriate as the majority of the islands should still consist of Fe atoms.

Figure 1 shows four $50 \times 30 \text{ nm}^2$ STM images which present the evolution of Fe films with thicknesses increasing from 1 to 4 ML. With the exception of Fig. 1(b), all the images were taken within 5 h after deposition. In Fig. 1(b), the image of 2 ML of Fe was taken after 30 h. While one cannot clearly see the substrate reconstruction in any of the images, as expected since the deposition of Fe is expected to destroy the reconstruction,¹⁷ it is apparent that while the aspect ratios of individual islands change for different thick-



FIG. 2. Island size along (a) [011], (b) $[0\bar{1}1]$, (c) area, (d) height, (e) density, and (f) surface coverage against nominal thickness for 0.3–4 ML of Fe. The black squares denote data taken within 5 h of deposition; the gray squares denote data taken after the 2 ML film had been allowed to relax. The dashed lines show a fit to curves of the form $x \sim t_{\rm Fe}^n$ using the data taken before the film was allowed to relax.

nesses, the islands still tend to be arranged in rows along the $[0\overline{1}1]$ direction. From images (c) and (d) (3 and 4 ML), it can be seen that the islands have stopped growing along the As dimers and that they have become more circular in shape at 3 ML and even elongated along the [011] direction at 4 ML, implying a change in the diffusion properties of the Fe atoms on the surface.

In situ MOKE measurements were performed on the films at RT, and the evolution of the magnetization was as reported previously.⁹ Only the 4 ML sample demonstrated ferromagnetism, and it showed the expected uniaxial anisotropy with the easy axis along the [011] direction,¹¹ although as will be discussed later, a 3.4 ML film showed superparamagnetism.

Figure 2 shows the trends in the average island size along the [011] and $[0\overline{1}1]$ directions, the island area, island height, island density, and surface coverage (defined as area × density) for the thickness range of 0.3–4 ML. The data points from 0.3–1 ML were taken from Ref. 7. The black squares denote measurements on the as-deposited sample, whereas the gray squares correspond to measurements made after the 2 ML film had been allowed to relax for 30 h. The dashed lines are fits to a power law,

$$x \sim t_{\rm Fe}^n,$$
 (1)

where x is the data (e.g., size, area, etc.), t_{Fe} is the nominal Fe thickness, and n is an exponent, which (using scaling arguments) may give information about the diffusion properties and the growth mode. The data used to fit the curve were the unrelaxed data, i.e., that from 0.3–2 ML, and then the curve was extrapolated to higher thicknesses.

From Fig. 2(a), it can be seen that the average island size grows very slowly along the [011] direction remaining close to the width of the GaAs reconstruction (2.4 nm). Along this direction, the island size follows a power law, with n

=0.20±0.02 for the 0.3–2 ML, and continues to be a good fit for the 2–4 ML thickness regime with little change due to the relaxation at 2 ML. This slow growth is in contrast to the behavior along the [011] direction, shown in Fig. 2(b), where the island size peaks at around 2 ML and then for the thickness range of 2–4 ML falls again. While, for the thickness range of 0.3–2 ML, the island size roughly follows a power law with n=0.36±0.14, there is a substantial deviation for the 4 ML film and a large difference between the unrelaxed and the relaxed films at 2 ML. The evolution of the island area shown in Fig. 2(c) reflects the changes in the island size along the [011] direction.

The island height, shown in Fig. 2(d), remains fairly constant in the range of 0.3–2 ML and falls as the film relaxes, then by 4 ML, it has increased back to the value observed at 2 ML, so the films tend to be rougher after deposition. This suggests that the same numbers of atomic layers are being observed by the STM tip. The island density, shown in Fig. 2(e), falls up to 2 ML and then increases for thicker films. The coverage c (defined as area \times density) shown in Fig. 2(f) has a very strong power law behavior of up to 3 ML, where $n=0.52\pm0.05$, and it shows little change between the unrelaxed and the relaxed films at 2 ML, unlike both the island density and area. At 4 ML, the coverage falls to about 60%, which is interpreted as small islands growing on top of larger unseen islands, which are merging and approaching percolation, and hence the start of Fe on Fe growth, rather than the break up of the previously existing islands.

Having observed changes in the average island properties with time at 2 ML, we wanted to investigate what happened to the distribution of island sizes. Hence, Figs. 3 and 4 show histograms of the island size and height taken within 5 and 30 h after the deposition of 2 ML of Fe. During the time between measurements, the sample was kept in the STM under UHV conditions and at RT.

From Fig. 3, it is apparent that the behavior of the island sizes along (a) [011] and (b) $[0\overline{1}1]$ is different. Along the [011] direction, the change in the island size distribution with time is quite small, whereas there is a significant change along the $[0\overline{1}1]$ direction. In the $[0\overline{1}1]$ direction, the islands have become larger and have a broader distribution of sizes.

As can be seen from the histogram of island heights presented in Fig. 4, initially there is a wide range of heights from 0.1 to 0.5 nm (1–3 ML) with an average height of 0.2 nm. However, after 30 h, the height distribution has become more uniform and favors a value of 0.15 nm (\approx 1 ML); thus, the sample has become smoother. Although, there are still a few islands with a height of several monolayers.

Encouraged by these results, we wanted to answer the question of whether or not this relaxation process has any influence on the magnetic properties of the Fe films. We approached this through two methods of MOKE in the superparamagnetic regime and BLS in the ferromagnetic regime. The MOKE loops shown in Fig. 5 were taken on a 3.4 ML film 18 h apart the first being immediately postdeposition. To estimate the magnetic volume of the island, a Langevin function was fitted to the raw data using the method described in Ref. 9. These yield values of 21.5 ± 0.8 nm³ for the film immediately after deposition and 21.1 ± 1 nm³ after



FIG. 3. (Color online) Histograms of island sizes along (a) [011] and (b) $[0\overline{1}1]$. Both histograms show data for two different times after deposition of a 2 ML Fe film: black circles indicate results from less than 5 h after deposition and red triangles indicate results from approximately 30 h after deposition. The lines are guides for the eye.

19 h, so within errors, there is no change in the magnetic volume of the islands. We note that the magnetic volume of the island is significantly larger than the volume that can be estimated by the values from the average island area and height at 4 ML as observed by STM (see Fig. 1). This is to



FIG. 4. (Color online) Histogram of island heights, as in Fig. 3. Black circles indicate results from less than 5 h after deposition and red triangles indicate results from approximately 30 h after deposition. The lines are guides for the eye.



FIG. 5. (Color online) Normalized MOKE loops from a 3.4 ML taken immediately after deposition (thin line) and after 18 h (dashed line); the data taken immediately after deposition were fitted to the Langevin function shown (thick line). No changes are seen within experimental errors during the course of 19 h.

be expected as the STM information is coming primary from the top morphology of the islands. Furthermore, it is well known from the literature that the superparamagneticferromagnetic transition in this system is, in fact, a percolation phase transition, a critical phenomenon characterized by the abrupt onset of interisland exchange coupling during film coalesecence at $t_c \sim 3.7$ ML.^{11,18} One would therefore expect a steep increase in the magnetic volume in a very narrow range of thicknesses just before t_c .

Two ferromagnetic films with nominal thicknesses of 4.25 and 6.2 ML were measured with *in situ* BLS directly after deposition and about 11 h later. Typical BLS spectra for the 4.25 ML film with the magnetic field applied along the [011] direction (magnetic easy axis) are shown in Fig. 6(a). Due to the reduced Fe thickness, only Damon-Eshbach surface modes¹⁹ were observed, exhibiting the characteristic Stokes– anti-Stokes intensity asymmetry. In the measurements taken after 11 h, the spin-wave peaks are shifted to higher frequencies ν and the peak linewidth $\Delta \nu$ (full width at half maximum) is decreased. Qualitatively, the same time dependence was observed for the 6.2 ML sample [Figs. 6(b) and 6(c)]. Both the changes in ν and $\Delta \nu$ can be related to the smoothening of the magnetic film, as will be discussed in detail in the following section.

IV. DISCUSSION

From the STM images shown in Fig. 1 and the data in Fig. 2, it is clear that the substrate reconstruction has a role in determining the island structure. Despite the fact that the actual reconstruction is destroyed in the early stages of Fe growth,⁷ the surface is reminiscent of the reconstruction. The average island size along the [011] direction is constrained to the separation between the As dimers for the 2×6 reconstruction, even after 4 ML of Fe have been deposited,



FIG. 6. (a) BLS spectra for a 4.25 ML Fe film, taken immediately (solid line) and 11 h (dotted line) after deposition with the magnetic field applied along the easy axis ([011]). The curves are offset for clarity. (b) The change in frequency ν with time for films with thicknesses 4.25 ML (\bullet) and 6.2 ML (\Box) and (c) the change in linewidth $\Delta \nu$ for the same thicknesses.

whereas the average size along the $[0\overline{1}1]$ direction changes dramatically.

In the early stages of growth (i.e., <1 ML of Fe), diffusion and island growth are clearly preferred along the $[0\overline{1}1]$ direction. It appears from Fig. 2 that this remains true up to 2-3 ML. Beyond this, the islands stop growing along the [011] direction and start to reduce in size. This suggests that the diffusion properties of the Fe surface have changed. From an inspection of the images in Fig. 1, it can be seen that for 3 and 4 ML, there are more islands spreading across several As dimer rows than in the 1 and 2 ML images. Further evidence that the growth and/or diffusion properties change around 3 ML is that, as seen in Fig. 2, the island density increases, while the island area and the nominal coverage decrease. This suggests that the changes are caused by the onset of Fe on Fe growth, which at greater thicknesses will lead to square islands.²⁰ The extra crosslinking between neighboring As dimer rows is the start of the film coalescing. This is consistent with the low temperature magnetic data of Bensch et al.,¹¹ which extrapolate that the Fe film first becomes ferromagnetic at 2.5 ML at 10 K. This together with the observation of superparamagnetism for films with a nominal thickness below 3.8 ML of Fe,9 we believe that percolation occurs gradually over a thickness range of 2.5-4 ML.

The time dependence we have seen could have several causes, including diffusion and oxidation. We believe that oxidation can be ruled out as a major cause for several reasons. Firstly, the oxygen partial pressure in the chamber is below our detectable limit of 1×10^{-11} torr; this corresponds to an estimate of 1 l of oxygen present after 30 h. This is

expected to have little effect on the magnetization of Fe,²¹ and oxide growth does not occur for this dose of oxygen.²² Secondly, the magnetization of the islands in the superparamagnetic regime should be very sensitive to the presence of oxygen. So given that we see from the STM data that the islands are changing in shape without changing in volume, this suggests that any change in the magnetic volume determined by MOKE should be due to oxidation. That there is no significant change in volume determined from the MOKE loops suggests that oxidation is not very significant.

The as grown surface morphology is unlikely to be the equilibrium surface, and it is therefore likely to change as diffusion occurs. Thus, we have looked at the surface morphology at different times at a nominal thickness of 2 ML. The inset of Fig. 1(b) shows an image of a 2 ML film taken within 5 h of deposition, whereas the main image was taken after 30 h. It can clearly be seen that there are considerable changes over 30 h. The islands have grown along the [011] direction. Now, considering the properties of the average island from Fig. 2, the island density, while the island area has increased in such a way that our measure of the coverage has remained constant, and the height are reduced. From the distribution of islands sizes (Fig. 3), it can be seen that smaller islands have disappeared and that the larger islands have grown. Most islands after 30 h have a height of around 1 ML (Fig. 4), whereas the as-deposited surface is far rougher with many islands having a height of 2 or 3 ML. Thus, it appears that the film has relaxed by atoms moving from the top of the islands to the sides thereby creating larger islands. This suggests that there is an energy barrier for the process of descending to a lower layer at the step edge, such as the Ehrlich-Schwoebel (ES) barrier.²⁰

The ES barrier is known to exist for homoepitaxial²³ and heteroepitaxial^{20,23} growth of Fe. These different modes give rise to different exponents in Eq. (1). Along the [011] direction, the islands grow steadily according to a power law with an exponent of 0.20 ± 0.02 that within errors is the same as found for homoepitaxial Fe on Fe growth by Stoscio *et al.*²³ However, for the [011] direction, there is a poorer fit to a power law with an exponent of 0.36 ± 0.14 . Intriguingly, within the large errors, the exponent would fit the case where strain leads to bonds breaking, which tends to occur with heteroepitaxial growth as found by Thürmer *et al.*²⁰ and Stroscio *et al.*²³ Looking at the scaling arguments, this anisotropic behavior arises due to differences in the chemical bonding of the Fe, which changes the diffusion properties along the [011] and [011] directions.

The [011] direction from our STM measurements shows far more changes than the [011] direction (see Figs. 2 and 3) and seems to be a preferred direction for diffusion. This is likely to be because of the two related factors: chemical and topological. It is known that Fe prefers to bond to itself or As rather than Ga,⁸ leading to the Fe growing along the As dimers in the [011] direction rather than crossing the Ga dimers, despite the extra strain that appears to exist for growth in this direction. However, the As dimers are raised up above the Ga dimers, so the Ga dimers form a trench between the As dimers.¹² This leads to it being easier to diffuse along the trenches than across them. It is interesting to note that for 1 ML of Fe on the 4×2 reconstruction, these same factors combine to produce islands orientated along the [011] direction, but the easy axis of the UMA remains along the [011] direction.¹⁰ It would be intriguing to investigate the scaling parameters for the growth of Fe on the 4×2 reconstruction. Since if the inequivalence persisted, it would suggest that the strain leading to the breaking of Fe-Fe bonds may be the cause of the UMA.

In a recent study on the influence of film roughness on the magnetic properties of thin fcc Co(001) films grown on Cu,²⁴ a significant reduction in cubic anisotropy was found for the rougher films. When measuring along an easy cubic axis, this led to a decrease in spin-wave frequency. Furthermore, a sizable linewidth broadening was seen. Both observations were attributed to the magnetic configuration of the Co films with the magnetization **M** following the sample morphology resulting in a tilt away from the easy axis. This tilt leads to an increase in the total anisotropy energy of the magnetic layer, as compared to the case of a perfectly flat film where all spins are aligned in plane, effectively rendering the easy axis "less easy" and causing a decrease in spin-wave frequency. Furthermore, local variations of the alignment of M (and, consequently, the magnetic anisotropy energy) due to film roughness give rise to additional allowed spin-wave modes, resulting in an increase in spin-wave peak linewidth. While, in the experiments discussed in Ref. 24, the Co roughness was induced by growth on a rough substrate (leading to an "undulating" Co film shape), the situation is slightly different in the present case. The film roughness observed with STM in our studies is predominantly due to the 3D growth mode of Fe on the GaAs(100)-2×6 surface. However, the main arguments given in Ref. 24. still apply, given the small thicknesses of the Fe layers investigated (just above the percolation threshold). It has been shown theoretically²⁵ that magnetic film roughness will affect both the cubic and the uniaxial anisotropies. The magnitude of this effect was found to depend purely on the roughness parameter $4\sigma/\xi$ (i.e., the average slope of the film roughness), with σ the roughness amplitude and ξ the roughness correlation length (see Fig. 1). From our STM measurements [Figs. 2(a) and 2(d)], we get $4\sigma/\xi=0.12\pm0.03$ for the 4 ML thick film within 5 h of deposition, which is of the same order as the value found for the rough Co films studied in Ref. 25 (0.28±0.03). Thus, the high degree of film roughness present in the ultrathin Fe layers under investigation is expected to alter the orientation of **M**, leading to a tilting of spins away from the easy axis, similar to the case of the Co/Cu system discussed above. Therefore, the measured changes in ν and $\Delta \nu$ are in good agreement with the island relaxation found by STM.

V. CONCLUSION

To summarize, we have studied the growth of Fe on the GaAs(100)-pseudo- 4×6 reconstructed surface for nominal thicknesses from 1 up to 6 ML using both STM and BLS. From the thickness studies, we have found that the reconstruction appears to play a significant role in determining the morphology of the film even after it is destroyed by the growth of the Fe islands. The island growth largely proceeds along the [011] direction and structural percolation appears to occur between 2 and 3 ML. Time dependent STM studies show anisotropic diffusion again favoring the [011] direction. This preference is likely determined by the initial reconstruction and the preference of Fe to bond with As or itself rather than Ga. Time dependent STM and BLS studies were performed and a smoothening effect was observed in both. From the STM measurements, it was observed that the average Fe island became larger, and the islands became more uniform in height, thereby reducing the films roughness. From the BLS measurements, the spin-wave frequency was observed to shift to a higher frequency and the linewidth decreased with time, both indicators of a smoother film. Thus, we have observed that changes in the morphology of an Fe film affect the magnetic properties of the film.

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