Magnetic phases of thin Fe films grown on stepped Cr(001)

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Magnetic phases of Fe films grown on curved Cr(001) with steps parallel to [100] are studied using the surface magneto-optic Kerr effect (SMOKE). We found that the atomic steps (1) induce an in-plane uniaxial magnetic anisotropy with the easy magnetization axis parallel to the step edges, and (2) generate magnetic frustration either inside the Fe film or at the Fe-Cr interface, depending on the Fe film thickness and the vicinal angle. For thickness greater than 35 Å, the Fe film forms a single magnetic domain and undergoes an in-plane magnetization switching due to the competition of the step-induced anisotropy and the Fe-Cr interfacial frustration. For thickness less than 35 Å, the Fe film forms multiple magnetic domains at low vicinal angle, and transforms into a single domain at high vicinal angle. A magnetic phase diagram in the 30-45 Å thickness range was obtained using a wedge-shaped Fe film. [S0163-1829(99)02918-5]

I. INTRODUCTION

Since its discovery in the Co/CoO system,¹ the exchange bias effect was believed to occur exclusively in systems where the antiferromagnet (AF) surface is uncompensated (the total surface moment is nonzero). However, a recent experiment showed that exchange bias also exists in the com*pensated* Fe/FeF₂(110) system² (where the total AF surface moment is zero). Using a total-energy calculation, a recent theoretical simulation³ was able to show the existence of the exchange bias in compensated ferromagnet (F)/AF systems. Moreover, this simulation predicts that the ground state of a ferromagnetic layer on a compensated AF surface should have the F and AF magnetic moments aligned 90° to each other. Several groups have reported the observation of this 90° coupling, $^{4-7}$ and the physical mechanism behind this 90° coupling was explored by an experiment performed on the Fe/stepped Cr(001) system.⁷ Cr is an AF with an incommensurate spin-density wave (SDW). In the 123-311 K temperature range, the SDW is transverse with the spins aligned in the same direction within each (001) plane but antialigned with respect to the neighboring (001) planes.⁸ The (001) surface of Cr, then, is uncompensated. However, when an atomic step is introduced on the surface, the (001) plane directly below the surface will be exposed. Since this plane has spins pointing in the opposite direction as the topmost layer, the effect of the atomic step is to compensate the AF surface at the step site. The advantage of using stepped Cr(001) as the compensated AF surface is that the degree of the magnetic frustration that occurs at the Fe/Cr interface⁷ can be made tunable by simply varying the step density or the miscut vicinal angle (α). The introduction of the atomic steps also causes a step-induced uniaxial anisotropy^{9,10} that can be used to detect the 90° coupling.

In this paper, we extend our previous work⁷ to include different thickness regimes to construct a magnetic phase diagram for Fe films grown on stepped Cr(001). In Ref. 7, the in-plane 90° magnetization switching as a function of the step density was attributed to the competition between the Fe-Cr 90° coupling and the step-induced magnetic anisotropy. In this paper, we further confirm this competition mechanism by an experiment in which we observe a shift of the 90° magnetization switching upon varying the step induced anisotropy. In the thin Fe film regime, we observe a multidomain structure as a result of the magnetic frustration. This multidomain phase is favored for low vicinal angle only, and a transition into a single domain phase was observed with increasing step density.

II. EXPERIMENT

A Cr(001) single crystal disk of 10 mm diameter was used as the substrate. Half of the crystal was kept in the (001)orientation while the other half was polished into a curved shape with the step edges parallel to the [100] crystallographic direction. The curved shape provides a continuous range of the vicinal angle (α) from 0° to 10°. Details of the substrate preparation are presented elsewhere.⁷ Auger electron spectroscopy (AES) was used to check the cleanness of the substrate. The AES results show that a few cycles of sputtering and annealing remove all contamination except for a small amount of nitrogen which comes from the bulk Cr. It has been reported that it is very difficult and almost impossible to obtain an absolute nitrogen-free Cr surface.¹¹ In the literature, the low-energy electron diffraction (LEED) pattern has been used as a gauge for the Cr substrate cleanness. In some cases, a Cr crystal with a (1×1) LEED pattern was used as an indication of a clean substrate.¹² In other cases, however, the (1×1) LEED pattern was attributed to the effect of the residual nitrogen at the surface, ¹³ and a (2×2) LEED pattern was identified as a signature of a cleaner Cr surface.¹⁴ Our Cr substrate was cleaned for 3-4 weeks with cycles of Ar ion sputtering and annealing. A (1×1) LEED pattern was first observed after a week's worth of sputtering and annealing. Further cleaning of the substrate eventually resulted in a stable (2×2) LEED pattern. Thus we followed the criteria of Ref. 14 to clean the Cr substrate. All Fe films were grown at 480 K to provide a comparison with previous work on this system, where a substrate temperature of 480 K was used during the Fe film growth to achieve a smooth film surface with minimal substrate-overlayer intermixing.¹⁵

Hysteresis loops of the Fe films were obtained with in situ

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FIG. 1. (a) The splitting field (H_S) of hard axis loops as a function of vicinal angle (a) at T = 480 K for several Fe film thicknesses $(d_{\rm Fe})$. (b) $H_S d_{\rm Fe}$ vs α . The universal curve indicates that H_S follows a $1/d_{\rm Fe}$ dependence.

surface magneto-optic Kerr effect (SMOKE) measurements using a He-Ne laser as the light source. The polarization of the incident beam is within the incident plane (p polarized). For all films studied, no polar loops were observed so that the Fe magnetization remains in the film plane. Therefore all hysteresis loops in this paper are from longitudinal measurements. For measurements on the curved surface, the reflection angle of the SMOKE laser beam was used to determine the local vicinal angle. A slit was used on the path of the reflected beam to improve the vicinal angle resolution to better than 0.25° .

III. RESULTS AND DISCUSSION

The step-induced magnetic anisotropy of the Fe on curved Cr(001) system has been studied by measuring the hysteresis loops at 480 K which is above the Néel temperature (T_N) of bulk Cr $(T_N = 311 \text{ K})$.⁷ It was found that the atomic steps induce an in-plane uniaxial magnetic anisotropy with the easy magnetization axis parallel to the step edges. The splitting field (H_S) of the hard axis loops, which is proportional to the strength of the step-induced magnetic anisotropy, was found to increase with increasing α . In the present work, the H_S vs α relation was studied for different Fe film thicknesses by growing an Fe wedge on the curved Cr [Fig. 1(a)]. It is interesting to note that the splitting field H_S is roughly linear in α which is different from the Fe/stepped Ag(001) [Ref. 9] and Fe/stepped W(001) (Ref. 10) systems which show a quadratic dependence of the H_S on α . Detailed discussion on the



FIG. 2. SMOKE loops for 40 Å Fe film at 140 K. The magnetization easy axis switches from perpendicular to the step edges for $\alpha < 4^{\circ}$ to parallel to the step edges for $\alpha > 5^{\circ}$.

 α dependence is beyond the scope of this paper as our focus is the effect of the magnetic frustration. The strength of the step-induced magnetic anisotropy decreases with Fe film thickness ($d_{\rm Fe}$). In Fig. 1(b) we plot $H_S d_{\rm Fe}$ vs α for all Fe films studied. All curves in Fig. 1(a) roughly fall into a universal curve, indicating that the step-induced anisotropy follows a $1/d_{\rm Fe}$ dependence. The interfacial character of this effect shows that the step-induced anisotropy is localized to the step edges, and no significant strain effect contributes to the anisotropy.

We then cooled the sample to 140 K to study the effect of interaction. From magnetic remanence the Fe-Cr measurements,⁷ we know that there are two thickness regimes for Fe on Cr(001). For thickness above 35 Å, the magnetic frustration occurs at the Fe/Cr interface and the Fe film forms a single domain. For thickness below 35 Å, the magnetic frustration occurs within the Fe film and the Fe film forms multiple magnetic domains. We first discuss the single-domain regime. For the purpose of completeness, we include our previous results for the hysteresis loops of a 40 Å Fe film grown on stepped Cr(001) (Fig. 2). At high vicinal angle, the Fe film forms a single domain with the magnetization parallel to the step edges. At low vicinal angle, the Fe film also forms a single domain but with the magnetization perpendicular to the step edges. This 90° magnetization switching as a function of the vicinal angle α was attributed to the competition between the Fe-Cr 90° coupling and the step-induced magnetic anisotropy.⁷ At low α , the Fe-Cr interaction dominates the step-induced anisotropy so that the Fe magnetization is perpendicular to the step edges. At high α , the step-induced anisotropy dominates the Fe-Cr interaction so that the Fe magnetization is parallel to the step edges. The 90° magnetization switching then occurs at a critical vicinal angle where the 90° coupling compensates the stepinduced anisotropy. To further confirm this competition mechanism, we purposely modified the strength of the stepinduced anisotropy and studied the corresponding change of the critical vicinal angle. It is known that adsorp-



FIG. 3. SMOKE measurements at 480 K with applied magnetic field perpendicular to the step edges for a 40 Å Fe film with (right column) and without (left column) 0.12 ML Au decoration. The greater splitting field after the Au decoration indicates an enhancement of the step-induced magnetic anisotropy.

tion of submonolayer amounts of metals on stepped magnetic films can change the strength of the step induced anisotropy.¹⁶ We deposited 0.12 ML Au on the 40 Å Fe film at 480 K and measured the hard-axis hysteresis loops before and after the Au deposition (Fig. 3). The increased splitting field after the Au deposition clearly shows that the step induced anisotropy has been increased. Since the Fe-Cr interaction occurs at the interface, far away from the deposited Au, we anticipate that only the step-induced anisotropy has been modified by the 0.12 ML Au. Therefore, as a result of the increased step-induced anisotropy, we expect a lower critical vicinal angle at which the 90° magnetization switching should occur. After lowering the temperature to 180 K, we found that the critical vicinal angle for the 90° magnetization switching indeed shifts to a lower value than that without the Au (Fig. 4). This result further confirms that the 90° magnetization switching is a result of the competition between the Fe-Cr 90° coupling and the step-induced anisotropy.

We then studied the thin film regime (<35 Å). At low temperature these Fe films exhibit multiple magnetic domains on a nominally flat Cr(001) substrate.¹⁵ Figure 5 shows the hysteresis loops at 140 K for a 30 Å Fe film grown on curved Cr. Different magnetic phases can be observed at different vicinal angles. For $\alpha < \sim 2.5^{\circ}$, the hysteresis loops exhibit splitting with low remanence for magnetic fields applied both parallel and perpendicular to the step edges. Since the hysteresis loop is an averaged result within the SMOKE laser spot (~ 0.2 mm), the low remanence in both directions indicate that the 30 Å Fe film consists of multiple magnetic domains in this vicinal angle range. This multidomain structure has been identified in thin Fe films grown on nominally flat Cr(001) as a result of the presence of random atomic steps. The appearance of multiple domains in the thin film regime can be understood in terms of the Fe-Fe and Fe-Cr



FIG. 4. SMOKE measurements at 180 K for the sample in Fig. 3. The 90° magnetization switching after the Au decoration shifts to a lower vicinal angle due to the enhancement of the step induced anisotropy.

interactions. For thicker Fe films, the Fe-Fe interaction dominates the Fe-Cr interaction so that magnetic domains are formed at the Fe-Cr interface to result in a single domain Fe film. For thinner Fe films, the Fe-Cr interaction dominates the Fe-Fe interaction so that magnetic domains will be formed inside the Fe film and result in low remanence.¹⁵ On the other hand, the Fe-Fe interaction energy scales as the Fe film thickness which is independent of the vicinal angle, but the Fe-Cr interaction energy scales as the step terrace length which is inversely proportional to the vicinal angle. Thus the



FIG. 5. SMOKE loops measured at 140 K for a 30 Å Fe film. For $\alpha \leq 2.5^{\circ}$, a multidomain phase is observed. For $\alpha \geq 2.5^{\circ}$, the behavior is the similar to the 40 Å film.



FIG. 6. Phase diagram at 140 K for Fe films grown on curved Cr (001). α_{C1} marks the transition from multidomains into single domain, and α_{C2} marks the 90° magnetization switching from perpendicular to parallel to the step edges. The arrows indicate the magnetization directions of the Fe film in different regimes.

appearance of multiple domains inside the Fe film should be less favored upon increasing the vicinal angle. This behavior was indeed observed for higher vicinal angle in the 30 Å Fe film. For $2.5^{\circ} < \alpha < 5^{\circ}$, the split loop for magnetic field applied perpendicular to the step edges becomes a square loop with full remanence, indicating a transition of the Fe film from a multidomain structure into a single-domain structure with the Fe magnetization perpendicular to the step edges. For $\alpha \ge 5^{\circ}$, we again observe the 90° magnetization switching from a direction perpendicular to one parallel to the step edges as in the 40 Å Fe film. Therefore there are two transitions for the 30 Å Fe film upon increasing the vicinal angle α : the one at lower α marks the transition of the Fe film from multiple domains into a single domain; the second one at higher α marks the 90° magnetization switching as in the 40 Å Fe film.

To obtain a better understanding of the α and $d_{\rm Fe}$ dependence of the two transitions, we constructed a magnetic phase diagram using a wedged Fe film which included both 30 and 40 Å of Fe. For operational convenience, we define the transition vicinal angles from the remanence of the loops with the magnetic field applied perpendicular to the step edges: the first transition angle α_{C1} corresponds to a change of the remanence from zero to one, and the second transition angle α_{C2} corresponds to a change of the remanence from one to zero. We then located the α_{C1} and α_{C2} for different Fe film thicknesses (d_{Fe}) to construct the phase diagram at 140 K (Fig. 6). It should be noted that the transition between different regions is not always sharp. Any discrepancies between the phase diagram (Fig. 6) and Figs. 2 and 5 are due to this fact. First we turn our attention to the α_{C1} transition. For $d_{\rm Fe} = 30$ Å, α_{C1} is located at $\sim 3^{\circ}$. The α_{C1} decreases rapidly with increasing d_{Fe} and goes to zero at $d_{\text{Fe}} = 35 \text{ Å}$ so that for $d_{\rm Fe}$ > ~35 Å the multidomain phase does not exist. This agrees with the results of the remanence experiments of Ref. 7. The behavior of α_{C1} can be qualitatively understood in light of the picture of step induced magnetic frustration presented by Berger and Hopster.¹⁵ The Fe-Cr interfacial frustration energy per step is proportional to the terrace length L(i.e., the distance between steps), where $\alpha \sim 1/L$. The energy per step for frustration within the Fe film is proportional to $d_{\rm Fe}$. Therefore the multidomain structure should be favored for low $d_{\rm Fe}$ and low α . That is why the multidomain phase appears at the lower-left corner of the phase diagram. We now look at the behavior of α_{C2} . Figure 6 shows that α_{C2} changes only slightly as a function of the Fe film thickness. Since the α_{C2} transition represents the critical vicinal angle at which the frustration induced 90° Fe-Cr coupling equals the step-induced anisotropy, the weak dependence of α_{C2} on $d_{\rm Fe}$ implies that the energy terms for the 90° coupling and the step-induced anisotropy must have a similar thickness dependence. From Fig. 1, we know that the step induced anisotropy follows a $1/d_{\rm Fe}$ dependence. Thus the 90° coupling should also have a $\sim 1/d_{\rm Fe}$ dependence. This is expected since the 90° coupling comes from the Fe-Cr interfacial frustration and an interfacial effect should scale as $1/d_{\rm Fe}$. The twisting of the Fe moments in the normal direction of the film will modify the $1/d_{\rm Fe}$ dependence⁷ which accounts for the slight variation of α_{C2} upon increasing the Fe film thickness.

Finally, we would like to mention that for Fe films thinner than ~20 Å, no clear transitions similar to α_{C1} and α_{C2} can be identified. For this reason we have not included the d_{Fe} <20 Å regime in the phase diagram. We speculate that in this thickness regime, the magnetic multidomains at low α continuously grow in size as α increases. Confirmation of this speculation relies on the domain imaging study which is beyond the scope of the present work.

IV. CONCLUSION

The experiments presented in this work serve as a confirmation for both the frustration picture presented by Berger and Hopster¹⁵ and the 90° coupling picture of Ref. 7. The presence of a multidomain phase at low vicinal angles for Fe film thickness thinner than ~35 Å confirms the transfer of the magnetic frustration from the Fe-Cr interface at higher thickness to the inside of the Fe film at lower thickness. When the magnetic frustration is located at the Fe-Cr interface, a 90° magnetization switching occurs as a result of the competition between the 90° coupling and the step-induced anisotropy. By changing the strength of the step-induced anisotropy, we were able to confirm this competition mechanism. We also confirmed the ~ $1/d_{\rm Fe}$ dependence of the step-induced anisotropy and implicitly for the 90° coupling.

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