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Role of the antiferromagnet in exchange-biased $\text{Fe}_3\text{O}_4/\text{CoO}$ superlattices (invited)

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Using neutron diffraction techniques, we have directly characterized the antiferromagnetic spin structure of CoO in exchange-biased $\text{Fe}_3\text{O}_4/\text{CoO}$ (001) superlattices. The CoO antiferromagnetic order is long-range and extends coherently across intervening ferrimagnetic Fe_3O_4 layers. The ordering is influenced significantly by not only epitaxial growth constraints but also by coupling to the Fe_3O_4 layers. The antiferromagnetic order in these $\text{Fe}_3\text{O}_4/\text{CoO}$ (001) samples exhibits significant dependence on field cooling preparations. The CoO spins show a strong tendency to align perpendicular to the net ferrimagnetic moment with some changes in the average domain size associated with field cooling. The CoO spin structure in the exchange-biased state illustrates important aspects of recent biasing theories but with unusually large domain sizes. © 1998 American Institute of Physics. [S0021-8979(98)17811-8]

I. INTRODUCTION

Recently, there has been a renewed interest in studying magnetic systems exhibiting exchange anisotropy or exchange biasing.¹⁻⁷ First discovered in the 1950s,⁸ these systems typically consist of ferromagnetic (FM) materials in contact with antiferromagnetic (AFM) materials. Due to *exchange*-coupling between the FM and AFM layers, a host of unusual magnetic behavior is observed, including a field shift or *bias* in the FM hysteresis loop after field cooling through the AFM ordering temperature (T_N). Despite over 40 years of experimental and theoretical effort, there is still considerable debate about the mechanisms for this exchange-biasing effect. In particular, recent theories have concentrated on the detailed role of AFM domain wall formation as an essential element to understanding the exchange anisotropy.⁹⁻¹¹ The nature of the AFM and FM coupling at the interface is also under discussion, as well as the ideal AFM spin configuration for maximal biasing effect. In most experiments, the interfacial behavior of the antiferromagnet is usually inferred from bulk measurements or from the FM response.^{6,7} In contrast, elastic neutron diffraction provides a method to determine directly the nature of the AFM order in such systems.¹²

In this article, we discuss neutron diffraction results that directly focus on the CoO AFM behavior in exchange-biased $\text{Fe}_3\text{O}_4/\text{CoO}$ (001) superlattices. The growth of crystalline superlattices is possible because the spinel Fe_3O_4 and rock salt CoO structures have similar bulk lattice parameters ($a_{\text{Fe}_3\text{O}_4} = 8.398 \text{ \AA}$ and $2a_{\text{CoO}} = 8.508 \text{ \AA}$) and have complementary oxygen sublattices in the (001) plane.^{4,13} By growing superlattices with thin layers and multiple repeats, we can highlight the interfacial region which is of interest for exchange-

biasing. In addition, the bulk properties of both CoO and Fe_3O_4 have been studied extensively.^{14,15} Bulk CoO orders antiferromagnetically below $\sim 291 \text{ K}$ in ferromagnetic sheets that alternate sign along $\langle 111 \rangle$ "propagation" directions, leading to distinct $\{111\}$ domains. Bulk Fe_3O_4 orders ferrimagnetically below $\sim 858 \text{ K}$ with a net moment of $4.2 \mu_B$ per unit cell. As demonstrated previously, the $\text{Fe}_3\text{O}_4/\text{CoO}$ (001) system exhibits substantial exchange-biasing, despite the fact that the CoO (001) *interface* is expected to be compensated with no net moment.⁴ Thus, the $\text{Fe}_3\text{O}_4/\text{CoO}$ system is a good model to study the role of the antiferromagnet in producing biasing.

For superlattices of this system, we find that the CoO AFM ordering is long-range and propagates coherently through intervening Fe_3O_4 layers. The Co moment directions are constrained by sample epitaxy and influenced as well by perpendicular coupling to the ferrimagnetic Fe_3O_4 layers. Upon field cooling and field cycling, subtle changes in the AFM domain sizes are observed. These features are illustrative of the complex role of the antiferromagnet in exchange-biased systems.

II. EXPERIMENTAL DETAILS

Two $\text{Fe}_3\text{O}_4/\text{CoO}$ superlattices, $[\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (30 \text{ \AA})]_{50}$ and $[\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (100 \text{ \AA})]_{50}$, were grown by oxidic molecular beam epitaxy (MBE) onto 1.6 cm diameter [001] MgO substrates. Reflection high energy electron diffraction and x-ray scattering measurements show that the deposition results in epitaxial [001] superlattices with rocking curve full widths at half maximum (FWHM) of $\sim 0.2^\circ$ and interfacial widths of $\sim 2 \text{ \AA} \pm 1 \text{ \AA}$. For comparison, a third

FIG. 1. Schematic showing the reciprocal space reflections discussed in the text as well as the directions in which the samples were field cooled. The scan directions through each peak are designated with small arrows.

superlattice with nonmagnetic MgO as spacer layers $[\text{MgO} (30 \text{ \AA})/\text{CoO} (30 \text{ \AA})]_{333}$ was also grown by MBE onto an $[001]$ MgO substrate.

After preliminary characterization by x-ray analysis, neutron diffraction data were obtained on three triple-axis spectrometers (BT-2, BT-9, and SPINS) at the National Institute of Standards and Technology. For unpolarized measurements (BT-2 and BT-9), a pyrolytic graphite (PG) monochromator and analyzer were used to select elastically scattered neutrons of energy 14.8 meV. Typical collimation of $40' - 48' - 48'$ resulted in resolution in the growth direction of ~ 0.012 to $0.030 \text{ \AA}^{-1}$ for all of the reflections of interest. For polarized measurements (SPINS), a supermirror polarizer and an Al coil spin flipper were positioned before and after the sample to select all four neutron spin cross sections, $[(++)]$, $[(--)]$, $[(+-)]$, and $[(+)]$, with polarization efficiencies greater than 91%.¹⁶ The + and - signs denote the neutron polarization before or after scattering from the sample.

For these experiments, the samples were oriented vertically with the $[110]$ in-plane axis and $[001]$ growth direction defining the scattering plane as shown in Fig. 1. The superlattices were cooled in a 14 kOe field applied in several directions within the sample growth plane. The specific reflections studied and the directions of the scans are also illustrated in Fig. 1.¹⁷ In these superlattices and consistent with bulk structure factor calculations,^{12,14} the $\{111\}$ class of reflections have the strongest contribution from the CoO AFM order. The (331) and (113) reflections provide additional information about the CoO magnetic ordering, whereas the (222) reflection has only a structural contribution from the CoO. The Fe_3O_4 structural and magnetic order contributes to all these reflections and is solely responsible for the (220) reflection. For the three superlattices, the (222) satellite reflections were nearly resolution limited in the growth axis direction, indicating that the structural coherence is long range.

III. RESULTS AND DISCUSSION

A. Zero field characterization

The AFM order of the superlattices was first characterized in zero field. Figure 2(a) shows typical neutron diffrac-

FIG. 2. Neutron diffraction scans along the $[001]$ Q_z direction through the (a) (111) and (b) (220) reflection. The circles ($\circ$) refer to the $[\text{MgO} (30 \text{ \AA})/\text{CoO} (30 \text{ \AA})]_{333}$ at 15 K, and the triangles ($\triangle$) refer to the $[\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (30 \text{ \AA})]_{50}$ superlattice at 28 K, both normalized to the peak values. The MgO/CoO data has been displaced from the origin by a factor of 0.2 for clarity. Solid lines denote fits to Gaussians. The dashed lines in the (111) scan show the broad Gaussians associated with the Fe_3O_4 component. The double-headed arrows indicate the width of the broad features in the $[\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (30 \text{ \AA})]_{50}$ reflection.

tion scans of the (111) reflection along the $[001]$ direction. As illustrated in the figure, the line shapes differ quite significantly for the MgO/CoO sample as compared to the exchange-biased $\text{Fe}_3\text{O}_4/\text{CoO}$ samples.

For the unbiased MgO/CoO superlattice, the (111) structural reflection is forbidden such that the diffracted intensity originates entirely from the AFM CoO ordering. The broad line shape in this case indicates that the magnetic order has limited coherence along the growth axis direction. Considering the full width at half maximum and the instrumental resolution, we find that the magnetic coherence length is $\sim 35 \pm 5 \text{ \AA}$ and is thus restricted to a single CoO layer. This result is not surprising given that the MgO spacers are nonmagnetic and that the superexchange interaction in bulk CoO is short range.¹⁴ The temperature dependence of the AFM ordering is also consistent with bulk CoO, as the intensity of the (111) reflection decreases to zero at $T_N = 300 \pm 10 \text{ K}$, as compared with the bulk AFM CoO $T_N \sim 291 \text{ K}$.

In contrast, for the two $\text{Fe}_3\text{O}_4/\text{CoO}$ superlattices, the (111) reflection senses the CoO AFM order as well as the structural and magnetic ordering of the Fe_3O_4 . In these samples, the scattering contributions from the two oxides can be distinguished by the differences in line shape and temperature dependence. As illustrated in Fig. 2(a) for the $\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (30 \text{ \AA})$ sample, the $\text{Fe}_3\text{O}_4/\text{CoO}$ (111) reflection in the growth direction shows a two-component line shape with a broad and narrow contribution.

The magnitude of the broad component is not sensitive to temperature for the range of $T = 10$ to 600 K which is well below the ordering temperature of bulk Fe_3O_4 (858 K). As shown in Fig. 2(b), the (220) reflection, which is solely due to the Fe_3O_4 layers, is also quite broad. For both samples, these broad peak widths give coherence lengths of ~ 100 Å along the growth axis direction which correspond to the width of a single Fe_3O_4 layer. These data indicate that the registry of the Fe_3O_4 spinel lattice is not retained across the intervening CoO interlayers. This lack of coherence is not surprising due to the nonunique alignment of the Fe tetrahedral sites in the spinel relative to the rock salt CoO template at each $\text{Fe}_3\text{O}_4/\text{CoO}$ interface. Thus, the differences in the two crystal structures is expected to result in some type of structural disorder.¹⁸ In particular, the existence of stacking faults in the Fe_3O_4 layers has been observed and modeled extensively in $\text{Fe}_3\text{O}_4/\text{NiO}$ superlattices.¹² These structural faults thus lead to the broadened peak widths observed in Fig. 2. Note that these reflections have both a magnetic and structural component from the Fe_3O_4 layers. Due to the broadening from structural features, the magnetic coherence length of the Fe_3O_4 is masked and may in fact still be long range.

As the broad component of the (111) reflection tracks the Fe_3O_4 structural and magnetic order, the narrow component measures the CoO AFM order. We note that the magnitude of the narrow component is sensitive to temperature. Upon heating, this intensity decays smoothly to zero at temperatures near or above the bulk CoO T_N value of 291 K. Specifically, the AFM ordering temperatures as determined by this method are 450 ± 15 K for the $[\text{Fe}_3\text{O}_4(100 \text{ Å})/\text{CoO}(30 \text{ Å})]_{50}$ superlattice and 325 ± 15 K for the $[\text{Fe}_3\text{O}_4(100 \text{ Å})/\text{CoO}(100 \text{ Å})]_{50}$ superlattice. The apparent increase in the AFM ordering temperature with increasing relative Fe_3O_4 thickness is consistent with observations in a series of $\text{Fe}_3\text{O}_4/\text{CoO}$ superlattices as well as for $\text{Fe}_3\text{O}_4/\text{NiO}$ and NiO/CoO samples.^{12,19,20} These changes are in agreement with mean-field simulations of the exchange coupling between the Co and Fe spins at the superlattice interfaces.¹² In addition to alterations in the CoO ordering temperature, we note that the CoO line width is much more narrow than for the MgO/CoO superlattice [Fig. 2(a)]. Thus, in both $\text{Fe}_3\text{O}_4/\text{CoO}$ samples, the AFM order is long range along the growth axis direction. Considering the full width at half maximum (FWHM) deconvoluted by the instrumental resolution, we find that the magnetic coherence lengths are always greater than 300 Å and in some cases greater than 800 Å. This long-range order may result from local exchange coupling to the Fe_3O_4 at the superlattice interfaces. Apparently, the alternation of the Fe_3O_4 moments preserves the phase of the CoO AFM order from one layer to the next. Scanning in the growth plane, we find that the CoO AFM order is also long range.

B. Moment direction determination

To determine the AFM moment directions within each {111} domain, we examined the three superlattices with polarized neutron diffraction. Figures 3(a)–3(c) shows typical polarized neutron diffraction data of the (111) reflection scanned in the [001] direction for the three samples taken at

FIG. 3. Polarized neutron diffraction scans of the (111) reflection along the [001] direction for the (a) $\circ$ $[\text{MgO}(30 \text{ Å})/\text{CoO}(30 \text{ Å})]_{333}$, (b) $\triangle$ $[\text{Fe}_3\text{O}_4(100 \text{ Å})/\text{CoO}(30 \text{ Å})]_{50}$, and (c) $\square$ $[\text{Fe}_3\text{O}_4(100 \text{ Å})/\text{CoO}(100 \text{ Å})]_{50}$ superlattices after cooling in a 14 kOe along the [110] direction to 78 K. The closed symbols denote the nonspin flip scattering which is sensitive to the CoO moment along $[1\bar{1}0]$. The open symbols denote the spin flip scattering which is sensitive to the CoO moments along the $[11\bar{2}]$ direction. (d) Illustration of the spin structure as determined from the above diffraction data.

78 K. In these measurements with the sample orientation as in Fig. 1, the nonspin flip $[++]$ and $--]$ intensities are sensitive to the AFM CoO moment contribution parallel to the $[1\bar{1}0]$, while the spin flip $[+-]$ and $-+]$ intensities are sensitive to the AFM CoO moment component parallel to the $[11\bar{2}]$ direction. As illustrated in the figure, for all three superlattices, the non-spin flip contribution dominates the spin flip, indicating that the AFM moments associated with the (111) reflection lie along the $[1\bar{1}0]$ direction. The small amount of CoO spin flip scattering detectable is at the efficiency limit of the polarizing elements. Thus to within $\pm 2^\circ$, nearly all the Co spins within the alternating ferromagnetic sheets point in the $\pm[1\bar{1}0]$ direction for the (111) domain, as illustrated in Fig. 3(d).²¹ This moment orientation persists to the AFM ordering temperature and did not change as a function of field cooling in three different directions within the growth plane, namely in the $[1\bar{1}0]$, $[110]$, and $[100]$ directions. The intensities observed for the (331) and $(\bar{1}\bar{1}3)$ antiferromagnetic reflections confirm that the AFM moments are essentially confined to the (111) plane.

For this (111) domain, the alignment of the Co moments along this single $[1\bar{1}0]$ direction within the sample growth

FIG. 4. Neutron diffraction scans along the $[001]$ Q_z direction through the (111) reflection after zero field cooling and field cooling in 14 kOe field along the $[1\bar{1}0]$ direction to 78 K. Data are normalized to the zero field cooled data. The circles ($\circ$ and $\bullet$), squares ($\square$ and $\blacksquare$), and triangles ($\triangle$ and $\blacktriangle$) refer to the $[\text{MgO}(30\text{ \AA})/\text{CoO}(30\text{ \AA})]_{333}$, $[\text{Fe}_3\text{O}_4(100\text{ \AA})/\text{CoO}(30\text{ \AA})]_{50}$, and $[\text{Fe}_3\text{O}_4(100\text{ \AA})/\text{CoO}(100\text{ \AA})]_{50}$ superlattices, respectively. Open symbols refer to the zero field cooled data, and closed symbols refer to the field cooled data. Dashed lines indicate the broad Fe_3O_4 contribution.

plane contrasts with the behavior of bulk CoO, which has three different moment directions ($[1\bar{1}3]$, $[1\bar{3}1]$, and $[3\bar{1}1]$) canted out of the (111) plane by 8° .¹⁴ These data suggest that the CoO spin structure in these superlattices is altered as a consequence of epitaxial growth constraints. We note that the bulk lattice parameter of CoO differs from that of Fe_3O_4 and that of the substrate MgO by $\sim 1.0\% - 1.5\%$.

C. Field-dependent domain characteristics

While the antiferromagnetic moment *directions* are influenced by structural features, the *number* of spins aligned in these directions is determined by the nature of the interfacial coupling as revealed by field-dependent measurements. Figure 4 shows the results at remanence after cooling the three superlattices from 320 to 78 K in a 14 kOe field applied in the $[1\bar{1}0]$ direction. The data are scaled against similar data obtained after cooling to 78 K in zero applied field.

For the MgO/CoO superlattice, the field cooling treatment leads to little change in the intensity of the (111) reflection relative to the zero field cooled state. The applied magnetic field appears to have little effect on the AFM CoO order. This lack of a field response is not surprising in that the applied field of 14 kOe is roughly an order of magnitude less than the typical fields in which many antiferromagnets undergo a spin flop transition and align mostly perpendicular to the applied field.²²

FIG. 5. The relative integrated intensity of the CoO contribution to the (111) reflection scanned along the $[001]$ direction after cooling from 320 K to 78 K in a 14 kOe field applied in different directions within the sample growth plane. The triangles ($\triangle$) refer to the $[\text{Fe}_3\text{O}_4(100\text{ \AA})/\text{CoO}(30\text{ \AA})]_{50}$ and the squares ($\blacksquare$) to the $[\text{Fe}_3\text{O}_4(100\text{ \AA})/\text{CoO}(100\text{ \AA})]_{50}$ superlattices. The data are normalized to data obtained after zero field cooling to 78 K as indicated by the solid line. For the 100 Å sample, the zero field data was obtained after randomizing the Fe_3O_4 magnetization by successively decreasing cycles of the field at 350 K. Lines between points are a guide to the eye.

In contrast, both of the $\text{Fe}_3\text{O}_4/\text{CoO}$ superlattices [Figs. 4(b)–4(c)] show a significant decrease in the (111) intensity upon field cooling in the $[1\bar{1}0]$ direction, as CoO spins are driven out of this (111) domain. Note that this change is associated with the narrow CoO contribution and not the broader Fe_3O_4 component. This field effect is large relative to that observed for nonmagnetic MgO spacers and indicates the importance of the coupling of the AFM CoO moments to the Fe_3O_4 layers.

The nature of this coupling is apparent when considering the effect of field cooling in different directions within the sample growth plane. Figure 5 shows the integrated CoO intensity at the (111) reflection in remanence after field cooling along the $[110]$, $[100]$, and $[1\bar{1}0]$ directions. The field cooling data have been normalized against comparable zero field cooled data which has been set to 0.5. For both $\text{Fe}_3\text{O}_4/\text{CoO}$ samples, the AFM (111) intensity is *highest* upon field cooling in the $[110]$ direction, as CoO spins are driven *into* the (111) domain. From the polarized beam data discussed in the previous section, we have determined that the AFM spins in the (111) domain are lined up along the $\pm[110]$ direction irrespective of field treatment. Thus, the preferred orientation of the AFM spins in the (111) domain is *perpendicular* to the net ferrimagnetic moment, which is aligned along the $[110]$ applied field direction. The extent to which this perpendicular arrangement is preferred is different for the two samples, as the field cooling effect is more pronounced for the $[\text{Fe}_3\text{O}_4(100\text{ \AA})/\text{CoO}(100\text{ \AA})]_{50}$ superlattice than for the $[\text{Fe}_3\text{O}_4(100\text{ \AA})/\text{CoO}(30\text{ \AA})]_{50}$ sample. This difference is also consistent with magnetization measurements on these samples. The $[\text{Fe}_3\text{O}_4(100\text{ \AA})/\text{CoO}(100\text{ \AA})]_{50}$ superlattice showed a much larger field shift for the hysteresis loop at 78 K as compared to the $[\text{Fe}_3\text{O}_4(100\text{ \AA})/\text{CoO}(30\text{ \AA})]_{50}$ sample (1300 ± 100 Oe vs 550 ± 50 Oe).²⁰

The direct observation of field-cooling dependent changes in the AFM (111) intensity agrees with most current models of exchange-biasing, to the degree that the nature of

FIG. 6. Changes in the (111) peak intensity upon field cycling in the $\pm[1\bar{1}0]$ direction after field cooling to 78 K in +14 kOe along the $[110]$ direction. The triangles (Δ) refer to the $[\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (30 \text{ \AA})]_{50}$ and the squares ($\square$) to the $[\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (100 \text{ \AA})]_{50}$ superlattices. The numbers and arrows refer to the order and direction of the field cycling. Solid lines are guides to the eye.

the antiferromagnet is essential to the effect. In particular, the observed perpendicular coupling between the antiferromagnetic CoO and ferrimagnetic Fe_3O_4 moments has been predicted previously by Hinchey and Mills²³ and connected to exchange-biasing more recently by Koon.¹¹ The coupling contrasts with most models of exchange-biasing which assume that the antiferromagnet always couples collinearly with the ferromagnet.⁷⁻¹⁰

Upon field cycling after field cooling in the $[1\bar{1}0]$ direction, we also observe some changes in the (111) peak intensity, as shown in Fig. 6. While field cooling in 14 kOe in the $[110]$ direction drives CoO spins out of the (111) domain (Fig. 4), the AFM spin structure is not completely frozen for both samples (Fig. 6). The partial return of the AFM spins into the (111) domain occurs at field values which are similar in magnitude to the coercive fields of the two samples [$H_c \sim 6000 \pm 100$ Oe for $\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (30 \text{ \AA})$ and $\sim 3200 \pm 100$ Oe for $\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (100 \text{ \AA})$ at 78 K]. As evident in Fig. 6, the $[\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (100 \text{ \AA})]_{50}$ superlattice shows less recovery upon field cycling than the $[\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (30 \text{ \AA})]_{50}$ sample and is hence more effectively “frozen” into a spin configuration. We note that in contrast to the 30 Å sample, for the 100 Å CoO superlattice, memory of the field cooled direction is retained upon heating and cooling above the AFM ordering temperature, indicating again the coupling to the Fe_3O_4 layers. To the degree to which this AFM response tracks the biasing, these differences are also consistent with the larger exchange-biasing fields and higher temperatures for the onset of biasing for the $[\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (100 \text{ \AA})]_{50}$ sample.^{5,20}

However, the domain formation in these $\text{Fe}_3\text{O}_4/\text{CoO}$ superlattices is more subtle than that proposed by many of these models. Figure 7 shows the full widths at half maximum for the narrow CoO (111) reflection in remanence after field cooling in different directions within the sample growth plane. For both samples the widths for zero field cooling match within error those obtained after field cooling in the

FIG. 7. The full width at half maximum (FWHM) for the (111) CoO contributions scanned along the $[001]$ direction after cooling to 78 K in a 14 kOe field applied in different directions within the sample growth plane. The triangles (Δ) refer to the $[\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (30 \text{ \AA})]_{50}$ and the squares ($\blacksquare$) to the $[\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (100 \text{ \AA})]_{50}$ superlattices. The dashed line indicates the instrumental resolution limit. Lines between points are a guide to the eye.

$[110]$ direction. The larger errors associated with the widths for the $[1\bar{1}0]$ orientation reflect the decrease in CoO (111) intensity for these scans. After deconvolution with the instrumental resolution, the FWHM is inversely proportional to the AFM domain size along the growth axis direction. For the $\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (30 \text{ \AA})$ sample, the average coherence length in the growth axis direction varies from $\sim 310 \pm 20$ Å for field cooling in the unpreferred $[1\bar{1}0]$ direction to $\sim 700 \pm 100$ Å for field cooling in the preferred $[110]$ direction. Field cooling thus leads to changes in the average domain size. However, in the case of the 100 Å sample, the widths are nearly resolution limited for all field directions with growth axis coherence lengths of greater than 800 Å. Thus, in both samples, domain walls do not occur at every superlattice interface. Scanning the (111) reflection along the $[110]$ direction in the growth plane, we find similarly large domain sizes across the sample plane.

In addition, upon field cycling, we observe changes in the average domain size of less than a few percent for the $[\text{Fe}_3\text{O}_4 (100 \text{ \AA})/\text{CoO} (100 \text{ \AA})]_{50}$ sample which has larger exchange-biasing fields.^{5,20} This contrasts with most recent models of exchange biasing in that we do not observe domain walls forming in the antiferromagnet near each $\text{Fe}_3\text{O}_4/\text{CoO}$ interface upon field reversal. However, small twists or incomplete domain walls may form in the antiferromagnet which are not within the sensitivity of our measurement technique. Alternatively, the ferrimagnetic Fe_3O_4 may exhibit domain wall formation upon field cycling. In $\text{Fe}_3\text{O}_4/\text{NiO}$ multilayers, polarized neutron reflectivity experiments showed different magnetization depth profiles for the Fe_3O_4 layers before and after field reversal, suggesting such domain wall formation.²⁴ Further experiments are underway to explore this possibility for these $\text{Fe}_3\text{O}_4/\text{CoO}$ superlattices.

IV. SUMMARY/CONCLUSION

We have demonstrated the complex nature of the AFM spin structure in $\text{Fe}_3\text{O}_4/\text{CoO}$ superlattices. The spin arrangement of the CoO is influenced by both growth constraints

and a preferred perpendicular coupling to the net Fe_3O_4 moments. In general, the CoO domains are long range and extend coherently across intervening Fe_3O_4 layers. Upon field cooling, the AFM spins “freeze” into large domains which do not change size substantially upon field cycling. These results illustrate the importance of the details of the AFM spin structure in exchange-biased systems.

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