ANTIFERROMAGNETIC SPIN REORIENTATION IN EXCHANGE-BIASED Fe_3O_4/NiO SUPERLATTICES

The magnetic hysteresis loop of a ferromagnetic material can be displaced along the field axis as a result of magnetic exchange coupling to an antiferromagnet. This "exchange-biasing" phenomenon was discovered over 40 years ago in oxidized Co particles [1] and has been observed in a variety of thin films and multilayers. Artificial spin-valve structures with exchange-biased layers show great promise for applications as magnetoresistive sensors in read heads. Recent theories [2-4] make specific predictions about the origin of exchange biasing and the response of the antiferromagnetic layer.

High-angle neutron diffraction is an ideal probe as the antiferromagnet gives rise to distinct reflections of magnetic origin. We have performed neutron diffraction studies of exchange-biased [001] Fe_3O_4/NiO superlattices on the SPINS and BT-9 triple-axis spectrometers. These measurements confirm that the NiO layers retain their bulk antiferromagnetic structure in which ferromagnetic {111} planes alternate direction along each <111> axis. Our data show that exchange biasing is associated with domain walls that form and "freeze" within the antiferromagnetic NiO layer. Upon field cooling into the exchange-biased state, magnetic domains lock within the NiO layers and do not change with subsequent application of magnetic fields. In contrast, the antiferromagnetic domain sizes in unbiased samples prepared by cooling in zero field depend sensitively on the strength of the applied field.

We focus here on a Fe_3O_4(6 nm)/NiO(11 nm) superlattice deposited using oxygen plasma-assisted molecular beam epitaxy. Measurements of the magnetic hysteresis loop show little evidence of exchange biasing after cooling in zero field. Strong exchange biasing is induced upon field cooling the superlattices from high temperatures. For our superlattice, the biasing field at 30 K is 0.043 T after field cooling from 550 K.

For the diffraction experiments the sample was oriented as shown in the inset of Fig. 1. Vertical magnetic fields, H, were applied in the sample plane. In this configuration, the (l l l) and ($\bar{l}\,\bar{l}$ l) NiO antiferromagnetic reflections lie in the scattering plane. Using a horizontal-field magnet, the (l $\bar{l}$ l) and ($\bar{l}$ l l) reflections could be accessed by rotating the sample 90° about the growth axis. Figure 1 shows a typical growth-axis (00*l*) scan through the (111) reflection for the superlattice. Structural stacking faults [5] at the NiO/Fe_3O_4 interfaces limit the coherence of the Fe_3O_4 structural and magnetic order to a single Fe_3O_4 layer. As a result, we can easily separate the broad Fe_3O_4 and narrow NiO contributions to the reflection and track the latter as a function of field.

The neutron scattering data reveal that the magnetic domain sizes in the antiferromagnetic NiO depend on the presence or absence of exchange biasing. Figure 2 shows the full-width-at-half-maximum (FWHM) for the (111) NiO peak scanned along the (00*l*) and (*ll*0) directions after cooling in a 6 T field (i.e., exchange-biased state) and cooling in zero field (i.e., unbiased state). After field cooling, the FWHM of the NiO reflection scanned along the (00*l*) growth direction shows no dependence on field.

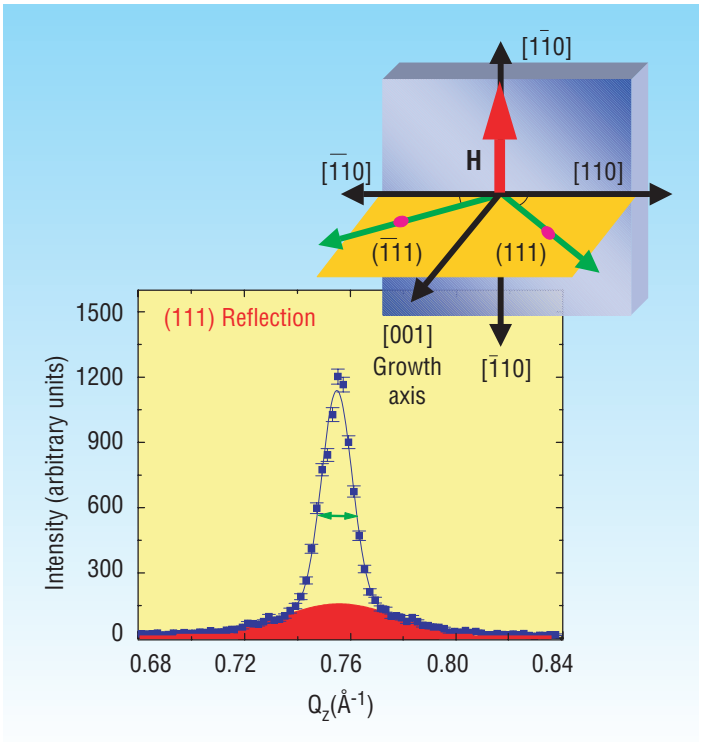

FIGURE 1. Zero-field growth-axis (00*l*) scan through the (111) reflection for the Fe_3O_4(6 nm)/NiO(11 nm) superlattice after cooling to 78 K in a 6 T field parallel to [1$\bar{1}$0]. The broad Fe_3O_4 peak is shown in red, and the remaining scattering is from the NiO. The green arrow marks the FWHM of the NiO peak. The inset shows the scattering diagram.

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The antiferromagnetic domain size along the growth direction, determined from the inverse of the FWHM, remains constant near 750 Å after field cooling. This contrasts with the pronounced field-dependence of the FWHM observed after cooling in zero field (Fig. 2). In this state, the FWHM is smallest near zero field, but approaches the constant field-cooled value when the magnitude of the field is greater than 2 T. The corresponding domain size varies from approximately 1200 Å in zero field to 800 Å in high fields. Growth-plane (*ll*0) scans through the NiO reflection show a similar difference between the field dependence of the FWHM after cooling in zero field and cooling in a 6 T field (Fig. 2).

Upon field cooling, domain walls both parallel and perpendicular to the growth direction lock into the antiferromagnetic layer, presumably due to the exchange coupling between the NiO antiferromagnetic moments and the Fe_3O_4 moments that are aligned parallel to the cooling field. After cooling in zero field, the domains are larger, but reversibly decrease in size in high fields (Fig. 2) as the magnetic frustration increases. Consistent with several theoretical predictions [2,3] we believe that exchange biasing may originate from magnetic frustration that leads to "frozen" domain walls in the field-cooled state.

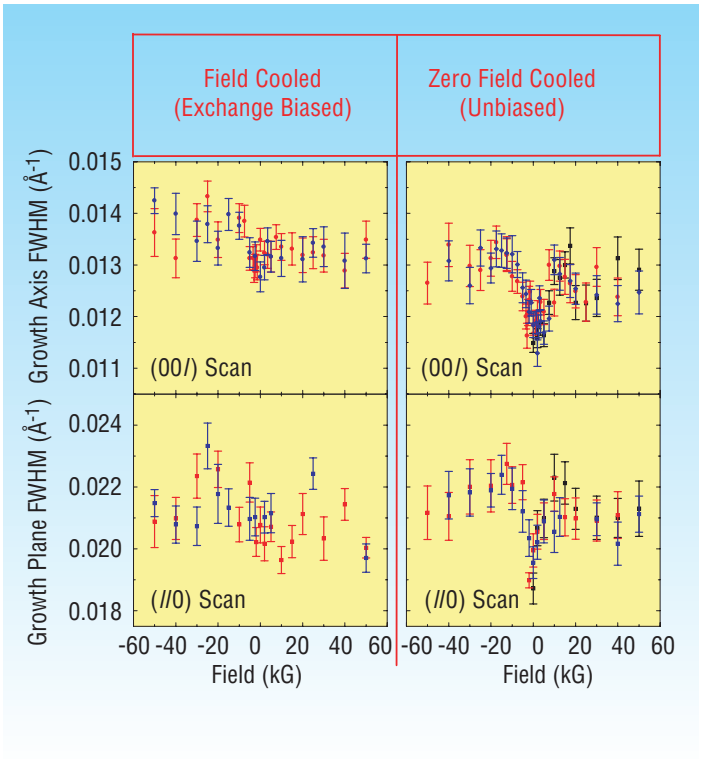

FIGURE 2. Full-width-at-half-maximum (FWHM) of the (111) NiO reflection as a function of field at 78 K after cooling in a 6 T field and after cooling in zero field. The top plots show the FWHM of the peak along the (00*l*) direction. The bottom plots show the FWHM from (*//*0) scans. The black, blue and red data are from each field cycle.

We also observe that the magnitude of the ordered NiO moments in all four of the {111} domains in the Fe_3O_4/NiO superlattices depends upon applied field. After cooling in a large vertical field, the intensity of the antiferromagnetic (111) NiO reflection reversibly decreases as the field is increased. (The behavior is qualitatively similar after cooling in zero field.) Analogous field-cooled measurements of the $(1\bar{1}1)$ NiO peak were performed in a horizontal field and surprisingly show a comparable intensity decrease. Some of the NiO moments thus seem to disappear out of all four {111} domains. We speculate that these NiO spins may become disordered as a result of the high internal fields. Simultaneously, some of the NiO moments reorient perpendicular to the Fe_3O_4 magnetization direction in high fields [6]. While this "spin-flop" response is favored by some theoretical models [4], it does not appear to be directly responsible for the exchange biasing for these samples.

Future studies will focus on the characteristics and origin of the "spin-flop" response of the NiO spin structure to large magnetic fields. In addition, we will further explore the differences between the antiferromagnetic domains for the exchange-biased and unbiased conditions.

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