Neutron Reflectometry Studies on Magnetic Stripe Domains in Permalloy/Superconductor bilayers

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We explored changes in magnetic domain structures in a magnetic layer due to the onset of the superconductivity of an adjacent superconductive layer using neutron reflectometry. Magnetic domain structures in 1 μm thick permalloy (Py) films were studied as functions of magnetic field, temperature and under the influence of the onset of superconductivity in a neighboring layer. Bragg peaks in the off-specular scattering were observed at low fields following saturation with an in-plane field, which are attributed to the quasi-parallel magnetic stripes along the field direction. During the magnetization reversal from saturation, the stripe pattern shows increases in the period, the transverse coherence length (i.e., perpendicular to the stripes) and the amplitude of the out-of-plane magnetization component. The coherence length of the magnetic stripes is anisotropic in the remnant state with the longitudinal coherence length (i.e., along the stripes) being larger than the transverse one. The stripe period shows a weak temperature dependence between 300 K and 3 K, but no abrupt change in the period is observed when the temperature crosses the superconducting critical temperature.

INTRODUCTION

Interesting stripe patterns occur in many systems [1], for example, zebras, smectic liquid crystals, aligned copolymers and magnetic thin films etc. Stripe patterns in magnetic thin films are unique because of their electronic rather than steric origin and high tunability [2]. The tunability of the magnetic domain pattern is beneficial because it not only enables potential applications in logic and memory devices [3-4], but also allows modification of the properties of layers directly coupled to the magnetic layer. For example, in artificial ferromagnetic/superconducting (FM/SC) hybrids, magnetic domain walls have been used to spatially confine the superconductivity [5-7], while magnetic domains have been used to guide and pin vortices in the adjacent superconducting layer [8-12]. Most studies focus on how superconductivity is influenced by the magnetic structure of the ferromagnetic layer, such as effects from stray fields [8,10] or induced exchange fields [13,14], inverse proximity effect [15], and induced triplet superconductivity [16,18]. In these studies, it is typically taken for granted that the magnetic configuration of the FM layer remains intact upon the onset of superconductivity in the adjacent layer, because the energy scale associated with magnetization is typically much larger than that of superconductivity. However, the long range effects from the interactions of FM stray fields with SC screening currents can change magnetic domain patterns. Thus, recently there is increased interest in exploring how the onset of superconductivity modifies the magnetic structures in FM/SC bilayers. Several techniques have been employed, including X-ray magnetic circular dichroism [19], SQUID magnetometry [20] and magneto-optical imaging techniques [21,23]. Here neutron reflectometry has been used to probe changes of the magnetic stripe domains in permalloy (Py) films in Py/Nb and Py/MoGe hybrids as a function of magnetic field and temperature. These specific hybrids are of interest as previous work has found a significant influence from the magnetic domain structure on the superconducting vortex dynamics in Py/MoGe hybrids [9,10] and on vortex formation in Py/Nb bilayers [24,25]. However, whether the onset of superconductivity in turn modifies the magnetic structure has not been investigated in these systems. Here, we have employed neutron reflectometry to determine changes in magnetic domain structures in Py films as functions of magnetic field, temperature and under the influence of the onset of superconductivity in a neighboring Nb (or MoGe) layer.

EXPERIMENTAL TECHNIQUES

Neutron reflectometry allows to probe variations of the film properties as a function of depth [26] and to determine in-plane correlations with sensitivity to the orientation of the magnetization vector [24,29]. For the specular neutron reflectivity, the incident angle of the neutron beam relative to the sample surface is equal to...
the reflected angle (\(\theta_i = \theta_f\)) and thus the wavevector transfer \(Q\) is equal to \(Q_z\) (see Fig. 1). Hence, specular reflectivity is determined by the depth profiles of both the chemical structure and the magnetization along the film stacking direction \(z\) [20, 30]. Off-specularly reflected intensity (\(\theta_i \neq \theta_f\)), originating from in-plane correlations along \(x\), has a non-zero in-plane component of the wavevector transfer (\(Q_x \neq 0\)) [31]. Correlations along \(y\) result in scattering out of the reflection plane (\(Q_y \neq 0\)), which is typically referred to as Grazing Incidence scattering (GIS, either diffraction or small angle scattering) [31, 32]. As the magnetic scattering cross-section is zero when \(Q\parallel M\), the specular polarized neutron reflectivity can only determine the magnitude and orientation of the magnetization components within the plane \((M_x, M_y)\), however off-specular and grazing incidence scattering are sensitive to modulations of \(M_z\). Further sensitivity to the direction of the magnetization is obtained by comparing spin-flip and non-spin flip intensities. Note that spin-flip intensities are solely determined by magnetization components that are perpendicular to the neutron spin quantization axis. This axis, i.e., polarization direction, is typically determined by the direction of the applied magnetic field.

At remanence after in-plane saturation, the domain structure of a thick (~1 \(\mu m\)) Py film consists of the quasi-periodic stripes with spatially oscillating in- and out-of-plane components of magnetization, as illustrated by a magnetic force microscopy (MFM) image in Fig. 1 [2, 33, 34]. This pattern, forming above a certain critical film thickness, is the result of a growth-induced perpendicular anisotropy [35]. The stripe domains form predominantly along the original saturating field direction, yet contain both disclination and dislocation defects. These defects are important for stripe ordering and limit the coherence length of the domain pattern [1]. The contrast in the MFM image of a Py film in Fig. 1 arises from the variation of the out-of-plane magnetization component that gives rise to a modulation of the scattering potential transverse to the stripe direction, thus can effectively scatter neutrons [36]. In an analogy, the stripe pattern behaves like a one dimensional micrograting for neutrons, and gives rise to Bragg scattering peaks in off-specular or grazing incidence directions, the positions of which depend on the period of the grating. Previously, GIS has been used to study the out-of-plane magnetization of magnetic stripe domains [27, 32, 37] and magnetic vortex cores [38] with correlations lengths below 100 nm. Here, we evaluated the off-specular scattering using neutron reflectometers, which characterizes larger correlations (> 500 nm [32]) and has so far only been used to probe domains with an in-plane magnetization or structural correlations of the surface [29, 39]. In a typical neutron reflectometer, the beam is relatively divergent along \(y\) to maximize the intensity, rather than being highly collimated in all directions as required for resolving grazing incidence scattering.

**SAMPLES AND INSTRUMENTS**

The nominal structures of the two studied samples are Nb (100 nm)/SiO\(_x\) (10 nm)/Py (1 \(\mu m\))/native SiO\(_x\)/Si substrate, and Mo\(_{70}\)Ge\(_{21}\) (40 nm)/SiO\(_x\) (20 nm)/Py (1 \(\mu m\))/native SiO\(_x\)/Si substrate, which are referred as the Nb/Pt and MoGe/Pt samples below, respectively. The Py (Ni\(_{70}\)Fe\(_{21}\)) and MoGe layers were grown by dc sputtering at room temperature at a base pressure of 1.5 \(\times 10^{-7}\) Torr. The Nb films were grown in a dedicated dc sputtering system at a pressure of 5.8 \(\times 10^{-9}\) Torr. A SiO\(_x\) layer was deposited between the FM and SC layers in order to suppress the proximity effect. The superconducting critical temperatures \((T_C)\) are 6.2 K [9] and 9.0 K [21] for the MoGe/Pt and Nb/Pt films, respectively. Superconducting MoGe and Nb films have quite different penetration depths, thus they might display different screening effects on the stray fields from the underlying Py films.

Neutron reflectivity experiments were performed using the Asterix reflectometer at the Lujan Neutron Scattering Center at Los Alamos National Laboratory and the Magnetism Reflectometer beamline at the Spallation Neutron Source at Oak Ridge National Laboratory. Both instruments use the time-of-flight technique and have position sensitive detectors, allowing the reflected intensity to be determined for a range of \(Q_z\) and \(Q_x\) values, respectively, with one setting of the incident angle \(\theta_i\). The
RESULTS

Field Dependence

Experiments were initially performed above $T_C$ of the superconductors and at various applied magnetic fields in order to establish the sensitivity of the neutron reflectometry to the magnetic stripe domains within the Py layer. Above $T_C$, the characteristics of Py determined for either sample can be considered to be typical for both, because their nominal thicknesses are the same and only the top superconducting layers are different. Data was first collected at room temperature with an in-plane magnetic field $H_y = 5$ kOe, which is far above the saturation field, then consecutively at $H_y = 50$ Oe, 10 Oe, and -10 Oe. This field history is expected to align the magnetic stripes to the polarization direction of the neutron beam, as shown in Fig. 1. Figures 2(a) and 2(b) show the reciprocal space intensity maps from the Nb/Py sample at 5 kOe and -10 Oe, respectively, measured on the Magnetism Reflectometer with $\theta_i = 0.4^\circ$. At low fields, there is indeed off-specular scattering near $Q_x$ of $\sim 6 \times 10^{-3}$ Å$^{-1}$ that is absent at 5 kOe, which indicates that the off-specular scattering is related to the magnetic domains within the plane of the film. To enable determination of the peak position and width of the off-specular scattering more clearly, the measured scattering intensities were integrated along $Q_z$ and the 5 kOe data was subtracted as a background from the low field data, effectively removing any contribution from the chemical structure. The results, plotted as a function of $Q_x$, are shown in Fig. 2(c). From these curves, the peak position and width of the off-specular peaks were determined via fitting with a Gaussian function, from which subsequently the period and the transverse (i.e., perpendicular to the stripes) coherence length are calculated, as shown in Fig. 2(d). The period can be determined with a relative uncertainty of about 0.5-1.5%, depending on the field. The measured period is around 1 µm, which is close to the value being calculated based on the film thickness, magnetic anisotropy, exchange constant, and saturation magnetization. In the range of 50 Oe to -10 Oe during the descending field scan, the period increases about 3.3 nm per Oe (a relative change rate of $\sim 0.3\%$ per Oe). A similar trend of the field dependence has been observed by magnetic force microscopy and reproduced by two-dimensional micromagnetic simulations. Since neutron scattering is a non-local probe, it allows not only to determine the average period of the stripes, but also to extract a statistical average of the coherence lengths of the stripe domains. The transverse coherence length of the stripe pattern is estimated by the Scherrer equation, $L \sim 0.89 \times 2\pi / \sqrt{\beta_B - \beta_0}$, where $\beta_B$ and $\beta_0$ are the full width at half maximum of the off-specular Bragg peak and the instrument resolution, respectively. The instrument resolution of $3 \times 10^{-6}$ Å$^{-1}$ was estimated by the peak width of the specular reflection along $Q_x$ at 5 kOe. The obtained values of $L$ range from 4 µm to 8 µm. Note that this coherence length is not limited by that of the neutron source, which is about 25 – 50 µm along the transversal direction for this experiment.

Figure 2(e) shows the peak height and the calculated magnetic scattering amplitude, which increase as the field decreases from saturation. In the first order Born approximation, the Bragg peak intensity $I \propto N^2 \times a^2$, where $N$ is the number of the scattering objects within the coherence length and $a$ is the scattering amplitude of each object. Here the scattering amplitude is determined by the amplitude of the out-of-plane magnetization modulation.
Counts

40

80

120

0

is visible in the first case but is absent after the experiments were performed at room temperature on the orientations with respect to the neutron beam. The existence of the Py/Nb sample in two different stripes coherence length were determined by comparing the scattered intensities for the two orientations of the sample close to $Q_x = 0$. There is a significant amount of diffuse scattering around the specular reflection after the sample is rotated by 90°, i.e. when the magnetic stripes are parallel to the reflection plane, as shown in Fig. 4(b). From the width of the diffuse scattering, the longitudinal coherence length of the magnetic structure along the stripe direction is estimated to be 13 μm, which is roughly twice the transverse one. The origin of the anisotropy of the coherence length is very intriguing because for a perfect and infinitely large stripe pattern, the coherence length is isotropic and infinite in any direction. The origin of the anisotropy is likely caused by the magnetic structural defects. To our knowledge, the coherence length anisotropy of magnetic domain patterns has not been discussed in the literature, but it is certainly worth future investigation.

Temperature Dependence

The temperature dependence of the stripe domain period was investigated in both the MoGe/Py and Nb/Py samples. Figure 5(a) shows the temperature dependence of the off-specular Bragg peak position from the MoGe/Py sample. These experiments were performed using Asterix, with an incident angle of 0.8°. The sample was first saturated in an in-plane magnetic field of 540 Oe at 300 K and then the field was reduced to 8.5 Oe. Data were collected at each temperature during cooling from 300 K to 3.2 K. The period shows a weak temperature dependence. It gradually decreases about 1.5% when the temperature decreases from 300 K to 100 K, then barely changes below 100 K. There is no indication of an abrupt change in the period when the temperature crosses the $T_C$ of MoGe, which is 6.2 K. The Nb/Py sample was studied at the Magnetism Reflectometer in the vicinity of $T_C$, but now with fine temperature steps, as shown in Fig. 5(b). The experiments started from 10 K, and then cooled down to 7.2 K, the lowest temperature achievable during the experiments, and then warmed back to 9.6 K, with a step size of 0.4 K. Since the temperature

![Graph](image_url)
range was small during the experiments, all instrumental parameters, except the temperature, were kept fixed to avoid potential uncertainty from re-alignment between consecutive runs. Similar to the results from the MoGe sample, there is no significant or abrupt change of the off-specular Bragg peak position when the temperature crosses the $T_C$ of Nb, which is 9.0 K. From these two experiments it can be concluded that there is a slight decrease in the period of the stripe pattern in Py when temperature changes from 300 K to 100 K, and the upper limit of the relative change is $\sim$1% when the temperature cross the $T_C$ in the two samples.

It is worth noting that in the analysis of the off-specular scattering, the intensity along $Q_z$ was integrated, thereby eliminating any depth sensitivity. Therefore, these results do not rule out the possibility that the domain pattern at the top surface Py layer, closest to the superconductor, changes when the temperature crosses $T_C$. There is in fact a slight difference between the specular polarized neutron reflectivities above and below $T_C$ of the MoGe/Py sample. Figure 6(a) shows the specular reflectivities $R^+$ and $R^-$, measured with polarization of the incident neutrons parallel or antiparallel to the applied field, respectively at 7.5 K. Figure 6(b) shows the spin asymmetry, which is defined as, $\frac{R^- - R^+}{R^+ + R^-}$, at 7.5 K and 3.2 K. The spin asymmetry is slightly different for the two temperatures, indicating the in-plane magnetic induction of the sample has slightly changed upon crossing $T_C$. The specular reflectivity is affected by the depth profile of the in-plane components of $B$, thus the change in the spin asymmetry could be due to a change in the magnetic domain pattern, and/or a change in the profile of $B$ due to field penetration and vortices in MoGe.

For this experiment, quantitative analysis of the specular data is very challenging as it would require a full dynamic scattering theory to take into account contributions to the magnetic induction from the domains, stray fields at the surface of the domains [27], as well as variations of $B$ in MoGe below $T_C$. Due to these complications and the small change in the spin asymmetry, such an analysis would unlikely be able to determine the magnetization change, thus was not attempted. Overall, the off-specular results show that the long range effects, associated with interactions of the FM stray fields with the SC screening currents, barely changes the average magnetization domain patterns in the Py films.
reflectivity of a neighboring superconducting layer. There-
fore, the long range effects, associated with interactions
of the FM stray fields with the SC screening currents,
barely modify the average magnetization configurations in
these samples, although changes at the surface of Py
cannot be ruled out.

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FIG. 6. (a) Specular polarized neutron reflectivity measured
at 7.5 K; (b) Spin asymmetry above (7.5 K) and below (3.2 K)
the superconducting transition temperature of the MoGe/Py
sample.

SUMMARY

In summary, off-specular scattering resulting from the
out-of-plane magnetization components of magnetic
stripe domains in thick permalloy films in ferromag-
netic/superconducting hybrid structures has been suc-
cessfully observed in neutron reflectometry experiments,
which illustrates the feasibility of such studies. The stripe
pattern in Py is found to be anisotropic in the remnant
state, with the longitudinal coherence length (i.e. along
the stripes) being larger than the transverse one (i.e.
perpendicular to the stripes). The period, the trans-
verse coherence length, and modulation amplitude of the
out-of-plane magnetization component depend strongly
on the field amplitude, consistent with expectations. A
weak temperature dependence of the period is observed
between 300 K and 100 K, however a significant change
of the stripe pattern could not be observed when the
\( T_C \) of a neighboring superconducting layer. There-
fore, the long range effects, associated with interactions
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