Unconventional domain wall magnetoresistance of Nb-Ni-Nb planar structures below superconducting transition temperature of Nb

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(Dated: 23 April 2019)

Spin singlet Cooper pairs convert into spin-triplet Cooper pairs on passing through a magnetic inhomogeneity along the direction of propagation at the superconductor/ferromagnet(S/F) interface. In superconductor-ferromagnet hybrid spintronics structures the aspect of singlet-to-triplet conversion of Cooper pairs naturally becomes important, because in S-F structures Cooper pairs come in close proximity of the domain walls in the ferromagnetic materials, which are the purest forms of magnetic inhomogeneity in nature. Therefore, in addition to the usual S-F proximity effect, the domain wall specific transport properties also may show significant changes. Here we have studied one of the characteristic properties of domain walls, known as domain wall magnetoresistance (DWMR), in patterned Ni stripes in proximity with a Nb overlayer. In the normal state of Nb, the measured DWMR correlated well with the number of domain walls in the Ni stripes, imaged separately using Kerr microscopy. Interestingly, below the superconducting transition temperature of Nb, the DWMR of Ni layer shows an unconventional decrease in the field range where the number of domain walls becomes maximum. We have discussed this unconventional feature of DWMR from the perspective of singlet-triplet conversion through intrinsic domain walls.

PACS numbers: Valid PACS appear here
Keywords: Suggested keywords

I. INTRODUCTION

Spin singlet Cooper pairs, when injected across a superconductor(S)-ferromagnet(F) interface, decay within a distance of a few nm in the ferromagnet due to the strong magnetic exchange experienced by the Cooper pairs. Therefore, a complete synergy between superconductor and spintronics is possible only through the generation of long-range, spin triplet Cooper pairs at carefully engineered S-F interfaces. It has been predicted more than a decade ago that magnetic inhomogeneity at the S-F interface is the key ingredient for generation of triplet supercurrent from singlet supercurrent. In S-F systems with homogeneous ferromagnetic interface, Cooper pairs undergo spin mixing giving rise to short range $S_z = 0$ singlet and triplet components with a proximity length of the order of 1 to 10 nm. Introduction of magnetization non-collinearity at the S-F interface leads to spin rotation along with spin mixing which converts short range $S_z = 0$ triplet component into the long range $S_z = \pm 1$ triplet component. When a Cooper pair with $S_z = \pm 1$ triplet component propagates through a ferromagnetic layer, the exchange field of ferromagnet no longer has a pair-breaking effect on it.

In this direction, a long range supercurrent was reported in Josephson junctions with half metallic ferromagnetic CrO$_2$ barriers. However, the results were less reproducible due to the random nature of inhomogeneous magnetic states at the SF interface. Later on, a series of experiments were reported demonstrating the generation of triplet supercurrent in SFS Josephson junctions by carefully engineering the SF interface to be magnetically inhomogeneous. Recently, a series of experiments have been carried out to generate triplet supercurrent in Josephson junctions with textured ferromagnets, bilayer and trilayer ferromagnetic regions, spin injection, and via spin-active interfaces, by creating a misalignment of magnetic moments at the SF interface. Evidences of induced triplet superconductivity have also been reported using scanning tunneling spectroscopy of SF bilayers, Andreev spectroscopy in SF junctions, conductance measurements, and critical temperature measurements of S-F spin-valve. In a recent report by Robinson et al., an in-plane Bloch domain wall was created artificially in Gd by using non-parallel alignment of the Ni layer moments in a Ni-Gd-Ni trilayer in the Nb-Ni-Gd-Nb Josephson junction. An enhancement of supercurrent was observed in this structure by modifying the low temperature DW state in Gd. The magnetic inhomogeneity has been created artificially in all these reports.

One of the purest forms of magnetic inhomogeneities exist as the ferromagnetic domain walls (Bloch and Neel) in ordinary ferromagnetic materials. It is therefore natural to wonder if domain walls also convert singlet to triplet Cooper pairs. Infact, theoretically, it has been predicted more than a decade ago that a magnetic do-

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main wall (DW) at the SF interface can produce triplet Cooper-pairs. There is, however, fundamental difficulty in implementing this concept because, a domain wall exists only at the interface of two ferromagnetic domains. In order to utilize the domain wall as a singlet to triplet converter, singlet Cooper pairs need to be pumped from one side of the domain wall to the other side. However, the ferromagnetic exchange field in the domains forbids the presence of spin-singlet Cooper pairs. Therefore, it is not practical to perform a measurement of current, directly verifying singlet-triplet conversion process of domain walls in S/F structures.

In this report, we have taken a simple, indirect approach to verify whether domain walls can cause singlet-triplet conversion. As shown in Fig 1(a), we have measured the DWMR above and below $T_c$ of a Nb-Ni bilayer stripe by making a gap in the Nb layer. In this geometry, during the magnetization reversal process, domain walls move in the ferromagnetic layer under the superconducting layer. It is well known that the appearance of domain walls in constricted ferromagnetic regions, such as stripes, causes an increase in resistance due to spin accumulation near domain walls, usually termed as domain wall magneto-resistance (DWMR). Typically DWMR follows the magnetic hysteresis curve maximizing near the coercive field of the ferromagnet. Unlike previous reports on S-F bilayers, where the S layer carries all the current, in this geometry the gap in the S-layer (Fig. 1(a)) forces the current to pass through the Ni layer. As a result, it becomes possible to measure the effect of Copper pair diffusion on the magnetoresistance of domain walls. In the normal state of Nb we are able to correlate the measured DWMR of this planar structure (with a gap in Nb layer) with number of domain walls in the Ni layer. However, in the superconducting state of Nb we observe an unconventional decrease in DWMR in the field range where the number of domain walls become maximum. We have discussed this unconventional behavior from the perspective of proximity effect at the S-F interface, domain wall superconductivity, vortex locking-unlocking effects, and generation of intrinsic domain wall induced triplet correlations.

II. EXPERIMENTAL DETAILS:

Nb-Ni thin films with thickness of 55 nm and 100 nm were prepared at room temperature using dc-magnetron sputtering of high purity(99.999%) niobium and nickel targets on cleaned Si-SiO$_2$ substrates. Deposition of the films was carried out at a base pressure of $1 \times 10^{-9}$ mBar in an ultra high vacuum chamber. Optical lithography and reactive ion etching techniques were used to fabricate the Nb-Ni-Nb planar structures as shown in Fig 1 (a). Four probe electrical transport measurements were carried out using Cryogenic PPMS. The voltage was measured by reversing the current and averaging over five measurements. The temperature was stable within a range of ±3 mK range. Magnetization measurements were carried out using Vibrating sample magnetometer. The magnetic domains in patterned structures were imaged by magneto-optical Kerr microscope in longitudinal mode for an in-plane magnetic field applied along the length and width of the stripes.

III. RESULTS

A. Domain wall magneto-resistance in Nb-Ni stripes:

We have lithographically patterned stripes of width 6 $\mu$m from Nb/Ni bilayer thin films as shown in the schematic of Fig.1(a). The thickness of the niobium layer was chosen to be ~55 nm which is above the coherence length of niobium (~40nm). Ni has been chosen as the ferromagnet layer because of its high exchange field
and the consequent low singlet pair coherence length (~4 nm)\textsuperscript{46}. The thickness of nickel layer was chosen as 100 nm, which, from earlier reports\textsuperscript{30,47}, exhibits Bloch domain walls. A 3 \(\mu\)m wide gap in the top Nb layer, as shown in the Fig.1 (a), was created in the central part of the stripe which was 6 \(\mu\)m wide. The large gap in the Nb layer excludes any possibility of Josephson coupling between the two Nb ends through singlet supercurrent. Fig. 1(b) shows the schematic diagram of a Bloch domain wall in the Ni layer, emphasizing the magnetic inhomogeneity. As discussed earlier, the gap in the superconducting Nb layer promotes diffusion of Cooper pairs into the ferromagnetic Ni layer at temperatures below superconducting transition (\(T_c\)), as shown in Fig. 1(b). In the absence of the gap, the superconductor would completely bypass the Ni layer, as shown in the supplementary Fig. S2. In fact, we find that the geometrical gap in the superconducting layer promotes diffusion of Cooper pairs well beyond the gap dimensions. In Fig. 1(c), we show the residual resistance in the superconducting state of such a Nb/Ni stripe with voltage measured at three different separations (varying between 1 mm and 1.5 mm) in the gap region, keeping the bias current contacts unchanged as shown in the inset of Fig. 1(c). Transition temperature in all cases was 8.5 K as shown in another inset of Fig. 1(c). Clearly, larger separation of the voltage contacts leads to a larger resistance below the transition temperature of Nb. One can argue that the observed effect is due to charge imbalance, which is a nonequilibrium phenomenon arising when the quasiparticle current in a normal metal converts to supercurrent in a superconductor. According to previous reports\textsuperscript{50,51}, the characteristic feature of charge imbalance is an increase in resistance below the superconducting transition which is not the case here. Therefore, the possibility of charge imbalance is excluded in the present experimental geometry. Although the major part of the resistance below \(T_c\) of Nb, in this geometry, comes from the Ni in the gap region, Fig. 1(c) shows that there is some contribution to resistance from Ni beyond the gap indicating diffusion of Cooper pairs beyond the gap region.

In Fig. 2, we present the magneto-transport behavior of the Nb/Ni planar structures at temperatures above the superconducting transition in presence of an in-plane applied magnetic field. These measurements are performed for a sample different from the one shown in Fig. 1(c). Panel 2(a) shows the response for magnetic field parallel to the length while panel 2(b) shows the response in a field perpendicular to the length of the stripe. Henceforth, keeping in mind that magnetic field is always applied in the plane of the substrate, throughout the text, we will refer to these two field configurations as parallel and perpendicular field, respectively. Intrinsic magneto-resistance of nickel was observed in parallel field (In Fig 2(a)), where the MR minima typically corresponds to the coercive field\textsuperscript{14}. This was found to be same irrespective of change in temperature value up to 30 K, due to very high Curie temperature\textsuperscript{34,46} of nickel. In perpendicular field configuration, MR peaks were observed with peak amplitude of 5.9 \(m\Omega\) at the coercive field, as shown in Fig 2(b). The peak amplitude was found to be same for various temperatures up to 15 K. In addition, we note that these peaks in MR, in perpendicular configuration, appear at field values corresponding to the minima in MR in parallel configuration, shown in panel 2(a). Usually, in a ferromagnetic film, the number of domain walls is maximum near the coercive field\textsuperscript{14}. Therefore, the origin of MR peaks in perpendicular configuration can be correlated to the number of domain walls. In order to verify this, we performed Kerr microscopy of nickel stripes of similar thickness in parallel and perpendicular configuration. As shown in panel 2(c), only two domain walls were observed in parallel configuration for fields applied near the coercive field. On the other hand, large number of domain walls were observed in perpendicular configuration near the coercive field as shown in panel 2(d). Therefore, in the perpendicular configuration, the DWMR contributions of a large number of domain walls add up to give the observed peaks in the MR curves. Whereas in the parallel configuration, due to the low number density of domain walls, no DWMR peaks were observed. The observation of a clear DWMR peak in the perpendicular field configuration ensures that, in this measurement geometry, an injected current passes through a large number of domain walls in the nickel stripe. Therefore, below

FIG. 2. Domain wall magneto-resistance of Nb-Ni-Nb planar structures for temperatures above \(T_c\) of Nb. (a) MR curves for magnetic field applied along the length of stripes show the intrinsic magnetoresistance of nickel for different temperatures above \(T_c\). (b) MR curves for magnetic field applied perpendicular to the length of stripes show the domain wall magneto-resistance (DWMR) in addition to intrinsic MR of nickel. The MR curves are similar for different temperatures above superconducting transition for magnetic field applied along and perpendicular to the length of stripes. (c) Longitudinal Kerr microscope image for magnetic field applied perpendicular to the length of stripes of nickel shows the two domain walls propagating towards the centre. (d) Kerr microscope image for magnetic field applied along the short axis of stripes shows the multiple domain walls in nickel. Both the Kerr measurements have been done at room temperature. Red color and blue color corresponds to the domains orienting in opposite direction.
the $T_c$ of Nb in the gapped Nb-Ni bilayer stripes, it is more probable that a significant number of diffused singlet Cooper pairs will encounter a domain wall directly. Therefore, this is a suitable measurement configuration to look for any signatures of singlet to triplet conversion through domain walls upon decreasing the temperature below superconducting transition.

B. Magneto-transport below transition temperature

In Fig. 3, we present the magneto-transport behavior of the Nb/Ni planar structures at temperatures below the superconducting transition in perpendicular configuration. Fig. 3(a) shows the magnetoresistance (MR) curve measured at 8K which is very close to superconducting transition ($T_c \sim 8.3K$). Two distinct MR peaks were observed whose amplitudes were found to be more than 50 times higher than the normal state DWMR shown in Fig.2(b). Since this measurement was done very close to the superconducting transition, the Nb layer is expected to respond to domain wall stray fields very sensitively. Therefore, the origin of MR peaks observed at this temperature is the usual suppression of superconductivity due to the out of plane component of domain walls present in nickel as observed in earlier reports. The overall domain wall stray field maximizes near the coercive field, the MR peaks, in this case appear near the coercive field. At lower temperatures, superconductivity is less sensitive to the out of plane stray field of nickel domain walls. Therefore, these peaks indicating suppression of superconductivity should diminish at lower temperatures. This is what we observe in the MR data measured at 7 K, as shown in Fig. 3(b). Moreover, we observe a DWMR peak with peak amplitude of 0.2 mΩ at 7K as shown in the inset of Fig. 3(b). The DWMR peaks corresponds to the coercive fields similar to the RH curve in the normal state as shown in Fig. 2(d).

Fig. 3(c) and Fig. 3(d) show the MR measurements at a temperature of 6K with magnetic field swept in negative (+ve to -ve field) and positive (-ve to +ve field) directions, respectively. Since the effect of domain wall stray field was minimized below 7 K as shown in Fig.3(b), the DWMR peaks, which is a much smaller effect, was possible to observe. However, we note a significant change in the nature of the DWMR peaks compared to the corresponding DWMR peaks above $T_c$. In the normal state of Nb, in the negative (positive) sweep of magnetic field, the DWMR peak was observed at the negative (positive) coercive fields (Fig. 2(b)). In Fig. 3(c) and (d), however, we observe the DWMR maxima on the opposite side, i.e. for negative (positive) field sweep the peak value appears in the positive (negative) field ranges. It can be seen in Fig. 3(c) that while decreasing the magnetic field from positive saturation the DWMR effects start showing an increase in resistance as soon as domain activity sets in. However, below certain field (in the range of field where the number of domain walls become significant) the increase in resistance is curtailed. Fig. 3(d) shows the same feature while increasing the field from negative saturation field. The sharp spikes represent jumps in resistance due to abrupt re-configuration of vortices as reported earlier. Below $T_c$ the domain structure remains similar to the structure above $T_c$ apart from a decrease in domain size. Earlier report on Nb/Garnet bilayer has shown that superconductivity shrinks the domains but the domain structure remains same. Therefore, the decrease in resistance can not be explained due to change in domain structure with temperature. Also, the field dependent decrease in resistance can not be explained in terms of Nb-Ni proximity effect, which is present at all field values. The observed decrease in the DWMR, which is a property of the Ni layer, may be explained due to singlet-triplet conversion through domain walls in the Ni layer which reduces the effective resistance. Here we note that unlike planar Josephson junctions, the large ferromagnetic gap of $\sim 3 \mu m$, in this case, forbids any direct coupling between the superconducting electrodes at the gap. By studying the conductance-voltage characteristics, one can study the proximity effect at the interface of S-F hybrid, which is not the purpose of the present work. Rather, the purpose of this work is to look at the changes in DWMR due to a direct proximity of the domain walls with the Cooper pairs, which is possible to measure via magnetotransport measurements.

In order to explain the curtailed DWMR effect, in Fig. 4, we schematically show the different resistances seen
by the current at temperatures above and below the superconducting transition. For temperatures above the superconducting transition, both Nb and Ni layers are normal metals, and they carry the total current in parallel according to their resistance values. A current going through the Ni layer encounters the domains and domain walls of nickel. Therefore, the effective resistance seen by the current in Ni layer consists of resistance of domains $R_D$ and domain walls $R_{DW}$. It is well known from theory\textsuperscript{31–33} that the current passing through domain walls causes spin accumulation and hence gives rise to DWMR peaks in the MR curves as shown in Fig. 2(b).

For temperatures below the superconducting transition, Nb is fully superconducting and the resistance, $R_{Nb}$ of the niobium electrodes become zero. The singlet Cooper pairs which diffuse directly into the domain walls may convert into triplet pairs\textsuperscript{31–33}. The spin-diffusion length of Ni ($\sim 21 \pm 2$ nm)\textsuperscript{34} limits the decay length of triplet Cooper pairs beyond 23 nm, as predicted theoretically\textsuperscript{34}. These triplet Cooper pairs do not cause spin accumulation and therefore, may reduce the effective resistance observed in Fig. 3(c) and 3(d). Moreover, the size of magnetic domains are of the order of microns as shown in Fig. 2(b). Therefore, the singlet Cooper pairs diffusing into magnetic domains under niobium electrodes de-pair into normal electrons due to very short coherence length ($\sim 4nm$) of singlet Cooper pairs in Nickel\textsuperscript{34}. All these normal electrons give rise to spin accumulation and hence, the DWMR in nickel. The comparison of (supplementary) Fig. S1(a) and Fig. S1(b) shows that number of domain walls increases as one decreases the field from saturation and approaches the coercive field. This may give rise to an increase in singlet-triplet conversion and hence a decrease in resistance at a positive field on sweeping the field from positive saturation to negative saturation as shown in Fig. 3(c).

The current dependence of DWMR peaks at temperatures above and below the superconducting transition further supports the evidence of triplet correlations in this Nb/Ni/Nb planar structure. Fig. 5(a) and Fig. 5(b) shows the MR curves for different currents at a temperature of 10 K and 6 K. The curves at 6 K and 10 K have been shifted along the y-axis for clear comparison of DWMR peaks. The DWMR was found to be same irrespective of injected current at a temperature of 10 K, as expected\textsuperscript{31}. But for a temperature of 6 K i.e. below $T_c$, the DWMR was found to decrease with increase in source current as shown in Fig. 5(b). The inset of Fig. 5(b) shows the $\Delta R_{6K}$ for 6K data and $\Delta R_{10K}$ for 10 K data with respect to bias current. Here, $\Delta R_{6K}$ and $\Delta R_{10K}$ have been defined as the difference between the DWMR peak amplitude at a particular bias current with respect to DWMR peak amplitude at 100 $\mu$A at a temperature of 6 K and 10 K, respectively. We notice that $\Delta R_{10K}$ is zero for all the bias current values which means that DWMR peak amplitude is independent of applied cur-
rent at a temperature of 10 K. On the other hand, $\Delta R_{6K}$ increases with increase in bias current which means that the DWMR peak amplitude is decreasing with increase in applied current at 6K. This decrease in DWMR peak amplitude may be explained from the fact that total number of Cooper pairs increase with an enhancement in source current. Thus, the number of Cooper pairs crossing the domain walls also increases giving rise to more effective conversion of singlet Cooper pairs to triplet Cooper pairs. The number of singlet Cooper pairs in the Nb layer and the normal electrons in the Ni layer also increases with increase in source current. It is clear from Fig. 5(a) that DWMR is independent of the number of normal electrons passing through it. Thus the increase in number of singlet Cooper pairs in the Nb layer and the normal electrons in the Ni layer, have absolutely no effect on DWMR. Only the increase in singlet-triplet conversion contributes to the change in DWMR observed in Fig.5(b). Hence, there should be no spin accumulation at the domain walls due to the long range triplet Cooper pairs. In this way, the increase in triplet Cooper pairs will lead to a decrease in net spin accumulation and hence DWMR. This further gives an evidence for the generation of triplet correlations in the present experimental geometry.

IV. DISCUSSION

We have analyzed the observed anomalous decrease in the DWMR as a signature of singlet-triplet conversion through intrinsic domain walls of Ni layer in the field range of hysteresis loop where number of domain walls maximizes. Similar anomalous features in MR have also been observed in an earlier report\textsuperscript{13} on Nb/BaFe\textsubscript{12}O\textsubscript{19} bilayers where the authors have ascribed the dips in MR to the onset of domain and domain wall superconductivity. However, we must point out that these measurements were done close to the transition temperature where domain and domain wall stray field can have a large effect. This possibility can be excluded in our case because, our data (Fig.3) shows that below 7 K the stray field of domain walls no longer has any effect on MR. The observed changes in DWRM below $T_c$ can not be explained by usual proximity effect at the S-F interface because, interface proximity effect is not field dependent, unlike the case here. Another possible effect that may cause an apparent decrease in magneto-resistance in hybrid S-F systems is the vortex locking effects\textsuperscript{12}. With increasing bias current, the Lorentz force on any possible vortices in the Nb layer would increase. Therefore, if vortex locking would have been the cause of the observed resistance drop in the DWMR peaks (Fig.3(c)), the corresponding field values would have been different at all bias currents, unlike Fig. 5(b). However, the fact that the decrease in DWMR starts around the same field value for all bias currents indicates that the decrease is connected to the magnetization dynamics of the Ni layer which remains unaffected by the bias current.

V. CONCLUSION

In conclusion, we have studied DWMR for temperatures above and below the superconducting transition in a Nb/Ni/Nb planar structure. We observed unconventional features in DWMR for temperatures below the superconducting transition. In a Nb-Ni bilayer stripe geometry, by carving a gap in the Nb layer, we have been able to measure the DWMR of the Ni layer below $T_c$. In the conventional S-F bilayer geometries, used in the earlier reports, current is predominantly confined to the superconducting layers and the intrinsic DWMR property would not be possible to measure. Upon singlet-triplet conversion, the triplet Cooper pairs may reduce the effective resistance in the Ni stripe (below $T_c$) which showed up as a drop in the DWMR peak in the domain activity range of the hysteresis loop. We have excluded other possibilities leading to the observation of similar drop in MR, such as domain and domain wall superconductivity, S-F proximity effect, and vortex locking-unlocking effects. Although the triplet super-current generated by domain walls is not directly accessible, our discovery would motivate further studies in the field of superconducting spintronics. If gaps of dimensions close to the domain wall width can be fabricated then a single domain wall can be pinned at the gap to achieve a triplet Josephson junctions in a planar geometry which would be useful for the field of superconducting spintronics.

ACKNOWLEDGMENTS

We acknowledge the funding from National Institute of Science Education and Research(NISER), DST-Nanomission (SR/NM/NS-1183/2013) and DST SERB (EMR/2016/005518) of Govt. of India

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