Reentrant superconductivity in HoNi$_5$-NbN-HoNi$_5$ nanostructures

Gyanendra Singh$^1$, P. C. Joshi$^1$, Z. Hossain$^1$ and R. C. Budhani$^{1,2(a)}$

$^1$ Condensed Matter - Low Dimensional Systems Laboratory, Department of Physics, Indian Institute of Technology Kanpur - Kanpur - 208016, India
$^2$ CSIR-National Physical Laboratory - New Delhi - 110012, India

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Abstract – Superconductivity (S) and ferromagnetism (F) are probed through transport and magnetization measurements in nanometer scale HoNi$_5$-NbN (F-S) bilayers and HoNi$_5$-NbN-HoNi$_5$ (F-S-F) trilayers. The choice of materials has been made on the basis of their comparable ordering temperatures and strong magnetic anisotropy in HoNi$_5$. We observe the normal state reentrant behavior in resistance vs. temperature plots of the F-S-F structures just below the superconducting transition in the limited range of HoNi$_5$ layer thickness $d_{HN}$ ($20 \text{ nm} < d_{HN} < 80 \text{ nm}$) when $d_{HN}$ is fixed at $\pm 10 \text{ nm}$. The reentrance is quenched by increasing the out-of-plane ($H_{\perp}$) magnetic field and transport current where as in-plane ($H_{\parallel}$) field of $\leq 1500 \text{ Oe}$ has no effect on the reentrance. The origin of the reentrant behavior seen here in the range $0.74 \leq T_{Curie}/T_C \leq 0.92$ is attributed to a delicate balance between the magnetic exchange energy and the condensation energy in the interfacial regions of the trilayer.

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Introduction. – The antagonistic order parameters of superconductor (S) and ferromagnet (F) lead to several fascinating effects in the transport and magnetic properties of thin film S-F heterostructures [1,2]. It has been noticed that there is a large suppression of the superconductivity at the S-F interface due to the strong pair breaking effect of the ferromagnet via spin-flip scattering and/or spin rotation. The ferromagnetic layer is also affected by the presence of the superconductor as the Cooper pairs entering the F region acquire a center-of-mass momentum due to the exchange field of the F. This adds an oscillating term to the Cooper pair wave function inside the ferromagnetic region [3]. The experimental studies on S-F bilayers [4–7], trilayers [8–10] multilayers [11–14] and S-F-S junctions [15–20] show effects such as the direct and inverse proximity effect, reentrant superconductivity and critical-temperature oscillations.

In addition, the phenomenon of long-range triplet pairing is possible when the coupled ferromagnetic layers have inhomogeneities in magnetization, which can be due to domain walls, spiral magnetism and spin scattering at the interface [19–21]. In the case of a F-S-F trilayer, for certain angles between the magnetization vectors of the F layers, a triplet pairing can be induced in the F layers [22,23]. As a result, the Cooper pair can survive in the ferromagnetic region up to a length scale of normal metal coherence length ($\xi_N$).

In most of the S-F hybrids studied till date, the F order sets in at a temperature ($T_{Curie}$) higher than the transition temperature ($T_C$) of the superconductor and thus the nucleation of the superconducting state is subjected to a robust exchange field of the ferromagnet. An equally important and perhaps much more illuminating option is to have a ferromagnet whose $T_{Curie}$ is lower than the $T_C$ of the superconductor. If the superconducting layer is sufficiently thin, one may see reentrant superconductivity due to the appearance of the ferromagnetic order just below the superconducting $T_C$. The heterostructures of NbN and HoNi$_5$ offer an ideal system to study the suppression of the F order due to superconductivity because NbN becomes superconducting at $\sim 16 \text{ K}$ and the bulk ferromagnetic ordering temperature ($T_{Curie}$) of HoNi$_5$ is $\sim 5 \text{ K}$. In our previous paper on NbN/HoNi$_5$ bilayers [24], we have reported a spectacular flux flow induced peak effect in the magnetoresistance $R(H)$ of the system at temperatures below which HoNi$_5$ is ferromagnetic. This observation was attributed to a spin reorientation transition induced by the in-plane magnetic field. These bilayer samples, however,
do not show any direct signatures of the competition between the F and S order parameters. In contrast, a trilayer film of HoNi$_5$/NbN/HoNi$_5$ displays a dramatic effect of the antagonism in $R(T)$ and $M(T)$ measurements as the system is cooled through the superconducting and magnetic transition temperatures. The most striking signature of the competition is a reentrant superconducting transition in the $R(T)$ data of the trilayers when the NbN films are made sufficiently thin such that the perturbation from the F layer is strong enough to suppress superconductivity over a limited range of temperature below the $T_C$ of NbN. We have established a critical range of $T_C$ of the trilayer in which the reentrance is seen. The reentrant behavior is also seen prominently in the $M(T)$ data. We attribute this phenomenon to the competition between superconducting condensation energy and ferromagnetic exchange energy just below the superconducting transition of the layer. We discuss that the exchange energy of HoNi$_5$ which grows as we decrease the temperature from $T_C$ suppresses the order parameter of NbN over a limited range of temperature. To the best of our knowledge we are presenting the first observations of reentrant superconductivity in S-F heterostructures of $T_{Curie} < T_C$, while it has been observed prominently in bulk superconductors such as ErRh$_4$B$_4$, HoMnO$_3$S$_8$ and HoNi$_2$B$_2$C [25–29].

Experimental details. – Thin films of NbN, HoNi$_5$ and their heterostructures were deposited on (100) cut MgO substrates using the pulsed-laser deposition technique. For HoNi$_5$, a stoichiometric polycrystalline target was ablated in 0.1 mbar neon environment and then a NbN film was deposited in 0.1 mbar $N_2$ pressure on top of the HoNi$_5$ layer. For the deposition of the top HoNi$_5$ layer, the nitrogen was flushed out and the growth was carried out at 0.1 mbar of neon. The growth temperature for all three layers was 200°C. The thickness $d_{N}$ of the bottom and top HoNi$_5$ layers was kept the same but varied from 20 to 80 nm in different samples and the NbN thickness ($d_{NbN}$) was changed from 10 to 30 nm for given $d_{HN}$. Further details of the growth condition can be found in previous reports [30,31]. For the measurements of the electrical transport, the samples were patterned into 15 $\mu$m wide lines by using a tungsten shadow mask and the Ar$^+$ ion milling technique. A schematic diagram of the sample geometry is shown in the upper inset of fig. 1(a).

Results and discussion. – We first present the results of X-ray reflectivity measurements on HoNi$_5$/NbN bilayers of $d_{HN} = 50$ nm to quantify the extent of the interface roughness. These data are shown in fig. 1(a). The best fitting of a genetic algorithm (solid line) yields a roughness of 0.7 and 1.3 nm for NbN/HoNi$_5$ bilayer with 30 and 10 nm thick NbN, respectively. The superconducting transitions as seen in resistivity measurements on HoNi$_5$/NbN-HoNi$_5$ trilayers of NbN thickness 10 and 30 nm are displayed in the lower inset of fig. 1(a). The trilayer with 30 nm NbN shows a sharp transition with critical temperature ($T_C$) of $\sim 11$ K. On reducing the thickness to $\sim 10$ nm, the onset of $T_C$ shifts down to $\approx 6$ K. This drop, however, is truncated by a valley and then upturn of $R(T)$ on reducing the temperature below $\approx 5.5$ K. The resistance goes through a peak at $\approx 5$ K followed by a sharp drop at lower temperatures. This behavior is similar to the reentrant phenomenon seen in bulk samples of HoNi$_2$B$_2$C, ErRh$_4$B$_4$ and HoMnO$_3$S$_8$ [25–29]. A magnified view of the reentrant nature of the $R(T)$ of the trilayer with 10 nm thick NbN is shown in panel (b) of fig. 1. The $R(T)$ is separated into three regions: a normal metallic behavior for $T > 6$ K, reentrant superconductivity (red bar) in the range 5 K $< T < 6$ K and zero resistance state for $T < 5$ K. No such reentrant behavior was seen in the trilayers with

![Fig. 1](image-url)
30 nm thick NbN. In the inset of fig. 1(a), we also display the \( R(T) \) data for a bare 10 nm thick NbN film. From a comparison of curve A and B, it is clear that \( T_C \) is suppressed drastically in the trilayer for a fixed NbN layer thickness.

At this stage it is important to recall the magnetic properties of HoNi\(_5\), which shows bulk ferromagnetic ordering at \( \sim 4.5 \) K. The magnetism in this compound is highly anisotropic due to 4f electrons of holmium. The easy axis of magnetization lies in the \( ab \)-plane of the hexagonal unit cell, but the magnetization for the field along the \( c \)-axis does not saturate at field as high as 15 T [32]. The HoNi\(_5\) thin films grown on (100) MgO investigated by our group [30] are polycrystalline in nature with grain size of \( \sim 10 \) nm. To establish magnetism and superconductivity in the HoNi\(_5\)/NbN/HoNi\(_5\) trilayer with 10 nm thick NbN, we have measured the temperature-dependent magnetization \( M(T) \) after zero-field cooling the sample and then applying a 100 Oe field along the out-of-plane direction. The trilayer used in the magnetization measurements was grown along with the sample used for transport measurements in the same run. The \( M(T) \) shown in fig. 1(c) rises sharply below 5 K, goes through a peak and then drops to a negative value, indicating strong diamagnetism. A magnified view of \( M(T) \) in the temperature range from 3 to 7 K is shown in the inset of the figure along with the \( M(T) \) of a bare HoNi\(_5\) film on MgO [30]. The \( T_{\text{Curie}} \) extracted from the behavior \( M \) in the critical regime \( \sim M_0(1 - T/T_{\text{Curie}})^3 \) is \( \approx 5.5 \) K and \( \beta \approx 0.52 \). As we see in fig. 1(b), the onset of superconductivity is at \( \approx 6 \) K. However, before a fully diamagnetic state could develop, the fluctuating Cooper pair density experiences the pair breaking field of the HoNi\(_5\) and hence there is no substantial change in the rising part of the \( M(T) \) curve. However, at still lower temperature the condensate becomes robust and a competition between diamagnetism and ferromagnetism leads to a peak in \( M(T) \) at \( \approx 3.5 \) K. On further lowering the temperature, the diamagnetism of superconducting NbN clearly dominates the ferromagnetic response of the system.

The appearance of the reentrance in trilayers also depends on the thickness of each HoNi\(_5\) layer. As seen in fig. 2(a), when \( d_{HN} \) is sufficiently thick (\( \approx 80 \) nm), the \( T_C \) of NbN is greatly suppressed with no evidence of reentrance. Also when \( d_{HN} \) is small (\( \approx 20 \) nm), the \( T_C \) of NbN remains robust with no sign of entry into the normal state on cooling below \( T_C \). In fig. 2(b) we show the variation of \( T_{\text{Curie}} \) of the trilayer as a function of the total thickness \( d_F = (d_{F1} + d_{F2}) \) of HoNi\(_5\) layers. Here we would like to emphasize that reporting \( T_{\text{Curie}} \) as a function of total thickness, though it appears unconventional is none the less justified as the magnetometer sees the response of the combined system. \( T_{\text{Curie}} \) is extracted from \( M \) vs. \( T \) data acquired by applying the out-of-plane magnetic field in the same manner as used for a plain HoNi\(_5\) film (see fig. 1(c)). \( T_{\text{Curie}} \) is defined at the inflection point of straight line fit to \( M(T) \) data. \( T_{\text{Curie}} \) drops by \( \approx 1.4 \) K on lowering \( d_F \) from 160 nm to 40 nm. This interesting observation is consistent with the drop in the ordering temperature seen when films are made sufficiently thin. Such behavior is seen in films of magnetic alloys and oxides when the extent of disorder increases on reducing the film thickness [33]. If there is no disorder, the ordering temperature should not be sensitive to reduction in thickness unless we approach fundamental limits of long-range ordering in 2-dimension. From these data a very interesting correlation evolves between the \( T_{\text{Curie}}/T_C \) of the F-S-F configuration and the observation of the reentrance. The later is seen when \( 0.74 \leq T_{\text{Curie}}/T_C \leq 0.92 \) (see inset of fig. 2(b)).

In order to get further insight of the reentrant behavior of superconductivity, we have measured the resistivity of the sample in the transition region in a magnetic field applied along the out-of-plane (\( H_\perp \)) and in-plane (\( H_\parallel \)) directions (perpendicular to the bar) of the trilayer. The results of the in-plane and out-of-plane field measurement are shown in fig. 3(a) and (b), respectively. The superconducting transition in the \( H_\perp \) configuration is much more sensitive to the field; it becomes significantly broader as we increase the field from 0 to 1400 Oe. An enlarged view of the behavior of minimum and peak in

**Fig. 2:** (Color online) (a) \( R(T) \) of HoNi\(_5\)/NbN/HoNi\(_5\) trilayers at zero field with five different thicknesses of HoNi\(_5\) \( \sim 20, 40, 50, 60, 80 \) nm. A 100 \( \mu \)A current is applied during the measurements. The NbN thickness was kept constant to \( \sim 10 \) nm for all the samples. (b) Variation of the magnetic ordering temperature of the trilayer as a function of the total thickness of the HoNi\(_5\) layer. The inset shows the dependence of \( T_{\text{Curie}}/T_C \) on \( d_{F1} + d_{F2} \). The shaded area range shows where the reentrance is seen.
Fig. 3: (Color online) (a) Temperature-dependent resistance of a trilayer with 50 nm thick HoNi$_5$ and 10 nm thick NbN at constant in-plane magnetic field varying from 0 to 1400 Oe. The inset shows a larger view of the data for the reentrant region. (b) $R(T)$ with field applied along an out-of-plane direction. A 100 μA current is applied along the bar for these measurements. The upper inset shows a clear view of the reentrant behavior as a function of $H_\perp$. The peak and dip in $R(T)$ are marked by $R_P$ and $R_{min}$, respectively. The lower inset shows the calculated value of $\Delta R = R_P - R_{min}$ and $T_{min}$ as a function of $H_\perp$.

$R(T)$ which characterize the reentrant transition is shown in the inset of fig. 3(b) for several values of $H_\perp$. The changes in the minimum have been quantified in terms of $\Delta R = R_P - R_{min}$ and $T_{min}(H_\perp)$, and are plotted in the lower inset of fig. 3(b). The $\Delta R$ and $T_{min}(H_\perp)$ drop monotonically with the applied field. The suppression of the peak here can be understood in terms of the reduction of the superconducting condensation energy with increasing the $H_\perp$. $H_\perp$ also leads to a large number of vortices in the system. The thermally activated flux flow can lead to the broadening of the resistive transition in the tail region of the curve.

The reentrant behavior shows a strong dependence on the current used for the resistivity measurements. In fig. 4(a) we show the $R(T)$ data in zero field for current varying from 1 μA to 500 μA. The changes in the $R(T)$ with current in the temperature regime where the two order parameters compete strongly, is quite different from the behavior seen under $H_\perp$ field (fig. 3(b)). Here at the lowest current, there is very little evidence of a minimum in the $R(T)$ below the onset of superconductivity. We only see a shoulder below which the resistance drops precipitously to zero. As the current is increased from 1 μA to 100 μA, the minimum in the $R(T)$ becomes pronounced and $T_P$ shows a small shifts to lower temperatures (see fig. 4(b)). A further increase in current to 500 μA leads to saturation of $\Delta R$. However, for $I \geq 500$ μA a flattening of the minimum and shift of the $T_P$ to lower values is observed (not shown here). The $R(T)$ in the tail portion of the transition ($T < T_P$) becomes progressively broader on increasing $I$ from 1 μA. While the reason for this strong current dependence is not understood fully, it is certainly not a heating effect. To further clarify the issue
of sample heating, we have measured current vs. voltage (I-V) curves at different temperatures across the superconducting transition in forward and reverse directions. We observe no hysteresis in I-V’s which could have resulted from a thermal lag between the temperature seen by the sensor and the actual temperature of the sample. A similar current dependence of the reentrant behavior has been reported by Rathnayaka et al. in HoNi$_2$B$_2$C samples [26]. There authors have reported an interesting correlation between the magnitude of the current dependence and the ratio of magnetic and superconducting transition temperature. The effect is seen when $T_m/T_C \geq 0.6$. The value of $T_m/T_C$ in our case is $\approx 0.88$. Furthermore, our observation of very little reentrant behavior seen at low currents is also similar to the behavior observed in the HoNi$_2$B$_2$C crystal [26].

We close the discussion by proposing a possible scenario for the observation of reentrance in superconductivity in our magnetic thin-film heterostructures. It is important to point out here that our observation of reentrant superconductivity as a function of temperature appears different from the so-called reentrant behavior recently reported in Nb-Cu$_{41}$Ni$_{59}$ bilayers as a function of the CuNi layer thickness [6,7]. Such observations have been made earlier as well [4,5]. This arises because the superconducting pair amplitude (PA) is oscillating inside the F, and depending on the thickness of the F layer [1,2], the PA at the interface region of the F can be very large or nearly zero, leading to higher or lower $T_C$, respectively. A sudden entry into (or out of) the normal state can also occur if the magnetization vectors of the F layers transit from a parallel to an antiparallel configuration as a function of temperature. These spin accumulation [34] and Cooper pair averaging effect have been addressed in the past [35]. However, such magnetization reorientation transition is more likely to occur in a magnetic field than by temperature.

A more intuitive explanation for the RES can, however, be given in terms of the proximity effect in a normal (N) and ferromagnetic metal. The decay of the superconducting order parameter in N and F layers is given by the length scale $\xi_N = (hD_N/2\pi k_B T_C S)^{1/2}$ and $\xi_F = (hD_F/2\pi k_B T_C S)^{1/2}$, respectively [5]. Here $\xi_N$ and $\xi_F$ are coherence length, and $D_N$ and $D_F$ are diffusion coefficients in the normal and ferromagnetic metal, respectively. The other relevant length scale in the problem is $\xi_F = (hD_F/E_{ex})^{1/2}$, which defines the decay of the S order parameter in the F layer. It is the measure of the length scale over which superconductivity is induced in the F layer. The exchange splitting of the conduction band $E_{ex}$ is related to the exchange integral $I$ and the magnetic moment $\mu_F$ as $E_{ex} = I \mu_F$ [36]. Since $E_{ex}$ goes to zero at $T_{Curie}$, $\xi_F$ diverges on warming the sample toward the magnetic ordering temperature. This divergence is however cut off by $\xi_N$. This is the unique feature of our experiment because $T_{Curie} < T_C$. As long as $T_{Curie} < T < T_C$, the superconductivity in the effective thickness of $d_S + 2\xi_N$, where $d_S$ is the thickness of the superconducting layer, leads to a drop of resistance. But as soon as $T \approx T_{Curie}$, this effective thickness starts decreasing not only because $\xi_F < \xi_N$ but also because of the pair breaking effects of the magnetic layer inside the superconducting film. The consequence of this would be a rise in the resistance. We have estimated the diffusion coefficient $D_F$ of HoNi$_5$ from the specific-heat data [37,38] and measured resistivity of a 40 nm thick HoNi$_5$ film, which is $\approx 280 \mu \Omega$cm. Since the elastic mean free path for this value of resistivity is already of the order of interatomic distance, we expect a marginal increase of the resistivity of the thinner HoNi$_5$ film due to a size effect [39]. Band structure calculations of $E_{ex}$ for HoNi$_{5}$ [40] suggest a value of 0.5 eV, which yields $\xi_F(0) \approx 0.3$ nm. Since for a strong ferromagnet like Fe, $\xi_F$ is only $\approx 1$ nm [41], we believe that this calculation overestimates $E_{ex}$. $\xi_N$, on the other hand, is 2.7 nm. As the correlation length in HoNi$_5$ reduces on going through the magnetic transition, a reentrance into the normal state is expected. However, at still lower temperatures, the order parameter in the S film becomes robust enough to short circuit the HoNi$_5$ films and the resistance would start approaching the zero value. These semi-quantitative arguments are consistent with the results in fig. 1.

Recently Wu, Valls and Halterman [42] have proposed an interesting possibility of observing a temperature-dependent reentrant superconductivity in a F-S film if the F layer has a spiral magnetic order, such as that exists in holmium. The magnetic structure of HoNi$_5$ is not fully understood, although magnetization measurements show strong anisotropy with the ab-plane of hexagonal unit cell being the easy plane. The HoNi$_5$ film grown on the MgO substrate shows inhomogeneities in magnetization due to a different orientation of nanostructures, as we have discussed in previous report [30]. It is possible that the inhomogeneous magnetization of HoNi$_5$ allows a reentrant behavior in the Wu, Valls and Halterman sense [42]. This effect is presumably accentuated by the presence of HoNi$_5$ on both sides of the thin NbN layer.

**Conclusion.** In summary, we have carried out a detailed study of the electronic transport in F-S-F thin films where the F and S order parameters are of comparable strength. We see a robust reentrant superconductivity over a critical range of the ratio of the magnetic and superconducting transition temperature of the F-S-F trilayer ($0.74 \leq T_{Curie}/T_C \leq 0.92$). The RES seen here is different from the more common reentrant behavior observed as a function of the sample geometry. Many features of the RES reported here are similar to those found in bulk magnetic superconductors consisting of ternary and quaternary alloys of rare earths [28,29].

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