Enhanced vortex pinning in Nb using proximity effect through organic molecules

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Abstract

While the superconductor proximity effect is well understood in layered superconductor/normal-metal junctions, its understanding is quite limited in systems involving nanoparticles (NPs) and molecules. In recent studies, a unique inverse proximity effect phenomenon was found in which the critical temperatures of Nb films surprisingly increased upon the chemical attachment of gold NPs.

Concomitantly, the tunneling density of states on and around the gold NPs was significantly modified, showing either zero-bias peaks or the development of proximity gaps in the NPs. These results seem to be related to the molecule-mediated coupling strength. Here, we study the strong molecular coupling regime of such an architecture, for which proximity gaps are induced in Au NPs. We show that significant pinning is induced in a periodic array of Au NPs coupled to a superconducting surface via organic molecules. The pinning potential in this case is stronger than the potential achieved through the direct proximity of Au or Ni islands to the superconducting surface. A matching field magnetoresistance signal can only be identified using the hybrid Au/organic-linker/Nb system. In this case, the matching vortex lattice density is higher than the saturation number. These results suggest that the NP-Nb electrical coupling through the molecules induces a resonance behavior, which modifies the local pairing amplitude.

Superconductivity is a condensation phenomenon involving electronic pairs. As such, superconductivity involves two imperatives: the binding of electrons into pairs and the establishment of phase coherence between the pairs. Phase coherence can also occur between adjacent layers via a mechanism reminiscent of the superconductor (SC) proximity effect (PE). In 1976, Ginzburg [1, 2] proposed the use of layered metal–organic–metal hybrids to explore the possibility of increasing the critical temperature, $T_C$, by the ‘exciton mechanism’. The excitonic mechanism in hybrid systems has been studied theoretically by various authors, who have suggested similar geometries to ours (see below) [3]. Although the formation of bound excitons for pairing is not essential, and it is electron–hole interactions that are ultimately involved, the ‘exciton mechanism’ label has remained for this area of study. Often, the basic model of pairing forces is based on a total dielectric function approach, and some researchers claim that no coupling is possible [4]. These problems with the formalism seem to arise due to the neglect of local field effects [5]. Recently, using a hybrid superconductor–molecule–NP geometry, it was shown that nanostructured components inherently have local fields that permit coupling, which are lacking in the more conventional metal–semiconductor geometries [6]. This nanostructured geometry provides an opportunity for new types of interactions. In a recent study of such a system, two experimentally unique PE-related phenomena were found, in which $T_C$ and the critical current of a Nb film increased upon chemically linking gold NPs [6]. Concomitantly, the tunneling density of states (DOS) around the gold NPs was significantly modified, showing either zero-bias peaks or proximity-induced gaps. These
unexpected PE phenomena, which are relevant to the emerging field of molecular electronics [7], are not yet well understood.

The superconductor PE occurs when a superconductor is placed in good electrical contact with a normal metal. The resulting critical temperature of the superconductor is then reduced, and signs of weak superconductivity are induced in the normal metal [8, 9]. The PE is well understood for ‘conventional’ superconductors and is known to occur via Andreev reflections. A hole impinging on the superconductor-normal interface from the normal side is retro-reflected as an electron, consequently destroying a Cooper-pair in the superconductor (and diminishing the order parameter). The overall outcome can be thought of as the transfer of a Cooper pair from the superconductor to the normal metal, where it induces superconducting correlations up to the point at which the electron and hole lose phase-coherence [9]. The PE requires, in general, good electrical contact and Fermi wave vector matching. Nevertheless, and quite surprisingly, proximity effects were also observed for non-metallic systems, such as Anderson insulators [10], molecules [11] and hybrid superconductor quantum-dot devices [12, 13]. However, only recently [14] was the direct manifestation of PE in the local DOS of hybrid systems containing organic molecules observed.

Control over the local DOS and order parameter is instrumental for creating artificial vortex pinning centers, an issue that continues to attract considerable attention also from the application viewpoint [14–17]. The dynamics of vortex formation is governed by the competition between the vortex pinning potential and the Lorentz force that compels the vortices to move and thereby dissipate energy. In general, artificial pinning defects change the properties of the superconductor by locally suppressing the superconductor order parameter, or condensation energy, in the vicinity of the defects, thus providing attractive potential for vortices. Ordered arrays of engineered artificial pinning sites have profound effects on the magnetic and magnetoresistance properties of superconducting films. In particular, in matching fields where the corresponding vortex lattice density is commensurate with that of the pinning array, a drop in the resistance can be observed. Since the density of vortices increases linearly with the magnetic field, a periodic (as a function of applied magnetic field) drop in the magnetoresistance is expected under such commensurate conditions, e.g., whenever integer numbers of the vortex density are equal to the pinning-site density. Numerous methods have been implemented to produce periodic pinning sites by modifying the condensation energy landscapes, including the deposition of arrays of magnetic and non-magnetic dots [15, 18] and the establishment of varying film thicknesses [19], film corrugation [20], anti-dots or holes [21–23] and lattice defects [24]. In matching field conditions, the critical current in proximity to the dot is increased [25, 26] and the total inter vortex forces are minimized according to the Abrikosov triangular lattice [27]. However, a prerequisite for the observation of effects related to any type of artificial pinning site (ordered or not) is that their pinning force should exceed that of the natural (defect-related) pinning of the host superconductor. Interestingly, no matching field effects were observed for Nb films upon which periodic normal or magnetic islands were deposited (without modulating the film thickness) without changing the film shape, probably due to the large density of strong intrinsic (defect-related) pinning sites.

Here, we study the matching field magnetoresistance effect in arrays of gold NP clusters linked to Nb via organic linker molecules. We found a significant increase in the pinning potential for this hybrid system compared to that of similar arrays of gold or Ni (magnetic) islands in direct contact with the Nb film. These results may also explain the $T_c$ enhancement by relating it to an efficient pinning potential [28].

As noted above, owing to the strong intrinsic pinning centers of Nb, measuring a matching field magnetoresistance effect using Nb films is not simple. All previous studies [15, 29–33] performed with magnetic or non-magnetic islands deposited the islands prior to SC film evaporation; thus, the SC film showed surface corrugation, or the SC thickness was modified. Changing the Nb intrinsic pinning appeared to be essential for the observation of the matching field effect [14]. The pinning effect with magnetic islands was stronger than that with non-magnetic ones due to the corresponding stray field [16]. The strong random intrinsic pinning in Nb films masks all artificial pinning effects induced solely by over-layer surface modifications. Even for periodically corrugated Nb films, matching field effects are difficult to measure, and factors such as the film thickness (and its modulation) and temperature (with respect to the critical temperature), along with the radii of the underlying dots and spacing between them, need to be carefully controlled [15, 20, 29, 34].

In the present work, we modified only the surface of an Nb film without altering its morphology using two methods that form an Abrikosov-like pinning lattice. The first was achieved by directly evaporating metallic or magnetic islands on top of the Nb film (see figure 1(a)). The 2nd method involved adsorption of circular clusters of Au NPs via chemical linking to the Nb (see figure 1(b)). The linking of the Au NPs was done via 3-mercaptopropryltrimethoxysilane (MPS) organic molecules. The circular regions were 200 nm in dia., forming a triangular lattice of 600 nm periodicity (see figure 2). Within each circle, the distance between the 10 nm diameter Au NPs was less than $\xi$, the Nb SC coherence length (~40–60 nm), so that the areas of reduced order parameter around adjacent Au NPs overlapped. Thus, each circle could be considered as one vortex pinning site. The estimated MPS molecule length was less than 1 nm, as calculated from typical bond lengths.
Eighty nanometer Nb films were sputtered on a Si wafer, all having a critical temperature of $\sim 8.5$ K. The relatively high $T_c$ of the (pristine) Nb films indicates that the intrinsic defect density in the film was not very high. For the aforementioned second type of samples, a film was then lithographically patterned with RIE etch to have a junction of $100 \times 100 \mu \text{m}^2$ with a four-probe configuration. In the junction area, the film was covered with PMMA resist upon which a triangular array of 200 nm diameter holes with 600 nm spacing was patterned using e-beam lithography. Following PMMA development, 5 $\mu\text{l}$ of an MPS-Au NPs solution was drop-casted on the pattern, but the MPS molecules covalently bound to the surface only in the Nb regions in the exposed circles. The solution consisted of 3.75 Wt% gold NPs attached to MPS and dispersed in ethanol (purchased from Nanopartz, Inc. USA), was further diluted 1:50 in anhydrous ethanol (Sigma Aldrich Co. Israel). After several minutes, the sample was rinsed with ethanol to remove the unlinked NPs followed by an acetone wash to remove the PMMA, which were processes that did not detach the linked Au NPs. Figure 2 shows scanning electron microscopy (SEM) images of the pinning array configuration containing Au NPs dots linked by MPS molecules to the SC surface in a triangular lattice, and each 200 nm dot contains dozens of 10 nm NPs with 600 nm spacing between dots.

In addition to the hybrid samples, two direct evaporation samples were produced (first method described above), where triangular arrays of Au or Ni (magnetic) islands 20 nm thick, 200 nm in dia. and separated by 600 nm spacing were fabricated on the Nb surface using conventional e-beam lithography and a lift-off method (figure 3).

The primary matching field condition that complies with the dot-lattice configuration in all our samples can be calculated from the corresponding lattice constant, $a_{\Delta}$, using the relation $a_{\Delta} = \frac{4}{3} \frac{\phi_0}{\pi} \left( \frac{\phi_\theta}{\phi_0} \right)^2 = 1.075 \left( \frac{\phi_\theta}{\phi_0} \right)^2$. Taking $\phi_0 = 2.07 \times 10^{-15}$ T m$^2$, which is the superconducting flux quantum, and $a_{\Delta} = 600$ nm, we find that the primary matching field for which the number of vortices is equal to the number of pinning sites is $B_1 \cong 66$ G. At this field, all pinning sites are ideally occupied by one vortex, leaving no free vortices; thus, the vortex lattice is strongly pinned as a whole, and a dip in the magnetoresistance is anticipated. Matching field effects are also expected at other vortex-site commensurate conditions, $B_m = m66$ G, where $m$ is usually a small integer number or a simple fraction (e.g., $\frac{1}{2}$) [25]. The radius of the dots (100 nm) was chosen to fulfill the condition $\frac{\phi_\theta}{\phi_0} \sim 1$, which
is beneficial for the observation of matching field effects [15, 35], where $\xi$ is approximately 40 nm for clean, bulk Nb and 60 nm in the dirty limit [36].

All samples were zero-field cooled to the base temperature before magnetoresistance measurements were performed. The measurements were done in a closed loop He system, Oxford’s SpectromagPT 7 T, with an electrical four probe configuration using rectangular current pulses every 0.2 ms with a delay time of 0.1 ms to reduce joule heating of the sample. The temperature stability was better than 2 mK at the experiment temperature range.

To detect the matching field effects, particularly in magnetoresistance measurements, the artificial pinning potential should exceed that of the random pinning sites. It is therefore advantageous to measure the magnetoresistance near the critical temperature of the film, where the intrinsic random pinning of the SC is less effective and the vortex mobility is higher [37] so the effect of the periodic artificial pinning sites may be detected. This is particularly important in the case of sputtered Nb films, which have strong intrinsic pinning that commonly masks the effects of artificial pinning centers.

We first present the results measured on the two control samples described above, namely, triangular lattices comprising 200 nm islands with 600 nm spacing between them, one sample with Ni islands and the other with Au islands evaporated directly on the Nb surface. These two samples allow us to compare magnetic and non-magnetic arrays of the same geometry. Note that unlike in previous experiments, the metals were evaporated onto the Nb surface, thus eliminating the effects of Nb film corrugation. Figure 3 presents the magnetoresistance measurements done on the Ni (figures 3(a) and (b)) and Au (figures 3(c) and (d)) island arrays. All magnetoresistance measurements on these samples did not exhibit any matching field effects despite being performed at a temperature very close to $T_C$, and with relatively high current densities. These results match previous measurements in which the Nb film covered the islands [16], and they indicate that corrugation of the film is crucial for observing the matching field effect. Moreover, even the magnetic Ni islands, which are expected to significantly reduce the condensation energy on Nb in their vicinity due to their stray field, could not promote matching field effects without corrugation at such small volumes [16].

Figure 3. Typical magnetoresistance measurements obtained from the triangular arrays comprising 20 nm thick Ni and Au islands 200 nm in diameter with a 600 nm lattice constant deposited on Nb. (a) Magnetoresistance measurements on the Ni array performed with current densities, $J$, in the range 12–33.25 kA cm$^{-2}$ (increasing from bottom to top) at temperature $T = 0.98T_{C}$. (b) SEM image of the Ni island array. (c) Magnetoresistance measurements on the Au island array taken with $J = 12–24$ kA cm$^{-2}$ and $T = 0.98T_{C}$. (d) SEM image of the Au island array.
The absence of a matching field in the samples with Ni or Au island arrays deposited directly on the superconducting Nb surface shows that the intrinsic pinning potential of the Nb films is stronger than that induced by the metallic (magnetic or non-magnetic) islands [33].

In contrast, and quite surprisingly, the magnetoresistance curves acquired from the samples in which Au NPs were chemically linked to the Nb surface in clusters to form a triangular periodic array of the same parameters (figure 2) do exhibit clear matching field effects. Here, shallow minima with 66 Gauss increments are observed in the magnetoresistance measurements, as demonstrated by figure 4. The detected effect appears for different temperatures and current densities, both with increasing and decreasing of the external magnetic field applied perpendicular to the samples (figures 4 and 5). In all samples, we can detect the first- and second-order magnetoresistance minima at ~66 Gauss and ~132 Gauss, respectively. These are seen most clearly in figure 4(b), while in others, they appear shallower. Interestingly, in figure 4(c), the second principal minimum can hardly be observed, possibly due to the lower temperature (further away from $T_c$) at which this curve was acquired. It is important to note that the MPS molecules adsorbed on the surface of the Nb film in the same system configuration without Au NPs attached to them did not show any signatures of a matching field effect, as shown in the inset of figure 4(a).

In addition to the two principal (integer in $B_{m}$) magnetoresistance minima discussed above, in some cases, we could also observe clear evidence of the half-integer effect. This is demonstrated in figure 5, where the magnetoresistance curve exhibits a kink at $1/2B_{m}$ (~30 Gauss) followed by a series of 5 (shallow but clear) dips in $1/2B_{m}$ increments, up to $3B_{m}$. The inset of figure 5 demonstrates the expected linear dependence of the magnetic fields at which the minima were observed on their serial number, with good agreement to $B_{m} = 68$ Gauss, which corresponds well to the sample design.
The minima observed in the magnetoresistance measurements are notable but shallow in many cases. The main reason for such a behavior is our configuration design, where the artificial pinning centers are located on top of the SC film and not beneath it, as reported in numerous papers on the subject. Creating a pinning array under the SC saturation number, which is given by the total effective pinning strength.

reported in

Saturation number, which is given by the total effective pinning strength.

example, the distance between NPs within a cluster may yield variations in the pinning potential, which weakens to the Au NPs within a cluster, their density and density cluster lattice and the edge roughness of individual clusters. When estimating the pinning potential related to the Au NPs within a cluster, their density and density fluctuations should also be taken into account. For example, the distance between NPs within a cluster may yield variations in the pinning potential, which weakens the total effective pinning strength.

On the other hand, the number of dips in the magnetoresistance curve shown in figure 5 is higher than the saturation number, which is given by \[ n_s = \frac{d}{4\pi T_0} \] (\(\sim 2\) in our case), where \(d\) is the dot diameter. The saturation number defines the expected number of vortices that can be pinned beneath a single dot before repulsive forces between vortices exceed the pinning potential. The high number of dips, which is higher than the saturation number in our case, implies strong pinning, which may pin vortex lattices denser than the artificial dot structure. The situation of interstitial pinning of vortices may point to a different pinning mechanism enhancement of the pair potential beneath the dots compared to within the interstitial regions and could favor additional regions for weak vortices pinning [6, 13].

The results presented in figures 3–5 are very intriguing. While no matching field affects are detected with arrays comprising Au or Ni islands directly deposited onto the Nb surface, a significant effect is observed with Au NPs linked to the Nb surface via MPS molecules, although the molecules move the NPs away from the surface. This indicates that the presence of the organic MPS linker enhances the pinning strength, but at the same time, the Au NPs are also needed (figure 4(a), inset). While it is true, that the effect was measured only at small temperature and current regimes, we note that showing that the pinning forces induced through the organic molecules by the nanoparticles, are strong enough to overcome intrinsic pinning of Nb is surprising at any temperature range. This implies that these molecules add another transport route that may enhance the proximity effect and thus further reduce the pairing amplitude in the Nb, in addition to conventional proximity effect taking place in ‘conventional’ metal/SC interfaces. One such mechanism is the multiple Andreev reflections process via disorder, which was found to enhance the proximity effect (and Josephson coupling) in systems comprising an Anderson insulator coupled to a superconductor [10]. Such a process can also occur within the MPS molecules, resulting in further reduction of the pairing amplitude of Nb in the regions where the MPS-Au NPs exist, thus enhancing the pinning strength there. In this respect, it is important to note that our previous tunneling spectroscopy measurements show that MPS molecules can support the proximity effect between Nb and Au NPs, where the latter developed a mini-gap in the quasiparticle DOSs [6, 13, 45]. The proximity of the pair wavefunction through the molecular electronic states mediated by vibrational resonance modes of the molecules can influence the pair potential. In such a multiple Andreev reflection system, the electrons and holes acquire a different phase, which may create strong pinning center for vortices. The Andreev reflection near the Au NPs seems to occur by resonance energy transition through the organic molecules at the chemical potential of the molecule and the Fermi energy of the superconductor.

In the current work, we showed that significant pinning is induced by Au NPs coupled to a Nb superconducting surface using organic molecules. The pinning potential in this case was stronger than that achieved through Au or Ni islands directly coupled to the superconducting surface. The matching field magnetoresistance effect was detected only for Nb films on which periodic arrays of clusters consisting of Au NPs were coupled by MPS organic molecules to the Nb. Such an effect was not observed for Au and Ni island arrays of the same geometry or for MPS clusters without Au NPs. These results point to a resonance behavior between the NPs and Nb through the organic molecules and provide further evidence that the superconductor proximity effect in the nanoparticles/molecule/SC system is surprisingly rich. We attribute the enhanced matching field resonances to the multiple Andreev reflection mechanism occurring in the Nb/linker-molecule/Au-NP junction, which effectively suppresses the Nb superconducting order parameter. For applications, the studied effect supplies a simple procedure for enhancing the critical current in superconducting thin films.

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