Little-Parks Oscillations in a Single Ring in the vicinity of the Superconductor-Insulator Transition

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We present results of measurements obtained from a mesoscopic ring of a highly disordered superconductor. Superimposed on a smooth magnetoresistance background we find periodic oscillations with a period that is independent of the strength of the magnetic field. The period of the oscillations is consistent with charge transport by Cooper pairs. The oscillations persist unabated for more than 90 periods, through the transition to the insulating phase, up to our highest field of 12 T.

Introduction. Transport properties of amorphous superconducting films are strongly influenced by Cooper pairing, Coulomb repulsion and disorder. The interplay of these effects leads to the very interesting physics of Superconductor-Insulator transition (SIT), which is now routinely observed in dirty metallic films[1–3]. This quantum phase transition can be driven by variation of disorder[4], thickness[5], magnetic field (B) [6], composition and carrier concentration[7].

One of the central questions regarding the physics of the SIT is to which extent Cooper pairing is relevant in the insulating phase terminating superconductivity. From the theoretical side, there are two complementary approaches. The so-called Fermionic theory of suppression of superconductivity[8], being quite successful in describing the reduction of the transition temperature $T_c$ via Coulomb interaction, including full suppression of superconductivity, does not take into account the effects of Cooper pairing in the normal, or insulating, state of the film. The alternative approach considers competition of Anderson localization and superconductivity[9–11] and, contrary, admits activated transport by Cooper pairs in the insulating regime[12].

Experimentally, the importance of Cooper pairing in the insulating state can be probed by both tunnelling spectroscopy and transport measurements. In the first approach, one directly measures the superconducting gap in the insulating phase, which indicates the presence of localized Cooper pairs[13–15]. The second approach (which we adopt in this paper) is based on specifically addressing effects, which are related to the crucial property of the Cooper pairs - their ability to maintain coherence at macroscopic distance. This idea can be traced back to one of the first indications to the importance of the Cooper-pairing principle - Little-Parks experiment[16]. Since then, a series of experiments was performed following the same logic[17–20].

Recently, following the experiment of J. M. Valles Jr. group[21], we applied this idea to amorphous indium-oxide ($a$:InO) films[22]. We used a self-arranged array of holes to create a sample comprised of a network of rings of a disordered superconductor. Our measurements demonstrated the existence of oscillations with a period consistent with elementary charge of 2e (Cooper pairs) in the insulating regime. However, as in other experiments[17–20], we were able to detect only a few oscillations, due to their decay with B. The reason of this decay was not clear and, in principle be twofold: 1) intrinsic effect of magnetic field, which quickly destroys spatial coherence of the Cooper pairs on the scale of the elementary cell of the array and 2) effect of fluctuating size of the individual loops of the array, which smears out oscillations at larger fields. In addition, it was not possible to exclude the possibility of Josephson array physics[23].

The aim of the present work is to extend our work[22] to the case of a single ring, in order to clarify both questions. We concentrated on the direct vicinity of the disorder-induced SIT transition in $a$:InO. We found that oscillations not only exist in a single ring both below and above SIT, but persist up to the highest fields available (12 T).

Fabrication. To define the structure, we used the ultra high resolution Electron Beam Lithography (EBL). In order to minimize the size of $a$:InO contacts directly adjoin to the structure, we had to implement the EBL process twice with on overlay precision of less than 20 nm between phases: in the first step we produced the inner Ti/Au contacts, followed by the fabrication of the $a$:InO ring (using a second EBL step). Each time thermally oxidized silicon wafer ($Si/SiO_{2}$ with typical value of the surface roughness less than 1 nm; oxide layer 300 nm and resistivity less then 5 mΩ·cm), was spin-coated with bilayer of poly(methyl methacrylate) (PMMA) electron-beam resist of two different molecular weights. The desired structure was exposed in the resist using a EBL system JEOL JBX-9300FS. Photolithography was used to prepare four or six-point Ti/Au electrical (outer) contacts. $a$:InO film was e-Gun evaporated in ultra high vacuum system ($2.5 \times 10^{-7}$ Torr; Thermionics) from high purity (99.999%) $In_2O_3$ pellets in residual $O_2$ pressure $\sim 1.5 \times 10^{-5}$ Torr.

For structural determination we deposit one more test-
sample along with the experimental one. From the scanning electron microscopy (SEM), we conclude, that the internal diameter is 50 nm and for the external diameter \(d_e\) we have 150 nm. The external diameter of the disk was 320 nm. Accuracy of this measurements was \(\pm 2\) nm. The SEM images of the obtained structures are shown in the Fig. 1a and 1b (one of four experimental samples exhibiting oscillations is shown). Atomic force microscopy (AFM) images showed the thickness variation about 10\%, i.e. the thickness is 30 \(\pm 3\) nm. \(\alpha: \text{InO}\) is known to form relatively uniform films\cite{24}, so we expect our structures to be uniform as well.

After gentle lift-off, the sample was mounted on the sample holder, electrically connected with Au wire. Finally, the sample is immersed into a Kelvinox TLM (Oxford Instruments Inc.).

**Experiment.** We implemented two- and four-probe techniques. The signal from the sample was amplified by low-noise, home-built differential voltage pre-amplifier and measured using EG&G 7265 DSP Lock-in Amplifiers at low frequency 1.8 Hz. In order to minimize heating of the structure, we used low excitation current \(\sim 1\) nA.

We first measured the dependence of the resistance \((R)\) of the \(\alpha: \text{InO}\) disk on temperature \((T)\) at \(B = 0\) T. The result is shown in Fig. 1a. As \(T\) is lowered below 4 K the resistance drops abruptly from \(1.4k\Omega\) to 50\%. We note that despite such a sharp drop of the resistance, it saturates at measurable value and remains finite down to the \(T = 50\) mK. In this regime, the sample demonstrates quadratic positive magnetoresistance at low field turning into a negative magnetoresistance at \(B > 2\) T.

Next, we measured the resistance of the ring as a function of \(T\). Contrary to the disk and films, it does not show any sudden change in the resistance down to the lowest temperature. However, it demonstrates non-monotonous magnetoresistance, similar to that of the disk. We show \(R\ vs\ T\) traces at different values of magnetic field in Fig. 1c.

On a large scale of \(B\), the disk and the ring demonstrate similar behaviour. They exhibit the familiar high-\(B\) phenomenology that we are accustomed to in our previously studied \(\alpha: \text{InO}\) films\cite{23}, although, in this case, it is less developed. In Fig. 2 we plot \(R\) isotherms over our entire \(B\) range. The crossing point of the isotherms at \(B_c = 0.8\) T identifies the ‘critical’ \(B\) of the magnetic field tuned SIT, followed by the prominent magnetoresistance peak at \(B = 8\) T. We believe that relative smallness (compared to measurements on macroscopic films) of the resistance variation with \(B\) and \(T\) is due to mesoscopic nature of our sample. Another effect of the finite size, related to the loop geometry, is clearly seen on the Fig. 2 small, about \(\sim 1\%\) by magnitude, oscillations of resistance as function of magnetic field appear, which will be in the focus of the remainder of this Letter.

We start our analysis of these oscillations with the region of low \(B\). On the plot of \(R\ vs\ B\) (Fig. 3), more than ten oscillations are seen, superimposed on a parabolically rising background. The oscillations period \(\Delta B \approx 0.15 \pm 0.02\) T can be read from this figure. It is independent of \(T\), indicating that it is determined by the geometry of the ring. Trajectory of a particle of a charge \(2e\) encompassing \(\Phi_0 = h/2e\) in a field of 0.15 T is shown on the Fig. 1b and is consistent with the flux periodic-
ity in integer units of $\Phi_0$. For better characterization of oscillations, it is convenient to define normalized oscillating part $\alpha(B) = (R(B) - R_o(B))/R_o(B)$, where $R_o(B)$ is smooth part of the $R(B)$ dependence (averaged over several oscillations).

Our central result is related to the behavior of $\alpha(B)$ at high $B$. It is presented in Fig. 4, where we plot $\alpha(B)$ of our ring for the entire range of $B$ at $T = 150$ mK. Oscillations are clearly visible throughout the range, up to our highest $B$. This result is quantified in a table, shown inset on the Fig 5, where, we show the period of the oscillations as determined by counting the peaks in the interval of 1 T on several ranges of $B$. Different rows correspond to different ranges of $B$: the first row, for example, is for the range [-0.5 T, 0.5 T]. It is clear that the oscillations have similar periodicity at different values of $B$.

Finally, we characterize the $T$-dependence of the amplitude of the oscillations. As a quantity characterizing the amplitude of the oscillations, we choose $\sqrt{\langle \alpha^2 \rangle_B}$, where averaging over the entire range of $B$ is implied (the results are qualitatively the same if averaging over other range is performed). The $T$-dependence of this quantity is shown in Fig. 5. It is consistent with our intuition: with increasing $T$, coherence length of the Cooper pairs decreases and oscillations disappear at $T \sim 1.2$ K.

Discussion. Our main observation is magnetoresistance oscillations of a constant period throughout the available interval of $T$ and $B$, consistent with a flux periodicity, corresponding to elementary charge $2e$. We argue that this magnetoresistance is most likely due to electron-electron interaction in the Cooper channel, that is, undeveloped Cooper pairing. We now turn to the analysis of the possible origins of these oscillations.

We are aware of two physically distinct mechanisms that can lead to such oscillatory magnetoresistance: the electron-electron interaction in the Cooper channel and weak (anti)localization (WL). The first effect is expected to be most prominent close to the superconducting transition, as is measured in the Little-Parks scheme. In the vicinity of $T_c$ the resistance of the ring is determined by thermal phase slips, which are influenced by magnetic flux penetrating the ring. Detailed theoretical analysis of this effect was performed in Ref. [19], where it was demonstrated that periodic flux-dependence of activation energy of the phase slips in such a ring allows to explain magnitude of experimentally observed oscillations in the vicinity of temperature-induced superconducting transition of the LaSrCuO rings. For the metallic regime outside the transition region in the vicinity of $T_c$, Kulik predicted, based on the Ginzburg-Landau approach, that this effect should also be noticeable. It appears due to the presence of fluctuating Cooper pairs, which can be rather long-lived, $\tau_{GL} = \pi/\ln T/T_c$, and in this respect is due to paraconductivity. Later, Larkin demonstrated that further away from the transition Maki-Thompson correction would be dominant in magnetoresistance and, hence, will give dominant contribution in Little-Parks type of oscillations.

Interestingly, in the experiment of Shablo[30], where oscillations of the resistance in the normal state were first observed, they were attributed to paraconductivity, and only later it became clear that it is more realistic that they are actually related to weak localization, which was not well understood. It gives another possible contribution to the observed oscillations, see also experimental study in Ref. [32]. This effect is not associated with electron-electron interaction, but its phenomenology is similar to that of interaction-induced one. Interestingly, this effect was also predicted to exist in the hopping conductivity regime, but it has the sign opposite to that
observed in our samples.

As was stressed already in the seminal work of ref. [31], the amplitude of oscillation in metals is usually determined by a factor $\gamma - \beta(T)$, where $\gamma$ is coming from weak localization part and depends on the symmetry class of the system ($\gamma = 1$ for weak spin-orbit impurity scattering, $\gamma = -1/2$ for opposite case) and $\beta(T)$ is an effective constant of Cooper interaction [29]. These two effects, although having the same periodicity, have rather different spatial scales: single-particle coherence length $L_\phi$ and coherence length of the Cooper pair $L_\xi$. Since we do not have any reliable estimate for $L_\xi$ in our sample, we will concentrate on ruling out the possibility of WL origin of the effect. The first argument concerns the ability of single electrons to maintain coherence on the size of the sample. Standard estimation [34] gives $L_\phi \approx 60$ nm at $T = 0.2$ K. This is an order of magnitude smaller than the circumference of our ring. Another argument holds on the fact that magnetic field not only imposes the phase on the interfering electrons, but also induces mass into the Cooperon, the effect which is relevant as long as the ring has finite width $w$ (which is especially large in our case). In general, it imposes certain restriction on the number of oscillations, which can be seen as follows: $N_{osc} \sim d_e/w$, in our case $N_{osc} \sim 3$. We resolve over 90 oscillations.

These arguments allow to exclude WL for the description of the observed effect. We emphasize that we defer an attempt for a theoretical explanation of our observation to a future publication, we nevertheless stress that in all likelihood, it is rooted in the stability of Cooper pairing deep in the insulating regime. This is especially interesting because our ring does not show strong superconducting trend. More detailed study of these oscillations can shed more light on the role of the Cooper interaction in mesoscopic $\alpha$:InO rings.

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